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PROCESS SUMMARY OF PUREX PLANT OPERATION JANUARY 1961 THRU DECEMBER 1961

AUTHOR

R. G. Geier
H. C. Rathvon

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PROCESS SUMMARY OF PUREX PLANT OPERATION
JANUARY 1961 THRU DECEMBER 1961

R. G. Geier - H. C. Rathvon

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 1/13/61 TO 2400 ON 1/19/61

1. Normal processing was commenced (HAF on) at 3.0 CF at 1800 on Jan. 16. Processing of "hot" HAF did not start until 0300 on Jan. 17. After being initially routed to the L-Cell Package, the 2BP was routed to the N-Cell ion exchange unit at 0700 on Jan. 18. Due to the relatively short period of operation, the report for this period will not include "typical" process data.

2. Flowsheet

(a) The start-up flowsheet on Jan. 16 was the same as at shutdown on Dec. 24 except for the following:

- (1) The HSR-HNO₃ was 6.4 instead of 9.2 flows.
- (2) The HAF flow was 111 instead of 75 flows, and the HAF specification was 2.40 #/gal U and 1.40 #/gal HNO₃ instead of 3.57 #/gal U and 0.60 #/gal HNO₃.
- (3) There was no 3WB flow.
- (4) The 2BP flow was routed to the L-Cell Package.
- (5) The IOC was 7.4 continuous flows. (No batch changes of G7.)
- (6) The IOF-KMnO₄ was 1.7 continuous flows. (No rapid additions of KMnO₄ after a G1 carbonate change.)
- (7) No water was added to the 3WF Tank.

(b) On Jan. 17 the 3WB was started at 36 flows, HAF was reduced from 111 to 75 flows, and the HAF was made 3.57 #/gal U and 0.60 #/gal HNO₃.

(c) On Jan. 17 the J1-HNO₃ flow was started at 4.0 flows.

(d) On Jan. 17 200 gallons of waste from the shutdown neptunium run (high Pu content) was added to each 4000-gallon batch of HAF.

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- (e) On Jan. 18 the 2BP was diverted to N Cell.
- (f) On Jan. 19 the J1-HNO₃ was reduced from 4.0 to 2.5 flows.
- (g) On Jan. 19 the 2IF-Fe was reduced from 0.20 to 0.15 flows.

3. Solvent Extraction

The plant start-up was exceptionally smooth, and as a result no unusual waste losses were encountered. The gamma levels of intercycle streams were initially very high, but this resulted from the December shutdown and subsequent neptunium recovery run. The gamma levels decreased sharply after start-up to values about 5 fold less than the December shutdown levels. At the end of the report period, the first batch of plutonium product through the ion exchange unit had been produced, and met specifications. Uranium product gamma ratios (before silica gel) were averaging about 25 per cent above the shipping specification.

At start-up the HS Column pulse frequency was operated at 96 cycles per minute with the new, all-stainless-steel cartridge in service. Because the HS Column static pressure was initially unusually high and the HSP gamma monitor was erratic, on Jan. 17 the frequency was reduced to 81 cycles/minute, a value resulting in a static pressure value normal for the previously installed zebra cartridge. The gamma monitor operated normally and the HA-HS Column IF appeared slightly better after the frequency reduction. Although adequate process data have not been acquired, thus far the new HS Column cartridge is apparently as good or better than a damaged zebra cartridge.

Other items of interest to solvent extraction are as follows:

- (1) About 6 tons of uranium waste from the UO₃ Plant was processed through the Final Uranium Cycle (K Cell) on Jan. 16. Uranium product was recycled to the 2IF for several hours after the rework in order to insure good 200 quality. Ferrous iron had been added to the original waste but not to the recycled product. As a result about 1 1/4 tons of uranium slightly exceeded the specification for Pu content and will require blending.
- (2) On Jan. 18 a leaky 3WF pump (TK-F10) was replaced without lost production.
- (3) The rework of waste from the recent neptunium recovery run (high Pu 2AW concentrated in E-F11) was commenced on Jan. 17. No process effect was noted.

4. Solvent Treatment

At the beginning of the report period flushing of TK-G1 was continuing in order to reduce the volume of solids hold-up. After oxalic-nitric flushes had reduced this hold-up to about 300 gallons, flushes of

20% HNO_3 followed by 5% NaOH -2% tartaric acid reduced the hold-up to zero.

Following plant start-up, the ICO activity increased from about 700 uc./gal. to the 3000 to 4000 uc./gal. range.

5. Waste Treatment and Acid Recovery

During the shutdown period work was completed on the diversion of steam condensate to the A30 Steam Condensate Crib. Also, trap pit work was completed, resulting in the elimination of condensate leaks and the replacement of the trap jumper to E-F11.

Waste resulting from the Np recovery run was processed commencing on Jan. 17 at the ratio of approximately one part of rework to 10 parts of HAF with no deleterious effects. In addition to the Np recovery wastes, this material included empty-cut and acid flushes of the HA-HS Columns and acid flushes of the E-H⁺ Concentrator.

The P2 Silver Reactor was regenerated during the shutdown period.

Condenser vent vacuum has been greatly improved since replacement of the E-H⁺ Concentrator, with vacuums of 45 to 50 inches of water vs. about 30 inches prior to the replacement.

6. Fission Product Recovery

Between Jan. 12 and Jan. 19 another series of strontium recovery runs was completed. The series consisted of four lead carrier sulfate precipitation runs; two oxalate precipitation runs on the combined carbonate product from the sulfate runs; and one concentration precipitation run on the combined oxalate product. A total of 129 kilocuries of Sr 90 in a volume of 1140 gallons was transferred to TK-002 in the 244-CR Vault. The composition of the material transferred was as follows:

	<u>Curies/Gallon</u>	<u>Lb./Gal.</u>
Sr 89	870	
Sr 90	110	
Ce 144	1600	
Zr-Nb 95	2600	
Ru-Rh	50	
HNO_3		0.329
Fe		/0.034
SO_4		/0.136

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The average age of the INW being processed through the Fission Product Recovery Test Plant has been gradually decreasing with a corresponding increase in the amount of heat generated in the centrifuge. The high heat generation rate has made carbonate cake removal difficult due to gassing of the centrifuge jet. Frequent back-flushing of the jet with cold water is necessary in order to remove the dissolved carbonate cake. This jet dilution has reduced the number of carbonate runs that can be accumulated in TK-E1 from 5 to 4.

As previously reported, the strontium solution received by HLO contained large amounts of precipitate. After adjustment with nitric acid, enough of the precipitate dissolved to permit ion exchange processing without filtration. Purification Run No. 2 with this material has been successfully completed. Based on preliminary analytical results, recovery is estimated to be approximately 80%.

Appendix --- Process Summary Through Jan. 22, 1961

Over the week end process performance improved markedly. The uranium product (from solvent extraction) gamma ratio declined to about 1.2, and the Pu product likewise was comfortably within specifications. At the same time the HAW Pu losses were declining.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 1/20/61 TO 2400 ON 1/26/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.5	4.1	-	-	0.003	0.09
Final	3.2	3.3	8.5	3.9	0.003	-
Ion Exchange	-	0.9	-	0.3	-	-
Overall	6.7	8.3	8.5	4.2	0.006	0.09

II. Production

A. HAF Processed: 239 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
1	3.6	42.0	3.6	E6
1a	3.6	42.0	5.0	F3
1b	3.6	42.0	0.9	F3
2	4.7	17.7	5.3	F3
3	4.9	9.7	6.9	F3
4	3.6		6.3	F3
5	2.6		6.5	F1
6	2.1		7.1	P1
7	2.3		6.9	P1
8	2.2	0.7	6.5	P1
9	2.4		6.7	P1
10	2.6	0.5	6.9	P1
11	2.8		7.1	P1
12	2.8	0.4	7.1	P1
13	2.2		7.0	P4
14	1.6	0.3	7.1	P4

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
15	1.2		7.2	P4
16	1.4	0.3	7.4	P4
17	1.3		7.1	P4
18	1.1	0.4	7.2	P4
19	1.3		7.5	P4
20	1.4	0.7	7.3	P4
21	1.4		7.1	P4
22	1.5	0.9	7.3	P4
23	2.0		7.2	P4
24	1.7	0.7	7.2	P4
25	1.4		7.0	P4
26	1.8	0.7	7.5	P4
27	2.3		7.4	P4
28	2.4		7.2	P4
29	2.7		7.3	P4
30	2.9	0.3	7.4	P1
31	3.0		7.2	P1
32	3.0	1.6	7.3	P1
33	3.2		7.8	P1
34	3.1	0.5	7.2	P1

2. Silica Gel

Gamma Ratios: Feed = Approximately 3.0
Product = 1.8

Tonnage: 205

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 1011*</u>	<u>% U in Pu</u>	<u>Fe, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
1				Recycled	to	E6
2	3.4	0.009		5	4	234-5
3	1.6	0.007		4	1	234-5
4	1.2	0.005		5	1	234-5
5				Recycled	to	E6
6	1.2	0.008		5	1	234-5
7	2.9	0.008		4	1	234-5
8				Recycled	to	E6
9	0.79	0.008		4	1	234-5
10	0.66	0.006		5	1	234-5
11	1.5	0.010		5	1	234-5
12	1.6	0.007		5	1	234-5
13	2.3	0.010		5	1	234-5
14	0.7	0.016		4	1	234-5
15	1.3	0.021		5	1	234-5
16	0.89	0.007		5	1	234-5

(Batches 1, 5 and 8 were predominantly out-of-spec Pu Product from L-Cell Package, which was reworked).

*Revised Plutonium Specification (See Note #5 below.)

D. Overall Waste Loss - % of Total Production:

U = 0.26%
Pu = 0.39%

E. Rework - % of Total Production:

U = 3.4%
Pu = 18.8% (Predominantly L-Cell Package flushes and material via L11 to HAF Make-up TK-E6; also, IWW Waste via TK-F15 to HAF Make-up TK-E6)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	861	101 and 104
Cell Drainage	249	104
FP Waste	182	104
Coating Waste	270	103-C

B. IWW Flows: 5.2

C. Tank Farm:

<u>Tank</u>	<u>Estimated Boil-Off Rate, Gal/Min</u>
101	10.0
102	5.0
103	0.3
104	12.7

Notes:

1. The HAF rate was maintained at 3.0 CF throughout the report period.

2. Flowsheet

- On January 20 the 2DF-HNO₃ was increased from 7.7 to 8.7 flows, and the 3WB-HNO₃ flow was stopped.
- On January 20 the HAO was increased from 80 to 91 flows.
- On January 23 the rework of waste from the neptunium recovery run via the HAF was completed.
- Beginning on January 23 the uranium metal feed solutions were made 0.0005 M Hg(NO₃)₂.
- On January 26 the HSR-HNO₃ was increased from 6.3 to 8.4 flows.

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3. Feed Preparation

During the report period 8.81 curies of iodine were emitted at an average dissolver metal "cooling" time of 115 days. This compared with 2.78 curies for the previous week. The iodine problem was being aggravated by intermittent charging of cars of less than 100-day metal. On January 26 the addition of mercuric nitrate to all feed batches was resumed.

4. Solvent Extraction

Both products met all specifications, and it was necessary to silica gel only 29% of the uranium product.

Reported high HAW neptunium losses led to an increase in the HAO from 80 to 91 flows on January 20 and subsequent increase in the HSR-HNO₃ from 6.4 to 8.4 flows on January 26. No particular effect on the losses was noted; however, these losses have been inconsistent with the high rate of Np accumulation noted in the 2DW stream. The HAW Pu loss averaged about 0.07% until January 23, but was increased up to two fold in order to improve First Cycle DF later in the report period.

The decontamination performance of the plant was good early in the report period and continued to improve until January 22. The DF of the plant started deteriorating while reworking the final 250 gallons of the waste accumulated during the last Np recovery run, and continued to deteriorate after the rework was completed on January 23. The following table shows the changes in gamma levels of some intercycle streams during the report period:

	$\frac{\text{ZrNb/AT}}{2\text{AF}}$	$\frac{\text{GR}}{2\text{DF}}$	$\frac{\text{GR}}{\text{K6}}$
Start	5.7×10^{-7}	3850	2.75
Low Point	9.5×10^{-8}	790	1.03
End	7.4×10^{-7}	5500	3.14

5. Plutonium Concentration

Since plant start-up, performance of the N-Cell Ion Exchange Facility has been good. The fluoride concentration in the XAF has been maintained at 0.01 M with an Al/F ratio of 2 and excess AMN added to the JWB. The N-Cell DF has generally remained at about 10, although this value was apparently increasing at the end of the report period.

A revised Pu product specification became effective at the beginning of the current operating period (January 17). Allowable gamma activity

is now specified in terms of gamma scans by the multi-channel analyzer. For practical purposes at the Purex Plant the specification is based on a $ZrNb/AF$ value equal to about 1.25 times the gamma value of the previous specification.

AF

6. Solvent Treatment

Following a gradual increase from 3,000 to 5,400 uc./gal., the IO gamma activity suddenly increased to about 8,000 to 10,000 uc./gal. on January 25. The increase coincided with a general increase in Zr-Nb levels throughout the solvent-extraction system.

Since start-up a jumper for sampling the organic phase at the top of the IO Column has been installed. With this sampler, the IF across TK-61 and the IO Column will be measured separately from the Turbomixer G7 (and centrifuge) IF. Operability tests of the sampler are in progress. Based on only one set of samples the gamma IF across each part of the recovery process is 2 to 3.

7. Waste Rework and Acid Recovery

On January 23 the rework of concentrated wastes from the shutdown Rp recovery run was completed with the recovery of about 250 lb. of U and about 1,200 g. of Pu.

The scheduled addition of 2,000 gal. of INW to TK-241-A-106, for the purpose of pre-warming this tank, was temporarily suspended. Necessary diversion box jumper changes were hampered by high radiation readings resulting from the accidental discharge of process wastes into the box.

8. Essential Materials

On January 19 considerable amounts of iron were found in the plant's caustic supply. The failure of the heating coil in TK-21 is believed to be the cause. Special arrangements were made to provide clean caustic for demineralizer regenerations and fission product needs, while the contaminated caustic will be used strictly for coating removal and neutralization of waste.

Appendix -- Process Summary Through January 29, 1961

Over the week end, process gamma levels gradually increased. A test was started to measure the effect of varying the pulse frequency on the performance of the new ES Column cartridge.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 1/26/61 TO 2400 ON 2/2/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma of</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.3	3.4	-	-	0.004	0.11
Final	3.1	3.5	8.6	6.1	0.002	-
Ion Exchange	-	1.3	-	0.5	-	-
Overall	6.4	8.2	8.6	6.6	0.006	0.11

II. Production

A. FAF Processed: 175 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
35	3.3		7.2	Pl
36	3.8	0.4	7.3	Pl
37	4.6		7.0	Pl
38	5.0	0.6	7.3	Pl
39	6.0		7.7	Pl
40	5.8	0.6	7.2	Pl
41	5.4		7.4	Pl
42	5.5	1.0	7.3	Pl
43	5.7		7.3	Pl
44	5.4	0.5	7.3	Pl
45	5.4		7.2	Pl
46	5.8	0.4	7.4	Pl
47	5.5		7.3	Pl
48	5.9	0.6	7.4	Pl
49	5.3		7.3	Pl
50	5.6	0.6	7.2	Pl

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
1	5.4		7.1	P1
2	5.4	1.1	7.1	P1
3	5.3		7.5	P1
4	5.8		6.8	P1
5	5.7		7.3	P1
6	6.0	0.2	7.5	P1
7	5.2		7.5	P1

2. Silica Gel

Gamma Ratios: Feed = 5.3
Product = 1.9

Tonnage: Approx. 120

C. Plutonium Product:

<u>Batch</u>	<u>Zr/Hf/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
17	0.83	0.007		4	1	234-5
18	1.0	0.003		5	2	234-5
19	1.5	0.005		4	2	234-5
20	1.4	0.006	470	4	1	234-5
21	2.2	0.012		5	2	234-5
22	2.4	0.011		5	2	234-5
23	1.8	0.006		5	3	234-5
24	2.2	0.010		6	3	234-5
1	2.3	0.010	962	6	2	234-5
2	2.8	0.009		5	2	234-5
3	2.5	0.005		5	2	234-5
4	2.6	0.009		5	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.01%

Pu = 0.04%

E. Rework - % of Total Production:

U = Negligible

Pu = 0.43%

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III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	462	104
Cell Drainage	6	101
FP Waste	11	101
Coating Waste	270	103C

B. IWW Flows: 4.0

C. Tank Farm:

<u>Tank</u>	<u>Estimated Boil-off Rate, Gal/Min</u>
101	10.5
102	6.3
103	0.3
104	10.4

Notes:

1. The HAF rate was maintained at 3.0 CF throughout the report period.

2. Flowsheet

Commencing on Jan. 29, batch changes of the Turbonizer Tank carbonate were scheduled every 24 hours. The volume change is 1600 gallons.

3. Feed Preparation

The addition of mercuric nitrate to all dissolver batches, beginning on Jan. 26, has resulted in decreased iodine emissions. The weekly iodine total was 6.06 curies, about 30% below the previous week.

4. Solvent Extraction

Gamma levels continued to increase early in the report period, and then levelled off at values near normal for uranium-bearing streams, and about 1.5 fold above normal for plutonium-bearing streams. The increased activity toward the plutonium streams apparently resulted from the high gamma level of the 100 stream.

The pulse frequency of the ES Column was varied to determine the effect on First Cycle decontamination performance. Decreasing the pulse frequency from 81 to 64 cycles per minute over an eight-hour

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period caused the 2AF gamma level to increase 1.5 to 2.0-fold, but no other effect was noted. Increasing the pulse frequency to 88 cycles per minute over an eight-hour period decreased the 2AF gamma level to its previous level. Further increases up to 94 cycles per minute had no noticeable process effect. Later in the week the HS Column pulse frequency was reduced to 71 cycles per minute, with reductions of 5 cycles per minute over an eight-hour period scheduled each day. No process effects were noted that could be related to this change. At the slow rate of change, however, it is possible that an effect would be obscured by other process variations.

5. Plutonium Concentration

N-Cell DF performance for the report period was excellent. DF's of 15 to 24 were obtained compared to approximately 10 for the preceding week. There were no flowsheet or procedural changes made to account for the added efficiency. The DF increase corresponded almost exactly with increasing 2BF gamma activity.

6. Solvent Treatment

The gamma activity in the IOC increased from 10,000 uc/gal to 19,000 uc/gal at mid week, then decreased to 13 - 15,000 uc/gal at the end of the report period. Samples taken at the top of the IO Column have indicated a gross gamma DF of about 3 for the TK-G1-IO Column system and a DF of about 1.5 to 2 for the Centrifuge and Turbomixer.

Recirculation of a portion of the IOF stream back to the IOF Tank G1 indicated an initial slight DF improvement.

The aqueous phase in the Turbomixer is currently being changed once every 24 hours (~ 1600 gallons) while maintaining normal IOC and IOD flows.

7. Waste Treatment and Acid Recovery

Extremely erratic specific gravity recordings for the IWF Tank F7 were checked with a manometer on the high pressure diptube and found to be originating as pressure surges up to 5 inches of water. Apparently, an accumulation of solids within the diptube stilling chamber within the tank was causing partial diptube plugging. A two-hour water flush of the high pressure diptube returned the specific gravity readings to normal.

Efforts to send LW waste to tank 241-A-106 have been postponed due to excessive radiation readings in the diversion box, as reported on page 10. Inability to stop the leak necessitated installation of the original routes from TK-F16 and TK-F18 to TK-241-A-101 so that the large backlog of waste in the building could be sent to storage.

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8. Fission Product Recovery

During the period from Jan. 19 through Jan. 27, a series of strontium recovery runs were completed. The series consisted of four lead-carrier sulfate precipitations, two oxalate precipitations, and one final concentration precipitation. A total of 78,500 curies of Sr 90 was recovered and stored in the E1 vessel. Following this series of runs, strontium recovery was suspended because of maintenance work underway in a diversion box. At the end of the report period, the product was still being held in TK-E1. The composition of the product solution was as follows:

	<u>Curies/Gal</u>		<u>Lb/Gal</u>
Sr 89	690	HNO ₃	0.36
Sr 90	90	Fe	0.01
Ce 141 - 144	290	SO ₄	<0.011
Zr-Nb 95	940		
Ru-Rh	12		

Decontamination factors for Zr-Nb and Ce have decreased and recovery efficiencies have increased since the routine processing scheme of four sulfate runs, two oxalate runs, and one concentration run was adopted. Investigation of the poor DF's led to a special cleanout flush of TK-E3 following the two oxalate precipitation runs. Any fission products remaining in the E3 vessel after the oxalate runs would be returned to the strontium product fraction during the subsequent concentration run. The cleanout flush removed 2.6 megacuries of Ce, 400 kilocuries of strontium, but very little Zr-Nb. The cause of the poor Cerium DF and the apparent improved strontium recovery are thus attributed to incomplete precipitate removal which leads to cross-contamination in the concentration run. The cause of poor Zr-Nb DF is still being investigated. Decontamination factors for the strontium held in TK-E1, product from which the cross-contamination was removed with a special flush, are compared to the previous series of runs in the table below:

	<u>E1</u>	<u>Previous Runs</u>
Ce	8	1.2
Zr-Nb	3	2.6
Ru	300	300

Appendix --- Process Summary Through Feb. 5, 1961

Over the week end the ES Column was operated for about a day at 65 cycles per minute, after which the pulse frequency was increased to 80 cycles per minute over an 8-hour period. The frequency increase resulted in an improvement in ES Column DF which was barely noticeable on the NSP gamma monitor but amounted to about a 40% decrease on the IRP gamma monitor.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 2/3/61 TO 2400 ON 2/9/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma of</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.3	3.2	-	-	0.004	0.09
Final	3.0	3.4	8.5	1.3	0.004	-
Ion Exchange	-	1.4	-	0.1	-	-
Overall	6.3	8.0	8.5	1.4	0.008	0.09

II. Production

A. HAF Processed: 175 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
8	5.4	0.1	7.0	P1
9	6.4		7.4	P1
10	7.1	0.4	7.5	P1
11	6.8		7.2	P1
12	6.6	0.3	7.1	P1
13	6.8		7.0	P1
14	6.9	0.5	7.5	P1
15	6.5		7.3	P1
16	6.2	0.8	7.1	P1
17	5.8		7.5	P1
18	5.6	1.4	7.3	P1
19	5.8		7.2	P1
20	6.2		7.3	P1
21	6.9		7.3	P1
22	6.3	0.6	7.3	P1
23	7.1		7.3	P1
24	7.2	0.4	7.2	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
25	6.8		7.2	P1
26	6.5	0.5	7.3	P1
27	6.6		7.2	P1
28	6.5	0.4	7.2	P1
29	6.7		7.1	P1
30	6.8	0.4	7.4	P1
31	6.4		7.3	P1
32	6.5	0.4	7.2	P1

2. Silica Gel

Gamma Ratios: Feed = 6.5
Product = 2.0

Tonnage: 149 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Pu, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Mr/Hr</u>	<u>Shipped To</u>
5	2.5	0.019	460	4	2	234-5
6	2.3	0.010		5	2	234-5
7	2.3	0.010		5	3	234-5
8	3.2	0.010		5	5	234-5
9	3.4	0.020		5	4	234-5
10	2.9	0.030	460	5	2	234-5
11	3.1	0.030		5	3	234-5
12	3.0	0.030		4	3	234-5
13	3.3	0.020		5	4	234-5
14	3.3	0.020		5	4	234-5
15	3.8	0.029	< 2050	5	4	234-5
16	3.2	0.015		5	4	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.06%

Pu = 0.14%

E. Rework - % of Total Production:

U = Negligible

Pu = 4.1% (L and E Cell sumps and flushes via TK-L11 to HAF Make-up Tank E6)

III. Waste Treatment

A. Volumes to UGS:

Source
Wash Solution

Gal/Ton
583

Waste Tank
104 →

A. Volumes to UGS: (Cont'd.)

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Cell Drainage	95	101A
FP Waste	96	101A
Coating Waste	270	101C

B. LW Flows: 3.9C. Tank Farm:

<u>Tank</u>	<u>Estimated Boil-Off Rate, Gal/Min</u>
101	10.6
102	3.5
103	0.5
104	10.8

Notes:

1. The HAF rate was maintained at 3.0 CF throughout the report period.

2. Flowsheet

- (a) On Feb. 3 the 2HF-HNO₃ was increased from 8.7 to 9.5 flows. It was returned to 8.7 flows on Feb. 5, and was reduced to 8.3 flows on Feb. 6.
- (b) On Feb. 6 the HNO₃ flow to TK-G1 was increased from 1.7 to 3.4 flows.
- (c) On Feb. 6 the frequency of carbonate solution changes (1600 gallons) in the Turbomixer Tank G7 was changed from every 24 hours to every 8 hours.
- (d) On Feb. 7 the IOS was reduced from 178 to 36 flows. On Feb. 8 fresh IOS was added continuously to the IO Column at 36 flows, and the recycle of IOW was stopped.
- (e) On Feb. 8 the IBS was increased from 17.0 to 18.6 flows. It was increased again on Feb. 9 to 19.7 flows.

3. Solvent Extraction

Gamma levels of intercycle and product streams remained essentially constant, except for an approximately 30% increase in the 2BP and plutonium product streams resulting from increased IDO activity. A IBS flow increase resulted in only a slight improvement in the 2AF, 2BP, and plutonium product gamma levels.

The HAW plutonium losses initially decreased two fold, from about 0.1%, but later increased to 0.2% as efforts were made to improve First Cycle BP by increasing HA Column saturation. The HAW neptunium losses also increased about 3 fold with the increased Pu loss.

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On Feb. 4, the HS pulse frequency was increased over an 8-hour period from 65 to 80 cycles/minute. The frequency increase decreased the IRP gamma level about 40%, but no changes were noted in the gamma levels of other streams. On Feb. 5 a further frequency increase was made to 84 cycles/min.

During the last report period it was noted that the C Columns required about 105% of normal extractant flow in order to maintain normal waste losses. It also appeared that the capacity of the C Columns was below normal. During the current report period the evidence became obvious. As in the case of plant experience of a year ago, a correlation with water demineralizer regeneration has been noted.

4. Solvent Treatment

The gamma activity in the IOO generally remained in the 15,000 to 20,000 uc/gal range except for one burst of activity to ~56,000 uc/gal, resulting from purging the IO Column interface. Apparently this burst was caused by the return of activity-laden interface "crud" to the process via the IOS pump. To overcome further experiences of this nature, the IOS stream was repiped to provide a continuous flow of fresh IOS to the column, with continuous flow of the column aqueous waste to the waste tank TK-G8. With this change the IOS flow was decreased by a factor of 5.

Other flowsheet changes which were made in efforts to decrease the IOO activity included the following:

- (a) The IOF recycle (back to TK-G1) was shut off.
- (b) The permanganate addition rate was doubled.
- (c) The Turbomixer TK-G7 carbonate was changed once per shift.

Only the carbonate change frequency had any effect on the activity levels.

5. Fission Product Recovery

Strontium recovery operations continued during the report period. Four lead-carrier sulfate precipitations were completed and the carbonate product was accumulated in TK-E1. At the end of the report period TK-E1 contained 1.72 megacuries of Sr 89-90.

Efforts to define the poor Zr-Nb DF continued. The first batch of raw LW was centrifuged to determine the amount of the Zr-Nb present as a precipitate. Without prior removal precipitated Zr-Nb would normally be carried with the strontium fraction in the first precipitation step. A DF of 1.4 was obtained across the centrifuge. The

subsequent lead-carrier sulfate precipitations were carried out under careful pH control. The first batch of carbonate product (from centrifuged IWW) had an over-all Zr-Nb DF of 9. Accumulated carbonate product from this run and three more precipitations on raw IWW, with careful pH control, had an over-all Zr-Nb DF of 3.3. The Zr-Nb DF between raw IWW and centrifuged IWW does not explain the DF decrease from 9 to 3.3. This test will be repeated during the next series of runs.

Appendix --- Process Summary Through Feb. 12, 1961

The Centrifuge G-22 failed on Feb. 10. The motor was checked and found to be satisfactory but the centrifuge bowl will not rotate.

Solvent-extraction processing continued without significant change.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 2/10/61 TO 2400 ON 2/19/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.4	3.6	-	-	0.003	0.20
Final	2.9	3.1	6.0	3.9	0.006	-
Ion Exchange	-	1.2	-	0.8	-	-
Overall	6.3	7.9	6.0	4.7	0.009	0.20

II. Production

A. NAF Processed: 225 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
33	5.9	0.4	7.2	P1
34	7.2	0.4	7.3	P1
35	6.7		7.4	P1
36	6.5		7.0	P1
37	6.9		7.3	P1
38	7.0	0.4	7.2	P1
39	7.4		7.2	P1
40	6.2	0.3	7.1	P1
41	6.1		7.1	P1
42	5.7		7.1	P1
43	6.1		7.2	P1
44	6.2		7.0	P1
45	6.6	0.1	7.0	P1
46	6.7		7.0	P1
47	6.2		7.0	P1
48	6.7		7.1	P1

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
49	6.3		7.2	P1
50	5.6		7.2	P1
51	5.8		7.2	P1
52	5.8		7.2	P1
53	6.0		6.8	P1
54	5.8		6.9	P1
55	6.0		7.1	P1
56	6.1	0.3	7.0	P1
57	5.7		7.2	P2
58	5.4		7.1	P2
59	5.3		7.3	P2
60	5.1	0.6	7.2	P2
61	6.4		7.2	P2
62	4.4	0.6	7.0	P2
63	4.8	4.3	7.4	P2
64	4.9		6.6	E6
65	8.5	10.5	6.6	E6
66	13.6	9.8	4.0	E6
67	12.4	4.4	6.8	P1
68	8.6		7.0	P1

2. Silica Gel

Gamma Ratios: Feed = 6.6
Product = 2.1

Tonnage: 175 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Mr/Hr</u>	<u>Shipped To</u>
17	3.6	0.016		5	4	234-5
18	3.4	0.017	1835	5	4	234-5
19	3.6	0.006		5	3	234-5
20	3.1	0.004		5	3	234-5
21	2.8	0.013		4	3	234-5
22	2.9	0.021	480	6	3	234-5
23			Reworked Via			E6
24	3.4	0.006		4	3	234-5
25	2.7	0.020		4	3	234-5
26	2.8	0.002	1750	5	3	234-5
27	2.5	0.001		5	3	234-5
28	2.6	0.001		5	3	234-5

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C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr/Br</u>	<u>Shipped To</u>
29	2.8	0.001		5	3	234-5
30	2.8	0.001	420	5	3	234-5
31	2.7	0.002		5	3	234-5
32	3.1	0.071		5	4	Holding
33	6.4	0.001		6	7	Holding

D. Overall Waste Loss - % of Total Production:

U = 0.9%

Pu = 0.22%

E. Rework - % of Total Production:

U = 7.1% (Uranium product recycle at shutdown)

Pu = 8.7% (Predominantly out-of-spec Pu product via TK-L11 to HAF TK-E6)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	746	104A
Cell Drainage	80	101A
FP Waste	72	101A
Coating Waste	270	101C

B. LW Flows: 4.7

C. Tank Farm:

<u>Tank</u>	<u>Estimated Boil-off Rate, Gal/Ton</u>
101	12.3
102	3.8
103	0.6
104	8.3

Notes:

1. The HAF rate was maintained at 3.0 CF until plant shutdown at 0545 on Feb. 20, except as follows:

(a) Between 2100 on Feb. 18 and 1230 on Feb. 19 the HAF flow was off because of failure of the IBS Column pulsar.

(b) Immediately prior to plant shutdown, approximately 17 tons of "cold" uranium product was processed as HAF.

2. Flowsheet

- (a) On Feb. 11 the volume of carbonate solution in the turbomixer tank was increased from 1600 to 2700 gallons. On Feb. 13 the frequency of the turbomixer tank carbonate changes was decreased from three times daily to once daily.
- (b) On Feb. 13 the HSS was increased from 56 to 63 flows. The flow was further increased to 71 and 74 flows on Feb. 15 and 16, respectively.
- (c) On Feb. 13 the 2AS was increased from 4.35 to 4.60 flows. On Feb. 17, this flow was returned to 4.35.
- (d) On Feb. 13 the 2BP was reduced from 1.60 to 1.46 flows.
- (e) On Feb. 14 the Gl-K₂CO₃ flow was increased from 3.4 to 4.7 flows.
- (f) On Feb. 14 the 3WB was increased from 37.5 to 39.8 flows.
- (g) On Feb. 16 the IBS flow was reduced from 19.7 to 18.6 flows.

3. Solvent Extraction

The gamma activities of the 2AF and 2BP streams decreased about 3 fold, and the 2UF uranium product gamma level decreased 1.3 fold as a result of increasing the HSS from 56 to 74 flows over the period Feb. 13 - 16. The Pu product activity also responded, but was still higher than normal.

The HAW plutonium loss was maintained at about 0.2%. In spite of the high HA Column saturation and the increased HSS flow, the HAW neptunium loss improved 2 to 3 fold. The IBU plutonium loss gradually rose from 0.28 to 0.52 per cent apparently because of increased plutonium recycle to the HAF. The 2DW loss was decreased from about 10 to 6 per cent in preparation for the plant shutdown and scheduled neptunium recovery run.

Continued IC Column instability tendencies were correlated with routine regeneration of the No. 2 demineralizer unit, on an every-other-day schedule. Investigation of this problem is proceeding as described under Note 5, below.

Persisting high ICW and IOO gamma activity levels led to a special program of HAP and HSP sampling. Intent of these samples was to estimate the DF performance of the HS Column. On Feb. 11 a 100-ml. HAP was thieved from the top of the HA Column. Laboratory inspection showed the presence of a considerable concentration of black solids and an unusual green color to the supernate. A second bulk HAP sample, taken on Feb. 16, had the same appearance. HSP samples, both by normal

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Gilson sampling and by thief from the crane, appeared normal. Investigation of the solids is underway in the laboratory. Appreciable Pu, U, and Zr-Nb is associated with them.

A special sample thieved by crane from the HA Column pulse leakage pot on Feb. 16 was found to be all organic phase, analyzing as follows:

Gross gamma, uc./gal	- 2.4×10^6
Zr-Nb, uc./gal.	- 1.8×10^6
Bu, uc./gal.	- 8.4×10^4

This sample had been scheduled to investigate the possibility of HAW leaking via the pulse leakage line to TK-G1, and thereby contributing to the high IOO gamma activity. Lack of an aqueous phase and the absence of cerium indicate no HAW to be present. The activity level is high, however; assuming 0.2 gal/min. pulse leakage, about 10 - 20% of the input activity to the No. 1 Solvent Treatment System (TK-G1) is introduced via this source.

Emergency shutdown of the plant was necessitated by failure of the IBS Column pulser on Feb. 18. Following pulser replacement, processing of "hot" HAF was completed on Feb. 19 and scheduled shutdown of the plant was commenced.

4. Solvent Treatment

The gamma activity in the IOO decreased from 20,000 uc/gal. to about 12,000 uc/gal. during the report period, with the improvement correlating timewise with the general improvement in solvent-extraction performance noted above (Note 3). Solvent treatment system changes were also made during this period, but with no obvious and appreciable improvement resulting from any particular change.

- (a) Recirculation of IOF to TK-G1 was resumed, and varied to maximum flows.
- (b) The K_2O_4 addition to TK-G1 was increased to 3 times flowsheet.
- (c) The IO Column interface was purged to waste at a frequency of 3 times per week.
- (d) The IO Column pulse frequency was increased from 45 to 93 cycles/min.
- (e) The aqueous phase volume in the Turbomixer G7 was increased from 1600 to 2700 gallons and the change frequency was decreased from once per shift to once per day.

(f) The Turbomixer G7 speed was increased from 400 to 500 RPM.

To assist in the investigation of the possible introduction of HAW to TK-G1 via the HA Column pulse leakage line, a sample of TK-G1 aqueous phase (IOB) was isolated in TK-G8 and sampled. Laboratory investigation indicated no Am-Cm or Cs; hence, no HAW.

Laboratory investigation of a solvent sample from the top of the ID Column indicated no further DF from acid washing or centrifugation. Also, laboratory washing with carbonate yielded a DF of about 3, very close to the DF achieved by the Turbomixer. This evidence again points out the poor efficiency of the permanganate contactor TK-G1.

5. Water Demineralizer

During the report period, a correlation of poor "C" Column performance with regeneration of the No. 2 Demineralizer unit was noted. Investigations of this problem were conducted by operation of semiworks "C" Columns using Purex water samples and by normal water analyses. Semiworks studies of sanitary water (demineralizer feed), cation bed product water, and fully demineralized water (anion bed effluent) indicated the "C" Column problem to be caused by the anion resin bed of the No. 2 unit. This bed contains the Duolite resin which was not completely renewed during the demineralizer renovation program of April 1960. Replacement of this bed with Malsite WBR, the same brand in service in the No. 1 unit, was recommended.

6. Waste Treatment and Acid Recovery

The IW rate was below flowsheet primarily due to plugging Sp.Gr. dip-tubes and a tendency to overconcentrate rather than have excessive IW flow. Partial plugging in the waste line to Tank 241-A-101 was cleared by jetting Tanks F16 and F18 at the same time.

A faulty F16 Thermocouple was replaced with a new flexible jumper which will allow greater utilization of a single jumper design and reduce the number of spare units necessary for plant operation.

Because of Minor Construction work with tie-ins to the new cooling towers (emergency water source), the tank farm vapors were routed to the contact condensers.

Sodium analyses of recent samples taken from the underground storage tanks are tabulated below. Also tabulated are values calculated on the basis of building records.

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Tank	Na Concentration, M	
	Sample Result	Calculated
101	4.4	6.9
102	6.3	5.0
103	8.25	8.7
104	2.8	6.0

7. Fission Product Recovery

Following completion of the last lead-carrier sulfate precipitation run in the series reported last week, the E2 centrifuge failed while rotating at a normal 1500 RPM. "Cold" side testing of the machine revealed that the skimmers operated properly and were in the "out" position at failure; the electric motor had not failed; and the bowl would not rotate either forward or reverse even when the normal horsepower of the motor was doubled.

On Feb. 14 the E4 centrifuge was returned to service. This machine has not recently been used because of a failed neoprene hose to the spray nozzles, stuck skimmer, and noisy operation. However, a new spray jumper was installed remotely and the skimmer was freed by putting considerable force on the mechanism. Also, the noisy operation was corrected, at least in part, by replacing a loose and leaking feed jumper.

The carbonate product stored in TK-E1 was processed through the oxalate and concentration steps, and 1.37 megacuries of Sr 89-90 was sent to the 002 tank in the 244 CR Vault. The composition and decontamination factors achieved for the product solution were as follows:

Component	Curies/Gal.	DF	Lb./Gal.
*Sr 89-90	1240		
Ce 141-144	790	5	
Zr-Nb	880	5	
Ru-Rh	10	100	
HNO ₃			0.28
Fe			/0.003
SO ₄			/0.02

*Sr-89/Sr-90 ratio not yet available.

During the oxalate step, the feed was butted with nitric acid to test the effect of acid concentration on strontium recovery. The oxalate precipitation was carried out at 1.3 M nitric acid as compared to prior runs at 0.70 to 1.0 M. Analyses across the oxalate step and concentration step showed that 80 per cent of the strontium in the carbonate product was recovered as final product as compared to the usual 50 per cent. The over-all cerium DF between LNW and oxalate product was 14 while over-all cerium DF between LNW and final product was 5. The flush of the E3 vessel between the oxalate run and the concentration run removed 5.4 megacuries of cerium. The loss of DF between the oxalate run and the concentration run and the large amount of cerium removed from the E3 vessel with a single flush indicates some cross-contamination is still occurring. The number of E3 vessel flushes between the oxalate and concentration steps will be increased in the future.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 2/20/61 TO 2400 ON 2/28/61

During the report period, the plant was shut down and scheduled maintenance work was being accomplished. Process notes of interest for the period include the following:

1. Solvent Extraction

The plant was shut down on Feb. 19 and 20. The shutdown procedures were designed to prepare for a scheduled neptunium recovery run and for maintenance operations which were accomplished, as follows:

- (a) The HAW control valve (DOV) was replaced.
- (b) The 2AF pump was replaced.
- (c) The 2A Column, which originally contained Fluorothene Raschig Rings was replaced with a new column containing stainless steel nozzle plates. Prior to its replacement, the old column was flushed three times with 60% HNO₃ at about 70° C. After installation the new column was flushed with 10% sodium carbonate at 50° C. The new cartridge nozzle plates have 3/16-in. holes and 20% free area and are spaced at 2 and 3 inches in the extraction and scrub sections, respectively.

2. Plutonium Concentration

Following the shutdown on Feb. 20, N-Cell ion exchange columns were stripped, and the resin was removed and thrown away. Bags of waste resin were reading 300 mr/hr. which is abnormally high, apparently reflecting an unusual gamma burst at shutdown. This resin had been in use for 5 weeks. Dismantling and removal of the resin receiver, resin reservoir, and push tank was begun. Replacement with newly designed equipment will be completed during the shutdown.

3. Solvent Treatment

The gamma activity in the IOO was reduced from about 10,000 uc/gal. at plant shutdown to about 3,000 uc/gal. by recycling solvent in G Cell with equipment operating at a 3.0 CF. Since the permanganate head tank was being used for Head-End flushes, permanganate was used for only about 10 hours during the clean-up operation.

In preparation for its replacement during the shutdown period, the IO Column was flushed with two series of flushes as follows:

- (a) 5% oxalic acid - 2% HNO₃.
- (b) 5% caustic - 2% tartaric acid.

This column contained Fluorothane sieve/ plates. It will be replaced with a spare (which has previously been in service) containing stainless-steel nozzle plates.

To reduce the possibility of a loss of pumping capacity of the Tank G1 recirculation pump due to solids, a modified G1 pump was installed which has the pump intake approximately 6 inches from the tank bottom.

4. Demineralized Water

Because of recent problems involving the effect of demineralized water on "C" Column performance (see page 26), a complete water analysis was obtained of the effluents of both demineralizer units. The analyses showed that the cation units were removing all of the metallic ions. These analyses, coupled with the 300 Area column tests and the fact that the cation capacity remains in excess of 300,000 gallons, indicated that the cation beds are still performing satisfactorily.

The analyses also show that the #1 anion unit, containing Malcite WBR resin, is allowing 68% of the sulfate and 3% of the chloride to be transmitted into the product water. The pH of this effluent is a correspondingly low 3.5. The #2 anion is transmitting no sulfate or chloride and the pH is 4.6 (the slightly acidic pH being caused from weak acids). This unit is charged with Duolite A-30 resin.

From the analytical results and the "C" Column tests, the conclusion has been reached that the #2 anion unit is removing anions satisfactorily but is apparently introducing an unknown substance which affects column operation. On the other hand, the #1 anion unit is not noticeably contributing to the column problem, but it is not operating as advertised in removing anions.

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To better evaluate the situation arrangements have been made to send samples of all four resin beds and three water samples to the Malco Chemical Company for analysis and testing. As previously noted (p. 26), the decision has also been made to replace the #2 anion resin with new Nalcite WBR during the current shutdown.

5. Waste Treatment and Acid Recovery

Three tube bundle failures were detected during the shutdown: E4 East, after 11.5 months of service; F6 West, after 4.2 months of service; and F11 West, after 26 months of service.

In preparation for tube bundle changing, the E-F11 Concentrator has been flushed three times with 5% caustic. Failure of the empty-out jet in E-F6 Concentrator (possible hole in the jet dipleg) has prevented flushing in that vessel.

Approximately 3,410 gallons of IWW waste were transferred to Tank 106-A during the period Feb. 24 - 26, for warm-up purposes. The total IWW waste in this vessel is now 4,410 gallons.

The vent system filter was replaced with a unit of newer design which contains a top layer of one foot of 9 lb./cu.ft. 115K Fiberglass rather than the original one inch of 1.7 lb./cu.ft. AA Fiberglass. Below the top layer, the new unit contains one foot of 6 lb./cu.ft. 115K, 6 inches of 3 lb./cu.ft. 115K, and one foot of 1.5 lb./cu.ft. 115K on the bottom. The use of 115K Fiberglass for the top layer adds moisture resistance to the filter with a sacrifice of only about 10% of the efficiency.

The original condenser vent jet, rated at 288 scfm of air at 50 inches of water vacuum, was replaced with a new model, rated 350 scfm at the same vacuum. However, following installation, operability testing showed no noticeable vent vacuum improvement with the new jet. Consequently the new jet was removed for testing and the original was re-installed.

Other vent system maintenance included replacement of the F1 Condenser with a spare. This change was necessary because of severe corrosion of the original condenser damage.

6. Fission Product Recovery

A series of strontium recovery runs was performed just prior to the building shutdown. The series consisted of two lead-carrier sulfate precipitations, instead of the usual four, in order to mesh with cesium and other building maintenance schedules. On Feb. 25 during the

concentration run the E1 Centrifuge failed while rotating at 750 RPM. "Cold"-side tests indicated the failure was similar to the E2 Centrifuge failure described on page 27.

The strontium product solution processed prior to the centrifuge failure was sent to TK-002; the rest of the run was sent to waste. The product solution sent to TK-002 contained 156,000 curies of Sr 89-90 with the following composition and decontamination factors:

<u>Component</u>	<u>Curies/Gal.</u>	<u>DF</u>	<u>Lb./Gal.</u>
Sr* 89-90	380		
Ce-141-144	260	6	
Zr-Nb	400	4	
Ru-Rh	6	60	
HNO ₃			0.276
Fe			/0.004
SO ₄			/0.016

*Sr 89/Sr 90 Ratio not yet available.

The Sr 89/Sr 90 ratio for the production run reported last week was 10.9. Thus 126,000 curies of Sr 90 was sent to TK-002 last week. The strontium inventories as of 2/24/61 were as follows:

	<u>Sr 90 - Curies</u>
TK-003	144,000
TK-002	538,000
TK-F8	56,000

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 3/1/61 TO 2400 ON 3/9/61

During the report period the plant was shut down with respect to normal uranium processing. A scheduled neptunium recovery run was conducted and necessary maintenance and other operations were accomplished. Process notes of interest are presented below.

1. Feed Preparation

The A2 and B2 Silver Reactors were regenerated on March 2 and March 4, respectively. Since the last regeneration, A2 had processed the off gases from 569 tons of uranium; and B2, the off gases from 563 tons of uranium. Neither reactor had shown signs of iodine breakthrough at the time of shutdown, but regenerations were advisable in anticipation of the next scheduled run period.

A test was performed to establish the validity of the uranium and plutonium concentrations which are normally reported for the basic coating waste solutions. The probability of non-representative samples of such basic solutions has been theorized. In preparation for the test, each dissolver was rinsed in a routine manner after the second cut. After the rinse, the heel in each dissolver was covered with demineralized water. Meanwhile, the Coating Waste Tank D2 was cleaned by first, digesting with hot 2 M sodium hydroxide, and secondly, digesting with hot 2 M nitric acid. After the D2 Tank had been cleaned, the water covering the heel in each dissolver was jettied separately to the D2 Tank. Each of these three special rinses (one from each dissolver) was (a) sampled for uranium and plutonium, (b) acidified to 2 M nitric acid, and (c) resampled every three hours for U and Pu while digesting at elevated temperature. A comparison of these results to normal coating waste losses will be made and reported when all the data is available.

2. Plutonium Ion Exchange

During the current shutdown a new resin addition and push system was installed. The new system eliminates three air-operated Jamesbury valves. Sight glasses have also been lengthened to permit better

push control and minimize the addition and removal of resin. Other N-Cell maintenance included installation of a resin sampling line between the XC Column and the 3-way Durco valve. This will provide a means for determining if the resin is being stripped properly. Piping was also installed between the XAW sample pot and the jet to J1 which will permit continuous addition of ANN direct to the N-Cell backcycle waste stream.

3. Solvent Treatment

During the neptunium recovery run, flushing and removal of the IO Column was also scheduled. Consequently, for the recovery operation, solvent washing was conducted as necessary in the Turbomixer G7, only.

On March 3, the old IO Column (containing Fluorothene sieve plates) was replaced with a spare containing the following stainless-steel cartridge assembly:

- 10% free area nozzle plates
- 1/8-in. holes on 0.4-in. equilateral triangular spacing
- 4-in. plate spacing
- 0.05-in. nozzle indentations
- Nozzles pointing down

This spare had been in service prior to installation of the Fluorothene cartridge column during October of 1956. In preparation for its current installation, the spare was modified by adding a nozzle to the bottom disengaging section which will contain an interface float.

In preparation for start-up of normal processing, TK-G1 was flushed twice with 4000-gal. volumes of 5% oxalic acid - 2% nitric acid at 50 - 60° C. Tank calibration showed a volume discrepancy of only about 100 gallons after these flushes.

Also, prior to start-up, the IOO was reduced in gamma activity from nearly 10,000 uc./gal. to about 3500 uc./gal. by recycle through the treatment system.

4. Demineralized Water

During the shutdown all four resin beds were inspected. Measurements revealed that during the past ten months, the resin loss has been less than 4%. Samples of all four resin beds along with samples of sanitary water and water effluents from the No. 1 cation and anion units were taken and sent to the Malco Chemical Company for evaluation.

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On March 1 the resin from the No. 2 anion unit (Duolite A-30) was removed and replaced with 36 ft³ of new Malco WBR weak base anion resin. The new resin was backwashed, exhausted with sulfuric acid, and given a double regeneration prior to being put into service.

5. Neptunium Recovery Run

(a) Summary

The neptunium recovery run was started (3WB on) at 0300 on March 1 with an estimated 0.83 kg of neptunium available for recovery. The run was completed at 1800 (2BP off) on March 8 with the recovery of 0.74 kg. The standard "reducing" flowsheet was used in the Final Plutonium Cycle equipment (2A and 2B Columns).

The recovery operation was started and completed at a processing rate of ~ 60% of normal due to erratic operation of the 2B Column. Excessive 2BW losses required adjustments to the flowsheet. During all phases the 2BW losses remained 2 to 4-fold above normal despite corrective action such as increasing the L/V in the 2BP Column.

A laboratory analysis of 2BW and 2BP samples taken during Phase I showed the $Np E_2^{+}$ in the 2BW and 2BP streams were 0.24 and 0.024 respectively. The laboratory later contacted a 2BW sample (containing negligible uranium) with equal volumes of various aqueous solutions and obtained the following E_2^{+} data:

<u>Aqueous Phase</u>		<u>$Np E_2^{+}$</u>
Plant 2BX	Sulfamic	2.4
Plant 2BX - 0.005 M	Sulfamic Acid	0.62
Plant 2BX - 0.11 M	Ferrous Sulfamate	0.14
Plant 2BX - 0.05 M	H ₂ SO ₄	0.001

These data indicate that the high 2BW losses are related to the organic quality rather than the neptunium valence.

The gamma levels of the 2BP stream during Phase I were about 10 fold below normal while other streams gamma levels were normal. The lower 2BP gamma level suggest that the DF with the new stainless steel cartridge in the 2A Column may be superior to the old Fluorothene Raschig-Ring packing. Although the gamma level of the 2BP during Phase I was 10 fold below normal, the decrease of the 2BP gamma level during Phase II, and the final 2BP gamma level during Phase III were normal.

(b) Material Balance*

Np in Plant	0.84
Np in 3WB	0.83
Np Processed as 3WB	0.82
Np Load-out	0.74
Np Recycled	0.10

(c) Typical Solvent Extraction Losses*

	<u>% of 3WB Processed</u>
2AW	1.7
2BW	7.4

(d) Product Analytical Results

	<u>Np, g/l</u>	<u>HNO₃, M</u>	<u>% Contamination of Np Pu</u>	<u>G/Np, AT x 10⁷</u>
Principal Load-out	22.2	10.2	18.2	58
First Flush	9.29	11.3	6.8	76
Second Flush	2.71	10.9	26.2	86
Third Flush	0.41	11.3	19.4	102
Fourth Flush	0.63	11.9	19.6	44
Fifth Flush	0.96	13.1	10.4	40

*All losses recycled for recovery during normal processing operations.

6. Fission Product Recovery

During the report period the E Cell equipment was decontaminated in preparation for the Cesium recovery run. The flushes consisted of:

- (a) 3 M HNO₃.
- (b) 2% Tartaric Acid - 5% Sodium Hydroxide.
- (c) 5% Oxalic Acid - 3% Nitric Acid.

The E1 vessel received 6 M nitric acid flushes, in addition to those listed above, to dissolve solids observed in the load-out lines. At the completion of the chemical flushes, the last water flush transferred from E1 to E3 through a plastic tubing jumper in the load-out station had a surface reading on the tubing of 300 mr./hr.

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The 156,000 curies of Sr 89-90 sent to TK-002 (reported on page 32) had a Sr 89-90 ratio of 7.8. This brings the total Sr 90 inventory in TK-002 to 555,000 curies.

The failed B4 Centrifuge was removed on March 2 and a rebuilt replacement was installed on March 6.

Appendix --- Process Summary Through March 12, 1961

Start-up of normal processing was commenced at 2225 (HAF on) on March 10 at 3.0 CF. The new 2A Column initially flooded and required drastic pulse frequency reduction for correction. Also, it was necessary to replace the LWF pump (TK-F7) and the 2DF pump (TK-K1) over the week end.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 3/10/61 TO 2400 ON 3/15/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.7	4.2	-	-	0.004	0.07
Final	3.0	2.9	6.0	6.7	0.012	-
Ion Exchange	-	1.0	-	0.2	-	-
Overall	6.7	8.1	6.0	6.9	0.016	0.07

II. Production

A. HAF Processed: 122 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
1	4.9		3.3	R6
1a	4.9		3.5	P1
2	4.3	3.2	6.4	P1
3	2.5		7.0	P1
4	1.7	4.7	7.0	P4
5	1.9		7.0	P4
6	1.6	2.2	7.0	P4
7	1.0		4.8	R6
8	2.3		7.0	P2
9	2.7		7.1	P1
10	2.2		7.2	P4
11	2.2	3.9	7.3	P1
12	1.7		7.3	P4
13	2.0		7.0	P4

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
14	2.4	4.9	7.1	P3
15	2.8	3.8	7.3	P3
16	3.8	2.9	6.9	P3
17	3.6	2.3	6.9	P3
18	3.6	8.4	6.4	P1
19	3.7	4.9	6.8	P1

2. Silica Gel

Gamma Ratios: Feed = Approx. 3.5
Product = 1.7

Tonnage: 170 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Pu, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
1	2.0	0.035		2	4	234-5
2	1.9	0.009		4	2	234-5
3	1.7	0.005		5	1	234-5
4	2.4	0.004		5	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.54%

Pu = 2.4%

E. Rework - % of Total Production:

U = 6.6% (U Rework at startup via TK-K5 to HAF Make-up TK-E6)

Pu = 23.5% (Predominantly L-Cell Package Flushes and Sumps)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	1687	104-A
Cell Drainage	638	101-A
FP Waste	258	101-A
Coating Waste	270	107-C

B. LW Flows: 5.0

C. Tank Pans:

<u>Tank</u>	<u>Estimated Boil-Off Rate, Gal/Min.</u>
101	12.0
102	2.7
103	0.7
104	9.6

Notes:

1. (a) Normal processing was commenced (HAF on) at 2230 on March 10 at 3.0 CF. Following initial rework of "cold" uranium remaining from the shutdown, processing of "hot" HAF was commenced at 0830 on March 11.
- (b) On March 13 about 5 tons of uranium as 2DF was recycled to the HAF in order to permit replacement of a failed 2DF pump.
- (c) On March 13 the HAF rate was reduced to 2.4 CF. On March 14 the rate was returned to 3.0 CF and was continued at this level throughout the remainder of the report period.

2. Flowsheet

- (a) The start-up flowsheet was the same as that in effect at the Feb. 19 shutdown, except:
 - (1) The HSR-HNO₃ was 4.0 instead of 8.5 flows;
 - (2) The IOO temperature was 46° C instead of 48° C; and
 - (3) The 2BF flow was to the L-Cell Package instead of to the N-Cell Ion Exchange Unit.
- (b) On March 10 and 11 the 2AS flow was varied between 4.3 and 1.4 flows. On March 12 the 2AS flow was reduced from 4.3 to 3.1; on March 13 from 3.1 to 2.5.
- (c) On March 12 the 2AX was increased from 4.0 to 4.7 flows.
- (d) On March 12 the 2BF flow was diverted from the L Cell Package to N Cell.
- (e) On March 15 the 2DF-Fe was increased from 0.15 to 0.35 flows. The flow was increased to 0.53 flows on March 16, but was returned to 0.35 flows about 16 hours later.

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- (f) On March 16 the 2DF-HNO₃ stream was switched from Purex to UO₃ Recovered Acid.
- (g) On March 16 the IDO temperature was reduced from 46 to 44° C.
- (h) On March 16 the 2BP was increased from 1.54 to 2.32 flows.
- (i) On March 14 the uranium metal feed solution was made 0.0005 M Hg(NO₃)₂.

3. Feed Preparation

On March 14 the addition of 0.0005 M mercuric nitrate to each dissolver batch was resumed. This action was prompted by the rising iodine emissions which were caused by processing metal which had been "cooled" less than 105 days.

4. Solvent Extraction

The plant start-up and operation during the report period have been characterized by (a) high 2AW plutonium losses, (b) excellent HAW neptunium and plutonium losses, and (c) an increase in gamma levels approximately to the values typical for the last operating period.

The high 2AW losses were caused principally by lack of definition of optimum operating conditions for the new 2A Column cartridge (see p. 29), and also by occasionally low 2AF acidity. At plant start-up a severe flood developed in the 2A Column scrub section which was not broken until the pulse frequency had been reduced from 75 cycles per minute to 45 cycles per minute, and the 2AS flow had been temporarily reduced from 4.3 to 1.4 flows. At these conditions the column was stable, but excessive losses persisted. Consequently, the 2AX flow was increased from 4.0 to 4.7, and the 2AS flow was decreased to 3.1. Also, a condition of erratic and low 2AF acidity was corrected. The losses, while reduced, were still 2 to 3 fold above normal, so further 2AS flow reductions were in progress.

The excessive 2AW losses caused the plutonium levels of intercycle streams to increase about 2 fold. This increased plutonium level tended to aggravate the 2AW loss problem and also, when coupled with the higher 2AX flow, increased the plutonium hold-up in the 2B Column substantially. The 2B Column plutonium reflux was brought under control by increasing the 2BP from 1.54 to 2.32 flows. In addition, the high plutonium levels caused the IBU Pu loss to reach 1.0%, resulting in a temporary breakthrough of Pu to the uranium product. The uranium product, however, met the plutonium specification after increasing the flow of ferrous sulfamate to the 2DF.

Following plant start-up, the acidities of the HSS, HSR, and the 2AF were generally below normal, and control was poor. Ultimately this condition was improved by repair of the acid header pressure controller (Foxboro Consotrol). Even after control was restored, it was necessary to operate with rotameter settings almost double those in effect at the time of shutdown in February. This behavior was apparently a continuation of a pattern set in January and February during which time gradual rotameter increases of about 20% were necessary to maintain flowsheet acidities. As previously noted, 2AW losses responded to improved control of the 2AF acidity. Too, plutonium and neptunium losses were reduced to very low values in the HAW.

Other items of interest to solvent extraction were as follows:

- (a) Process control has been hampered since start-up by temporary loss of the 2AW alpha monitor, and the 2BF and 2EU gamma monitors.
- (b) The 1NF and 2DF Pumps were replaced because of leaking seals which developed during start-up. No production was lost while replacing the 1NF Pump, but replacing the 2DF Pump required recycling about 5 tons of uranium from the 2DF to the HAF stream.
- (c) The 2AF Tank, 2A Column, and 2B Column were flushed with a 60° C solution of 60% nitric after the Birch Recovery run and just prior to plant start-up.
- (d) The capacities of the IC and 2E Columns have returned to normal following the replacement of the No. 2 Anion demineralizer resin (see p. 35).
- (e) The addition of Permacol to the process steam at a concentration of 5 ppm was commenced on March 15.

5. Solvent Treatment

Shortly after start-up the gamma activity in the IOO rapidly increased to about 10,000 uc/gal. When rates were reduced to a 2.4 CF the activity dropped to about 8,000 uc/gal; however, after returning to 3.0 CF, the activity leveled out at about 10,000 uc/gal. During this period the solvent treatment gross gamma DF averaged about 3, nearly entirely across the TK-G1 contactor.

Flowsheet conditions were maintained throughout the week except IOS at 36 flows was added through the aqueous indicator dipleg in the top of the IO Column and discharged to waste without recirculation (as described on p. 19).

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Beckman radiation readings on the G3 Centrifuge indicate very little activity is being removed from the solvent by centrifugation since adopting the new IOS system and since replacing the IO Column.

6. Fission Product Recovery

(a) Cesium Recovery

Cesium recovery runs were initiated on March 9 with the pumping of 4,000 gallons of tank farm supernatant solution from the 103-A Tank to the Purex Fission Product Test Plant. The solution was centrifuged and cask loading was started late on March 9. Leaks in threaded stainless steel fittings in the fill lines were encountered which persisted despite repair efforts. Also, the Nyogen plastic portions of the fill lines collapsed under vacuum. A new fill line assembly was fabricated from Saran tubing with all stainless fittings welded and cask loading resumed on March 13. The in-canyon jet caused some difficulty through the remainder of the run, requiring occasional back-flushing and air-purging to maintain flow. Also, leaks around the Snap-tite fittings, which are part of the cask, caused difficulty. The solution enters the cask at the top and is withdrawn from the bottom of the cask through a dipleg. Under normal operation the air space above the solution in the cask is at 10 inches of mercury vacuum. Leaks around the connecting Snap-tite fittings allowed air to enter the cask with a resulting decrease in solution level in the cask. This required interruption of the filling operation to add water so that the Decalco would remain covered. During periods when all of the plumbing operated satisfactorily, solution was passed through the cask at the rate of 4.5 GPM with 10 inches of mercury pressure drop across the bed.

A total of 33,400 curies of cesium 137 were passed through the cask with 29,900 curies remaining on the Decalco in the cask. Breakthrough was easily detected by taking radiation readings on the inlet and outlet lines. Normal surface readings on the inlet and outlet lines were 25 to 30 and 5 to 8 R/hr., respectively.

Data obtained on the loaded cask thus far is as follows:

Cask Surface Temperature	- 10° C
Cask Solution Temperature	- 10° C
Cask Pressure	- 1 psi

Radiation Readings -

Dose Rate at 6 feet	- 5 Mr/Hr.
Dose Rate at 3 feet	- 14 Mr/Hr.
Maximum Surface Dose Rate	- 100 Mr/Hr.

(b) Centrifuge Failures

Since failure of the last E2 Centrifuge, considerable effort has been expended in efforts to define the cause of the relatively high failure frequency. HIO personnel had previously irradiated centrifuge oil and found it satisfactory under the radiation fields encountered in the canyon. Their tests were repeated with bearings in the presence of oil, and bearings in the presence of grease to determine if there might be pitting at the oil or grease interface or if they might be damaged by some other unknown phenomena. At doses up to 2×10^7 R no noticeable effects were observed. At doses of 1.5×10^8 R some brown discoloration of the bearing, at areas not covered with oil or grease, was noted.

A spare neoprene buffer ring, which in the centrifuge assembly supports the weight of the bowl and drive head assembly, was checked for radiation and thermal stability. It was concluded that the ring was satisfactory in the radiation fields encountered but would harden rapidly under normal operating temperatures. As the buffer hardens, greater load and shock is transmitted to the bearing. Chemical Development Operation recommended the neoprene rings be replaced with Hypolon, which has excellent radiation and thermal stability.

During run-in of the replacement E4 Centrifuge, it was found that the oil temperature climbed to 85° C at 1500 RPM operation and cooled to 65° C at 750 RPM operation. The observed temperatures are at the upper limit of the temperature range for which the lubricating oil is recommended, and above the temperature range for which neoprene is suitable. An oil that is satisfactory for higher temperature service was obtained and is being used in the replacement E4 machine. However, production commitments did not allow time to replace the neoprene buffer rings. The replacement E4 machine was equipped with a temperature element to monitor bearing temperature during canyon operation, but the instrument would not function after installation.

In addition to defining the causes of centrifuge failure, efforts have been underway to meet the immediate needs for replacement machines. An engineering study was completed which indicated that B-Plant centrifuges can be modified to be interchangeable with Purex centrifuges. The study also indicated that it was not practical to attempt to modify Purex organic centrifuges for this service.

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Appendix --- Process Summary Through March 19, 1961

On March 17 the processing rate was increased to 3.4 CF.

Plutonium levels in the intercycle streams continued above normal because of internal recycle. A program of reduction of 2AS flow and increase the 2A Column pulse frequency was commenced in an effort to further reduce 2AW losses.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 3/17/61 TO 2400 ON 3/23/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.3	4.0	-	-	0.002	0.02
Final	3.1	3.1	7.5	3.4	0.004	-
Ion Exchange	-	1.1	-	4.0	-	-
Overall	6.4	8.2	7.5	7.4	0.006	0.02

II. Production

A. HAF Processed: 214 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
20	4.2	1.6	6.7	P1
21	3.7	2.5	7.2	P1
22	3.4	1.4	7.0	P1
23	3.2		7.2	P1
24	3.5	1.8	7.1	P1
25	3.6		7.3	P1
26	4.1	3.2	7.1	P1
27	3.7		7.3	P1
28	4.2	2.8	7.3	P1
29	4.5		7.3	P1
30	5.2	4.3	7.4	P1
31	4.8		7.3	P1
32	4.9	1.4	7.2	P1
33	5.2		7.0	P1
34	5.8	3.2	6.8	P1

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
35	5.8		6.8	Pl
36	5.7	3.8	6.7	Pl
37	6.1		6.8	Pl
38	6.3	3.4	7.0	Pl
39	7.1		6.9	Pl
40	7.1	4.2	7.0	Pl
41	7.2		7.0	Pl
42	7.0		7.1	Pl
43	6.9		7.3	Pl
44	6.8	2.2	7.4	Pl
45	6.4		7.4	Pl
46	6.6	1.8	7.2	Pl
47	6.3		7.1	Pl
48	6.3	1.3	7.4	Pl
49	6.8		7.5	Pl

2. Silica Gel

Gamma Ratios: Feed = 5.4
Product = 1.9

Tonnage: 150 Tons

C. Plutonium Product:

<u>Batch</u>	<u>Zr/Nb AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Pu, ppbp U</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
5	2.0	0.004		5	1	234-5
6	1.5	0.003		5	2	234-5
7	1.7	0.002		6	1	234-5
8	1.1	0.009		5	2	234-5
9	1.1	0.006		6	1	234-5
10	1.1	0.009	426	5	1	234-5
11	1.3	0.011		5	3	234-5
12	1.4	0.012		5	1	234-5
13	1.1	0.014		5	3	234-5
14	1.7	0.025		5	3	234-5
15	3.6	0.029	381	5	4	234-5
16	3.0	0.029		5	6	234-5
17	2.2	0.019		5	4	234-5
18	1.3	-		5	3	234-5

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D. Overall Waste Loss - % of Total Production:

U = 0.12%

Pu = 0.33%

E. Rework - % of Total Production:

U = Nil

Pu = 2.8% (Predominantly L Cell Package flush) via TK-L11 to HAF
Make-up TK-E6.

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	544	104A
Cell Drainage	84	101A
FP Waste	-	101A
Coating Waste	270	107C

B. ISW Flows: 4.1

C. Tank Farm:

<u>Tank</u>	<u>Estimated Boil-off Rate, Gal/Min</u>
101	11.1
102	2.3
103	1.0
104	10.6

Notes:

1. On March 17 at 0900 the HAF was increased to 3.6 CF.

2. Flowsheet

- (a) On March 17 the production rate was increased from 3.0 to 3.6 CF. The HSR-HNO₃, HAO, HSS, IBS, 2DF-Fe, 2AS, 2AX, 2EP, and all N-Cell flow rates, however, were not changed. In addition, the IBX, IBXP, IBXP-HNO₃, 2AF-NO₂, 2AF, and 2DB flow rates were only increased to the equivalent of 3.2 CF.

- (b) On March 19 the 2AS was reduced from 2.08 to 1.47 flows.
(Note: Flows for this and other changes noted below are on the basis of CF = 3.6.)
- (c) On March 20 the 2EX temperature was increased from 46 to 50° C. The temperature was increased to 55° C on March 21, but was reduced to 52, 50, and 47° C on March 21, 22, and 23 respectively.
- (d) On March 20 the IBS flow was reduced from 15.4 to 14.1 flows.
- (e) On March 21 the IOO temperature was reduced from 43 to 41° C.
- (f) On March 22 the 2DS was increased from 52.5 to 62.5 flows.
- (g) On March 23 the HAO was increased from 75.0 to 78.0 flows.
- (h) On March 23 the HSR-HNO₃ flow was increased ~ 10%.
- (i) During the week it was noted that the ratio of aluminum to fluoride in the XAF has been 3.4 since Jan. 28 instead of 2.0.

3. Feed Preparation

Subsequent to commencing mercuric nitrate addition to the metal solution (page 41), the iodine emission rate has dropped to an average of 0.65 curies per day. This is well below the Purex control limit.

4. Solvent Extraction

The gamma activities of the 2AF and 2DF streams gradually rose about 1.5 fold following the rate increase to CF = 3.6 on March 17. The gamma activity of the plutonium product was not significantly changed, but the uranium product gamma level increased nearly 2 fold. The higher gamma levels apparently resulted from biasing the flowsheet (particularly lowering the HBS and 2DS flows) in order to obtain maximum production. Although the uranium product gamma ratio frequently exceeded 2.0 after silica-gel treatment, the residual activity was about 75% ruthenium, and the material was therefore suitable for shipment.

The 2DS flow was increased to the flowsheet value during the week in an attempt to improve a slightly excessive 2DU neptunium loss and the uranium product gamma level. The higher 2DS flow required a higher 2DX flow, and in order to insure the stability of the

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2E Column the 2EX temperature was temporarily increased from 45 to 55° C. By the end of the report period the 2EX temperature was reduced to 47° C. Neither the uranium gamma level or the 2DU neptunium loss immediately responded to the increased 2DS flow, but data at this time are insufficient for a good evaluation.

Excessive intercycle plutonium levels, notably in the 3WB and in the 2AF, caused above-normal IBU Pu losses during the report period. This condition was finally corrected on March 23 following the discovery of a "short circuit" of a portion of the XAF flow to the 3WB Tank J1 (Note 5, below).

Control of other process losses was good. HAW Pu and Np losses, in particular, were excellent. The 2AW plutonium loss was decreased over-all from ~ 6.0 to ~ 1.0 per cent by reducing the 2AS flow and increasing the 2A Column pulse frequency from 55 to 75 cycles/minute.

The black particulate material found in special, large-volume HAF samples on Feb. 11 and on Feb. 16 (see p. 24) was determined by the Process Chemistry laboratory to contain molybdenum sulfide (probably, disulfide). This compound is a common dry powder lubricant with general applications similar to graphite. Investigations of possible origins and possible process effects is continuing.

5. Plutonium Concentration

On March 21 the new high capacity XAF metering pump was successfully put into service. The old pump is being retained as a spare.

Mechanical operation of the newly installed resin receiver, resin reservoir, and push tank has been satisfactory.

On March 23 the cause of recent persistently high plutonium levels in TK-J1 (3WB) and in TK-J5 (2AF) was discovered to be originating in N Cell, and was corrected. A Jamesbury valve located on an XAF bleed line in N Cell was found to be partially open and was allowing XAF solution to be returned directly to TK-J1 via the N-Cell waste jet. Within a shift after closing the valve the J1 AT returned to a normal operational level.

6. Solvent Treatment

The gamma activity in the IOG decreased from about 10,000 uc/gal to 7,000 uc/gal during the report period. The over-all dF has been about 3.0 of which the Turbomixer has been contributing a factor

of about 1.2. Solids build-up in the G3 Centrifuge, as indicated by the G3 Beckman radiation indicator, has been very slight since installation of the column containing the stainless steel nozzle-plates and since the use of single contact IOS, as described on pages 19 and 42.

To reduce radiation levels in R Cell, Tank R8 was flushed with 5% oxalic acid - 2% nitric. The activity level was decreased by a factor of 3.

7. Demineralized Water

On March 20 the sulfuric acid mix tank was replaced with the spare duplicate lead-lined tank. The two tanks have been alternately undergoing relining between failures. The current failure occurred after only three months of service, and the frequency of tank replacement indicates that it is becoming increasingly difficult to recondition the tanks in a manner which will insure safe and adequate service.

Since the current shutdown when the Duolite A-30 resin in the No. 2 anion unit was replaced, the water quality as indicated by plant "C" Column performance has been exceptionally good.

8. Fission Products

Cesium recovery operations continued during the report period with the filling of two more ORNL SIT casks. The second and third casks were loaded with 30,200 and 31,000 curies of cesium 137, respectively.

Filling of the second cask was hampered by leaking Snap-tite fittings and poor canyon jet performance. A total of 9,900 gallons of solution was passed through the cask of which 2,800 gallons was feed. The difference of 7,100 gallons resulted from jet dilution, addition of water to displace the air inleakage around the Snap-tites, and back-flushing of the canyon jet.

The third cask was filled with very little difficulty. Some back-flushing of the canyon jet was necessary. However, the troublesome leaks around the Snap-tite fittings were eliminated by flooding the well in the cask with water. This placed a few inches of water over all of the Snap-tite connections.

The ORNL liquid level probes in the SIT casks have performed exceptionally well. The probe consists of a small electrical heater and a thermocouple located in a stainless steel tube. When the probe is

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not covered with solution, the probe heater has sufficient capacity (3 watts) to heat the probe to 100° C. When the probe is covered with solution, the probe temperature is cooled to 5 - 10° C above solution temperature. The thermocouple temperature is read by a capacitrol instrument which operates a low-level and high-level indicator light.

The first and second cask solution temperatures and cask pressures have been under surveillance since the casks were loaded and sealed. The cask temperatures are essentially ambient. The pressures in the first and second casks are 10 inches of water and less than one psig, respectively. From the experience to date with Snap-tite leaks, the pressure determinations may be in error. The only way the pressures can be measured is by attaching a Snap-tite fitting and a gage to the vent nozzle. However, pressures can be calculated from gas analyses if hydrogen and oxygen are the principal gases formed by radiolysis. A gas sample from the first cask had the following composition 100 hours after being sealed:

Gas and Mole Per Cent

CO ₂	0.33	CO	/0.01
H	0.80	H ₂	/0.01
O ₂	19.9	H ₂	0.82
N ₂	78.1		

This composition should yield a cask pressure of around 5 inches of water. The pressure gage on the cask at the time of sampling would not read less than one psig (27.7 inches of water).

Appendix --- Process Summary Through March 26, 1961

Process performance did not significantly change over the week end.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 3/24/61 TO 2400 ON 3/30/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.3	3.8	-	-	0.002	0.05
Final	3.5	3.2	9.0	2.6	0.004	-
Ion Exchange	-	1.1	-	0.1	-	-
Overall	6.8	8.1	9.0	2.7	0.006	0.05

II. Production

A. NAF Processed: 203 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
50	6.4		7.2	P1
51	6.5		7.3	P1
52	6.7	2.4	7.3	P1
53	6.5		7.4	P1
54	6.7		7.3	P1
55	6.9		8.1	P1
56	7.0	1.0	7.5	P1
57	6.9		7.4	P1
58	6.7	0.5	7.6	P1
59	5.8		7.2	P1
60	7.9	1.4	7.6	P1
61	7.4		6.9	P1
62	6.8	0.8	7.5	P1
63	7.0		7.4	P1
64	7.2	0.5	6.8	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbm U</u>	<u>Tons</u>	<u>Shipped To</u>
65	6.4		7.2	P2
66	6.4	0.1	7.2	P2
67	6.2		7.3	P2
68	6.9	1.0	7.0	P2
69	7.2		6.9	P2
70	6.8	0.5	7.0	P2
71	7.0		6.7	P2
72	7.8	0.4	7.0	P2
73	7.8		7.1	P2
74	7.0	1.1	7.3	P1
75	8.1		7.2	P1
76	7.7	1.4	7.3	P2
77	8.4		7.3	P1

2. Silica Gel

Gamma Ratios: Feed = 7.0
Product = 2.3

Tonnage: 132 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppbm Pu</u>	<u>No. Cans</u>	<u>Average Mr/Hr.</u>	<u>Shipped To</u>
19	1.0	0.031		5	1	234-5
20	0.9	0.050	450	5	2	234-5
21	0.9	0.059		5	2	234-5
22	1.1	0.041		5	0	234-5
23	1.3	0.030		5	2	234-5
24	1.7	0.003		5	2	234-5
25	2.1	0.010	450	6	2	234-5
26	2.0	0.012		5	2	234-5
27	2.2	0.023		5	3	234-5
28	2.6	0.024		5	3	234-5
29	1.8	0.010		5	2	234-5
30	2.1	0.010	450	5	3	234-5
31	1.8	0.009		5	2	234-5
32	2.1	0.013		4	3	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.06%

Pu = 0.16%

E. Rework - % of Total Production:

U = Negligible

Pu = 1.9% (Rework of sumps and L-Cell Package material via TK-L11 to HAF Make-up TK-B6)

III. Waste Treatment**A. Volumes to UGS:**

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	610	104A
Cell Drainage	63	101A
F.P. Waste	33	101A
Coating Waste	270	107C

B. INW Flows: 3.0 Flows**C. Tank Pans:**

<u>Tank</u>	<u>Estimated Boil-off Rate, Gal./Min.</u>
101	13.0
102	2.0
103	0.4
104	9.6

Notes:

1. The HAF was reduced from 3.6 to 3.2 CF on March 30 at 1030.

2. Flowsheet

(a) On March 24 the IBS was increased from 14.1 to 15.4 flows.

(b) On March 24 the HSR-HNO₃ flow was increased ~ 10%.(c) On March 25 the GI-HNO₃ flow was increased from 1.7 to 3.4. This was further increased to 5.1 flows on March 26.

(d) On March 27 the HSS flow was increased from 6.2 to 7.0 flows.

(e) On March 27 the HSR-HNO₃ was increased ~ 50%.(f) On March 30 the plant rate was reduced from 3.6 to 3.2 CF. Only the HAF, HAX, IBXP, IBXP-HNO₃, ICX, 2HF-HNO₃, 2HF, and 2DX flows were decreased.

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3. Feed Preparation

On March 30, a failed Teflon gasket on the charging chute lid of the A3 dissolver was replaced with a Teflon-asbestos remote gasket which was installed on the chute itself. A problem of insufficient vacuum during dissolving in the A3 dissolver was corrected by this change.

4. Solvent Extraction

The gamma level of the 2IF and the uranium product gradually increased about 1.2 fold. The Zr-Nb/Al level of the 2AF and plutonium product increased about 2 fold principally as a result of correcting the problem of Pu recycle (see p. 50) and thereby decreasing the plutonium level of intercycle streams to normal. The plutonium product continued to meet all specifications, however.

The HSB flow was increased from 62 to 70 flows, but the First Cycle decontamination performance was not significantly improved. At the end of the report period the plant rate was reduced from 3.6 to 3.2 CF, but the HSB and 2IS flows were not decreased. This in effect increased the HS-BA and 2D Column scrub flows about 13%.

Waste losses generally have been excellent. The IBU plutonium loss decreased from 1.0 to 0.6% after reducing the plutonium levels of intercycle streams late in the last report period. The 2AW loss was deliberately increased from 0.8 to 2.5% by a slight decrease in the 2AX flow to aid 2A Column decontamination performance. Increasing the 2A Column pulse frequency over the range of 72 to 80 cycles per minute did not significantly improve the 2AW loss. The HAW neptunium and plutonium losses are satisfactory, but both have increased about two fold during the report period. The above-normal (2 to 3 fold) 2DU neptunium loss persisted throughout the report period.

5. Solvent Treatment

The gamma activity in the IOO slowly increased from 7,000 to 9,000 uc./gal. (Zr-Nb/Ru ratio = 0.5) by mid-week, then dropped to ~ 6,000 uc./gal. over a 24-hour period. The initial increase correlated with a general increase of First Cycle (including ICW) gamma levels, and the decline correlated with increasing the TK-G1 permanganate addition rate, ultimately to three times flowsheet.

6. Fission Products

Cesium recovery operations were completed on March 25 with the loading of the fourth GRNL STT with 29,000 curies of cesium-137. The third and fourth casks were then sealed and prepared for shipment.

The first and second casks remained under surveillance through March 27. On March 27 the last set of gas samples were obtained and the casks closed. Gas analyses on the two casks were as follows:

<u>Time Elapsed Since Filling, Hr.</u>	<u>Gas Analyses, Vol. %</u>							
	<u>CO₂</u>	<u>A</u>	<u>O₂</u>	<u>N₂</u>	<u>CO</u>	<u>He</u>	<u>H₂</u>	<u>H₂O</u>
Cask I, #45-A								
100	0.33	0.80	19.9	78.1	0.01	0.01	0.82	0.10
216	0.01	0.77	19.3	77.3	0.01	0.01	2.66	-
288	0.02	0.73	19.0	71.7	0.01	0.58	7.94	-
Cask II, #48								
75	0.01	0.80	19.9	78.5	0.01	0.01	0.82	-
147	0.08	0.77	19.6	77.3	0.01	0.49	1.75	-

The maximum pressures observed were 0.36 psig on Cask I and 3 psig on Cask II. However, Cask I pressure data are known to be in error due to a leak around the liquid level probe. Cask II data and gas analyses indicate that pressure build-up during shipment will be less than one psig per day.

Following completion of cesium processing, the Purex Fission Product Test Plant equipment was flushed with water and 10% nitric in preparation for receiving the TK-003 contents for concentration and storage in TK-78. The next strontium-90 production will go to TK-003. TK-002 contents will be allowed to age.

Appendix --- Process Summary Through April 2, 1961

Over the week end the processing rate was reduced to about 3.0 CF. Late, on April 2 a process test of the use of hydrazine as a holding reductant in the IBX was commenced.

The HAPD 1A Cask containing 20,000 curies of strontium was received from HLO on March 31, and was placed under surveillance as a prerequisite for shipping. Over the week end the cask pressure levelled off at 8 - 10 psig. The cask temperature was 15° C.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 3/31/61 TO 2400 ON 4/6/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.3	3.8	-	-	0.003	0.02
Final	3.1	3.4	9.0	2.4	0.005	-
Ion Exchange	-	1.2	-	0.05	-	-
Overall	6.4	8.4	9.0	2.45	0.008	0.02

II. Production

A. HAF Processed: 181 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
78	7.0	1.1	7.0	P1
79	7.1		7.3	P2
80	6.6	0.7	7.0	P2
81	5.8		6.9	P1
1	5.2	0.7	7.0	P1
2	5.3	0.5	6.8	P1
3	5.0		7.0	P1
4	4.7	0.1	7.0	P1
5	4.5		7.1	P1
6	4.7	0.4	7.0	P1
7	4.2		7.1	P1
8	4.7	0.5	7.1	P1
9	4.4		7.2	P1
10	4.3	0.6	7.4	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
11	4.7		7.0	P2
12	4.3	0.7	6.7	P1
13	5.0		6.9	P1
14	4.8	0.6	7.0	P1
15	4.8		7.0	P1
16	5.0		6.9	P1
17	5.3		6.7	P1
18	3.4		7.0	P1
19	5.0		6.9	P1
20	5.3	0.8	6.9	P1
21	5.5		7.3	P1
22	5.2		6.8	P1

2. Silica Gel

Gamma Ratios: Feed = 5.1
Product = 2.4

Tonnage: 107

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
33	1.9	0.008		4	2	234-5
34	2.1	0.008		6	3	234-5
1	2.2	0.010	480	5	2	234-5
2	1.7	0.006		5	2	234-5
3	1.5	0.003		5	2	234-5
4	1.3	0.034		5	2	234-5
5	1.1	0.002		5	2	234-5
6	(2.4)*	0.005		4	2	234-5
7	(3.2)*	0.001		5	2	234-5
8	(3.5)*	0.004		5	2	234-5
9	(3.4)*	0.004		5	2	234-5
10	(3.2)*	0.005		5	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.08%

Pu = 0.12%

E. Rework - % of Total Production:

U = Negligible

Pu = 4.6% (Predominantly L-Cell Package Flushes)

*Results later found to be about 3 fold high due to contaminated sampler.

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	608	104A
Cell Drainage	133	101A
FP Waste	0	101A
Coating Waste	270	107C

B. IW Flows: 4.0

C. Tank Farm:

<u>Tank</u>	<u>Estimated Boil-off Rate, Gal/Min</u>
101	13.2
102	1.9
103	0.2
104	9.7

Notes:

1. The HAF rate was reduced from 3.2 to 3.0 CF at noon on March 31.

2. Flowsheet

- (a) On March 31 the plant rate was reduced from 3.2 to 3.0 CF. Only the HAF, HAX, IBXP, IBXP-HNO₃, ICX, 2AF-HNO₃, 2DF and 2DX flows were decreased.
- (b) On March 31 through April 1 the HSE was reduced from 83 to 75 flows.
- (c) On April 2 the IBXP, IBXP-HNO₃, 2AF-HNO₂, and 2AF flows were reduced to 3.0 CF.
- (d) On April 2 the 2EP was reduced from 2.32 to 1.70 flows.
- (e) On April 2 the IBX was made 0.005 M N₂H₄. The IBX was increased to 0.015 and 0.030 M N₂H₄ on April 4 and 6, respectively.
- (f) On April 2, 4, and 6 the 2AF-HNO₂ was increased from 2.8 to 3.1, 3.7, and 4.3 flows, respectively.
- (g) On April 5 the 2DE was reduced from 67 to 62 flows. It was returned to 67 flows on April 6.

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- (h) On April 5 the HEP-HNO₃ was increased about 15%.
- (i) On April 6 the IBS was reduced from 1.9 to 1.7 flows.

3. Solvent Extraction

Gamma levels in the intercycle streams generally declined about a third during the report period, apparently as a result of stepwise rate reductions to 3.0 CF. The improvement was not entirely reflected in the final products due to contamination of a sampler, in the case of the concentrated Pu product, and unstable operation of the Final Uranium Cycle columns during the latter part of the report period. Waste losses were excellent, except for a 3 to 4-fold increase in the neptunium loss to the uranium product. Again, this increase was at least partly caused by unstable 2D Column operation.

On April 2 a test of the use of hydrazine as a holding reductant in the IBX was commenced with the introduction of 0.005 M N₂H₄ to the IBX stream. This was followed by increases to 0.015 M and 0.03 M N₂H₄ on April 4 and April 6, respectively. The initial objective of the test was to demonstrate the absence of deleterious process effects by the addition of hydrazine; consequently no reduction in normal IBX sulfamate concentration was made. At the end of the report period, the following observations had been made:

- (a) No deleterious process effects had been noted; and
- (b) No hydrazine had been detected in the IEP, while a trend for increased ferrous ion concentration in the IEP was noted.

Instability of the Final Uranium Cycle columns was indicated by unusually low 2E Column static pressure during most of the report period and severe flooding of the 2D Column, commencing in the scrub section, early on April 5. In the case of the 2E Column, the major decrease in static pressure commenced late on April 3. The subsequent 2D Column flood was touched off by a temporary 2DF pump outage. A 2D Column pulse frequency reduction from 73 to 55 cycles/min. was necessary to bring the operation under control. Laboratory investigation of possible causes for the poor column behavior included dispersion and disengaging studies of the process streams, and solvent quality studies. No abnormalities were indicated. A review of HA Column experience during the scrub cartridge failure of January 1959 revealed similarities to the current 2D Column experience. Thus, failure of the 2D Column scrub cartridge (Fluorothene-plate zebra cartridge) is considered a possibility. This cartridge has been in service for nearly 4-1/2 years.

For purposes of comparison to special HAP samples taken during February (p. 24), another 100-ml HAP sample was thiafed from the crane on April 2. Visual inspection of this latest sample showed a considerable improvement

in appearance, with no unusual green coloration and with considerably less black particulate content. ES Column gamma DF measurements based on this sample and an HSP sample taken at the same time are tabulated below. Results for a pair of samples taken on Feb. 16 are also tabulated.

Sample	Uc./Gal.					
	April 2, 1961			Feb. 16, 1961		
	Gross	Zr-Nb	Ru	Gross	Zr-Nb	Ru
HAP	2.2×10^5	1.5×10^5	4.7×10^3	2.0×10^5	1.5×10^5	9.9×10^3
HSP	1.1×10^5	7.5×10^4	4.2×10^3	7.7×10^4	5.6×10^4	9.8×10^3

The change in ruthenium levels apparently was directly related to a corresponding change in IOO ruthenium content.

4. Solvent Treatment

The gamma activity in the IOO slowly decreased from 6,000 uc/gal to ~ 3,300 uc/gal during the report period. Although solvent extraction rates were reduced toward a 3.0 CF, G-Cell change rates and flows, including the three-times-flowsheet permanganate rate, were left at a 3.4 CF. Discontinuing IOF recycle on March 31 had no apparent effect on solvent activity. Solvent losses continue to be high (about twice normal), although less than the losses experienced at 3.6 CF.

5. Plutonium Concentration

An increase in the gamma activity in the Pu product on April 4 correlated with a change from the use of TK-L10 to the use of TK-L9 as the sampling and load-out tank. After PR Can thief samples indicated the routine TK-L9 gamma analyses to be erroneously high, an inspection of the L9 sampler revealed that the sample lines and sample surge pot were reading 40 mr./hr. Thieving of batches is continuing until the sampler is cleaned.

6. Waste Treatment and Acid Recovery

Testing of the INW Anion Exchange equipment revealed two ruptured DOV's -- the elutant line from the P & O Gallery to the ion exchange bed and the line from F12 to the bottom leg of the ion exchange bed. Two plugged in-trench lines from the wall at F12 to the wall at F16 were also detected. One of these lines has been freed by the application of water pressure.

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7. Fission Products Test Plant

The HAPO 1A Cask containing 20,000 curies of strontium-90 was received from HLO on March 31 and placed in B-Plant tunnel for observation of its temperature and pressure behavior after which duplicate samples of the radiolytic gas were obtained for analysis. After venting to the atmosphere, the cask was allowed to come to equilibrium a second time, the radiolytic gas was again sampled, and the cask was sealed for shipment. The equilibrium pressure was determined to be 8 psig at an ambient air temperature of 50° F. The equilibrium temperature inside the cask was 9° F above ambient. The composition of the radiolytic gas at equilibrium was as follows:

Cask Gas Analyses

<u>Gas</u>	<u>First Equilibrium Samples</u>		<u>Second Equilibrium Sample</u> ⁽¹⁾
CO ₂	1.06	3.24	1.05
A	/0.01	/0.01	0.13
O ₂	/0.01	/0.01	3.14
N ₂	4.35	7.09	17.3
H ₂ O	1.7	1.6	0.40
CO	0.11	/0.01	/0.01
He	/0.01	0.04	0.13
H ₂	92.8	88.1	77.9

(1) Presence of argon indicates partial contamination of sample with air. On an argon-free basis, the oxygen content is negligible.

A 200-gallon cask of crude strontium was returned from HLO because of excessive solids. The contents of the cask were transferred into the building and combined with P8 material. The combined material was concentrated, centrifuged twice, and used to refill two 200-gallon casks. Because of the numerous jet transfers involved, the material loaded into the cask was only 50 - 60 curies per gallon of strontium-90. Reports from HLO personnel indicate that the centrifugation was successful and the solution is free of solids.

An attempt to return TX-003 material (aged strontium) to the building was unsuccessful because of a leaking pump. The pump is being replaced and the transfer is rescheduled for late in the week.

Appendix --- Process Summary Through April 9, 1961

Performance continued without significant change over the week end. The presence of hydrazine in the IBX at 0.03 M had no effect.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 4/7/61 TO 2400 ON 4/14/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.4	3.9	-	-	0.003	0.035
Final	3.0	3.4	8.0	2.4	0.003	-
Ion Exchange	-	0.8	-	0.6	-	-
Overall	6.4	8.1	8.0	3.0	0.006	0.035

II. Production

A. NAF Processed: 199 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
23	5.0		6.9	P1
24	4.7	1.0	6.8	P1
25	4.6		6.8	P1
26	4.5	1.0	7.0	P1
27	5.0		7.0	P1
28	5.2		7.3	P1
29	5.8		6.7	P1
30	5.4		6.9	P1
31	5.0		7.2	P1
32	6.0		7.1	P1
33	5.0		6.5	P1
34	5.3	1.2	7.0	P1
35	4.6		7.0	P1
36	5.1	1.0	7.0	P1
37	5.0		6.7	P1
38	4.7	0.7	6.8	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
39	4.3		7.1	Pl
40	4.3	0.5	7.1	Pl
41	4.4		6.8	Pl
42	4.6	0.7	7.3	Pl
43	4.3		7.0	Pl
44	3.9	0.6	6.9	Pl
45	4.4	2.3	7.0	Pl
46	4.2	1.1	7.1	Pl
47	4.2	1.1	7.3	Pl
48	4.2		7.0	Pl
49	5.7		7.1	Pl
50	4.1	4.1	7.1	E6
51	-		5.9	E6
52	4.9		4.1	E6
52a	4.9		3.1	Pl
53	3.8		6.9	Pl

2. Silica Gel

Gamma Ratios: Feed = 4.7
Product = 1.9

Tonnage: 145 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppb Pu</u>	<u>No. Cans</u>	<u>Average Shipped Mr./Hr.</u>	<u>To</u>
11	2.8*	0.010		4	2	234-5
12	2.2*	0.001		4	3	234-5
13	4.3*	0.001		4	3	234-5
14	3.4*	0.002		5	3	234-5
15	1.4	0.006	440	5	3	234-5
16	1.5	0.003		5	3	234-5
17	2.1	0.006		5	3	234-5
18	2.1	0.010		5	3	234-5
19	2.3	0.012		4	3	234-5
20	3.2	0.010	500	4	3	234-5
21	2.2	0.007		5	4	234-5
22	2.1	0.007		4	3	234-5
23	1.8	0.002		6	3	234-5
24	2.0	0.003		5	2	234-5
25	1.8	0.002	460	5	3	234-5
26	4.0	0.090		4	3	234-5

*Results estimated to be about 3 fold high due to contaminated sampler.

D. Overall Waste Loss - % of Total Production:

U = 0.20%

Pu = 0.05%

E. Rework - % of Total Production:

U = 8.5% (Recycle from TK-R6 to HAF Make-up TK-R6 at shutdown)

Pu = 0.33% (L-Cell Pkg. flushes and sump rework)

III. Waste Treatment**A. Volumes to UGS:**

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	673	104 TK
Cell Drainage	160	101 TK
FP Waste	38	
Coating Waste	270	107-C

B. IWW Flows: 4.3**C. Tank Farm:**

<u>Tank</u>	<u>Est. Boil-off Rate, Gal./Min.</u>
101	13.7
102	1.9
103	0.5
104	8.9

Notes:

- The HAF rate was maintained at 3.0 CF until 1930 on April 14 at which time scheduled shutdown of the plant was started ("hot" HAF off).
- Flowsheet**
 - On April 10, hydrazine was removed from the IBX stream.
 - From April 10 through 13, the 2EX flow was reduced from 530 to 460 flows.
 - From April 11 through 13, the 3MB was increased from 40 to 49 flows.
 - On April 12, the 2DF ferrous sulfamate was reduced from 0.29 to 0.17 flows.

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3. Feed Preparation

During the report period a tendency for less than normal vacuum in the A3 Dissolver was noted. The charging chute for this dissolver had been recently fitted with a remote gasket made of Teflon-asbestos (see p. 56).

4. Solvent Extraction

Process performance continued without significant change up to the time of shutdown on April 14. Neptunium losses to the 2DU continued above normal (at about 6% of the input M). Pu losses to the HAW showed a mild rise to about 0.05%.

The addition of hydrazine to the IBX stream at 0.03 M was continued until April 10 at which time the test was terminated (see p. 61). Observations made during the course of the test are as follows:

- (a) The hydrazine had no effect on plant waste losses or decontamination performance.
- (b) Initially, analytical results indicated increased ferrous iron concentration in the IBP as the IBX hydrazine was increased stepwise to 0.03 M. However, this pattern did not persist.
- (c) At 0.005 and 0.015 M hydrazine in the IBX, no hydrazine was detected in the IBP. At 0.03 M in the IBX, a low level of hydrazine was detected in the IBP. The reason for this apparent destruction of hydrazine has not yet been satisfactorily explained. Laboratory data showed that there was no loss of hydrazine during the period of normal holdup in the IBX tanks. Also, laboratory data showed that hydrazine does not extract into the IBU and that nitrite preferentially attacks sulfamate ion before attacking hydrazine. It is possible that the hydrazine kill results from reduction of ferric to ferrous iron in the IBX-IBS system, although this pattern was not satisfactorily verified as indicated under (b), above.

The unusually low 2E Column static pressure previously reported (see p. 61) was caused by an instrumentation problem, rather than by column instability. The capacity of the 2D Column, however, continued to be unusually low and was demonstrated to be unaffected by the presence or absence of hydrazine in the process. For both conditions, scrub section instability was demonstrated to occur at a pulse frequency of about 59 cycles/minute. It is thus concluded that the plastic sieve plates in the scrub section have failed.

Analysis of the HAP sample which was thieved on April 2 (see p. 61), again showed that the black particulate material present was predominantly molybdenum.

5. Solvent Treatment

The gamma activity in the IOO remained at 3000 to 4000 uc/gal until the recycle of "cold" uranium was commenced at plant shutdown. Laboratory tests indicated solvent quality to be excellent.

6. Plutonium Concentration

On April 11, the contaminated I9 sample surge pot was replaced (see p. 62), and thief sampling of product batches was stopped.

During the report period, considerable difficulty was experienced in obtaining proper resin pushes. On April 13 and 14, XAW losses as high as 40% resulted when the pushes failed to move sufficient resin. Lack of sufficient resin in the system probably contributed to this difficulty.

7. Fission Product Test Plant

On April 7, the STF cesium casks were opened and each was purged with 50 cubic feet of argon to sweep out the explosive hydrogen mixture. The cask pressures were measured prior to purging and a gas sample obtained from the cask with the highest pressure. One of the casks had solution pressure against the vent nozzle. Seven gallons of solution were removed from the cask before the cask could be vented and purged. Cask pressures, gas composition, and cask solution analyses were as follows:

Cask Pressures

Cask 45A	< 1 psig
" 48	5.3 "
" 44	4.8 "
" 47	< 1 "

Cask Numbers

	<u>45A</u>	<u>48</u>	<u>44</u>	<u>47</u>
Pressure, psig	<1	5.3	4.8	<1

Gas Composition

Mol - %*

CO ₂	0.11
A	0.52
O ₂	17.3
H ₂	52.8
CO	<0.01
He	0.098
H ₂	27.3
H ₂ O	1.7
H ₂ O	0.015
NO	0.15

*Sampled 410 hours after filling cask.

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Cask Numbers			
<u>45A</u>	<u>48</u>	<u>44</u>	<u>47</u>

**Solution Analyses,
Curies/Gallon**

ZrNb	3.5×10^{-2}
Cs	8.3×10^{-3}
Ru	4.5×10^{-3}
Cs	7.9×10^{-3}
Sr	2.6×10^{-4}
H ₂ O ₂ Content, g./l.	0.026

After the SFT casks had been purged with argon the closures were all tightened and the lids secured. Radiation surveys were completed in preparation for off-site shipment. The results of the radiation survey were as follows:

Radiation Readings, Mr./Hr.

<u>Cask No.</u>	<u>Distance, 2 In.</u>		<u>Distance, 1 Meter</u>	<u>Distance, 3 Meters</u>
	<u>Top</u>	<u>Side</u>		
45A	10	70	11	
48	4	85	18	2
44	7	42	8	3
47	2	31	6	2
				2

Car No.

314016	8
314017	4

The SFT casks and the HAPO 1A cask left HAPO by special train April 8. The casks arrived safely at ORNL the evening of April 12.

The aged strontium in TK-003 was returned to the building on April 7 for concentration and storage in TK-F8. The TK-003 heel which could not be pumped to the building was transferred to TK-002. Jumper changes were completed for routing current production into TK-003.

Strontium production was resumed on April 10. The first series of runs will be made on LW stored in TK-F13. This material is estimated to have a Sr 89/90 ratio of 4 to 5.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 4/15/61 TO 2400 ON 4/27/61

During the report period the plant was shut down with respect to normal uranium processing. A scheduled neptunium recovery run was conducted and necessary maintenance and other operations were accomplished. Process notes of interest are presented below.

1. Feed Preparation

In preparation for the processing of non-representative irradiated uranium, the entire inventory of normal metal was processed prior to plant shutdown on April 14. This included the dissolution and processing of dissolver heels. Remaining dissolver rinse water which normally contains a significant Pu content was segregated by storing in the B3 Dissolver.

Because the asbestos-Teflon remote gasket on the A3 Dissolver chute did not give satisfactory service (see p. 67), a normal Teflon gasket was installed on the chute lid and the remote gasket was removed.

2. Solvent Extraction

The plant shutdown operations of April 14 and 15 were designed to prepare for a neptunium recovery run, for special uranium processing (high M/D/T), and for maintenance operations. In connection with the processing of the special uranium, stripping, flushing and neptunium recovery operations were designed to minimize the in-plant plutonium and to accumulate the remaining in-plant plutonium in the 3WB system. The plutonium collected in the 3WB system following the neptunium recovery run was scheduled for segregation for rework, after the special uranium processing operation.

Other shutdown operations of interest are described below:

- (a) The HA scrub-section zebra cartridge was removed on April 17, after 10 months of service, and was replaced with a new all-stainless-steel cartridge on April 26. The appearance of the

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old cartridge was typical, with broken and discolored plastic plates. Prior to installation of the new cartridge, plastic fragments were floated from the column with water. The new cartridge contains 75 nozzle plates, six per cent free area with nozzles pointing down, and 4 lower plates of 16 per cent free area. The nozzle plate spacing is 2 1/8 inches except for 3-inch plate spacing in the lower three feet of the cartridge. The new cartridge was degreased with 5% Na_2CO_3 solution at 50° C after installation.

- (b) The failed HSS- HNO_3 and IBXP rotameters were replaced. Removal of the HSS- HNO_3 rotameter showed that the rotameter was badly fouled with a black substance. A sample of this material will be obtained for chemical analyses. Apparently, this substance is also the cause for the recent large changes in the calibrations of the IBXP- HNO_3 and HSB- HNO_3 rotameter. Installation of a Cuno filter in the Purex Recovered Acid system on April 24 helped this situation.
- (c) The HAF system was replaced with a new system which will allow changing the HAF DOV and rotameter without stripping and emptying the HA Column.
- (d) The 3WP DOV was replaced (preventive maintenance).
- (e) The 2B Column was backflushed to the cell floor after the Np recovery run. Also, hydraulic tests of the 2AP system were made (see Neptunium Recovery Run, below).
- (f) The J5-2A-2B system was flushed with 60% HNO_3 at 60° C following the neptunium recovery run.

3. Plutonium Concentration

In preparation for the processing of non-representative irradiated uranium during May, the plutonium concentration facilities --- L-Cell Package, Pu Ion Exchange, and the PR Room --- were flushed to minimize plutonium blending. Based on the analytical results of the various flushes, the plutonium remaining in this equipment was estimated to be about 500 grams.

During the shutdown a complete N-Cell resin change was made. Maintenance items included (a) replacement of the leaking N6-to-N7 overflow line; and (b) installation of the necessary valving and piping for continuous resin addition and removal. Tie-in of the resin addition and removal system to R Cell remains to be done.

4. Neptunium Recovery Run

(a) Summary

The neptunium recovery run was started (3WB on) at 0200 on April 16 with an estimated 1.34 kg of neptunium available for recovery. The run was completed at 1900 (2BP off) on April 22 with a recovery of an estimated 0.21 kg. Most of the neptunium (estimated 0.88 kg) was irretrievably lost via the vessel vent system to underground storage. The standard "reducing" flowsheet was used in the Final Plutonium Cycle equipment 2A and 2B Columns.

The recovery operation was started at a processing rate of ~ 60% of normal but was increased to ~ 35% of normal after 24 hours. The 2BW losses were 2 to 4-fold above normal during Phase I and were not improved by making the 2BX stream 0.01 M-sulfamic and 0.01 M sulfuric acid. Phase II 2BW losses, however, were reduced about 10-fold by sulfamic and sulfuric acid in the 2BX stream. During Phase III the 2BW losses returned to their previous levels after removing the sulfamic and sulfuric acid. From these data it appears sulfate will reduce the 2BW neptunium loss in the absence of uranium. Since sulfamic acid had no beneficial effect, the organic quality rather than neptunium valence is responsible for the high 2BW neptunium loss.

The gamma levels were again about 10 fold below normal as in the previous run. The gamma levels of the product were above normal because the 2BP neptunium level was low, the length of time for the Phase II operation was short, and the length of time for the Phase III operation was long.

The loss of neptunium via the vessel-vent system to TK-F18 and underground storage was not discovered until after the conclusion of the run. As a matter of fact, this conclusion was based mostly on observations and failure to find the neptunium in other plant vessels. On April 26 tests were conducted in the 2A Column to determine the possibility of loss of 2AP via the vent system. Observation from the canyon crane revealed that a substantial carryover of liquid to the vent system was undoubtedly occurring at the conditions of the neptunium recovery flowsheet. This behavior prompted investigation of a possible flow restriction, so the line was backflushed by emptying water from the 2B Column via the 2BP distributor and the pulse leg to the cell floor. Further testing showed marked improvement in flow as a result of this treatment. Also, during further-flushing operations, dark-colored "crude" were observed coming from the 2B Column. Finally,

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testing of the 2AP line indicated a flow pattern judged to be normal and at least satisfactory for the normal processing flow-sheet. Any tendencies for 2AP carryover to the vent was observed to be aggravated by the relatively high pulse frequencies used with the new 2A Column. This column requires frequencies around 80 cycles/min. compared to 20 cycles/min. for the old Fluorothane Raschig ring column.

(b) Material Balance

	<u>Kg</u>
Mp in Plant	1.35
Mp in 3WB	1.34
Mp Processed as 3WB	1.33
Mp Load-out (Est)	0.21
Mp Recycled*	0.17
Mp Lost (Est)	0.95

(c) Typical Solvent Extraction Losses*

- % of 3WB Processed

2AW	1.5
2BW	4.8

(d) Product Analytical Results

	<u>Mp, g/l</u>	<u>HNO₃, M</u>	<u>% Contamination of Mp Pu</u>	<u>G/Mp, AT x 10⁷</u>
Principal Load-out	6.65	8.1	120	474
First Flush	3.77	10.4	148	600
Second Flush	**	12.3	**	**
Third Flush	4.54 ?	11.9	23 ?	87 ?
Fourth Flush	1.38	11.9	66	214

5. Solvent Treatment

During the neptunium recovery run, the No. 1 Solvent System was operated at the normal 1.44 C.F. except for occasional short shutdown periods.

After the recovery run, Tank G1 was flushed twice with 4,000 gallons of 5% oxalic - 2% nitric acid for 8 - 10 hours to remove MnO₂ from the vessel. Tank G2 and the IO Column were flushed with 5% NaOH - 2% tartaric acid and 5% oxalic - 2% nitric acid to facilitate replacement of the IO Column float, which had failed during the shutdown.

* Losses recycled for recovery during normal processing operations.

** Sample slurped before analyses were complete. Final load-out data .. quoted in text are Process Chemistry data.

Based on recommendations resulting from Process Chemistry investigations, a plant test was performed during the shutdown period to evaluate the operability of $Mg(OH)_2$ in the TK-G7 Turbomixer. A solution of 1 Wt. % $Mg(NO_3)_2 \cdot 6 H_2O$ in 1 M NaOH was added to the Turbomixer (TK-G7) and the G-Cell equipment was operated by spinning at a 3.0 C.F. Prior to commencing the test, the solvent activity level was very low as a result of operations during the Np recovery run. Consequently, test evaluation was planned only in terms of general operability, entrainment tendencies, and equipment decontamination. About four shifts of operation indicated no difficulties or unusual operating characteristics. An initial moderate increase in IOO activity may have been caused by equipment decontamination; however within the time of the test the original low level had been restored. Series of both Turbomixer samples and IOO samples have been submitted to Process Chemistry for further evaluation.

6. Waste Treatment and Acid Recovery

On April 21 the Acid Concentrator E-F6, a Purex Plant original equipment piece, was replaced because of leaks. Prior to its removal, the vessel was flushed with 5% NaOH - 2% tartaric, 5% oxalic - 2% nitric acid, and another caustic-tartrate flush.

Also, failure of the east tube bundle in F6 after 6 months of service necessitated its replacement. A new prototype titanium tube bundle was installed in the JWB Concentrator (E-H4), and the stainless-steel tube bundle replaced was installed in E-F6.

A temperature excursion has been noted in Tank 241-A-101 with the temperature reaching a peak of 201° C and remaining between 190 - 200° C for about a week. Efforts are continuing to control the temperature by water dilution.

7. Fission Product Test Plant

The first series of strontium recovery runs since cesium processing was completed on April 18 and 560,000 curies of Sr 89-90 was sent to the 003 tank in the 244 CR Vault. The composition and decontamination factors achieved for the product solution were as follows:

<u>Component</u>	<u>Curies/Gal.</u>	<u>DF</u>	<u>Lb/Gal.</u>
Sr 89 - 90	695		
Cs 141 - 144	622	4	
Zr-Hf	1040	3	
Ru-Rh	6	100	
 HNO ₃			0.532
Fe			0.016
SO ₄			0.016

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The Sr 89-90 ratio was reported as 6.3. This is not believed possible since the IW processed in this series of runs had been aged for several weeks in TK-F13. A ratio of 7.3 had been reported on this material in February. If this ratio is assumed correct, the current ratio is around 4. Based on the total number of Sr 89-90 curies measured in the IW, and assuming 1750 curies of Sr 90 per ton of uranium and 40 gallons of IW per ton of uranium, a Sr 89-90 ratio of 5.5 is calculated.

Laboratory studies by Chemical Research Operation on actual Purex carbonate product solution revealed no difficulty with strontium losses across the oxalate step. Plant losses across this step have been on the order of fifty per cent. These data indicate the strontium loss across the oxalate step in the Purex Plant may be due to material left in the E3 vessel at the conclusion of the sulfate runs. A special acid flush of the E3 vessel was made between the sulfate precipitations and the oxalate precipitations in the latest series of runs. Strontium recovery across the subsequent oxalate precipitation was greater than 90 per cent. A complete material balance on this series of runs was as follows:

<u>Stream</u>	<u>Per Cent</u>
IW	100
Sulfate Waste	14.0
Flush between Sulfate Precipitation and Oxalate Precipitation	16.0
Oxalate Waste	2.6
Flush between Oxalate Precipitation and Concentration Precipitation	6.3
Concentration Waste	11.5
Concentrated Product	<u>49.4</u>
	99.8

As shown in the above material balance, 22 per cent of the strontium was lost to flushes. Most of this loss is believed to be precipitate that is not transferred to the centrifuge. The E3 Agitator cannot be operated below a volume of 800 gallons. This means 30 to 40 per cent of a run is centrifuged without benefit of agitation. Tank E3 rinses are made to minimize the amount of precipitate left in the vessel; however the minimum agitator level of 800 gallons prevents agitating these flushes. Procurement of a special low-speed agitator with a longer shaft is being investigated.

Another series of strontium recovery runs was completed on April 28 and a total of 1.4×10^6 curies of Sr 89-90 was sent to the 003 tank in the 244 CR Vault. The composition and decontamination factors achieved for the product solution were as follows:

<u>Component</u>	<u>Curies/Gal.</u>	<u>DF</u>	<u>Lb/Gal.</u>
Sr 89 - 90	1870		
Cs 141 - 144	610	8	
Zr-Nb	1120	7	
Ru-Rh	21	120	
HNO ₃			0.283
Fe			0.006
SO ₄			/0.017

The Sr 89-90 ratio was reported as 16.4, however the calculated Sr 89-90 was approximately 10.

A complete material balance for this series of runs was as follows:

<u>Stream</u>	<u>Per Cent</u>
INW	100 /
Sulfate Waste	12.5
Flush between Sulfate Precipitation and oxalate Precipitation	6.0
Oxalate Waste	3.8
Flush between Oxalate Precipitation and Concentration Precipitation	7.3
Concentration Waste	6.1
Concentrated Product	<u>70.0</u>
	106.2

The agitator in the E5 vessel failed on April 23 and the series of runs was completed without agitation in E5. The lack of agitation in E5 did not seriously complicate the processing steps but it prevented accurate sampling of flushes and centrifuge supernatant solutions.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 4/28/61 TO 2400 ON 5/4/61

Due to process difficulties only about two days of the report period were spent in the processing of "hot" feed. Consequently, no data will be published in this report.

Notes:

1. (a) Normal processing was commenced at 2145 on April 29 (HAF on) at 3.0 CF. Subsequent checks showed the actual rate to be about 2.75 CF. Following initial rework of "cold" uranium remaining from shutdown, processing of "hot" HAF was commenced at 0145 on April 30.
(b) At 0515 on May 1, recycle of "cold" uranium product from TK-K6 to TK-B6 was started.
(c) At 0130 on May 4, processing of "hot" HAF was resumed. At 1900 on May 4, the processing rate was increased to 3.0 CF.

2. Flowsheet

- (a) The start-up flowsheet was the same as at shutdown on April 14 except (1) there was no 3MB flow, (2) the HAF was 1.25 M U and 3.0 M HNO₃, and (3) the HAF was 108 flows.
(b) Shortly after start-up the HA Column flooded and the HAX and HAF flows were reduced 8.5%. This in turn reduced the IBXF, 2IF and 2IX flows. Other flows (gpm) were not changed except the HES. The HES was reduced to 61 flows.
(c) On April 29 the IOS was reduced from 0.17 to 0.05 M HNO₃.
(d) On April 30 the 2IF-Fe was increased from 0.32 to 0.43 flows.
(e) On April 30 the 3MB was started at 41.4 flows and the HAF was reduced to 75 flows. In addition the HAF specification was made 1.80 M U and 1.05 M HNO₃.

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- (f) On April 30 the HSS was increased from 61 to 89 flows.
- (g) On May 1 the carbonate change frequency in the IOF Tank increased from six to four hours. This, in effect, increased the IOF-CO₃ from 20.8 to 31.4 flows.
- (h) On May 1 the IOF-K₂CO₃ flow was increased from 1.85 to 3.70 flows.
- (i) On May 1 the IOC was increased from 12.6 to 14.9 flows. In addition the carbonate solution in the G7 Turbonixer was changed every eight hours. These carbonate changes were equivalent to 35.2 flows of IOC.
- (j) On May 2 the JWB flow was stopped for about six hours. During this time the HAF was 1.25 M U and 3.0 M HNO₃ at 108 flows.
- (k) On May 3 the HSS was reduced from 89 to 61 flows.
- (l) On May 3 the ICX was reduced from 474 to 416 flows.
- (m) On May 4 the HAF and HAX rates were increased from 2.75 to 3.0 CF. This in turn increased the IBXF, 2IF and 2IX flows. Other flows (gpm) were not changed except the ICX. The ICX was increased to maintain the stream at 416 flows.

3. Solvent Extraction

Plant start-up was characterized by a number of process difficulties.

- (a) Initially both the HA and 2A Columns flooded. They were brought under control by pulse frequency reductions.
- (b) A burst of gamma and Pu into the uranium product was associated with the start-up of the Final Uranium Cycle.
- (c) About a day after start-up a severe gamma burst from the HA Column occurred simultaneously with starting the JWB flow.
- (d) During most of the report period, Pu losses to the HAW were high.

The HA Column flood originated in the scrub section, and it was necessary to reduce the pulse frequency from 82 to 68 cycles per minute to restore column stability. Although column operation was satisfactory after pulse frequency reduction the scrub section still cycled moderately. Too, the aqueous hold-up in the scrub section has remained substantially greater with the new stainless-steel cartridge than with the old zebra cartridge. During the report period the HA Column process conditions were varied to determine their effects on HA Column operation. Flooding was initiated at each of the following three sets of conditions:

MAX gpm	42	42	45
HAO gpm	11.8	11.8	11.8
HSS-HNO ₃ gpm	~ 0.9	~ 0.9	~ 0.9
HSS gpm	10.3	7.3	7.3
HA Pulse Frequency (Cycles/Min.)	78	80	77

By the end of the period the HA Column was operating at CF = 3.0 and at a pulse frequency of 75 cycles per minute. The HSS flow, however, was about 90% of normal. Although waste loss and decontamination performance were satisfactory, the cycling scrub section continued and made operational control difficult.

The 2A Column scrub section also flooded at start-up, and the flood was extremely difficult to break. Once the 2A Column was stabilized, normal operating conditions were restored within a few days without any re-occurrence of instability.

The start-up burst of gamma and Pu into the uranium product probably resulted partly from the shutdown operations of April 16. In addition, low saturation at start-up contributed. It is also possible that an organic phase in the 2DF and/or the initial processing of concentrated ICU originating from the recent Np recovery run may have contributed. This was the first time the Np ICU concentrate has been processed through the Final Uranium Cycle at start-up. Previously it had recycled to the HAF Make up Tank E6.

The start-up flowsheet featured no 3WB flow because of the low 3WB inventory, and the acid necessary to correct for this change was added directly to the HAF. However, when the 3WB flow was finally started on April 30, and the HAF flow and specifications were returned to normal, a severe gamma burst originated in the HA Column and rapidly spread through the solvent-extraction system. Processing of "cold" uranium was commenced as soon as possible; still the activity in the uranium product peaked at nearly 600 gamma ratio, and other gamma levels were correspondingly high. After reducing the gamma levels by recycling "cold" uranium for four days, normal processing of "hot" feed was resumed, and decontamination performance of the First Cycle was about normal. Just prior to the resumption of "hot" HAF processing, a second burst occurred as a result of purging the IO Column interface (See Note 4, below). The effect of this burst was mostly localized in the No. 1 Solvent Treatment System. Laboratory data on the 3WB stream during the gamma burst showed nothing abnormal. At present it is postulated that the enormous gamma burst was caused by sudden

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increase in the HA Column acidity at the time of 3WB introduction, although such process sensitivity is not normal. Although plant operating data showed no straight organic phase was added to the 3WB Concentrator, the following items may have been related to the severe gamma burst:

- (a) Organic leaked from the 2D Column to the 2DF and 3WF tanks during the plant shutdown.
- (b) The contents of the ICU Concentrator was concentrated ICU accumulated during the Birch recovery run. This solution for the first time was processed as 2DF rather ~~than~~ than HAF.

The HAW plutonium losses were high (0.25 - 0.5%) at start-up, but gradually declined to normal. The exact cause or causes is not known but is probably related to the following:

- (a) High sulfate in the 3WB following the 3p recovery run;
- (b) Plutonium recycle from L11;
- (c) Reduced efficiency in the HA extraction section due to below-normal pulse frequency.

Other losses were normal except for 2-fold below normal 2AW loss and a 4-fold above normal 2DU neptunium loss. The continued high 2DU neptunium loss is probably related to the damaged 2D Column scrub section.

On the theory that general HA Column scrub-section instability and the gamma burst associated with 3WB flow introduction were related, a test of operation without 3WB flow as made on May 2 while operating with "cold" uranium recycle. No change in HA Column stability was noted during this test.

4. Solvent Treatment

Recycling G-Cell solvent at a 3.0 CF prior to plant start-up levelled the IDO activity at 1500 to 2000 uc/gal. The gamma activity burst associated with the start of the 3WB flow to the HA Column (See Note 3, above) resulted in IDO gamma levels as high as 145,000 uc/gal. "Cold" uranium recycle reduced this level to about 3000 uc/gal. However, on May 3, prior to the return to "hot" feed, the IDO activity again peaked at about 140,000 uc/gal as a result of purging of the ID Column interface to waste. Laboratory investigation of these bursts showed the activity to be readily scrubbable with the usual reagents and no increase in the IDOPu Retention values. Additional work is in progress in an effort to explain the process behavior.

5. Fission-Product Test Plant

Another series of strontium recovery runs was in progress at the end of the report period. Four lead-carrier sulfate precipitations had been completed and the first oxalate precipitation was underway. Failure of the pH meter electrodes prevented accurate pH adjustment on the last three lead-carrier sulfate precipitations. Adjustment of pH on these runs was made solely on the basis of nitric acid analyses.

A 200-gallon cask was loaded with crude strontium on April 19. The material used to fill this cask was a concentrate prepared by thermally concentrating the material returned from the 003 tank along with the material stored in Tank F8. A total of 16.2 kilocuries of strontium 90 was loaded into the cask. The solution composition was as follows:

<u>Component</u>	<u>Curies/Gal.</u>	<u>Grams/Liter (HLO Analyses)</u>
Zr-Nb 90	109	
Ru-Rh	13	
Ce 141-144	792	
Sr 90	97	
Na		28.5
Pb		2.7
Fe		7.3
HNO ₃		65

Appendix --- Process Summary Through May 7, 1961

Following the return to "hot" feed early on May 4, process performance returned toward normal over the week end. The plutonium product was comfortably within specification and the uranium product decreased to about a six gamma ratio.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 5/5/61 TO 2400 ON 5/11/61

I. Typical Solvent-Extraction Performance by Cycles

	Gamma dF		% Recycle		% Loss	
	U	Pu	U	Pu	U	Pu
First	3.6	3.9	-	-	0.003	0.05
Final	2.7	3.0	8.0	1.4	0.004	-
Ion Exchange	-	1.4	-	0.07	-	-
Overall	6.3	8.3	8.0	1.47	0.007	0.05

II. Production (Data included from start-up on April 29)

A. HAF Processed: 241 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
1	9.0	124	2.2	K1
1A	9.0	124	2.9	P2
1B	9.0	124	2.2	K1
2	20.5	181	7.1	E6
3	16.4	141	7.1	E6
4	4.2	57.3	7.1	E6
5	182	25.0	7.1	E6
6	400	4.6	7.1	E6
7	455		7.1	K1
8	580	10.2	2.5	E6
9	477		7.1	E6
10	266	1.4	7.1	E6
11	143		7.1	E6
12	67.5	3.2	7.1	P2
13	32.7	4.0	7.3	P2

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
14	18.5	0.4	7.3	P2
15	12.1		7.4	P2
16	8.6	0.4	7.4	P1
17	7.3	3.2	7.5	P1
18	6.5	5.2	7.0	P1
19	6.1		7.3	P1
20	6.4	5.7	7.2	P1
21	6.2	5.7	7.5	P1
22	6.4		7.3	P1
23	6.1	0.5	7.3	P1
24	6.7	9.3	7.3	P1
25	6.4		7.0	P1
26	6.8	8.8	7.4	P1
27	5.8		7.4	P1
28	6.3	4.2	7.5	P1
29	6.1		7.6	P1
30	6.0		7.6	P1
31	6.2		7.6	P1
32	6.7	6.4	7.6	P1
33	6.6		7.6	P1
34	7.0	4.5	8.0	P1
35	7.0		7.6	P1
36	5.9	1.2	7.5	P1
37	5.4		7.6	P1
38	6.3	1.3	7.6	P1
39	5.0		7.5	P1
40	4.3	2.2	7.4	P1
41	4.9		7.5	P1
42	4.8	2.0	7.3	P1
43	5.0	1.5	7.0	P1

2. Silica Gel: Gamma Ratios - Feed = 6.2
Product = 2.4

Tonnage - 66 Tons

C. Plutonium Product:

<u>Batch</u>	<u>G/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
1	3.6	0.010		4	3	234-5
2	1.6	0.004		4	3	234-5
3	2.3	0.002		5	4	234-5
4	2.7	0.001		5	3	234-5
5	1.9	0.001		5	3	234-5
6	1.8	0.001		5	3	234-5

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C. Plutonium Product:

<u>Batch</u>	<u>27ND AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
7	1.4	0.024		5	3	234-5
8	1.5	0.001		5	3	234-5
9	1.3	0.001		5	3	234-5
10	1.5	0.001	400	6	2	234-5
11	1.4	0.001		6	1	234-5
12	1.7	0.001		5	2	234-5
13	1.1	0.001		5	3	234-5
14	0.8	0.001		4	1	234-5
15	0.7	0.001		5	1	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.60%

Pu = 0.76%

E. Rework - % of Total Production:

U = 29.4% (About 90% U product recycle from TK-K6 to HAF Make-up TK-E6;
about 10% U product recycle from TK-K6 to 2IF TK-K1.)

Pu = 24.3% (Predominantly L-Cell Package flushes via TK-L11 to HAF
Make-up TK-E6.)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	1084	104
Cell Drainage	175	101
FP Waste	248	101
Coating Waste	270	107C

B. PW Flows: 5.4

C. Tank Farms:

<u>Tank</u>	<u>Estimated Boil-Off Rate, Gal/Min</u>
101	13.8
102	0.6
103	0.6
104	8.9

Notes:

1. The HAF was continued at a nominal 3.0 CF throughout the report period. Rate checks showed the actual rate average 3.3 CF.
2. Flowsheet
 - (a) On May 5 the HAX-NO₂ was increased from 0.42 to 0.51 flows.
 - (b) On May 5 the 2DF-Fe flow was reduced from 0.40 to 0.30. On May 7 this stream was increased to 0.46 flows.
 - (c) On May 5 the IBXP-HNO₃ was reduced from 6.5 to 5.6 flows. This stream was further reduced to 5.2 flows on May 6 and then increased to 5.3 flows on May 11.
 - (d) On May 5 the Fresh Nitric Acid was changed from 60% HNO₃ manufactured by General Chemical Co. to 57% HNO₃ manufactured by California Chemical Co.
 - (e) On May 5 the Final Uranium Cycle rate was increased from 3.0 to 3.3 CF. The 2DF-Fe and 2DB flows, however, remained at 3.0 CF rates.
 - (f) On May 5 the HSS was increased from 56 to 66 flows. This stream was reduced to 56 and 50 flows on May 8 and 9 respectively.
 - (g) On May 6 the IBS was reduced from 18.6 to 17.4 flows.
 - (h) On May 8 the IBXP was increased from 18.2 to 19.5 flows.
 - (i) On May 9 the IOP-IDnO₄ was reduced from 3.3 to 1.8 flows.
 - (j) On May 10 the HAO was reduced from 91 to 72 flows. This stream was further reduced to 66 flows on May 11.
 - (k) On May 10 the 3WB was increased from 40 to 42 flows. This stream was further increased to 44 flows on May 11.
 - (l) On May 10 the 2AF temperature was reduced from 60 to 50° C.

3. Solvent Extraction

During the report period the decontamination performance of the HA-HS Columns was 2 to 4 fold better than the normals of recent months. This improvement is shown by the following analytical results for special large-volume HSP samples:

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Date (1961)	Uc./Gal.		
	GSC Gamma	Zr-Nb	Ru
Feb. 13	1.4×10^5	1.0×10^5	1.0×10^4
Feb. 17	7.7×10^4	5.6×10^4	9.8×10^3
Apr. 2	1.13×10^5	7.5×10^4	4.2×10^3
May 10	3.59×10^4	2.4×10^4	5.2×10^3

Current HAF is made up from special non-representative uranium irradiated to >800 MWDT (800 to 1130 MWDT) and "cooled" to about 122 days (103 to 240 days). Calculations for this special metal show the mean gamma activities of concern (Gross Gamma, Zr-Nb, and Ru) are about equal to those for normally irradiated uranium which has been "cooled" 95 to 100 days. Thus the improved decontamination performance of the HA-ES Columns is attributed to the new HA Column scrub section cartridge.

Miscellaneous observations of the HA Column performance characteristics since changing the scrub cartridge are as follows:

- (a) The scrub-section differential pressure, indicative of aqueous hold-up, has increased markedly compared to recent normal values. Aqueous hold-up of this magnitude has not been achieved since July 1958.
- (b) The HA scrub-section cycling continued during the report period but was slightly improved by lowering the ESS and HAO flows. The cycling behavior, the high aqueous hold-up, and the low flooding frequency (compared to HLO studies) for this cartridge may be indicative of a tendency for aqueous wetting rather than organic wetting of the stainless steel surfaces. Poor response of the ESS-H₂O, HAF, and HAW rotameters and/or control valves, which are apparently sticking, also contribute to the cycling.
- (c) Varying the ESS from 50 to 66 flows had no effect on decontamination performance.
- (d) Since decreasing the ESS and HAO flows, the HSP and IBXP specific gravities have increased to values which in the past have correlated with excellent First Cycle decontamination performance.

The decontamination performances of the Final Plutonium and Final Uranium Cycles were below normal. The DF across the 2A Column was improved about 2 fold by reducing the 2AF acidity and the 2AX flow. Reducing these flows in turn increased the 2AW plutonium loss from 0.3 to 1.7%. The Final Uranium Cycle DF, however, remained below normal probably because of the damaged scrub-section cartridge.

Waste losses were excellent except for the 2DU neptunium loss. The internal recycle of plutonium via the IBU and IBSU streams gradually increased early in the report period. This resulted from the increased plutonium level of the HAF (>800 MWDT). The recycle of plutonium via the IBU and IBSU were restored to normal by adjusting the IBXP (IBX) and IBS flows.

4. Solvent Treatment

The gamma activity in the IOO generally remained at 2,000 to 3,000 uc/gal during the report period. A temporary decrease in batch change frequency, necessitated by scheduled waste diversion box work, caused a corresponding increase to 4,200 uc/gal.

Efforts to reduce recent high solvent losses included reduction of IO Column pulse frequency modification of the Tank G1 change procedure so that the interface is jettted only once a day. Returning change cycles in Tanks G1 and G7 to Flowsheet should also reduce losses.

Laboratory analyses of IOO samples, taken during the recent shutdown testing of $Mg(OH)_2$ washing (p. 74), showed less than 0.001 g/l Mg. Organic samples from the Turbomixer TK-G7, taken while operating with $Mg(OH)_2$, showed the same low level of Mg.

5. Waste Treatment and Acid Recovery

Operation of the Waste and Acid Recovery Section has been satisfactory since installation of the new Acid Concentrator (E-F6). Excessive 2NW flow has been reduced, resulting in stable operation of equipment with lower steam and water usage than required when it was necessary to jet F6 to Tank F7 during previous run periods.

Prewarming of Tank 241A-106 was continued by transferring INW wastes representing 70 tons of production to this vessel between May 9 and May 11. On May 11 wastes from F16 and F18 were then routed to Tank 241-A-102 while wastes from Tanks B8 and C8 remain routed to Tank 241-A-104.

6. Fission Product Test Plant

The series of strontium recovery runs reported in progress last report period was completed and 702,000 curies of Sr 89-90 were sent to TK-003. The composition and decontamination factors for the product solution were as follows:

	<u>Curies/Gal.</u>	<u>DF</u>	<u>Lb./Gal.</u>
Sr 89-90	970		
Zr-Nb	570	5	
Ce 141-144	400	5	
Ru-Rh	5	200	
Fe			0.005
SO ₄			0.017
HNO ₃			0.723

The Sr 89 to 90 ratio was measured as 11.7. This agrees well with the calculated value of 12.

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A complete material balance for the series of runs is tabulated below:

	<u>Curies</u>	<u>Percent</u>	
		<u>Input</u>	<u>Output</u>
<u>Sulfate Precipitations</u>			
IWW	1,900,000	100	
Sulfate Waste	470,000		24.7
Flush	87,000		4.6
Carbonate Product	1,400,000		<u>73.6</u>
	Totals	100	102.9
<u>Oxalate Precipitations</u>			
Carbonate Product	1,400,000	73.6	
Oxalate Waste	68,000		3.6
Flush	100,000		5.3
Oxalate Product	1,200,000		<u>63.4</u>
	Totals	73.6	72.3
<u>Concentration Precipitations</u>			
Oxalate Product	1,200,000	63.4	
Concentration Wastes	170,000		8.9
Concentration Product	702,000		<u>36.9</u>
	Totals	63.4	45.8
	Grand Total	100	84.0

As shown in the material balance the loss to sulfate waste was unusually high while the over-all loss across this step was about normal. This suggests a change in the characteristics of the precipitate. Two variables were involuntarily introduced in this series of runs which may have contributed to the change in loss behavior; the last three sulfate precipitations were made without pH control (see p. 81) and the DW was dilute because the plant was not at steady-state when this batch of DW was accumulated.

The material balance was good across the first two steps but poor across the concentration step. Since an 8 - 9 per cent loss across this step is not unusual, it is believed the product samples were in error. The quantity of strontium 89-90 in the product solution was probably close to 1,000,000 curies.

On May 9 a cask of crude strontium was loaded out for the high-level cells. The material was the same as the last two casks (p. 81) except the concentration of Sr 90 was further increased to 117 curies/gal by evaporation.

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Pumping of TK-002 crude strontium to the Hot Semiworks for their first run was completed on May 6. The latest available analyses from the Hot Semiworks indicate 180,000 curies of strontium 90 was transferred to the Hot Semiworks. Using these new analyses the remaining inventory in TK-002 is estimated to be 370,000 curies of Sr 90. The Sr 89 to Sr 90 ratio was 2.48 as of May 12. This is based on radiochemical analyses confirmed by mass spectrographic analyses.

Appendix --- Process Summary Through May 14, 1961

Over the week end the processing of non-representative irradiated uranium was completed and processing of "normal" material was resumed. The transition was made without plant shutdown. The solvent extraction system was stripped with respect to plutonium by recycle of "cold" uranium to the HAF for about 15 hours. The 2BP flow was diverted to the L-Cell Package long enough to permit stripping and emptying Pu from the ion exchange system.

Process performance over the week end was very good. Some difficulty was experienced with apparent HAW valve plugging.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 5/12/61 TO 2400 ON 5/18/61

I. Typical Solvent-Extraction Performance by Cycles

	Gamma dF		% Recycle		% Loss	
	U	Pu	U	Pu	U	Pu
First	3.9	4.0	-	-	0.002	0.03
Final	2.7	3.3	9.0	2.7	0.009	-
Ion Exchange	-	1.1	-	0.1	-	-
Overall	6.6	8.4	9.0	2.8	0.006	0.03

II. Production

A. HAF Processed: 188 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
44	5.3		7.5	P1
45	5.3		8.0	P1
46	5.2	0.6	3.4	E6
46a	5.2		3.4	P1
47	4.8		6.2	P1
48	3.8	0.7	6.5	P1
49	3.6		7.3	P1
50	2.9	0.1	7.1	P1
51	3.0		7.8	P1
52	2.6	0.7	7.7	P4
53	3.4		7.0	P1
54	3.3	0.8	7.0	P1
55	3.9		6.9	P1
56	4.0	0.7	6.9	P3
57	2.6		6.9	P4
58	3.8	0.8	6.9	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, updn U</u>	<u>Tons</u>	<u>Shipped To</u>
59	2.3		7.1	P4
60	1.3		7.1	P4
61	2.1		7.0	P4
62	2.3	0.9	6.9	P4
63	2.2		7.0	P4
64	2.0	0.5	7.0	P4
65	3.3		6.9	P1
66	2.1	0.6	7.5	P4
67	2.0		7.1	P4
68	2.8	0.6	7.1	P1
69	3.9		7.0	P1
70	3.1		7.4	P1

2. Silica Gel

Gamma Ratios: Feed = 3.3
Product = 2.5

Tonnage: 68 Tons

C. Plutonium Product:

<u>Batch</u>	<u>²⁴⁰Am/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, pump Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
16	0.7	0.001		5	1	234-5
17	0.7	0.002		5	2	234-5
18	0.7	0.001		5	1	234-5
19	0.9	0.028		2	1	234-5
20	0.7	0.007		5	1	234-5
21	0.7	0.005		4	2	234-5
22	0.6	0.001		4	3	234-5
23	-	0.005		5	-	Rework
24	1.5	0.001		4	3	234-5
25	1.4	0.004		5	1	234-5
26	1.2	0.008		5	1	234-5
27	1.1	0.007		4	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.06%
Pu = 0.25%

E. Rework - % of Total Production:

U = 6.6% (U Product from TK-P2 and TK-K6 to HAF Make-Up TK-E6)
Pu = 3.3% (L Cell Package, Miscellaneous PR Room Flushes and Sump Material via TK-L11 to HAF Make-Up TK-E6)

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III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	513	104
Cell Drainage	30	102
FP Waste	83	102
Coating Waste	270	107C

B. INW Flows: 6.6

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	14.3
102	0.5
103	0.6
104	9.0

Notes:

1. The HAF was continued at an average 3.3 CF throughout the report period. During the period from 0830 to 2310 on May 12, "cold" uranium was recycled as HAF.

2. Flowsheet

Flowsheet changes summarized below are on the basis of 3.0 CF operating rate.

- (a) On May 12 the 2AF temperature was reduced from 50 to 40° C.
- (b) On May 13 the Final Uranium Cycle was decreased from 3.3 to 3.0 CF. The 2DS and 2DF-Fe flows (gpm), however, were not changed. This, in effect, increased the 2DF-Fe and 2DS from 0.26 and 5.7 to 0.29 and 6.3 flows, respectively.
- (c) On May 13 the 3WB was reduced from 44 to 40 flows.
- (d) On May 13 the HAO temperature was reduced from 42 to 32° C.
- (e) On May 13 the 2DF-HNO₃ was reduced from 9.0 to 7.7 flows. This further reduced to 6.5 flows on May 14.
- (f) On May 15 the XAF was reduced from 0.01 to 0.005 M F⁻. The ratio of Al to F⁻ was maintained at 5.

- (g) On May 15 the IBS was reduced from 17.4 to 16.2 flows.
- (h) On May 15 the IBXP was increased from 19.2 to 20.1 flows.
- (i) On May 16 the IBX was made 0.015 M H_2O_2 . This was increased to 0.030 M on May 17.
- (j) On May 16 the 2AF- NO_2 was increased from 2.62 to 3.62 flows. This was further increased to 3.92 flows on May 18.
- (k) On May 17 the 2DF-Fe was reduced from 0.29 to 0.23 flows.
- (l) On May 18 the HAO was increased from 64 to 77 flows.
- (m) Between May 12 and 18 the IBXP- HNO_3 was gradually increased from 5.30 to 6.40 flows.

3. Solvent Extraction

Processing of the nonrepresentative uranium was completed early on May 12 and the transition to "normal" metal was accomplished without plant shutdown by recycling "cold" uranium for about 15 hours. Compared to operation with the nonrepresentative material, gamma levels of inter-cycle and product streams decreased 1.5 to 2 fold with the return to "normal". The calculated differences in the gross gamma, Zr-Nb, and Ru activities between "normal" HAP and the "special" HAP, however, were only about 1.2 fold. The additional DF apparently resulted from purging the HA-HS Columns with "cold" uranium recycle, and from a gradual improvement in HA Column scrub-section stability and efficiency.

The HA Column scrub-section performance improved throughout most of the report period. Column operation became more stable and the aqueous hold-up in the scrub section decreased, based on scrub-section differential pressure readings. Most of the differential pressure change occurred over an approximately two-shift period beginning late on May 16. Subsequently, on May 17 the pulse frequency of the column was increased from 75 cycles per minute to a normal 81 cycles per minute. In contrast to previous experience with this scrub-section cartridge, the frequency increase had little effect. It is postulated that the change in the HA Column scrub section operation resulted from (a) the change in the wetting characteristics of the new stainless-steel cartridge or (b) gradual breaking of a flooding condition that had started at the time of plant start-up.

Special samples of HAP and HSP were taken on May 16 to measure the distribution of HA-HS Column DF between the two columns:

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Stream	Uc./Gal.		
	Gross (GSC) Gamma	Zr-Nb	Ru
HAP	3.4×10^{34}	2.2×10^4	5.0×10^3
HSP	1.5×10^4	8.2×10^3	3.9×10^3

Compared to February and April analyses, these numbers indicate a seven to nine-fold increase in Zr-Nb decontamination performance with the entire change resulting from HA Column improvement.

Waste losses during the report period were generally excellent. However, the 2DU neptunium loss continued to remain 2 to 4 fold above normal, and the HAW neptunium loss on the average was slightly higher than during the previous week. The HAO flow was increased to reduce the HAW neptunium loss, and the 2D pulse frequency was increased six cycles per minute to reduce the 2DU neptunium loss. It is questionable whether either loss was significantly changed. The IBU plutonium loss was about 1.5 to 2.0 fold below normal as a result of the increased L/V and uranium saturation in the IBX Column.

Other items of interest to solvent extraction were as follows:

- (a) The IBX stream was made 0.03 M N_2H_4 in two steps. No process effects were noted.
- (b) Some difficulties were encountered with the HAW flow early in the week. Exercising the valve ultimately gave satisfactory control, but the HAW valve loading pressure continued to remain above normal.
- (c) The 2D Column pulse frequency was essentially returned to normal. Although the column did not flood, the column was slightly erratic and the 2D Column scrub section differential pressure was substantially above normal.
- (d) The HAX-nitrite flow was lost briefly on one occasion, and the flow became slightly erratic on other occasions. The flow difficulties resulted from trapping nitrous oxide fumes in the "cold"-side piping.

4. Plutonium Concentration

On May 12, during the transition from processing of nonrepresentative uranium and after solvent-extraction plutonium levels had dropped to about 20 fold below normal, the 2BP flow was temporarily diverted to the L Cell Package while the N-Cell ion exchange unit was stripped and emptied insofar as possible. An estimated 2 kg. of special-run Pu remained in the N-Cell-PR system.

Although N-Cell operation has been generally good since the May start-up, some push problems are still being encountered. Push lengths and times have been quite erratic and present indications are that the resin which backs into the push tank through the push line clings to the push probe and interferes with its proper operation.

5. Solvent Treatment

The gamma activity of the IOO has remained at the 2,000 - 3,000 uc/gal range during the report period, while the ²¹⁴Pb activity has been generally less than 20,000 uc/gal. Attempting to reduce solvent losses which have been averaging greater than 800 gallons per day, the IO Column pulse frequency was cut 5 cycles/min and the frequency of jetting the TK-G1 interface during carbonate changes was reduced. Losses have apparently decreased by about a factor of two with no increase in gamma activity due to the changes. The pulse frequency of the 20 Column was also cut 5 cycles/min.

Appendix --- Process Summary Through May 21, 1961

Process performance continued without significant change over the week end. The hydrazine concentration in the IBX was increased from 0.03 to 0.04 M.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 5/19/61 TO 2400 ON 5/25/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.9	4.3	-	-	0.002	0.02
Final	2.8	3.1	9.0	2.5	0.003	-
Ion Exchange	-	1.1	-	-	-	-
Overall	6.7	8.5	9.0	2.5	0.005	0.002

II. Production

A. HAF Processed: 173.0 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
71	2.9		6.8	Pl
72	3.3	1.0	7.2	Pl
73	3.0		7.3	Pl
74	2.9	2.0	7.2	Pl
75	2.7		7.2	Pl
76	3.0	2.7	7.4	Pl
77	2.8	3.9	6.9	Pl
78	2.7	3.9	7.2	Pl
79	2.8		7.2	Pl
80	2.4	1.8	7.1	Pl
81	3.0		7.2	Pl
82	2.4	1.4	7.1	Pl
83	2.8		7.7	Pl
84	2.4	1.5	7.1	Pl
85	2.6		7.1	Pl

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, prod U</u>	<u>Tons</u>	<u>Shipped To</u>
86	2.2	2.0	7.2	P4
87	2.5		7.5	P1
88	2.6	1.5	7.2	P1
89	2.4		7.2	P1
90	2.4	2.3	7.6	P1
91	2.5		7.5	P1

2. Silica Gel

Gamma Ratios: Feed = 3.3
Product = 2.5

Tonnage: 232 Tons*

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, prod Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
28	1.2	0.004		5	3	234-5
29	1.2	0.025		5	2	234-5
30	1.0	0.024		4	1	234-5
31	0.8	0.011		5	1	234-5
32	0.7	0.004		4	3	234-5
33	1.2	0.013		4	3	234-5
34	1.3	0.003		6	1	234-5
35	0.8	0.003		4	3	234-5
36	0.7	0.002		5	1	234-5
37	0.6	0.009		5	1	234-5
38	1.2	0.009		5	1	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.045
Pu = 0.35

E. Rework - % of Total Production:

U = Negligible
Pu = 2.05% (L10 Flushes and SLD Sump from TK-L11 to HAF Make-Up TK-36)

*Includes 159 tons sent to Redox for silica-gel processing.

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III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	598	104
Cell Drainage	128	102
FP Waste	40	102
Coating Waste	270	107C

B. I/W Flows: 5.4

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal./Min.</u>
101	13.9
102	0.6
103	0.9
104	8.6

Notes:

- The HAF was continued at an average 3.3 CF throughout the report period except for an emergency shutdown from 1840 to 2200 on May 25.
- Flowsheet
 - On May 19 the ZDS was increased from 62.5 to 66.5 flows.
 - On May 19 the ZDF ferrous sulfamate was decreased from 0.26 to 0.19 flows. It was returned to 0.26 flows on May 21. On May 25 the ZDF ferrous sulfamate was increased to 0.53 flows.
 - On May 19 the IBX was increased from 0.03 to 0.04 M N_2H_4 . The N_2H_4 concentration was further increased to 0.05 M on May 22.
 - Between May 19 and 25 the 2AF $NaNO_2$ flow was varied from 2.78 to 5.56 flows. On May 25 this stream was set at 4.56 flows.
 - Between May 19 and 24 the IBXP- HNO_3 was gradually increased from 6.3 to 7.6 flows. On May 24 the IBXP- HNO_3 was reduced to 7.1 flows.
 - On May 21 the ZDF- HNO_3 was increased from 6.5 to 7.0 flows. It was further increased to 7.7 flows on May 22. On May 23 and 25 the ZDF- HNO_3 was reduced to 6.5 and 5.6 flows respectively.
 - On May 22 the HAO was increased from 76 to 91 flows.

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- (h) On May 22 to 24 the HSR-HNO₃ was increased from 5.5 to 7.5 flows.
- (i) On May 23 the IBX sulfamic acid concentration was reduced from 0.034 to 0.026 M. On May 24 it was further reduced to 0.017 M.
- (j) On May 23 the 2AF temperature was increased from 40 to 55° C.
- (k) On May 23 the IOB carbonate was reduced from 19.2 to 14.4 flows.
- (l) On May 23 the IOC and IOD were reduced from 11.9 to 6.0 flows.
- (m) On May 23 the 2AX was increased from 3.8 to 4.3 flows for about 24 hours. On May 25 it was again increased to 4.3 flows for about 8 hours.
- (n) On May 23 the IBXP was reduced from 20.1 to 18.4 flows and then increased to 19.2 flows on May 24.

3. Solvent Extraction

Solvent-extraction performance continued to be excellent during the report period. Although the gamma levels of intercycle streams increased slightly, the product gamma activities were virtually unaffected. The intercycle activity increase resulted from a policy of gradual reduction of HA Column saturation to maximize neptunium recovery. Thus, neptunium recovery in the HA-HS system also continued to be good and HAW plutonium losses correspondingly decreased 2 to 3 fold during this period.

Testing of hydrazine in the IBX was continued with the gradual increase of hydrazine concentration to 0.05 M and a reduction in sulfamate concentration from 0.034 M to 0.017 M. The effects of these changes have been difficult to evaluate because of some instrument problems and erratic 2AW Pu "losses". The 2AW losses did not consistently respond to normal corrective measures, i.e., increases in the 2AF HNO₃, 2AF NaNO₂, and/or 2AX flows. At present it is postulated that the NaNO₂ - N₂H₄ reaction may be slower than expected, with the holdup time in the 2AF Tank perhaps borderline. It does appear that hydrazine in the IBX does result in lower Pu concentration in the IBSU and a higher ferrous-to-ferric ratio in the IBP.

The entire plant was shut down for about 3 hours by a power failure on May 25. The emergency systems performed as expected except for failure of the instrument air compressor to start, due to low voltage. The start-up which followed was uneventful, with a very slight gamma burst hardly worth noting.

4. Solvent Treatment

Except for the first two days of the report period when gamma levels were 2,000 to 4,000 uc/gal, the gamma activity in the IOC has remained low at about 1500 uc/gal. In an effort to reduce solvent losses, change times in TK-G1 were extended such that the IOC flow was reduced from 19.2 to 14.4 flows and the IOC-IOD rate was reduced from 11.9 to 6 flows. These changes, along with jetting the TK-G1 interface only once per day vice previous 4 times daily, have reduced solvent losses by approximately 30%.

5. Demineralized Water

On May 23, as part of the monthly water quality control program, a demineralized water sample was sent to 321 Bldg. for column performance analysis. It was subsequently reported that the water was the best of any tested to date, and was comparable to distilled water in column performance.

On May 20 large amounts of barnacle-type solids were removed from the HSS rotameter. Since cleaning, the rotameter bob still occasionally becomes stuck by single large pieces. Previous experience indicates that this material originates in TK-223, the demineralized water head tank, and is composed principally of aluminum and silica.

6. Fission Product Test Plant

(a) Strontium Recovery Runs

A series of strontium recovery runs was started on May 13. The first two lead-carrier sulfate precipitations were completed normally. The third run proceeded normally until the carbonate cake removal step. Then, despite repeated efforts to dissolve and flush the cake from the centrifuge with acid, the Beckman and temperature readings indicated the cake remained in the bowl. Previous experience had shown that "stubborn" cakes could be removed with oxalic acid. However, carbonate product solution stored from the first two runs in TK-E1 made special oxalic acid flushes difficult. Instead, it was decided to proceed to the oxalate precipitation step in an attempt to clean out the centrifuge while making the strontium and cerium-rare earth separation. The oxalate successfully reduced the activity in the centrifuge; however, little of the strontium associated with the third carbonate cake was found in the oxalate product.

The cake removal difficulty was subsequently determined to be due to a partially plugged upper spray system which caused the pressure relief valve on the spray pump to open and reduce the pressure on

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the sprays below normal operating pressures. Although solution could be pumped slowly into the centrifuge at reduced pressure, it was not effective in removing the cake.

The oxalate product solution was stored until May 28 when the final concentration step was completed. A total of 840,000 curies of Sr 89-90 was sent to TK-003 on May 29. The chemical analyses and decontamination factors for the product solution were as follows:

	<u>Curies/Gal.</u>	<u>DF</u>	<u>Lb./Gal.</u>
Sr 89-90*	920		
ZrNb	920	3.6	
Ce 141-144	1100	2.4	
RuRh	34	3.0	
Fe			0.005
SO ₄			0.017
HNO ₃			0.34

*The Sr 89 to 90 ratio was 9.84. The poor cerium DF is apparently due to cross-contamination. The cerium DF through the oxalate step was 6.9.

A complete material balance for the series of runs is as follows:

		<u>Percent</u>	
	<u>Curies</u>	<u>Input</u>	<u>Output</u>
<u>Sulfate Precipitations</u>			
LW	2,100,000	100	
Sulfate Waste	220,000		10.5
Flush (Estimate - Not Measured)	210,000		10.0
Carbonate Product	1,300,000		61.9
Totals		100	82.4
<u>Oxalate Precipitations</u>			
Carbonate Product	1,300,000	61.9	
Oxalate Waste	300,000		14.3
Flush	99,000		4.7
Oxalate Product	1,200,000		57.0
Totals		61.9	76.0
<u>Concentration Precipitations</u>			
Oxalate Product	1,200,000	57.0	
Concentration Waste	73,000*		3.5
Concentration Product	1,050,000*		50.0
Totals		57.0	53.5
Grand Total		100	93.0

*Corrected for 15 days of decay between sulfate runs and concentration runs.

The high loss across the carbonate step is believed due to the difficulty in removing the carbonate cake from the third sulfate run. The excess strontium found in this step (14 per cent more than carbonate product) nearly balances the strontium shown to be left in the centrifuge from the sulfate material balances.

(b) Strontium Shipment

The HAPO 1A Cask containing 25,000 curies of strontium-90 was received from 300 Area on Tuesday, May 16. It was left vented until noon the next day awaiting new secondary closures. At noon on May 17, the cask was sealed with the old closures because the new replacement closures were not yet available. The rate of pressure build-up was linear at 16 psig/day. When a pressure of 30 psig was reached the cask was vented and a gas sample obtained for analyses. The cask was sampled twice more before being shipped as described later. The analyses on the three samples were as follows:

<u>Component</u>	<u>Volume Per Cent</u>		
	<u>5/19</u>	<u>5/20</u>	<u>5/25</u>
CO ₂	0.66	0.63	0.45
A	0.01	0.01	0.01
O ₂	2.70	3.44	3.49
N ₂	3.20	3.05	1.46
H ₂ O	1.70	0.38	3.24
CO	0.35	0.26	0.01
H ₂	-	-	0.01
H ₂	91.4	92.2	91.36

The cask was sealed for the second time on May 19 and observed to increase in pressure at the rate of 24 psig/day. The cask was again vented early on May 20 and the gas sampled in preparation for installing the new secondary closures. However, the closures did not fit and the cask was not sealed again until late that night.

After the third sealing the pressure built up at the rate of 24 psig/day until a pressure of 45 psig was reached. The slope then decreased to the original rate of 16 psig/day. On May 25 the cask was vented after reaching a pressure of 90 psig. The third gas sample was obtained at this time. The cask was sealed a fourth time for 15 hours and a pressure build-up rate of 29 psig/day was observed.

From the above experience with the cask it was concluded that the rate of pressure build-up increased each time the cask was vented and sealed, probably because of the accumulation of radiolytic

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decomposition products in solution. In an attempt to prepare a cask suitable for shipping it was decided to wash out these radiolytic decomposition products and increase the void volume, both of which should lower the rate of pressure build-up.

On May 26 the cask was washed with 20 gallons of distilled water and the void volume increased to 2 gallons. The cask was sealed and reached a pressure of 30 psig on May 31. At this time the rate of pressure build-up was decreasing with a build-up rate of 2 to 3 psig/day observed on May 31. The cask was released for shipment on Thursday, June 1.

The reasons for the difference in behavior between this shipment and the first shipment are not known. The differences between the two shipments are tabulated below for reference.

Comparison of HAPO 1A Shipments 1 and 2

<u>Shipment #1</u>	<u>Shipment #2</u>
1) 520 BTU/Hr.	620 BTU/Hr.
2) Unused Decalso	Used Decalso
3) Stored vented 4 days before sealing	Stored vented 1 day before sealing
4) Void volume 1/2 liter (estimate)	Initial void volume 1-1/2 liters (estimate)
5) Loading: 1st half from NH_4NO_3 2nd half from HEDTA Copper loaded into cask and quantitatively eluted	All from NH_4NO_3
6) Ambient temperature 50° F	Ambient temperature 90 - 100° F

(c) Miscellaneous

Other items of interest to fission product recovery were:

- (1) The contents of TK-002 and TK-003 were combined in TK-002 on May 20 before the run reported above was added to TK-003.
- (2) Between May 18 and May 22, 170,000 curies of Sr-90 was transferred to Hot Semiworks.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 5/26/61 TO 2400 ON 6/1/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.7	4.0	-	-	0.003	0.02
Final	2.8	2.9	11.0	1.5	0.004	-
Ion Exchange	-	1.4	-	0.1	-	-
Overall	6.5	8.3	11.0	1.6	0.007	0.02

II. Production

A. HAF Processed: 195 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppm U</u>	<u>Tons</u>	<u>Shipped To</u>
92	2.5		7.1	P1
93	2.6		7.1	P1
94	3.0	4.7	7.0	P1
95	2.8		7.1	P1
96	3.1	2.4	7.2	P1
97	3.2		7.3	P1
98	3.3	2.4	7.1	P1
99	3.3		7.3	P1
100	3.2	2.3	7.1	P1
101	3.1		7.4	P1
102	2.9	1.8	7.4	P1
103	3.2		7.8	P1
104	2.8	0.9	7.4	P1
105	3.4		7.2	P1
106	3.4	2.2	7.1	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
107	3.7		7.6	Pl
108	3.8	2.2	7.3	Pl
109	3.6		7.5	Pl
110	3.6	3.1	7.5	Pl
111	3.8		7.5	Pl
112	3.7		7.7	Pl
113	3.9		7.4	Pl
114	4.1	3.0	7.7	Pl
115	4.2		7.5	Pl
1	4.5	1.8	7.5	Pl
2	3.3	1.2	7.5	Pl
3	4.1		7.8	Pl
4	4.6	2.0	8.0	Pl
5	4.7		7.8	Pl
6	4.3	0.4	7.8	Pl

2. Silica Gel

Gamma Ratios: Feed = 3.6
Product = 2.1

Tonnage: 140

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
39	1.3	0.005		5		234-5
40	3.0	0.001		4		234-5
41	3.3	0.001		6		234-5
42	1.5	0.002		4		234-5
43	1.4	0.001		6		234-5
44	2.0	0.003		5		234-5
45	1.0	0.001	450	3		234-5
46	2.7	0.003		6		234-5
47	1.4	0.001		5		234-5
48*	2.0	0.006		3		234-5
49	1.5	0.008		5		234-5
1	7.1	0.030			Recycled to E6	
2	2.3	0.001		4		234-5

*Batch Number 48 was shipped to 234-5 on the basis of "GSC" gross
gamma/AT.

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D. Overall Waste Loss - % of Total Production:

U = 0.04%

Pu = 0.17%

E. Rework - % of Total Production:

U = 6.9% (Out-of-Spec. "U" Product from TK-P2 to 2DF TK-K1 through Final Uranium Cycle, only)

Pu = 11.6% (Predominantly out-of-spec. Pu Batch #1 Recycled via L11 to HAF Make-up Tank E6)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	392	104
Cell Drainage	85	102
FP Waste	18	102
Coating Waste	270	107C

B. IWW Flows: 6.4

C. Tank Farm:

<u>Tank</u>	<u>Estimated Boil-Off Rate, Gal/Min</u>
101	14.5
102	4.2
103	0.4
104	7.3

Notes:

1. The HAF was continued at an average 3.3 CF throughout the report period except for an emergency shutdown from 2330 on May 30 to 0630 on May 31 which was caused by HAF pump failure.

2. Flowsheet

(a) On May 26 the 2AX was increased from 3.8 to 4.6 flows. It was returned to 3.8 flows on May 30.

(b) On May 26, 28, 29, and June 1 the 2AF NaNO₂ addition was reduced to 4.3, 4.2, 4.0, and 3.5 flows, respectively. Prior to the first reduction this rate has been 4.6 flows.

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- (c) On May 29 the IBX sulfamic acid concentration was reduced from 0.017 to 0.009 M. The addition of sulfamic acid to the IBX stream was stopped on May 31.
- (d) On May 26 the HAF, HAX, IBXF, ICX, 2DF, 2DX, and 2EX flows were increased from 3.0 to 3.1 CF. These flows were further increased to 3.2 CF on May 27. On May 30 these flows were returned to CF = 3.1. During these changes, the other solvent extraction flow rates, in gallons per minute, remained constant.
- (e) On May 29 the 2DF- HNO_3 , 2DF, 2DX, and 2EX flows were increased from 3.2 to 3.4 CF. The 2DS and 2DF ferrous sulfamate flow rates, in gal./min., remained constant during these changes.
- (f) On May 29 the 3WB was increased from 38 to 41 flows. On May 31 the 3WB was further increased to 43 flows.
- (g) On May 29 the HAX NaNO_2 addition rate was increased from 0.44 to 0.48 flows.

3. Solvent Extraction

Solvent-extraction performance continued to be excellent during the report period. Gamma levels continued the gradual rise previously reported (p. 99) as the HA Column saturation reduction was continued in the interests of neptunium recovery. The only activity increases of concern, however, occurred in the Pu product as a result of changing from one product load-out tank to another, and as a result of a process gamma burst associated with a short emergency shutdown on May 30.

The test of the use of hydrazine in the IBX was continued with the complete removal of sulfamic acid from the IBX. As previously noted (p. 99), evaluation of the test has been difficult because of the erratic pattern of 2AW "losses". In any event, an anticipated lowering of the 2AW "losses" as a result of decreasing the sulfate (from sulfamic acid) content of the 2AF has not materialized. Some further study of this problem is planned.

Other items of interest to solvent-extraction were as follows:

- (a) A seven-hour production outage resulted from failure of the HAF pump late on May 30. A noticeable gamma burst was associated with the start-up following this outage as a result of HA-E3 Column stripping, to avoid neptunium losses, and as a result of HAX and HAF instrumentation problems.
- (b) Rework of UO_2 Plant off-standard URE via the Final Uranium Cycle at about 2 tons per day was commenced on May 29 and was continued without incident during the report period. The Final Uranium Cycle Operation was maintained with this material alone during the HAF outage noted above.

4. Solvent Treatment

Significant reduction in solvent losses has resulted from recent decrease of the TK-G1 carbonate change frequency and decrease of the frequency of jetting the TK-G1 interface. Previously reported high solvent losses (pp. 87, 95) have now been returned to about normal (0.3%). Meanwhile, the IOO activity has been maintained at about 2,000 uc./gal., with about 80% of this activity being ruthenium.

5. Acid Recovery and Waste Treatment

The temperature in Tank 241-A-101, which had increased from about 198° C to 207° C during May, apparently was responding to further water additions and had decreased about 4° C during the last two days of the report period. Temperature increases in this tank originally commenced in late March, increasing from 128° C to 198° C by the first week in May (see p. 74). Recent water additions have increased the tank liquid level from about 285 inches to about 330 inches.

Purge water flow to the I/W Concentrator (E-F11) instrument diptubes has been recently increased by installation of larger control orifices. It is expected that better specific gravity control and better I/W flow control should result.

6. Fission Product Test Plant

The last series of strontium recovery runs for the SNAP 7 program was started on May 29. Three lead carrier sulfate precipitations had been completed by the end of the report period.

Appendix --- Process Summary Through June 4, 1961

Gamma levels of intercycle streams increased about 50 per cent over the week end as a result of an instrumentation problem associated with an attempted HAX increase.

An apparent restriction to the vapor and/or condensate flow from the I/W Concentrator F11 resulted in excessive pressure in the concentrator, reduced I/W processing capacity, and an approximate 10-fold increase in the Absorber acid activity level. This problem is being investigated.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 6/2/61 TO 2400 ON 6/8/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.7	3.9	-	-	0.005	0.02
Final	2.8	3.2	12.0	2.0	0.005	-
Ion Exchange	-	1.2	-	0.1	-	-
Overall	6.5	8.3	12.0	2.1	0.010	0.02

II. Production

A. HAF Processed: 190 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
7	3.2		7.6	P1
8	3.2		7.6	P1
9	2.7		7.6	P1
10	2.6	1.3	7.7	P1
11	2.7		7.8	P1
12	2.6	1.4	7.7	P1
13	2.6		7.5	P1
14	2.7	1.7	7.6	P1
15	3.1		7.6	P1
16	4.0	4.3	7.6	P1
17	4.1		7.7	P1
18	4.3	1.8	7.1	P1
19	5.2		7.4	P1
20	5.1	4.1	7.4	P1
21	5.5		7.8	P1

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
22	5.5		7.6	P1
23	6.1		7.1	P1
24	5.5	3.9	7.6	P1
25	5.6		6.9	P1
26	6.5		7.6	P1
27	8.4		7.8	P1
28	9.7	4.6	7.7	P1
29	10.0		7.7	P1
30	10.7	4.9	7.6	P1
31	9.5		7.8	P1

2. Silica Gel

Gamma Ratios: Feed = 5.2
Product = 2.0

Tonnage: 137

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
3	1.9	0.002		6	2	234-5
4	1.4	0.023		5	3	234-5
5	1.1	0.019	△470	5	2	234-5
6	0.9	0.009		5	2	234-5
7	1.3	0.006		4	3	234-5
8	1.4	0.002		6	1	234-5
9	1.4	0.003		5	2	234-5
10	1.0	0.029	△1000	5	2	234-5
11	1.2	0.002		5	2	234-5
12	1.8	0.129		5	2	234-5
13	3.7	0.272		5	1	234-5
14	5.3	0.093		5	6	234-5
15	2.4	0.072		5	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.11%
Pu = 0.13%

E. Rework - % of Total Production:

U = Negligible
Pu = 15.0% (Includes out-of-spec. Pu and miscellaneous L-Package flushes
recycled via L11 to HAF Make-Up TK-E6)

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III. Waste Treatment

A. Volumes to UCS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	428	104
Cell Drainage	33	102
FP Waste	56	102
Coating Waste	270	107C

B. DW Flows: 8.6

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	10.8
102	6.9
103	0.5
104	8.3

1. Notes

The HAF was maintained at a 3.3 CF throughout the report period except for a 5-minute outage on June 6 caused by a temporary failure of the HAF pump.

2. Flowsheet

- On June 2 and 3 the 2AF-nitrite was reduced from 3.60 to 3.27 flows.
- On June 2 the 2DF ferrous sulfamate was reduced from 0.52 to 0.34 flows.
- On June 2 the 3WB was reduced from 45 to 42 flows, but was increased to 43.5 flows on June 3.
- On June 3 the BSR-HNO₃ was increased from ~ 7.8 to ~ 8.1 flows, but was returned to ~ 7.8 flows on June 4.
- Between June 3 and 5 the DW-H₂O was reduced from 215 to 106 flows.
- On June 5 the HAX, HAF, HAX-nitrite, IBXF, ICX, 2DF, 2DX, and 2EX were increased from 3.1 to 3.2 CF.
- On June 5 the 3WB-HNO₃ was started at 2.0 flows. It was increased to 3.0 flows on June 6.

- (h) On June 6 the 2AX was reduced from 3.2 to 2.2 flows.
- (i) On June 6 the 2AS was reduced from 1.7 to 1.0 flows. It was increased to 1.3 and 1.5 flows on June 7 and 8, respectively.
- (j) On June 7 the addition of 0.0005 M $\text{Hg}(\text{NO}_3)_2$ to the metal solution was commenced.

3. Solvent Extraction

Although the gamma activities of intercycle streams increased about two fold during the report period, the plutonium product continued to meet specifications. The uranium product also met specifications after silica-gel treatment, but was approaching the maximum gamma level suitable for silica-gel feed at the end of the report period. The increased gamma levels at least partly resulted from continued reduced HA Column saturation (see also pp. 99, 107). In addition, appreciable gamma bursts resulted from

- (a) A defective instrument which caused doubled HAX flow and HA-HS Column flooding for a brief period on June 3; and
- (b) Loss of HAF flow for about five minutes on June 6.

Near the end of the report period the HA Column saturation was increased very slightly to improve the decontamination performance.

Waste losses were generally normal. The HAW neptunium and plutonium losses, however, were exceptionally low because of the low HA Column saturation. The plutonium and neptunium levels in the uranium product, on the other hand, were both two to three fold above normal in spite of a high 2DF ferrous sulfamate flow. These above-normal 2DU losses are attributed to the damaged 2D Column scrub section.

The 2AX flow was reduced to increase the 2AW plutonium loss from about 0.6 to 2.0%. The 2AS flow was also initially reduced simultaneously in order to maintain the 2A scrub-section L/V constant. When the 2AX flow reached the minimum controllable flow the 2AW loss was still less than 2.0%. A 2AW loss of 2.3% was achieved by increasing the 2AS flow back to 90% of normal. At week's end, following the adjustment of the 2AW loss the hold-up in the 2AF Tank J5 was gradually decreased from about 40 to 20 minutes. Reducing the hold-up in turn was apparently increasing the 2AW plutonium loss.

Other items of interest to solvent extraction were as follows:

- (a) The rework of "Waste" UNH from the UO_3 Plant was completed without incident on June 3.

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- (b) The uranium content of the 2BP and plutonium product suddenly increased and remained about 50-fold above normal on June 7. The 2AP uranium analyses, although erratic, have not shown an increase of this magnitude.

4. Solvent Treatment

The gamma activity in the No. 1 Solvent System reflected the plantwide increased activity due to lower EA saturation. However, even though the ICW increased from ~ 7,000 to 20,000 uc/gal the 100 increased from ~ 2,000 to 2,400 uc/gal.

5. Waste Treatment and Acid Recovery

(a) Pressurization of the IWV Concentrator F11

Commencing late on June 2, attempts to moderately increase the boil-off rate from the IWV concentrator F11 resulted in severe pot pressurization, with some initial measurements showing as high as 12 in. of water pressure at the bottom of the concentrator tower. Further attempts to increase boil-off rate apparently caused F11 bottoms (IWV) to actually rise into the tower section via the tray risers. The seal pot instrumentation indicated increased specific gravity (above the normal sp.g. of water), and drastic F11 steam flow reductions caused sudden sharp increases in the F11 overflow rate and receiver (TK-F12) liquid level. During one period of apparent tower holdup, a positive pressure of 55 in. of water was measured at the bottom of the tower. A review of operating data showed that this loss of pot vacuum had been gradually occurring since about May 8, at which time the vacuum was normal at about 16 in. of water.

Investigation of the pressurization problem included the following:

- (1) On the theory that a gross tube bundle leak might be causing the trouble, both bundles were checked by pressurizing with water. No leaks were indicated.
- (2) On the theory that the trouble might be due to leaks in the IWV condensate line to E-F6 (2WV Concentrator), the F6 steam flows (and, therefore, the F6 pot vacuum) were varied. No effect on F11 was noted.
- (3) Pressure readings were taken with the F11 steam flow reduced to insure a pot vacuum:

<u>Measuring Point</u>	<u>Vacuum, In. of H₂O</u>
Condenser Vent	50
Top of F11 Tower (via spray ring)	41
Between 1st and 2nd tray from top of F11 tower	36
Bottom of tower	5

The major pressure drop is thus indicated to be across the tower trays.

Attempts to correct this problem included flushing of the tower with 500-gal. increments of hot water. No changes resulted. A test of the use of an antifoam agent is planned, and chemical flushing of the tower during the next shutdown is anticipated. In the meantime, it is necessary to operate F11 with about 35% less steam flow, and with corresponding increased IWW flow and decreased acid recovery.

(b) Miscellaneous

The in-trench line from nozzle FT-40 to FT-30 which had been plugged with IWW solution from the IWW Anion Exchange Test was finally freed after several weeks of water and steam flushing.

The temperature in Tank 241-A-101 decreased from 200° C to 171° C during the report period while the liquid level was held at 335 inches.

6. Fission Product Test Plant

The last series of strontium recovery runs for the Snap 7 program was completed on June 7. A total of 1,090,000 curies of Sr. 89-90 was sent to TK-003 on June 8. The chemical analyses and decontamination factors for the product solution were as follows:

	<u>Curies/Gal.</u>	<u>DF</u>	<u>Ld./Gal.</u>
Sr 89-90	1360		
ZrNb	450	12	
Ce 141-144	740	6.6	
RuRh	ND*	-	
Fe			0.054**
SO ₄			0.017--
HNO ₃			0.243
Sr 89/90 Ratio			11.8

*Nondetectable.

**Believed to be a "flyer".

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The reasons for the unusually high Zr/Nb DF are not known. Analyses of oxalate product show a Zr/Nb DF of 9 which confirms the final DF observed.

A complete material balance for this series of runs is as follows:

	<u>Curies</u>	<u>Percent</u>	
		<u>Input</u>	<u>Output</u>
<u>Sulfate Precipitations</u>			
ISW	2,200,000	100	
Sulfate Waste	230,000		10.5
Flush	26,000		1.2
Carbonate Product	Not Sampled	-	-
	Totals	100	-
<u>Oxalate Precipitations</u>			
Carbonate Product	Not Sampled	-	
Oxalate Waste	320,000		14.5
Flush	85,000		3.9
Oxalate Product	1,100,000	-	50.0
	Totals	-	68.4
<u>Concentration Precipitations</u>			
Oxalate Product	1,100,000	50.0	
Concentration Waste	65,000		3.0
Concentration Product	1,090,000	-	49.5
	Totals	50.0	52.5
	Grand Total	100	82.6

The above material balance indicates the measured losses across the sulfate step are probably low. The high loss to the oxalate waste is typical of a poor cleanout between the sulfate and oxalate runs.

One set of Hypalon buffer rings for the E-Cell centrifuges has been received and tested. Hypalon was recommended by HLO Chemical Equipment Development Operation as a substitute for the presently used Neoprene buffer rings because of its greater resistance to thermal and radiation degradation. The mechanical performance of the Hypalon rings was satisfactory. As soon as additional rings are available, one will be submitted for thermal tests.

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Other items of interest to fission product recovery were:

- (a) A low speed long shaft agitator for TK-23 is expected to be available in July.
- (b) The HAPO 1A Cask arrived in Oak Ridge on June 7. The cask showed no signs of leakage and the pressure was greater than 60 psig (limit of gauge used).

Appendix --- Process Summary Through June 11, 1961

Over the week end, process performance continued without significant change. On June 10, the addition of hydrazine to the IBX was stopped and the use of normal IBX (with 0.04 M excess sulfamic acid) was commenced.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 6/9/61 TO 2400 ON 6/15/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dP</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.7	3.8	-	-	0.004	0.20
Final	2.7	3.0	13.0	11.6	0.003	-
Ion Exchange	-	1.3	-	0.1	-	-
Overall	6.4	8.1	13.0	11.7	0.007	0.20

II. Production

A. HAF Processed: 185 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu,ppbm U</u>	<u>Tons</u>	<u>Shipped To</u>
32	9.8	5.5	7.6	P1
33	9.9		7.6	P2
34	9.1	6.8	7.8	P2
35	7.8		7.6	P2
36	7.9	5.9	7.6	P2
37	7.5		7.7	P2
38	7.9	5.5	7.7	P1
39	7.8		7.8	P1
40	7.6	4.2	7.8	P1
41	7.2		7.8	P1
42	7.4	4.7	7.9	P1
43	5.8		7.4	P1
44	6.0	3.3	7.7	P1
45	5.1		7.7	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
46	5.1	64	7.5	P2
47	5.3	201	7.8	P2
48	4.4	140	7.6	P2
49	4.1	62	6.7	P2
50	3.5	13	7.4	P1
51	3.6	15	7.4	P1
52	3.5	14	2.3	E6
52a	3.5	14	5.2	P1
53	4.3	7	5.7	P1
54	4.3	5	7.4	P3
55	4.4		7.5	P3

2. Silica Gel

Gamma Ratios: Feed = 6.1
Product = 2.4

Tonnage: 11.5

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
16	2.1	0.091		5	3	234-5
17	1.9	0.080		5	2	234-5
18	1.6	0.080		5	2	234-5
19	2.1	0.119		5	4	234-5
20	2.3	0.160	1360	5	1	234-5
21	29.7	0.840		3	29	E6
22	-	-		-	-	E6
23	19.3	0.830		4	21	Holding
24	16.7	0.830		4	17	Holding
25	16.4	0.020		-	-	E6
26	198	0.001		-	-	E6
27	133	0.014		6	190	Holding
28	64.9	0.021		4	80	Holding
29	35.9	0.006		5	50	Holding

D. Overall Waste Loss - % of Total Production:

U = 0.07%
Pu = 0.44%

E. Rework - % of Total Production:

U = 1.3% (U Product from ZEU TK-K5 to HAF Make-up TK-E6)
Pu = 20% (Out-of-spec. Pu Product to HAF Make-up TK-E6 via TK-L11)

III. Waste Treatment

A. Volumes to UCS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	447	104
Cell Drainage	54	102
FP Waste	114	102
Coating Waste	270	107C

B. IWF Flows: 7.3

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	10.0
102	7.5
103	0.3
104	3.7

1. Notes

The HAF rate was maintained at 3.3 CF throughout the report period except for a three-hour outage early on June 13 during a partitioning failure.

2. Flowsheet

- (a) On June 9 the ZDF ferrous sulfamate was increased from 0.27 to 0.36 flows. On June 11 and 12 the ZDF ferrous sulfamate was increased to 0.44 and 0.87 flows, respectively. On June 14 it was reduced to 0.53 flows.
- (b) Between June 9 and 14 the IWF-H₂O flow was gradually decreased from 106 to zero flows.
- (c) On June 10 the HES was increased from 45 to 50 flows.
- (d) On June 11 the hydrazine addition (0.05 M) to the IBX was stopped. At the same time the IBX was made 0.034 M sulfamic acid.
- (e) On June 11 at 0930 the ZBP stream was diverted from N Cell (anion exchange) to the L-Cell Package. The ZBP flow was returned to N Cell at 0930 on June 15.

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- (f) On June 12 the IBS was increased from 16.1 to 20.7 flows; it was decreased to 18.2 flows on June 13.
- (g) On June 12 the IBXP was increased from 17.4 to 21.7 flows; it was decreased to 18.8 flows on June 13.
- (h) On June 12 the 2AF-nitrite (3.12 flows) was stopped for a four-hour period (during partitioning failure). The 2AF-nitrite was restarted on June 13 at 3.12 flows.
- (i) On June 13 Fresh HNO_3 (57%) was started to the 2AF Tank at 2.24 flows.
- (j) On June 13 the JWB was increased from 40 to 43 flows; it was returned to 40 flows about twenty hours later.
- (k) On June 14 the IAF stream was made about 1.5 ppm in antifoam for a 16-hour period.
- (l) On June 14 the Final Uranium Cycle rate was increased from 3.3 to 3.5 CF. The 2DF-ferrous sulfamate and 2DS flows (gpm), however, were not increased.

3. Solvent Extraction

Solvent-extraction performance was marked by

- (a) Excessive plutonium losses to the HAW commencing on June 10; and
- (b) A loss of partitioning on June 12, caused by the omission of flowsheet sulfamic acid in two consecutive batches of IBX.

The HAW plutonium losses were generally about 10 fold above normal, but peaked at about 2 per cent. HA Column saturation reduction resulted in no improvement. The loss pattern correlated with operating difficulties in the Plutonium Ion Exchange Facility, a pattern which has been previously noted. Apparently, off-standard ion-exchange performance leads to a product recycle pattern, either via XAW to JWB or via rework transfers to the HAF (L11 to E6), which causes the losses. Further laboratory investigation of this problem is in progress.

The testing of hydrazine as a stand-in holding reductant for excess sulfamic acid in the IBX was terminated on June 10. The test results successfully demonstrated the use of hydrazine for this purpose in the Purex Plant. However, the predicted beneficial affect of the sulfamate reduction on the 2AW "losses" was not conclusively shown.

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The 2AW loss pattern was generally inconsistent, as though it were controlled by some other factor. Prior to the removal of hydrazine from the IBX, the hold-up time in the 2AF Tank J5 was varied from 20 to 70 minutes to check the possibility of a hydrazine-nitrite kinetics problem. Here again the 2AW loss pattern was not consistent; an apparent initial response did not persist. It is concluded that further study of this particular 2A Column is necessary, using normal flowsheet IBX-IBP.

Other items of interest to solvent extraction were as follows:

- (a) Rework of uranium which exceeded the plutonium specification was started in the Final Uranium Cycle on June 14.
- (b) The quantity of uranium in the plutonium product, although improved, was still several fold above normal.
- (c) The HAF flow was lost twice for a brief time at the end of the report period. This caused severe HSP gamma bursts of only short duration.

4. Final Plutonium Concentration

On June 11 the ZBP stream was diverted to the L-Cell Package because of ion-exchange resin pushing difficulties. Resin was found to be tightly packed in the resin receiver and resin was also found ledged on the wrong side of the top hat screen. After product stripping, new resin was installed.

On June 15 the ZBP was diverted back to N-Cell. Start-up of the unit was marred by plugged XAF filters, indicating resin in the XAF Tank N1, and by leaking air cylinders. After diverting back to N-Cell, operation was again very poor with erratic pushes and essentially no push probe or CRC control. A shortage of resin in the unit prohibited maintaining a resin interface and also was undoubtedly contributing to much of the other operational difficulties.

5. Waste Treatment and Acid Recovery

The pressure drop through the LW Concentrator (E-F11) tower continued to increase during the report period, with a corresponding decrease in concentrator capacity. Flushing the tower with hot water resulted in only an initial moderate improvement. On June 14 the addition of 1 to 2 ppm of antifoam to the LW was commenced; however, this addition was terminated a day later, before any effects could be noted, because of the concern of a possible effect on recovered acid quality.

6. Fission Product Test Plant

The first series of strontium recovery runs since completing strontium recovery for the Snap 7 program was processed through the oxalate precipitation step and stored in TK-25. The oxalate product contained 1.77 megacuries of Sr 89-90 with the following fission product analyses and decontamination factors:

	<u>Curies/Gallon</u>	<u>DF</u>
Sr 89-90	630	
ZrNb	260	8.5
Ce 141-144	110	15.5
RuRh	4.5	130

A material balance for this series of runs is as follows:

	<u>Curies</u>	<u>Percent</u>	
		<u>Input</u>	<u>Output</u>
<u>Sulfate Precipitations</u>			
IWW	2,600,000	100	
Sulfate Waste	300,000		11.5
Flush	44,000		1.7
Carbonate Product	1,900,000	-	73.1
Totals		100	86.3
<u>Oxalate Precipitations</u>			
Carbonate Product	1,900,000	73.1	
Oxalate Waste	160,000		6.2
Flush	180,000		6.9
Oxalate Product	1,800,000		69.2
Totals		73.1	82.3
Grand Total		100.0	95.5

The improved decontamination factors are believed due to improved flushing between the sulfate and oxalate steps and a change in the characteristics of the IWW. Nitric acid flushes were used previously between the sulfate and oxalate steps. In this series of runs a 2% NaOH - 2% tartrate flush was used. Caustic-tartrate is believed to be more efficient in removing lead sulfate precipitates which interfere with the oxalate strikes. The acidity and flow of the IWW were increased since the concentrator tower plugging problem was

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encountered. The improved ZrNb DF observed in this and the last series of runs may be due to less precipitated ZrNb in the LW resulting from this change in concentrator operation. Precipitated ZrNb in the LW is collected with the product in the sulfate strike.

The oxalate precipitations are usually performed in two batches. During this series of runs the first batch was digested at 90 degrees and centrifuged at 25 degrees. The second batch was digested at 30 degrees and centrifuged at 25 degrees. No change in cerium DF was noted as a result in precipitating at a cooler temperature, however, the recovery efficiency in the first batch was 87% while the second batch was 99%. The difference in recovery may be due to measurement errors and the effect of the heel from the first batch and recovery in the second batch. The test will be repeated in reverse order.

After the contents of TK-002 and TK-003 were combined in TK-002 on May 29 (see page 103) the tank was agitated and sampled on June 9. The sample showed an inventory of 200,000 curies of Sr 90 instead of the expected 350,000 curies. A considerable amount of white solids was observed in the sample. Leaching the solids with various chemicals indicated another 140,000 curies of Sr 90 may be associated with the solids. The 002 Tank was agitated for 20 hours on June 16 and resampled. This sample showed 300,000 to 350,000 curies of Sr 90 (depending on the ratio of Sr 89/90 used) in the supernatant and up to 225,000 curies of Sr 90 associated with the solids. This latter figure would nearly account for the Sr HPID observed to date. It is not known how closely the amount of solids in the sample represent the amount of solids in the tank.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 5/16/61 TO 2400 ON 5/25/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.8	3.5	-	-	0.003	0.30
Final	2.6	3.4	8.0	1.8	0.002	-
Ion Exchange	-	1.1	-	15.0	-	-
Overall	6.4	8.0	8.0	16.8	0.005	0.30

II. Production

A. HAF Processed: 194 Tons

B. Uranium Product:

1. Solvent Extraction:

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
56	5.1	4.0	7.4	P1
57	6.1	3.1	7.4	P1
58	6.6	1.6	7.4	P1
59	6.7		7.4	P1
60	6.1	2.0	6.1	P1
61	4.1	1.0	7.3	P1
62	5.0	1.0	7.4	P1
63	6.9		7.4	P1
64	8.7	2.5	7.2	P1
65	10.0	2.8	7.3	P1
66	10.7	1.3	7.6	P3
67	8.8	1.8	7.5	P1
68	8.0		7.6	P1
69	7.9		6.1	E6
70	7.7	5.9	7.5	E6

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, pbpb U</u>	<u>Tons</u>	<u>Shipped To</u>
71	8.7		7.5	E6
72	-		7.8	E6
73	8.6	5.3	7.4	E6
74	7.6	6.0	7.0	E6
75	5.6		7.9	P1
76	2.2	6.4	7.9	P1
77	3.1		7.9	P1
78	3.7		7.8	P1
79	5.3		7.6	P1
80	7.4	6.6	7.7	P1
81	9.1		7.7	P1
82	11.6	9.9	7.7	P2
83	15.9		7.6	P2
84	11.7	4.1	7.7	P2
85	11.8		7.0	E6
86	11.8	3.4	7.1	P2
87	12.1		6.9	E6
88	12.6	3.5	4.1	E6
89	12.4		7.3	P2
90	10.8		2.0	E6
91	10.7		6.6	P2

2. Silica Gel

Gamma Ratio: Feed = 8.3
Product = 1.7

Tonnage: 125 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/MT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, pbpb Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
30						E6
31	3.6	0.001		5	3	234-5
32	5.2	0.004		5	5	Holding
33	15.2	0.103		4	19	Holding
34	9.3	0.007		5	5	Holding
35	5.2	0.002		5	4	Holding
36	29.5	0.122		4	33	Holding
37	6.9	0.002		5	4	Holding
38	2.1	0.001		4	2	234-5
39	1.7	0.001		5	2	234-5
40	54.0	0.002	5310	3	65	Holding
41	1.0	0.002		4	3	234-5

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, pump Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
42	129.0	-			-	E6
43	0.7	0.007		5	2	234-5
44	1.0	0.007		5	2	234-5
45	0.8	0.006		5	3	234-5
46	0.8	0.015		5	3	234-5
47	1.5	0.022		5	4	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.10%
Pu = 0.67%

E. Rework - % of Total Production:

U = 32.5% (Predominantly U from 2EU TK-E6 to HAF Make-up TK-E6)
Pu = 9.9% (Out-of-spec. Pu Product via TK-L11 to HAF Make-up TK-E6)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	760	104
Cell Drainage	115	102
FP Waste	21	102
Coating Waste	270	107-C

B. IHW Flows: 10.6

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	11.5
102	7.7
103	0.3
104	7.0

Notes:

1. Up until the time of scheduled shutdown at 1300 on June 24, the HAF rate was maintained at 3.3 CF with the following exceptions:

(a) On June 16 at 1630, the HAF flow was stopped for 11.5 hours due to HAF pump failure.

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- (b) From 1730 on June 19 until 1015 on June 21, "cold" uranium was recycled in order to lower Pu levels in the plant.

2. Flowsheet

- (a) On June 16 the 3.0 flows of 3WB-HNO₃ was stopped. On June 19 the 3WB-HNO₃ was started at 4.8 flows, and then increased to 7.7 flows on June 21. On June 22 the 3WB-HNO₃ was reduced to 3.1 flows.
- (b) On June 19 the Final Uranium Cycle rate was reduced from 3.5 to 3.3 CF. The 2DF ferrous sulfamate and 2DS flows (gpm), however, were not changed.
- (c) On June 20 the IBS was reduced from 18.2 to 17.1 flows.
- (d) On June 22 the 2DF-HNO₃ was increased from 5.9 to 6.3 flows.
- (e) On June 22 the 3WB was increased from 39 to 42 flows.

3. Solvent Extraction

Solvent-extraction performance was unsatisfactory principally as a result of problems with the Plutonium Anion Exchange Unit which directly caused high HAW plutonium losses and excessive plutonium levels in the intercycle streams. In an effort to correct the HAW loss problems the HA Column saturation was decreased, with loss of decontamination efficiency. The high Pu levels in the plant necessitated the recycle of "cold" uranium for about 40 hours during the report period. Additional loss of DF resulted from

- (a) A gamma burst associated with a 12-hour outage caused by HAF pump failure; and
- (b) Reduction of the 2D Column saturation in preparation for the scheduled neptunium recovery run.

The HAW plutonium losses generally were 0.2 to 0.4 per cent, but occasional losses as high as 2 per cent were encountered. The pattern was not affected by stopping plutonium recycle via the HAF or by reworking "cold" uranium as HAF.

On June 24, the preparation for plant shutdown and a neptunium recovery run started. The plant shutdown was completed on June 25. Highlights of the shutdown operation were as follows:

- (a) About 18 tons of "cold" uranium without plutonium recycle were processed as HAF in order to reduce the gamma and plutonium levels of intercycle streams.

- (b) The HA-HS was stripped and emptied in preparation for changing the HCR jet system.
- (c) The 2D Column was stripped and emptied in preparation for flushing and column change.

4. Final Plutonium Concentration

On June 19 approximately six gallons of 10 - 20 mesh Permutit SK resin was added to the N-Cell system in order to bring the inventory to normal. This resin is twice the size of normal N-Cell resin, but was added because no normal resin was available.

After the resin addition an interface was established and with pushes being manually controlled, N-Cell operation improved and normal operation was maintained to the end of the report period. No ill effects, such as high XAW losses or decrease in dF were observed after the larger resin was added. The only apparent change in operation was a shorter resin fall-out time.

5. Waste Treatment and Acid Recovery

In an attempt to reduce the F11 tower pressure drop, the continuous addition of hot water via the tower spray was commenced on June 21. No change in pressure drop was noted.

6. Fission Product Test Plant

Fission product processing has been suspended until the aged strontium stored in TK-F8 can be transferred to 244-CR storage. The material in TK-F8 is being held as "back-up" feed for the high-level cells in the event of difficulties in loading the HAP0 1A Cask or in shipping the HAP0 II Cask. Tanks 002 and 003 contain aged strontium for the SNAP program. Strontium 89/90 ratio specifications do not allow blending of TK-002 and TK-003 or adding "green" strontium to either tank.

A large-volume sample was obtained of the TK-F8 contents and the behavior of the F8 solids and the TK-002 solids was compared. Although the solids in both samples appeared visually to be the same, no appreciable amount of strontium was found on the F8 solids. This is in sharp contrast to the studies performed on TK-002 solids, (see page 123). This suggests that the strontium on TK-002 solids might be returned to the solution phase by boiling in 3 - 4 M nitric acid. Laboratory leach tests on TK-002 solids with up to 4 M nitric acid at room temperature, however, have been unsuccessful.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 6/26/61 TO 2400 ON 7/6/61

During the report period the plant was shut down with respect to normal uranium processing. A scheduled neptunium recovery run was conducted and necessary maintenance and other operations were conducted. Process notes of interest are presented below.

1. Solvent Extraction

(a) Vent Jumper for 2A Column

Following scheduled shutdown of normal processing on June 24, and prior to the start of the neptunium recovery run, a new vent jumper arrangement was installed on the 2A Column and was successfully tested. The new vent jumper includes a slab-shaped de-entrainment section, 36 in. x 12 in. x 2 in., which is filled with 3/4-in. stainless-steel Raschig rings. The de-entrainment section is also jumpered to drain to the 2AP stream. According to design "ground rules", the slab dimensions obviate any requirement for scheduled routine flushing of this equipment for critical mass control.

For testing of the new vent jumper arrangement, a temporary line was installed on the de-entrainment section to permit visual observation of entrainment. The 2A Column was then operated at various conditions of scrub-section volume velocity and pulse frequency. Ultimately, 2AX and 2AS rates 20% and 30%, respectively, above normal neptunium recovery rates were attained at a pulse frequency 25 to 35 cycles/min. above the normal for neptunium recovery. Relative to normal plutonium processing, these conditions are equivalent to capacity factors of about 15 and 5 for 2AX and 2AS, respectively, at a pulse frequency 25 to 35 cycles/min. above normal. No loss of liquid through the new de-entrainment section was noted.

(b) New 2D Column

Also following shutdown of normal processing, preparations were immediately commenced for replacement of the 2D Column with a spare column containing a remote scrub-section cartridge of stainless-steel nozzle plates. The old column was flushed with two series of hot, dilute caustic-tartrate and oxalic- HNO_3 , and finally with water. The new 2D Column was set in place on June 29. Subsequently, it was flushed with 5% sodium carbonate after which the scrub-section was pre-treated with solvent. This latter treatment was aimed at avoiding the aqueous wetting problem that had been previously experienced with a cartridge of the same type which had been installed in the HA Column on April 26 (see pp. 78, 86, 93). The pretreatment operation involved a solvent soak of the scrub-section plates followed by solvent displacement and air-drying of the plates. Operability checking of the new column after installation revealed that instrument line revisions which are required to permit column differential pressure measurements were not installed on this spare. Consequently, at the end of the report period the column was being removed for the necessary changes. The new scrub cartridge contains 66 nozzle plates, six per cent free area with nozzles pointing down, and 3 louver plates of 16 per cent free area. The nozzle plates are spaced at 2 inches in three groups of 22 plates each, separated by the louver plates.

(c) Miscellaneous

Other items of interest to solvent extraction included the following:

- (1) A new in-cell HSR jet routing was installed. This design avoids a leakage problem which was caused by a badly corroded canyon wall nozzle.
- (2) The leaking west (left-hand) tube bundle in the ICU Concentrator E-J8 was replaced on July 5.
- (3) Approximately 1200 gallons of solvent detected in the 3WF Tank F10 were transferred to waste on July 6 after prior sampling indicated negligible product content.

2. Plutonium Concentration

During the June-July shutdown the following work was done in N-Cell ion exchange facility:

- (a) Stripped and removed resin.

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- (b) Replaced the ZEP heat exchanger which apparently was leaking.
- (c) Installed a screen and 1/2" orifice in the push line between the push tank and the resin reservoir. These changes were made to improve push control.
- (d) Examined all screens for tears and holes.
 - (1) The XAW screen was found to be torn away from its clamping bar and resin was packed between the screen and metal. Resin was also found in the XAW line. It must be assumed that resin had been lost from the system and had been carried to TK-J1 during the last operating period. A new XAW spool piece and screen were installed.
 - (2) A small tear in the XAF screen was found and the screen was replaced.
- (e) Replaced the air cylinder to a Durco valve which was leaking badly.
- (f) Added new resin. Approximately 25% of the resin added was the larger 10 - 20 mesh with the balance 20 - 40 mesh.
- (g) Made preparations for processing plutonium rework through N Cell prior to the normal plant start-up.

3. Waste Treatment

To correct a problem of excessive differential pressure in the plate-section of the LW Concentrator E-F11 (see p.113), chemical flushing with hot dilute caustic-tartrate were conducted beginning on June 28. Initial flushes involved filling the tower section insofar as possible. Later during the report period when other operations precluded such deep flushing, additional flushes were poured through the tower. On June 30, E-F11 operability testing indicated possible improvement in behavior, but test interpretation was inconclusive because of very low condenser-vent vacuum.

On July 1 a contamination spread outside of the Purex building resulted from failure in the trap pit of the trap jumper for the 2W Concentrator E-F6 west tube bundle. The contamination occurred after start-up of E-F6 during E-F11 operability testing.

On July 4 a spray jumper was installed in the Demister which is located in the E-F6 vapor line. Intent of this addition is to improve the decontamination performance of the Demister by spraying water at controlled rates over the Demister pad. Demister drainage is routed to the 2W Concentrator (E-F6) pot.

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4. Neptunium Recovery Operation

(a) Summary

A neptunium recovery run was started at 1530 on June 26 and was completed (2BP off) at 1000 on July 4. After product concentration, 1.74 kg. of neptunium were loaded out, giving 85% recovery based on the 3WB processed.

Slight processing abnormalities were encountered with respect to product losses. During Phase I the 2BW losses were 3 to 6 fold above normal, and were not reduced by reasonable 2BP - 2BX flow increases.

During Phase II, however, the 2BW losses returned rapidly to normal. The 2AW losses were about 10 fold high for a short period of time following the transition to Phase II. Simultaneously, a burst of gamma activity through the system was noted. The gamma activity and the 2AW losses were associated with the processing of the 3WB heel at the end of Phase I. At the same time a slight layer of organic on a 2AF sample was reported by the laboratory.

A new 2A Column vent jumper (see Note 1, above) successfully prevented any product losses via this route. TX-F13, the collection tank for the vent system, was sampled and analyzed routinely.

Solvent treatment operations were normal with the No. 1 Treatment System operated at 1.44 CF when necessary. The gamma activity of the IOO was 450 uc./gal. at completion of the operation.

(b) Material Balance*

	<u>Kg</u>
Np in Plant	2.15
Np in 3WB	2.09
Np Processed as 3WB	2.04
Np Load-out (Est.)	1.74
Np Recycled	0.39
Np Lost	0.02

(c) Typical Solvent Extraction Losses*

<u>Stream</u>	<u>% of 3WB Processed</u>
2AW	2.8
2BW	8.3

*Losses recycled for recovery during normal processing operations.

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(d) Product Analytical Results

	<u>Np,</u> <u>g/l</u>	<u>MO₃</u> <u>M</u>	<u>% Contamination of Np</u>		<u>G/Np,</u> <u>AT x 10⁷</u>
			<u>Pu</u>	<u>U</u>	
Principal Load-out	33.7	9.88	16.5	12.5	37.3
First Flush	16.6	11.02	20.9	11.6	38.7
Second Flush	2.35	10.58	33.3	13.1	45.6
Third Flush	0.502	9.99	13.7	25.6	73.7
Fourth Flush	0.434	10.30	24.5	51.3	97.1

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 7/7/61 TO 2400 ON 7/27/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gross OF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.5	3.5	-	-	0.004	0.07
Final	3.0	3.2	10.0	3.4	0.005	-
Ion Exchange	-	1.4	-	0.3	-	-
Overall	6.5	8.1	10.0	3.7	0.009	0.07

II. Production

A. HAF Processed: 417 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gross Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
1	6.0	71	4.2	E5
2	107	74	6.7	K1
3	86	34	7.0	E6
4	57	151	6.9	E6
5	20.5	188	7.3	E6
6	17.4	113	7.4	E6
7	30.2		7.4	P2
8	22.2	46	6.9	P2
9	13.0	23	7.2	P2
10	7.8	11.6	7.2	P1
11	4.6	5.3	7.1	P1
12	3.0	2.2	7.2	P1
13	7.2		7.0	P1
14	7.5	9.2	7.1	P1

Rework

Rework

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pt,ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
15	5.8		7.2	P1
16	4.0	3.9	7.3	P1
17	2.8		6.3	P1
18	2.4	1.4	7.2	P1
19	2.0	0.6	7.5	P1
20	2.0	0.6	7.5	P4
21	2.3	0.4	7.4	P1
22	2.0		7.0	P4
23	2.1	0.4	7.3	P4
24	2.2	0.4	7.5	P4
25	2.2		7.2	P4
26	2.3	0.4	7.3	P4
27	2.2		7.2	P4
28	3.0	1.3	7.2	P1
29	3.0		7.3	P1
30	3.2	0.1	7.2	P1
31	6.0		7.4	P1
32	8.5		7.5	P1
33	9.5	8.9	7.3	P1
34	10.7	3.5	7.8	P1
35	9.5		8.2	P1
36	9.1	7.0	7.7	P1
37	11.6		7.9	P1
38	9.6	5.0	7.8	P1
39	8.4		7.8	P1
40	8.1	8.0	8.0	P1
41	6.2		7.3	P1
42	5.6	3.1	7.4	P1
43	5.5		7.3	P1
44	4.9	2.0	7.3	P1
45	4.4		7.4	P1
46	11.1	16.6	7.2	P1
47	27.4	35.5	6.4	E6)
48	24.5	28.2	5.4	P2) Rework
49	17.0	18.5	7.3	P2)
50	11.0	10.7	7.7	P1
51	6.7	4.1	7.0	P1
52	4.9	2.8	7.4	P1
53	4.6		7.4	P1
54	4.7	1.8	7.0	P1
55	3.9		7.3	P1
56	3.7	0.9	6.6	P1
57	3.5		7.5	P1

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
58	3.7	0.8	7.7	Pl
59	4.0		7.4	Pl
60	3.4	0.6	7.5	Pl
61	3.5		7.6	Pl
62	3.9	0.8	7.3	Pl
63	4.0		7.6	Pl

2. Silica Gel

Gamma Ratios: Feed = ~ 5.6
 Product = 1.15
 Tonnage = ~ 220 Tons

C. Plutonium Product:

<u>Batch</u>	<u>Zr-Nb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
1	2.9	0.040		4	3	234-5
2	7.9	0.033		4	10	Holding
3	2.0	0.019		5	2	234-5
4	1.3	0.014		6	2	234-5
5	3.3	0.031	2400	5	2	234-5
6	3.7	-		5	3	234-5
7	1.1	0.004		5	3	234-5
8	0.79	0.002		5	2	234-5
9	0.72	0.001		6	1	234-5
10	0.72	0.001	2550	5	2	234-5
11	0.88	0.001		5	1	234-5
12	1.0	0.005		5	2	234-5
13	0.92	0.001		5	2	234-5
14	1.2	0.003		5	3	234-5
15	1.3	0.001	4721	5	3	234-5
16	0.82	0.002		5	2	234-5
17	0.68	0.003		5	2	234-5
18	0.92	0.002		6	2	234-5
19						Recycle Via L11
20	7.9	0.007	4170	4	1	Holding
21	4.6	0.012		5	5	234-5
22	4.8	0.010		4	4	234-5
23						Recycle Via L11
24	3.1	0.009		5	2	234-5
25	1.9	0.010	2270	4	-	234-5
26	1.5	0.002		6	27	234-5
27	36.4	0.005		5	2	Holding

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D. Overall Waste Loss - % of Total Production:

U = 0.08%

Pu = 0.24%

E. Rework - % of Total Production:

U = 18.8% (Out-of-spec U Product from 2EU TK-K6 to HAF Make-up TK-E6, and U Storage TK-P2 to 2DF TK-K1 through Final U Cycle.)

Pu = 34.3% (Includes Out-of-spec Pu Product, Misc. L Cell Pkg. Flushes and Sumps, via TK-L11 to HAF Make-up TK-E6; and Out-of-spec Pu Product previously loaded out, via TK-L9 to XAF TK-N1 through Pu Anion Exchange Unit.)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	556	104
Cell Drainage	106	102 & 106
FP Waste	0	102 & 106
Coating Waste	270	107-C

B. IWV Flows: 6.6 Flows

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate Gal/Min</u>
101	12.1
102	9.0
103	0.2
104	5.7

Notes:

1. At the beginning of the report period scheduled shutdown operations were being completed. Plant start-up commenced at 0800 on July 11. The nominal start-up rate was 3.0; however, the actual rate was about 3.3 CF. Processing of "hot" HAF commenced at 1515 on July 11. Subsequent changes were made as follows:

(a) "Cold" uranium was processed from 1730 on July 12 until 1700 on July 13.

(b) HAF processing was stopped between 1200 and 2100 on July 15 because of a 2AF pump failure.

- (c) HAF processing was stopped for about 45 minutes at about 0200 on July 23 because of a power failure.
- (d) Commencing at 1245 on July 23, 6.4 tons of "cold" uranium was processed as HAF.
- (e) At 1030 on July 24, the HAF rate was reduced to 3.0 CF.

2. Flowsheet

Flows indicated below are on the basis of operation at 3.3 CF until July 24 and at 3.0 CF thereafter.

- (a) The start-up flowsheet on July 11 was the same as the shutdown flowsheet on June 25 except for the following:
 - (1) The uranium feed streams and corresponding extractant streams were biased about 10% high, resulting in other flows being about 10% less than standard flowsheet values.
 - (2) The 2BP stream was routed to the L-Cell Package rather than to N Cell.
 - (3) The IWF-H₂O was 151 flows. At the time of prior plant shutdown, this flow had been stopped.
 - (4) Water was being sprayed on the Demister pad at 3 gal/min. (22 flows). Demister drainage is routed to E-F6 bottoms.
- (b) On July 12 the 2DF ferrous sulfamate was increased from 0.54 to 0.67 flows.
- (c) On July 13 the IBS was reduced from 19 to 17 flows.
- (d) On July 13 (2300) the 2BP stream was diverted to N Cell.
- (e) On July 14 the HSS, HAX-nitrite, and the HSR-HNO₃ were increased (about 10%) to their flowsheet values.
- (f) On July 14 the 2DF-ferrous sulfamate was reduced from 0.64 to 0.53 flows. The stream was further reduced to 0.32 flows on July 17.
- (g) On July 14 the 2AF-nitrite was increased from 3.1 to 3.7 flows. It was returned to 3.1 flows on July 15.
- (h) On July 14 the 2AF-HNO₃ was increased from 1.4 to 1.9 flows.
- (i) On July 16 the IBXP was increased from 18.2 to 19.2 flows.

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- (j) On July 19 the ZDS was reduced from 66 to 48 flows.
- (k) On July 20 the ZDS was increased from 48 to 57 flows. This was further increased to 66 flows on July 21.
- (l) On July 22 the 3WB-HNO₃ was started at 2.1 flows.
- (m) On July 23 the ZDF-ferrous sulfamate was increased from 0.32 to 0.53 flows. It was reduced to 0.32, 0.27, and 0.23 flows on July 24, 25, and 26, respectively.
- (n) On July 23 the ZBP stream was diverted to the L-Cell Package for about 12 hours.
- (o) On July 24 the HA and HS Columns, the IBXF, and ICX flows were reduced from 3.3 to 3.0 CF. Other flows (gpm) were not changed. The Final Uranium Cycle was maintained at 3.3 CF by reworking P2 material.
- (p) On July 24 the 2.1 flows of 3WB HNO₃ was stopped.
- (q) On July 26 the IBS was increased from 17.8 to 18.6 flows.
- (r) On July 27 the Final Uranium Cycle was reduced from 3.3 to 3.0 CF. The ZDF-Fe and ZDS flows (gpm), however, were not decreased. Thus the rate reduction increased the ZDF-ferrous sulfamate and ZDS to 0.25 and 72 flows, respectively.

3. Feed Preparation

On July 24 the A-Cell dissolver facilities were shut down because of plugging of the A3 Silver Reactor. A plant rate decrease from 3.3 to 3.0 CF was necessary as a result. Flushing of the A3 reactor was in progress at the end of the report period.

4. Solvent Extraction

Decontamination performance and product qualities were generally normal. However, during plant shutdown-start-up periods short but severe gamma bursts of varying magnitude occurred, and this in turn affected product qualities. These shutdown-start-up periods are discussed briefly below:

- (a) Decontamination performance of the First Cycle during and immediately following the plant start-up on July 11 was very poor. The poor performance resulted primarily from inadequate process control caused by loss of the BAW sampler and several defective instruments. The decontamination performances of the Final Plutonium Cycle and Final Uranium Cycle were also initially poor. The poor Final Plutonium Cycle DF resulted from flooding the IBS and 2A Column.

The poor Final Uranium Cycle DF resulted primarily from operating the 2D Column unsaturated. This latter case was a control problem caused by unsatisfactory performance of the column differential pressure instrumentation. Shortly after start-up, "cold" uranium was recycled for 24 hours in order to reduce the gamma levels of intercycle streams.

- (b) On July 15 the 2AF Pump failed and the First Cycle and the Final Plutonium Cycles were shut down for about 12 hours. A severe but short gamma burst followed the plant start-up. However, the uranium product quality was not significantly affected, and the plutonium product met specifications after blending.
- (c) On July 23 an electrical storm caused a power outage for about 45 minutes. The plant start-up was followed by extremely severe gamma bursts in the product streams. Although the 2AF and 2DF gamma activities increased several-fold for a short period, the excessive product gamma levels resulted largely from flooding the 2A and 2D Columns.

The HAW loss was extremely high following the July 11 start-up, and segregation of HW for future rework was required. Since that time the HAW plutonium loss has been somewhat erratic but usually around 0.1%. The 2AW plutonium loss was excessive at start-up, but was restored to normal by increasing the 2A Column pulse frequency and 2AF acidity. During the latter portion of the report period the 2AW loss increased to about 2-fold above normal. Efforts to reduce the loss by increasing the 2A Column pulse frequency have thus far been unsuccessful.

The performance of the new 2D Column scrub section is comparable to the performance of a normal zebra cartridge scrub section. The new scrub section, however, is easily flooded, and when flooded recovery of stable operation is very difficult. During this operating period the 2D Column was flooded on three occasions. On each occasion it was necessary to shut down in order to break the flood. To date it appears the behavior of the new 2D scrub section is more similar to the 2A Column scrub section than the HA Column scrub section. This phenomenon is probably related to the particular acidities and aqueous/organic flow ratios.

Prior to the July 11 start-up, the 2D Column had been removed for installation of differential pressure instrumentation (see p. 130). After re-installation, the scrub section was pretreated for a second time, by soaking with solvent and then air drying. As previously explained, this treatment is aimed at preventing a tendency for aqueous wetting of the stainless-steel plates.

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5. Plutonium Concentration

(a) Shutdown Rework Operation

On July 8, while the plant was shut down, the rework of out-of-specification plutonium through N Cell was commenced. Between July 8 and July 13, sixteen cans were processed with no valence adjustments being made.

Based on integrated N2 sample results and F10 batch samples, the average loss for the first six cans reworked was ~ 20%. Analysis of XAW samples which were taken during periods of high loss indicated the loss to consist essentially of 100% Pu VI. Analyses of the rework feed material before processing showed an average Pu VI content of ~ 29% for the first six cans. These six cans were all from L-Cell Package loadouts.

Losses from the next ten cans reworked, however, were markedly lower, averaging only ~ 2%. Analyses of these cans showed little or no Pu VI. These ten cans were all from N Cell loadouts. It is concluded that the bulk of Pu VI present is lost to the XAW regardless of resin saturation or N Cell operating conditions and that Pu IV is recovered to the same degree as the Pu normally found in the 2BP stream. The reason for the absence of Pu VI in the ten N Cell cans is still not completely understood. Previous checks on N Cell loadouts have indicated the presence of Pu VI in the range of 15% to 25%. It is known, however, that the normal final concentration step shifts the valence equilibrium toward Pu VI. Apparently, after the loadout an equilibrium shift back toward Pu IV takes place.

The test has also demonstrated that the use of an orifice to control the flow of the rework solution to the XAF Tank N1 is too crude to be satisfactory. The absence of reliable liquid level instrumentation has added to the problem. Considering that no fluoride was being used in the XAF, the DP obtained was satisfactory.

The rework was suspended on July 12 with the start-up of normal plant processing.

(b) Normal Processing Performance

Following plant start-up, ion exchange operation was excellent with decontamination factors of greater than 20 realized. The use of a resin mixture --- 25% 10-20 mesh and 75% 20-40 mesh --- has not caused any problems.

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On July 23 one can of out-of-specification plutonium was successfully reworked through ion exchange, but further attempts were postponed because of the necessity for recycling other product material via the HAF Make-up Tank E6.

6. Solvent Treatment

Prior to plant start-up the IOF Tank G1 was flushed twice with oxalic acid-tartaric acid, after which tank calibration indicated a solids hold-up of about 200 gallons.

During the report period the IOO activity varied between 450 and 1800 uc./gal., with the periods of low activity occurring prior to plant start-up, and after a period of uranium recycle following plant start-up.

7. Waste Treatment and Acid Recovery

(a) E-F11 Pressurization Problem

Caustic-tartrate flushing of both the IWW Concentrator E-F11 and the IWF Tank TK-F7 were completed prior to plant start-up. TK-F7 flushing was conducted following reports of a black sludge in that vessel.

Operability boil-up testing of E-F11 prior to start-up did not conclusively prove the absence of further capacity restriction involving the F11 tower. However, pressure measurements over a tower section from a point between the first and second trays from the top and a point at the pot vapor space were indicative that the flushing had been successful. Prior to plant shutdown this pressure differential measured 31 inches of water at a condenser-vent vacuum of 50 inches of water. After flushing, the measurement was 10 inches of water at vent vacuum of 19 to 35 inches.

Following plant start-up, E-F11 operation was normal, with pot vacuum readings of about 20 inches of water at a condenser vent vacuum of 45 inches. The pot vacuum in the ZWW Concentrator E-F6 has been low, however, with the steam flow to this concentrator limited to about 75% of normal. However, the F6 capacity decrease is not continuing. Some increase in IWW flow has resulted from the F6 problem.

(b) Miscellaneous

Prior to plant start-up, the failed 4-inch carbon steel elbow in the F6 trap pit jumper (see p. 131) was replaced. Subsequently, the failed F6 west tube bundle was also replaced.

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Following plant start-up, control of I/W processing was hampered by failure of the E-F11 specific gravity dip tubes. On July 21, the failed dip tubes were replaced.

On July 17, waste containing an estimated 7 kg of Pu was transferred from storage in TK-F13 to TK-F8 for refluxing prior to its rework. This material had been accumulated prior to the recent processing of a special run of non-representative irradiated uranium. Mostly it contained waste from a neptunium recovery run plus N Cell and PR-Room flushes. Reflux was scheduled because of the inadvertent introduction of a small volume (ca. 200 gal) of fission product waste during TK-F13 storage. Transfer of this material out of TK-F13 permitted storage of about 500 g. of Pu in I/W which resulted from high HAW losses during the current report period.

On July 21, I/W flow to underground storage was re-routed from TK-241-A-102 to TK-241-A-106. Solvent treatment carbonate wastes continue to be routed to TK-241-A-104.

8. Fission Product Test Plant

The HAP0 II Cask was loaded with 120,000 curies of strontium-90 at the Hot Semiworks. Following loading and drying the cask was observed for temperature and pressure behavior. The temperature behavior was satisfactory, however, the pressure build-up rate could not be accurately defined because of a leaking Jamesbury valve.

On July 19 the HAP0 II Cask was transferred to the Purex loadout station for repair of the Jamesbury valve and final testing. A special lucite hood was fabricated and attached to the cask so the Jamesbury valve could be repaired by glove box techniques. The valve was removed, repacked and reinstalled without incident.

Following repair of the valve the cask was pressurized to 10 psig with helium to observe for leaks. The Snap-Tite connection at the Jamesbury valve leaked and could not be sealed by increasing the pressure to 30 psig. The Snap-Tite connection was removed and the pressure test apparatus screwed into the Jamesbury valve. Pressure and leak tests were completed at 20 psig with no significant pressure increase detected. All closures were checked by flooding the cask wells with water and observing for bubbles. No leaks were detected.

The cask's internal and surface temperatures and ambient air temperatures were monitored during the observation period. The temperature differentials were as follows:

Cask interior to air - 52° F
Cask surface to air - 27° F

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During the week of July 24 another transfer of strontium crude from TK-002 to the Hot Semiworks was completed. Based on storage tank inventory samples and the volume of solution pumped, 440,000 curies of Sr-90 was sent to Hot Semiworks. Hot Semiworks analyses show that less was received. TK-002 is being resampled to resolve the discrepancy.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 7/28/61 TO 2400 ON 8/3/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.5	3.5	-	-	0.004	0.15
Final	3.2	3.4	10.0	3.9	0.004	-
Ion Exchange	-	1.4	-	0.1	-	-
Overall	6.7	8.4	10.0	4.0	0.008	0.15

II. Production

A. BAF Processed: 181 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu,pphm U</u>	<u>Tons</u>	<u>Shipped To</u>
64	3.8	0.1	7.6	P1
65	4.1		7.2	P1
66	4.5	0.8	7.1	P1
67	4.5		7.1	P1
68	3.6	0.2	7.2	P1
69	3.7		7.2	P1
70	3.4	1.3	7.3	P1
71	3.3		7.2	P1
72	3.5	0.7	7.3	P1
73	3.3		7.3	P1
74	3.7		7.2	P1
75	3.0		7.2	P1
76	3.2	1.0	7.3	P1
77	3.3		7.2	P1
78	2.7	1.3	7.4	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
1	3.1	0.6	7.3	P1
2	3.8		7.1	P1
3	4.0		7.5	P1
4	2.7		7.1	P1
5	3.1	0.1	6.9	P1
6	3.2		6.9	P1
7	3.0		7.1	P4
8	3.6		7.3	P1
9	3.8	0.8	7.3	P1
10	4.0		7.4	P1

2. Silica Gel

Gamma Ratios: Feed = 3.5
Product = 1.5
Tonnage = 110 Tons

C. Plutonium Product:

<u>Batch</u>	<u>Zr-Nb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
28	4.9	0.002	1750	5	2	234-5
29	1.3	0.001		5	3	234-5
30	1.1	0.001		4	3	234-5
31	0.94	0.002		5	1	234-5
32	0.71	0.006	1750	4	2	234-5
33	1.2	0.021		5	1	234-5
34	0.98	0.026		5	3	234-5
35	0.80	0.041		5	3	234-5
1	0.98	0.035		5	2	234-5
2	0.96	0.021		6	2	234-5
3	2.4	0.010		4	3	234-5
4	2.1	0.006		4	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.14%
Pu = 0.55%

E. Rework - % of Total Production:

U = Negligible
Pu = 10.0% (Includes out-of-spec Pu Product & Misc. Sumps in TK-L11 to HAF Make-up TK-E6 and out-of-spec Pu Product via TK-L9 to XAF TK-N1 through Pu Anion Exchange Unit only)

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III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	450	104
Cell Drainage	58	106
FP Waste	0	106
Coating Waste	270	107-C

B. INW Flows: 5.5 Flows

C. Tank Turn:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	11.0
102	8.5
103	0.4
104	7.6

Notes

1. The HAF rate was continued at a nominal 3.0 CF throughout the report period. The actual rate averaged 3.1 CF.
2. Flowsheet
 - (a) On July 28 the 2DF-ferrous sulfamate was reduced from 0.25 to 0.22 flows.
 - (b) On August 1, the 2AF-HNO₃ was increased from 2.16 to 2.22 flows. It was further increased to 2.64 flows on August 2, but was returned to 2.32 flows on the same day.
 - (c) On August 2, the 2DS was reduced from 69 to 59 flows.
 - (d) On August 3, the 2AF temperature was reduced from 50 to 40° C. It was returned to 48° C on the same day.
 - (e) On August 3, the IBS was increased from 18.5 to 19.7 flows.
 - (f) On August 3, the 2EX temperature was increased from 44 to 47° C.
3. Feed Preparation

During the report period the plugged A3 Silver Reactor was cleared with a thiosulfate flush after which it was regenerated with silver nitrate. This was completed and the A Cell Dissolver was returned to service on Aug. 1.

4. Solvent Extraction

Process performance continued without significant change except for an increase in Final Plutonium Cycle gamma levels toward the end of the report period. On Aug. 2, a sharp increase in ZBP gamma content resulted from a 20% increase in 2AF HNO₃ flow. At the same time the 2AW losses dropped about five fold. These conditions did not persist, however, as the acidity was returned toward normal. On Aug. 3, the 2AF temperature was reduced from 50 to 40° C for about six hours. It was returned to normal, however, when instrumentation indicated the lower temperature was detrimental to both the 2AW loss and the ZBP gamma activity.

An attempt was made to improve the performance of the 2D Column scrub section by lowering the 2DS rate on the theory that this would permit higher pulse frequency. At the end of the report period, following a 2DS rate decrease of about 15%, the 2D Column behavior indicated little likelihood of a successful frequency increase. There were some indications of a slight loss of decontamination performance, also.

On Aug. 3 a small volume of dilute caustic-tartrate flush solution was overflowed via the jet system from TK-25 (Fission-Product Test Plant vessel) to the HAF Make-up Tank E6. The HAF batch thus contaminated was rebuffed to normal HNO₃ specifications and was made approximately 0.003 M in iron in order to preferentially complex the tartrate ion. Processing of this material proceeded without incident.

5. Final Plutonium Concentration

Performance of the plutonium anion exchange facility continued good. Five additional cans of out-of-specification Pu were successfully reworked with no major loss problem. To date all rework has been without chemical adjustment.

6. Solvent Treatment

The gamma activity in the IOO increased from about 1500 on the 26th to 2500 - 3000 uc/gal at the end of the report period. An increase from 1500 to 2800 was noted as a result of recycling IOS and delayed G1 changes due to diversion box work; however, the activity returned to ~ 1800 uc/gal after 48 hours. Another increase from 1900 uc/gal to ~ 4000 uc/gal was experienced when solvent was recovered from the G2 tank by pumping into the top of the IO Column via the IOS line. The gamma activity of the ICW stream has increased from ~ 10,500 at start-up to ~ 40,000 uc/gal.

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PROCESS SUMMARY OF FUREX PLANT OPERATION
FROM 0000 ON 3/4/61 TO 2400 ON 3/10/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.4	3.4	-	-	0.004	0.05
Final	3.0	3.2	10.0	3.6	0.005	-
Ion Exchange	-	1.5	-	9.0	-	-
Overall	6.4	8.1	10.0	12.6	0.009	0.05

II. Production

A. HAF Processed: 190 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
11	4.6		7.1	Pl
12	4.8	1.2	7.3	Pl
13	5.0		7.2	Pl
14	5.0	2.0	7.2	Pl
15	4.9		7.3	Pl
16	4.8		7.0	Pl
17	5.0		7.2	Pl
18	4.9	2.0	7.2	Pl
19	5.1		7.3	Pl
20	5.5	2.3	7.0	Pl
21	5.5	6.3	7.3	Pl
22	6.0	9.2	7.0	Pl
23	6.1	10.3	7.3	Pl
24	5.9	9.8	7.5	Pl
25	5.3	4.3	7.7	Pl

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
26	5.4	3.8	7.2	P1
27	5.6	1.9	7.5	P3
28	5.5		7.8	P3
29	5.3		7.6	P3
30	5.1	2.7	7.5	P1
31	5.7		7.7	P3
32	5.5	0.3	7.6	P3
33	6.1	3.0	7.3	P3
34	6.1	4.3	7.4	P1
35	6.5		7.3	P1
36	5.8		7.7	P1

2. Silica Gel

Gamma Ratios: Feed = 5.4
Product = ~ 2.0
Tonnage = 70

C. Plutonium Product:

<u>Batch</u>	<u>Zr-Nb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Pu, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
5	2.4	0.004	2300	5	3	234-5
6	4.7	0.011		5	4	234-5
7	3.2	0.009		5	3	234-5
8	2.2	0.025	530	5	2	234-5
9	1.7	0.004		5	2	234-5
10	1.9	0.010	430	5	3	234-5
11	1.7	0.003		5	3	234-5
12	2.3	0.003		6	3	234-5
13	2.2	0.004		5	3	234-5
14	1.8	0.007		6	3	234-5
15	1.8	0.007		5	3	234-5
16	2.3	0.007		6	3	234-5
17	2.5	0.004		4	1	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.14%
Pu = 0.20%

E. Rework - % of Total Production:

U = Negligible
Pu = 13.5% (Includes out-of-spec. Pu Product, Miscellaneous Sump and
Flushes via TK-L11 to HAF Make-up TK-E6, and out-of-spec.
Pu Product previously loaded out via TK-L9 to IAF TK-N1
through Pu Anion Exchange Unit only.)

III. Waste Treatment

A. Volumes to UCS:

<u>Source</u>	<u>Gal./Ton</u>	<u>Waste Tank</u>
Wash Solution	438	104
Cell Drainage	28	106
FP Waste	0	106
Coating Waste	270	107-C

B. LWW Flows: 5.7 Flows

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal./Min.</u>
101	13.2
102	4.8
103	0.8
104	8.2

Notes:

1. The HAF rate was continued at 3.0 CF until 1000 on Aug. 5, at which time the rate was increased to 3.3 CF.

2. Flowsheet

- (a) On Aug. 5, the plant rate was increased from 3.0 to 3.3 CF, except that the 2DF ferrous sulfamate, IBXP, IBXP-HNO₃, IBS, L-Cell, and N-Cell flow rates, in gal./min., remained unchanged.
- (b) On Aug. 7 the RSS was increased from 54 to 60 flows.
- (c) On Aug. 7 the 2AS was increased from 1.50 to 1.75 flows.
- (d) On Aug. 7 the 2DS was increased from 57 to 66 flows.
- (e) On Aug. 7 the 2DF-ferrous sulfamate was increased from 0.20 to 0.41 flows. It was decreased to 0.30 and 0.20 flows on Aug. 8 and 10 respectively, but was increased to 0.39 flows on Aug. 11.
- (f) On Aug. 8 the 3WB-HNO₃ was started at 2.6 flows. It was reduced to 1.9 flows on Aug. 10 and was stopped on Aug. 11.
- (g) On Aug. 8 the ICF-KMnO₄ was increased from 1.7 to 2.1 flows.

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- (h) On Aug. 8 the IOC-IOD flows were increased from 7.7 to 14.6 flows.
- (i) On Aug 9 waste rework (neptunium recovery 3WW) was added from TK-F8 to the HAF at a rate of 100 gallons per every other 4000 gallon batch of HAF. On Aug. 10 the 100-gallon addition was made to every HAF batch.

3. Solvent Extraction

Process performance continued without significant change during the report period. HAW plutonium losses gradually declined from about 0.1% to about 0.05%, apparently as a result of moderate HA Column saturation decrease. The IBU plutonium loss gradually increased from 0.45% to 0.80% as a result of the change in IBX Column L/V associated with the rate increase on Aug. 5.

Erratic behavior of the HSR-HNO₃ control system resulted in some difficulty in control of HAW acidity. This probably contributed to slightly high neptunium losses early during the report period.

Increasing the HES, 2AS, and 2DS rates on Aug. 7 had no significant effect on the decontamination performance of the respective columns. The 10% HES change increased the neptunium reflux in the HA-HS system by an estimated 200 grams.

The introduction of waste rework from TK-F8 to the HAF, commencing on Aug. 9, appeared to cause a moderate improvement in HA-HS Column DF performance. This rework material is largely the backcycle waste from a neptunium recovery run, plus PR-Room plutonium flushes. The batch contained about 6000 grams of Pu and had been refluxed in TK-F8 for 21 days.

4. Final Pu Concentration

At the beginning of the report period resin pushing became difficult and push pressures of 45 lb. were necessary to move the resin. Initial analysis of the problem attributed the difficulty to the presence of packed or damaged resin, and a partial resin change-out was begun. On August 4, however, it was discovered that the XAF valve was leaking during the push and allowing the push pressure to bleed back to the N1 Tank. After adjusting the XAF cylinder and valve, the push pressure was reduced to 25 lb. while still maintaining normal pushes.

Also, control of pushes by use of the push probe became somewhat unreliable during the report period. The sluggishness of response may be

due to resin fines clinging to the push probe.

During the report period 5 more cans of out-of-specification Pu were reworked through E Cell with no processing difficulties. No chemical adjustments were made on any of the cans as DK-2 analyses of the remaining L-Cell Package material show that the Pu^{VI} initially present at the time of loadout has now equilibrated back to essentially all Pu^{IV}. Eight weeks have elapsed since these cans were loaded out.

5. Fission Product Recovery

The aged strontium stored in TK-78 (see page 128) was transferred to TK-002 just prior to the last Hot Semiworks run. This left 140,000 curies of green strontium in the building in TK-E5. After Hot Semiworks completed the processing of TK-002 (see page 144), the strontium stored in TK-E5 was transferred to TK-002. TK-003 contents will be transferred and combined with TK-002 and comprise the feed for the next Hot Semiworks run. TK-002 should then contain 300,000 curies of Sr-90 plus the TK-002 heel from the last purification run. The Sr 89/90 ratio of the combined material, not considering the heel, is estimated to be 5.5 as of August 1.

The E-Cell equipment was decontaminated in preparation for cesium runs. Advantage was taken of the clean equipment to send HLO 700 gallons of centrifuged TK-103A supernatant solution for technetium and cesium research and development studies.

6. Demineralized Water

On Aug. 4 the lead-lined sulfuric acid mix tank was replaced with the spare because of excessive leaking. This is the fourth time this tank has been replaced during the last 18 months.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 8/11/61 TO 2400 ON 8/17/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.5	3.5	-	-	0.003	0.05
Final	3.0	3.3	10.0	5.2	0.004	-
Ion Exchange	-	1.2	-	1.5	-	-
Overall	6.5	8.0	10.0	6.7	0.007	0.05

II. Production

A. HAF Processed: 188 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu,ppm U</u>	<u>Tons</u>	<u>Shipped To</u>
37	6.0		7.5	P3
38	7.2	0.4	7.4	P1
39	6.5		7.7	P3
40	5.6	9.7	3.6	P3
41	5.7	7.6	4.6	P3
42	5.6	5.6	7.6	P1
43	5.6	5.7	7.7	P1
44	5.8	3.5	7.5	P3
45	5.4	2.9	7.5	P3
46	5.0	2.1	7.5	P1
47	5.0		7.7	P3
48	4.6	1.5	7.6	P3
49	4.5		7.8	P3
50	4.6	1.6	7.8	P3

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, pobj U</u>	<u>Tons</u>	<u>Shipped To</u>
51	4.4		7.6	P3
52	4.0	2.0	7.7	P3
53	4.8		7.6	P1
54	6.0	3.4	6.0	E6
55	5.9		6.3	E6
56	5.3	4.9	7.4	P3
57	4.5		7.6	P1
58	4.2	2.3	7.7	P1
59	2.9		7.0	P1
60	2.7	0.7	7.5	P1
61	3.2		7.5	P1
62	3.5	0.7	7.5	P1

2. Silica Gel

Gamma Ratios: Feed = 9.9
Product = 1.9
Tonnage = 111 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, pump Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
18	2.0	0.009		6	4	234-5
19	2.6	0.009		5	4	234-5
20	2.0	0.012		6	3	234-5
21	2.0	0.010		5	4	234-5
22	3.2	0.009		6	3	234-5
23	2.3	0.020		4	3	234-5
24	2.5	0.018		6	4	234-5
25	3.2	0.021	470	5	2	234-5
26	3.0	0.019		5	4	234-5
27	3.9	0.010		5	4	234-5
28	5.2	0.011		5	4	Holding
29	2.9	0.021		5	3	Holding
30	1.6	0.016	440	5	4	234-5
31	2.8	0.012		4	3	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.17%
Pu = 0.36%

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E. Rework - % of Total Production:

U = 6.9% (Predominantly "U" Product from 2EU TK-K6 to HAF Make-up TK-E6)

Pu = 30.0% (Includes out-of-spec. Pu Product and Misc. Sumps via the L11 to HAF Make-up TK-E6; and out-of-spec. Pu Product via the L9 to XAF TK-N1 through Pu Anion Exchange Unit only, and F8 Reflux Material to HAF Make-up TK-E6)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	373	104
Cell Drainage	29	106
FP Waste	-	-
Coating Removal	270	107C

B. IW Flows: 6.1 Flows

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	12.7
102	4.7
103	0.3
104	7.4

Notes:

1. The HAF rate was continued at 3.3 CF throughout the report period, except for a period of operation at 3.0 CF from 0930 on Aug. 15 until 1830 on Aug. 16. During the period of operation at 3.0 CF, 12.9 tons of uranium product was processed as HAF.

2. Flowsheet

(a) On Aug. 11 the 2DF-ferrous sulfamate was increased from 0.21 to 0.40 flows. It was further increased to 0.48 flows on Aug. 12, but was returned to 0.40 flows on Aug. 13. On Aug. 16 and 17 the 2DF-ferrous sulfamate was reduced to 0.36 and 0.27 flows.

(b) On Aug. 13 the 2AF-HNO₃ was increased from 2.1 to 2.2 flows. It was further increased to 2.3 and 2.5 flows on Aug. 15 and 16.

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- (c) On Aug. 14 the IBXP was increased from 19 to 20 flows.
- (d) On Aug. 15 the IOD-IOC was increased from 13.8 to about 18 flows.
- (e) On Aug. 16 and 17 the G7 Turbonixer Tank CO_2 solution was changed. Each change requires about 2200 gallons of IOC.
- (f) On Aug. 17 the 2DF-HNO_3 was reduced from 5.8 to 5.3 flows.

3. Feed Preparation

On Aug. 14, approximately 700 gallons of metal solution was accidentally transferred from A Dissolver to the Costing Waste Tank D2. Pending the installation of temporary routings to permit salvage of this material, scheduling of normal feed preparation operations was suspended. The plant rate was reduced 10% for about a day and about 13 tons of "cold" uranium was recycled as HAF. On Aug. 15 the metal solution in TK-D2 was transferred to TK-D3 and acidified. It is planned to hold this material for a few days at slightly elevated temperatures and then to attempt processing.

4. Solvent Extraction

Gamma levels declined slightly during the report period; however, the plutonium product gamma activity increased about 50%, apparently due to some problem associated with ion exchange or the PR Room. Both plutonium and neptunium losses to the HAW were under good control, as the improved gamma performance permitted HA Column saturation reductions. The improvement in HA-IS decontamination performance may have resulted from a "tonic" effect from continued waste rework from TK-F8, although such a relationship was not obvious.

The increase in Pu losses to the IBU noted during the previous report period was checked and commenced to decline following an increase in the IBXP flow.

Other items of interest to solvent extraction included the following:

- (a) Two momentary power outages on Aug. 14 caused the 2D Column to flood. It was necessary to shut off all the flows in order to break the flood.
- (b) Fresh 57% HNO_3 was added to the HA Column pulse leg on Aug. 13 at a rate of 0.04 gal/min. in order to reduce radiation readings in the P. and O. Gallery.
- (c) About 14 tons of "cold" uranium was recycled to the HAF due to a temporary shutdown of A Dissolver.

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5. Final Plutonium Concentration

During the report period twelve PR cans containing Pu for rework were successfully processed through N Cell without chemical adjustments.

For reasons not completely known, N-Cell DP decreased noticeably during the past week. Cross-contamination from the L9 rework tank, a resistance to cleanup of the Pu reworked in N Cell, or an increase in solids in the product have all been proposed as possible causes.

6. Waste Treatment and Acid Recovery

A pattern of decreasing capacity in the Waste Concentrator E-F11 has become evident as the vacuum at the bottom of the concentrator tower has been decreasing at about 0.5 inches of water per day. This compares to a decrease of about 1.0 inch per day during the last run period. The Acid Concentrator E-F6 has not shown this same decreasing capacity pattern.

Operation of the Waste Treatment and Acid Recovery equipment has been improved by establishing a set steam flow to the Waste Concentrator (E-F11) and adjusting the LWF rate to maintain the desired E-F11 specific gravity and LWF rate. Varying liquid level in the LWF Tank (TK-F7) is controlled by dilution water adjustments.

7. Sludge Sampling

Attempts to obtain a sample of the solids from the bottom of the TK-241-A-103 Waste Storage Tank on Aug. 8 and 9 were unsuccessful. On each of two attempts the sample probe broke at the same place, the threaded connection between the sample probe and the push rod. For the second sampler, this section was reinforced. Both probe sections remained in the storage tank.

Briefly the sludge sampling method involved the lowering of the probe, attached to the push rod, into the tank through a 12-inch riser. The assembly was lowered by motor crane until it rested on the tank solids. Then a hydraulic ram was used to push the probe through the solids. Based on as-built tank measurements, both probes hit a solid layer when the tip was $21 \pm 1/2$ inches from the bottom of the tank. Pushing the blunted tapered tip of the sample probe the first 2 inches into the solid with the hydraulic ram required a maximum of 5400 psig for the first probe and 6300 psig for the second. After the initial penetrations, the pressure rapidly declined to 800 psig; and the probes were inserted an additional distance, 15 and 12 inches, respectively for the two sampling attempts, before the pressures began to rise again. With the first probe the pressure increased to 2400 psig while pushing the tip another 2 inches at which point the probe broke. The tip was an estimated 2 inches from the tank bottom. The second probe broke when the tip was an estimated $5-1/2$ inches from the bottom at a pressure of 1550 psig.

Although it is not certain why the probes broke at the lower pressures after withstanding the initial high pressures, it is believed that the blunted tapered tip caused the probe to enter the solid at an angle, which in turn caused the probe to bend and eventually break at the weakest point - the threaded connection. Both attempts were carried out without other incidents and all of the auxiliary equipment was recovered and stored for future use.

After the second failure, it was decided to evaluate a rotary-type core drill before making another attempt to obtain a sample. Process Equipment Development, HLO, is presently working on the equipment and after "cold" testing, it is expected to be ready for use by Sept. 25, 1961.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 8/18/61 TO 2400 ON 8/25/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.5	3.5	-	-	0.005	0.07
Final	3.0	3.4	8.0	4.7	0.003	-
Ion Exchange	-	1.0	-	0.5	-	-
Overall	6.5	7.9	8.0	5.2	0.008	0.07

II. Production

A. HAF Processed: 192 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
63	3.5		7.9	P1
64	3.8	1.8	7.7	P1
65	3.8		7.5	P1
66	4.1	2.4	7.6	P1
67	4.3		7.6	P1
68	4.0		8.5	P1
69	4.1		7.6	P1
70	4.1		7.6	P1
71	4.4		7.8	P3
72	4.6		7.3	P3
73	4.9		7.5	P1
74	4.5	1.2	7.7	P3
75	4.7		7.8	P1
76	5.8	1.1	7.8	P3
77	5.4		7.8	P4

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, pphp U</u>	<u>Tons</u>	<u>Shipped To</u>
78	5.7	1.5	7.8	P1
79	5.9		7.1	P3
80	17.5	53.5	6.4	P2
81	42.2	132.3	6.8	E6
82	33.4	82.6	6.8	E6
83	23.1		7.9	P2
84	16.1	18.3	8.0	E6
85	11.3	18.8	8.9	P1
86	7.7	11.3	8.7	P1
87	5.6		7.2	P1
88	3.4	5.6	7.7	P1
89	2.9	4.4	8.2	P1
90	2.0	3.5	7.9	P4
91	1.2		7.0	E6
92	1.3	4.1	7.6	E6
93	4.9	3.5	7.1	P4
94	6.0	2.0	5.6	E6
95	6.1	2.9	7.9	P1

2. Silica Gel

Gamma Ratios: Feed = 4.6
 Product = 1.7
 Tonnage = 102 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, pphp Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
32	2.4	0.012		5	3	234-5
33	2.5	0.005		5	2	234-5
34	1.6	0.003		6	3	234-5
35	2.2	0.002		5	2	234-5
36	2.2	0.001		6	4	234-5
37	2.3	0.003		5	4	234-5
38	3.0	0.003		4	5	234-5
39	5.0	0.010		5	5	Holding
40	9.2	0.005		-	-	E6
41	10.5	0.300		-	-	E6
42	-	-		-	-	E6
43	-	-		-	-	E6
44	3.5	0.040		5	5	234-5
45	3.1	0.004	877	5	4	234-5

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C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, prep Pu</u>	<u>No.Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
46	2.3	0.001		5	2	234-5
47	2.1	0.001		5	2	234-5
48	2.7	0.002		5	5	234-5
49	2.6	0.002		6	4	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.30%
Pu = 0.47%

E. Rework - % of Total Production:

U = 16.7% (Predominantly U Product from TK-K6 to HAF Make-up TK-E6)
Pu = 45.0% (Includes out-of-spec Pu Product, Misc. Sumps and Flushes via TK-L11 to HAF Make-up TK-E6; also, out-of-spec Pu Product previously loaded out via TK-L9 to XAF Make-up TK-W1 through Pu Anion Exchange Unit, only)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	541	104
Cell Drainage	64	106
FP Waste	0	106
Coating Waste	270	107C

B. IWW Flows: 4.6 Flows

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	11.4
102	7.2
103	0.2
104	9.7

Notes:

1. The HAF rate was continued at 3.3 CF throughout the report period except for an approximately 3-hour period, e.g.: Aug. 22 during which the plant was shut down on an emergency basis because of a power

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outage. On Aug. 23 and 24 about 36 tons of "cold" uranium was recycled as HAF.

2. Flowsheet

- (a) On Aug. 22 the 2AF temperature was increased from 50 to 55° C.
- (b) On Aug. 22 the addition of F8 rework to the HAF stream was stopped.
- (c) On Aug. 24 the 2DF-HNO₃ was increased from 5.4 to 5.9 flows.
- (d) On Aug. 23, 400 gallons of metal solution rework was added to each of three successive (4000-gallon) batches of HAF. (See Note 3, below.)

3. Feed Preparation

On Aug. 23 the metal solution which had been reclaimed from TK-D2 and digested in TK-D3 (see p. 157) was successfully reworked by addition to the HAF.

4. Solvent Extraction

Solvent-extraction performance continued without significant change until interruption by a power outage on Aug. 22. As a result of this outage, the gamma activity in the plutonium product increased about 50%. At the same time the uranium product activity increased about 5 fold and both the plutonium and neptunium content in the uranium increased roughly 100 fold. The severity of the upset with respect to the uranium product resulted primarily from instability of the 2D Column scrub section while attempting start-up. Recovery from the process upset followed the recycle of about 36 tons of uranium product.

Prior to the process upset, some loss of Final Uranium Cycle decontamination performance resulted from reduction of the 2D Column saturation in preparation for the impending scheduled shutdown.

Shutdown of the plant was started on Aug. 25 and was completed on Aug. 27. Highlights of the shutdown operations were as follows:

- (a) About 16 tons of "cold" uranium was recycled to reduce the inter-cycle plutonium and gamma levels.
- (b) The HA-BE system was stripped and the organic phase was displaced in preparation for changing diluents.
- (c) The contents of the ICU Concentrator and 2DF Tank without shutting down the Final Uranium Cycle were processed for neptunium recovery.

- (d) The 2D and 2E Columns were stripped and emptied in preparation for flushing and changing the 2D scrub cartridge.
- (e) An organic layer (~ 1200 gals.) was removed from the 3WF Tank F10 (by transfer to the F13 Utility Tank).
- (f) Leak tests of the 3WB Concentrator tube bundles were negative.

Other items of interest to solvent extraction during the report period were as follows:

- (a) Rework of waste from TK-F8 by addition to the HAF (see p. 152) was completed without incident. During the latter part of this operation, occasional omission of the rework from the HAF caused no effect on the process. The final 500-gal. heel of this material was not processed directly. Instead, the heel was reworked in about 100-gal. increments with the transfer of each increment preceded by addition of about 100 gal. of 57% HNO₃ to TK-F8. This procedure was repeated 7 times.
- (b) The uranium product produced by the Aug. 22 power outage was reworked as 2DF and HAF. In addition the storage tank was thoroughly flushed with water to the 2DF stream in preparation for storage of the Purex solvent.
- (c) The 2D Column was extremely difficult to start up following the Aug. 22 power outage. Reducing the pulse frequency from 70 to 38 cycles failed to prevent flooding at start-up. In addition slow start-ups were also unsuccessful even at low pulse frequencies and following the complete removal of aqueous hold-up. A successful start-up was finally achieved by partially stripping the 2D Column (reduction of the organic Sp.G.), low pulse frequency, and a very slow start-up.
- (d) A 2-fold decrease in 2AW Pu losses was effected by increasing the 2AF temperature from 50 to 55° C, and by very small increases in the 2AF-HNO₃ and 2AX flows.

5. Final Plutonium Concentration

During the report period 5 cans of out-of-specification Pu were processed without chemical adjustment through the ion exchange equipment. On Aug. 18 one can of Pu containing 22% Pu^{VI} was chemically adjusted to 100% Pu^{IV} using ferrous sulfamate and was successfully reworked. Some solids were found in the sample of the adjusted solution and an analysis disclosed the majority to be resin beads and fines. A few solids were also found which had the white crystalline characteristics of sulfamates.

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Normal Pu product samples during the report period were also found to contain excessive solids which appeared to be resin beads. A possibility exists that the recent loss of N-Cell DF may be related to solids, and that the XCP screen may be ruptured. Shutdown plans call for inspection of the screen and boil-up flushes of the N-Cell Concentrator (N6) and receiver (N7).

6. Waste Treatment and Acid Recovery

Laboratory investigations of the problem of increasing pressure drop through the INW Concentrator (F11) tower were conducted as follows:

- (a) A sample of Purex INW taken Aug. 4 was boiled in the laboratory. No foaming was observed.
- (b) Samples of Purex fresh acid, Purex recovered acid, and laboratory CP HNO₃ were boiled and compared. A very slight foaming tendency was noted in the case of Purex recovered acid although the degree was judged to be not significant. Purex fresh acid was studied because of the fact that a change in acid vendors had immediately preceded the beginning of the F11 tower problem in May.

7. Fission Product Recovery

Attempts to load the first SIT cask began on Aug. 11. Initial inspection of the cask revealed that the guide bolts on the long leg of the SIT had been sheared and the screen in the vent nozzle was plugged. After repairing the cask, loading started on Aug. 15. The in-canyon jet did not operate properly and frequent air purging and backflushing was required to obtain flow. This difficulty was experienced with the previous cesium campaign also (see pages 43 and 51).

Loading of the first SIT was completed on Aug. 23 with a total of 36,000 curies of Cs loaded. Radiation readings on the inlet and outlet lines indicated total breakthrough was reached and that breakthrough occurs rapidly in less than a half hour.

Initial attempts to seal the cask were unsuccessful. The nozzle closures were modified by ORNL after the April shipment and the new closures would not seal. By returning the closures to their original design, the cask was finally sealed and leak tested with helium at 25 psig on Aug. 25. The cask was then vented, sealed and cask observation for temperature and pressure behavior started.

Loading of the second SIT was commenced on Aug. 26. Loading continued to be hampered by maloperation of the in-canyon jet.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 8/27/61 TO 2400 ON 9/7/61

During the report period, the plant was shut down with respect to normal uranium processing. A scheduled neptunium run was conducted and other shutdown processing and maintenance operations were performed. Process notes of interest are presented below.

1. Feed Preparation

During the report period the flow of drainage water from the ammonia scrubbers again backed up, resulting in a flow of this water to the dissolvers. This has been a reoccurring difficulty which has been recently increasing in frequency. On the suspicion that a flow restriction due to solids is involved, a sample was taken from the scrubber water catch tank, TK-216-A2, on Sept. 8. Even though an effort was made to sample a hypothesized sludge layer, no solids were observed in the sample.

Prior to plant start-up, the scrubber drain lines were individually water-flushed to the 216-A2 catch tank. In addition, the operating procedure was changed so that the catch tank was emptied as far as possible prior to starting an ammonia scrubber operation. Since these changes, no flow difficulty has been encountered.

2. Solvent Extraction

Because of its poor operating characteristics during the preceding run period the 2D Column scrub-section cartridge was replaced. Prior to its removal the 6% free-area nozzle-plate cartridge (see p. 130) was chemically flushed with hot dilute caustic-tartrate and oxalic- HNO_3 . The new scrub cartridge contains 66 nozzle plates, 10% free area with nozzles pointing down, and 3 louver plates of 16% free area. The nozzle plates are spaced at 2 inches in three groups of 22 plates each, separated by the louver plates. The new cartridge was flushed with 5% Na_2CO_3 at 50° C. In addition it was contacted with solvent and then air dried for 36 hours in an attempt to improve the wetting characteristics prior to start-up.

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Other items of interest include the following:

- (a) Following the neptunium recovery run (Note 6, below), the 2AF Tank and the 2A-2B Columns were flushed with 57% HNO_3 at 60° C.
- (b) Prior to the scheduled change of solvent in the plant (Note 4, below), the solvent-extraction system was flushed with Soltrol-170 diluent.

3. Final Plutonium Concentration

During the shutdown period the following items of interest to ion exchange processing were accomplished:

- (a) The push tank was replaced. The new tank has the push probe centered in the tank, rather than being next to the wall, in order to stop the resin from hanging on the probe. The screen in the push line, previously installed to protect the old push probe, was removed.
- (b) Testing of the resin jetting system was completed during the shutdown and the continuous resin removal and addition system is now ready for operational use..
- (c) The resin was changed, again to a mixture of 25% 10 - 20 mesh and 75% 20 - 40 mesh.
- (d) The XCP resin screen was replaced. This was done because of the increasing evidence of resin in the product during the last run period. Inspection of the screen which was removed did not show any tears or breaks. It is possible that a resin loss was occurring in a screen construction joint.

4. Solvent Treatment

Prior to the neptunium recovery run all the solvent in the No. 2 Solvent System and the Final Uranium Cycle was displaced to the No. 1 Solvent System in preparation for a complete plant solvent change. Operating the No. 1 System at the normal 1.44 C.F. during the neptunium recovery run provided excellent quality solvent for the run.

Following the neptunium recovery run, a complete change of process solvent was commenced. The purpose of this change was to evaluate the process behavior of Soltrol-170 (Phillips Petroleum) as a diluent (in place of Shell E-2342) on a test basis. Thus, all of the building solvent was displaced from columns and vessels and transferred to TK-P2 in the UMI storage area by routing from TK-G5 (100 Tank) to TK-K6 (20C Tank); then by pumping TK-K6 contents to TK-P2. All solvent treatment equipment was flushed with 5% oxalic - 2% HNO_3 and 5% NaOH - 2% tartaric acid, followed by water flushes. Following

these chemical flushes, the solvent treatment systems and the entire solvent-extraction system were flushed with fresh Soltrol-170; a total of 5000 gallons of diluent was used in two increments. The Soltrol-170 was then brought into the building directly from tank cars, butted to 30% TBP, and washed in the solvent treatment system prior to use.

Soltrol-170 was chosen to be used on a test basis as the first step in obtaining a more stable diluent because of its cost (less than 10% increase in total solvent cost) and laboratory studies which indicate superior stability compared to Shell E-2342. For the test, 48,000 gallons of Soltrol-170 was purchased.

Following chemical flushing of the solvent treatment system, calibration of TK-R1 showed the packed section to be free of solids, while the TK-G1 calibration showed a volume increase of 100 - 200 gallons, compared to the original calibration.

5. Waste Treatment and Acid Recovery

After the neptunium recovery run, both waste concentrators (E-F6 and E-F11) were flushed with about 10,000 gallons each of 7% NaOH - 2% tartaric acid via the chemical add line to the concentrator towers. Subsequent operability tests indicated the E-F11 tower pressure drop was returned to normal. However, little, if any, effect was noted in the case of E-F6. In the course of making the necessary jumper changes in connection with the F6 flush, a leak developed at the F6-to-F7 over-flow nozzle. The leak is appreciable but the exact location has not yet been determined.

A program to replace concentrator steam traps with in-cell condensate diaphragm-operated valves was commenced with the installation of the following condensate DOV jumpers:

- (a) E-F6 right-hand (east) bundle;
- (b) E-F11 left-hand (west) bundle; and
- (c) both E-B4 bundles.

These DOV's are sized to control the tube bundle pressures while permitting steam condensate removal. Pre-start-up operability testing of the new controls was accomplished successfully.

Approximately 1500 gallons of solvent which had been accumulated in TK-F10 from 2D Column upsets during the last run period were transferred to the TK-F13 Utility Tank. Carbonate washing of this solvent

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removed 62 grams of Pu and 260 lb. of U which were sent to TK-78 on Aug. 27 for refluxing and ultimate recovery. The washed solvent was ultimately transferred to underground storage after cribbing attempts were unsuccessful.

6. Neptunium Recovery Operation

(a) Summary

A neptunium recovery run was started at 1200 on Aug. 28 and was completed at 0600 on Sept. 2. After product concentration 2.09 kgs of neptunium were loaded out, giving 93% recovery based on the 3WB processed.

The run was extremely smooth and short. Also, product losses were excellent. The length of the run was shorter than normal due to the small 3WB volume and the lower 3WB uranium concentration. The small 3WB volume and lower 3WB uranium concentration apparently resulted from reducing the 2DW loss two days earlier than usual. The 2BW neptunium loss was about 3 fold less than in previous runs, and the time required for decontamination of the neptunium during Phase II was much shorter than normal. Both of these improvements are attributed to improved solvent quality which in turn resulted from blending the 200 and 100 inventories prior to start-up of the run.

(b) Material Balance*

	<u>Kg.</u>
Np in Plant	2.29
Np in 3WB	2.25
Np Processed as 3WB	2.23
Np Load-out (Est.)	2.09
Np Recycle	0.20
Np Lost	<u>0.01</u>

(c) Typical Solvent Extraction Losses*

<u>Stream</u>	<u>% of 3WB Processed</u>
2AW	2.2
2BW	1.1

*Losses recycled for recovery during normal processing operation.

(d) Product Analytical Results

	Np g/l	HNO ₃ M	% Contamination of Np		G/Np AT&LO7
			Pu	U	
Principal Loadout	46.6	8.94	2.8	0.04	5.3
First Flush	27.2	11.2	2.5	0.06	5.3
Second Flush	4.75	10.3	7.1	0.2	5.5
Third Flush	1.23	11.4	34	0.5	7.2
Fourth Flush	0.53	11.3	67	0.4	12.2

7. Fission Product Recovery

Loading of the ORNL STT casks continued during the report period. Loading was hampered by poor performance of the in-canyon loadout jet and the limitation of feed batch size imposed by the El vessel and existing routings. The amounts of cesium loaded thus far are as follows:

	Cs-137, Curies
Cask #1, STT-44	36,000
Cask #2, STT-45A	35,000
Cask #3, STT-48	32,000

Observation of the first two loaded casks for pressure and temperature behavior was completed. The behavior was the same for both casks; a pressure build-up rate of 0.6 psig per day and an internal solution temperature approximately equal to ambient air temperature.

The first two loaded STT casks were turned over to the AEC for shipment on Sept. 8. Just prior to shipping, the casks were vented and purged with argon gas to remove accumulated hydrogen.

In an effort to define the operating difficulty with the 10 GPM in-canyon loadout jet, a 10 GPM jet was obtained from spare parts and sent to Process Equipment Development Operation of HLO for performance testing. Preliminary data indicate that this jet is a factor of two below the BFP performance curve for discharge head. If the in-canyon jet characteristics are the same, the discharge head is not sufficient to handle solutions with a specific gravity much greater than 1.0. Cesium feed has a specific gravity between 1.20 and 1.30.

On Aug. 25 470 gallons of TK-002 strontium crude were pumped to Hot Semiworks for sampling purposes in preparation for the next purification

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run. This sample indicated TK-002 contained 500,000 curies of Sr-90 with the following compositions:

<u>Component</u>	<u>Curies/Liter</u>	<u>Molarity</u>
Sr-90	26.1	
Sr-89	71.3	
Ce-144	45	
Zr-Nb-95	8	
Ru-Rh-106	10.1	
Pm-147	6.5	
HNO ₃		0.62
Pb		0.005
Fe		0.019

On Sept. 6 Hot Semiworks started up their fourth purification run. Transfer of TK-002 contents to Hot Semiworks was completed on Sept. 9. The 244-CR Vault is now empty except for pump heels.

A sample of TK-103A supernatant solution was obtained from a level of 3 feet above the sludge. This sample will be used to evaluate ways to prepare suitable feed for a Decalco cesium cask by means other than centrifugation.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 9/8/61 TO 2400 ON 9/14/61

1. Normal processing was commenced (HAF on) at 3.0 CF at 1010 on Sept. 10. Processing of "hot" feed commenced at 1800 on Sept. 10. Plant performance had not levelled out at the end of the report period. Consequently, the report for this period does not include process data.

2. Flowsheet

- (a) The plant was started up at 3.0 CF on the same flowsheet that existed at the end of the August operating period except for the following:

- (1) The diluant in all organic streams was Soltrol-170 instead of Shell E-2342.
- (2) The IBX, IBXP, IBXP-HNO₃, IBS, L Cell and N Cell flows were started up at 3.3 CF.
- (3) The 2DF ferrous sulfate flow was also started at 3.3 CF.
- (4) The IOF-KMnO₄ flow was 1.7 rather than 2.1 flows.
- (5) The IOC was 7.7 rather than 18.0 flows.
- (6) The 2AF temperature was 60 rather than 55° C.
- (7) The 2EP was diverted to the L Cell Package.

- (b) On Sept. 10 and 11, the G7 Turbosix tank carbonate solution was changed. Each change required about 2200 gallons of 2.5 wt. % Na₂CO₃.
- (c) On Sept. 11, the 2EP stream was diverted to N Cell.
- (d) On Sept. 12, mercuric nitrate addition to the metal solution was stopped.

- (e) On Sept. 12, the ZDF-ferrous sulfamate was increased from 0.44 to 0.59 flows.
- (f) On Sept. 12 and 14, the ZEX was reduced to 510 and 486 flows, respectively. The original flow had been 535 flows.
- (g) On Sept. 13, the G5-HNO₃ was increased from 0.85 to 1.0 flows.
- (h) On Sept. 13, the ICF-KMnO₄ was increased from 1.7 to 2.3 flows.
- (i) On Sept. 13, the ZEP was increased from 1.62 to 1.74 flows.
- (j) On Sept. 13, the ZAF-HNO₃ was reduced from 1.8 to zero flows. The IBXP-HNO₃ was increased from 5.4 to 6.7 flows.
- (k) On Sept. 13, the HAX-NO₂ was increased from 0.53 to 0.56 flows. It was further increased to 0.67 flows on Sept. 14.
- (l) On Sept. 13, the ZDS was increased from 65 to 73 flows.

3. Feed Preparation

Prior to plant start-up, the XA Tower in the Back-up Facility was water-flushed to reduce the radiation levels. The levels were reduced by a factor of 3 to 4.

4. Solvent Extraction

Start-up of the solvent-extraction system was uneventful except for some initial instability of the 2A Column. The pattern of decontamination and recovery performance subsequently has been superior with respect to the HA-HS Column system but substandard with respect to the final cycles. The First Cycle DF has been about 50% higher than normal, even with HAW Pu losses at 0.02 to 0.05%. Meanwhile the Final Uranium Cycle and Final Plutonium Cycle decontamination factors have been only about 600 and 750, respectively. With good ion exchange and silica decontamination performance, however, initial product-batch gamma activity has been satisfactory.

The new 2D Column scrub cartridge is operationally very stable, but may be lacking in efficiency. In addition to low gamma DF, the losses of plutonium and neptunium to the uranium product have been excessive, with the Pu content near the specification limit.

Other items of interest to solvent extraction include the following:

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- (a) The nitrite concentration in the HSP stream has been below normal (essentially non-detectable). For this reason, the addition of NaNO_2 to the HA Column was increased from 0.53 to 0.67 flows.
- (b) The HA-HS Columns were shut down for approximately 30 minutes on Sept. 11 to permit repair (by impacting) of a leaky HSR jet steam jumper.

5. Final Plutonium Concentration

Ion exchange operation was hampered by failure of the CRC on Sept. 11. Excessive Pu losses to the XSW resulted until corrected by the installation of a new CRC probe and associated wiring on Sept. 14. This maintenance was accomplished without stripping and emptying of the ion exchange columns.

6. Solvent Treatment

The gamma activity in the IOO rapidly increased after start-up from 800 to 4500 - 5000 uc/gal. where it has remained for several days. Greater than 80% of this higher level is Zr-Mb. The ICW activity has levelled out at about 20,000 uc/gal. indicating a DF of only 4 - 5 across the No. 1 Solvent Treatment System, with the greatest DF across the TK-G1 and IO Column system ($\text{KMnO}_4\text{-CO}_3$ and HNO_3 contact). Increasing the KMnO_4 flow from 1.7 to 2.3 flows, changing the entire carbonate inventory in the Turbomixer (TK-G7) and increasing the aqueous hold-up in the Turbomixer vessel from ~ 3500 gallons to 5000 gallons had no apparent effect on solvent quality.

It is theorized that the IOO activity increase may have resulted from equipment flushing preceding the change to Soltrol-170 diluent during the last plant shutdown. Laboratory work on current IOO samples indicates excellent response to laboratory washing, with resulting good solvent quality.

7. Waste Treatment and Acid Recovery

Operation of the Waste Treatment and Acid Recovery System has been limited by poor condenser vent vacuum of 35 inches vs. the normal 45 - 50 inches. The E-F6 Acid Concentrator capacity is still limited to about 75% of normal, as was experienced during the previous run period.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 9/15/61 TO 2400 ON 9/21/61

I. Typical Solvent-Extraction Performance by Cycles*

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.6	4.0	-	-	0.002	0.03
Final	2.7	2.9	12.0	2.3	0.002	-
Ion Exchange	-	1.4	-	0.2	-	-
Overall	6.3	8.3	12.0	2.5	0.002	0.03

II. Production*

A. HAF Processed: 280 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
1	4.6	2.0	8.0	P1
2	4.4	1.5	4.4	Z6
3	3.4		6.7	P1
4	7.7	6.2	6.6	P1
5	4.5		6.7	P1
6	4.0	2.6	6.0	P1
7	4.2		7.4	P1
8	4.7	13.8	7.1	P1
9	4.9	13.0	7.5	P1
10	5.6	11.1	7.6	P1
11	5.8		7.8	P1
12	4.8	8.3	7.7	P1
13	4.5	8.6	7.6	P1
14	4.9	8.6	7.7	P1

*Data shown is since plant start-up on Sept. 10

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
15	5.0	1.2	7.9	P1
16	5.1	7.0	7.3	P1
17	5.4	7.0	7.2	P1
18	4.7	8.2	7.7	P1
19	4.5	9.8	7.3	P1
20	4.6	9.5	7.1	P1
21	4.7		7.6	P1
22	5.4	8.5	7.4	P1
23	6.5		7.7	P1
24	7.0	14.0	7.5	P1
25	8.5	20.0	7.5	P3
26	9.1	21.5	5.4	P1
27	8.1	14.8	7.2	P3
28	6.7	10.8	4.5	P1
29	6.0	7.7	7.0	P1
30	6.7		7.1	P1
31	7.3	12.3	7.7	P1
32	8.2	15.5	7.0	P3
33	7.6	13.8	7.2	P3
34	6.9	12.6	7.4	P3
35	6.1	10.5	7.3	P3
36	5.6	9.5	7.3	P3
37	5.5	13.6	7.6	P1
38	5.8	13.8	7.2	P3
39	5.4	13.8	7.4	P3
40	5.4	12.4	7.7	P3

2. Silica Gel

Gamma Ratios: Feed = 5.7
 Product = 1.1
 Tonnage = 230 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/NT x 10¹¹</u>	<u>% U in Pu</u>	<u>Pu, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
1	1.7	0.010		3	2	234-5
2	2.6	0.002		4	2	234-5
3	1.5	0.002		5	2	234-5
4	1.2	0.004		5	2	234-5
5	1.5	0.003	2400	5	2	234-5

C. Plutonium Product:

<u>Batch</u>	<u>Zr/Pu/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Pu, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
6	1.2	0.003		5	2	234-5
7	1.0	0.002		4	2	234-5
8	1.2	0.002		6	2	234-5
9	1.6	0.002		4	2	234-5
10	1.2	0.005	2350	5	2	234-5
11	1.3	0.009		5	2	234-5
12	2.0	0.013		5	1	234-5
13	1.6	0.009		5	2	234-5
14	0.7	0.007		5	2	234-5
15	0.3	0.009		4	2	234-5
16	0.5	0.007		5	2	234-5
17	0.9	0.007		5	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.23%

Pu = 0.53%

E. Rework - % of Total Production:

U = 4.1% (Out-of-Spec. U Product from U Storage TK-F3 to 2DF TK-K1 through Final U Cycle only and 2EU TK-K6 to HAF Make-up TK-E6 at shutdown)

Pu = 13.8% (Predominantly Out-of-Spec. Pu Product and Misc. Rework Material via L11 to HAF Make-up TK-E6)

III. Waste Treatment*

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	1010	104 and 106
Cell Drainage	217	106
FP Waste	54	106
Coating Waste	270	107C

B. LW Flows: 5.4 Flows

C. Tank Farms:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	11.1
102	6.3
103	0.5
104	10.6

*Data shown is since plant start-up on Sept. 10.

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Notes:

1. The HAF rate was maintained at 3.0 CF until 1330 on Sept. 16 at which time an increase to 3.3 CF was made. On Sept. 18, the HAF flow was stopped for four hours (0110 to 0510) for an HAF pump change. At 2100 on Sept. 18, the HAF rate was reduced to 3.0 CF.

2. Flowsheet

- (a) On Sept. 15, the HAX-~~NO₂~~ was increased from 0.65 to 0.67 flows. It was further increased to 0.85 flows on Sept. 17.
- (b) On Sept. 15, the 2DS was increased from 73.5 to 77.5 flows. It was further increased to 81.0 flows on Sept. 16.
- (c) On Sept. 15, the 2SX-H₂O was reduced from 510 to 480 flows.
- (d) On Sept. 16, the plant rate was increased from 3.0 to 3.3 CF. The IBXP, IBXP-HNO₃, IBS, 2DF-Fe, L-Cell and N-Cell flows (gpm), however, were not increased.
- (e) On Sept. 18, the IBXP-HNO₃ was reduced from 6.5 to 6.4 flows.
- (f) On Sept. 18, the 2DF-ferrous sulfamate was increased from 0.49 to 0.69 flows.
- (g) On Sept. 18, the 3WB was increased from 40 to 45 flows.
- (h) On Sept. 18, the 2BP was reduced from 2.72 to 2.45 flows.
- (i) On Sept. 18, the HAX and HAF were reduced from 3.30 to 3.16 CF.
- (j) On Sept. 19, the Final Uranium Cycle rate was increased from 3.3 to 3.6 CF, except the 2DF-HNO₃, 2DF-Fe, and 2DS flows (gpm) for an 8 hour period.
- (k) On Sept. 19, the addition of 0.0005 M Hg(NO₃)₂ to the metal solution was commenced.
- (l) On Sept. 20, the 2DS was increased from 77 to 81 flows. It was further increased to 84 flows on Sept. 21.
- (m) On Sept. 21, the 2DF-HNO₃ was decreased from 7.7 to 4.6 flows.
- (n) On Sept. 21, the 3WB HNO₃ was started at 2.9 flows.
- (o) On Sept. 21, the 2DF, 2DX, and 2EX flows were reduced from 3.30 to 3.16 CF.
- (p) On Sept. 21, the IOF-K₂Cr₂O₇ was reduced from 2.30 to 1.69 flows.

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3. Feed Preparation

On Sept. 21, A3 Dissolver was shut down because of failure of the dissolver thermohm.

4. Solvent Extraction

Solvent-extraction performance improved during the report period, except for the Final Uranium Cycle. The First Cycle DF improved about two fold, while HAW losses were maintained very low. The Final Plutonium Cycle DF improved one third or more, apparently as a result of increasing the 2A Column pulse frequency. The Final Uranium Cycle performance suffered, however, with respect to gamma, plutonium and neptunium decontamination.

Since plant start-up the 2D Column performance has been substandard with respect to gamma, Pu, and Np DF. At the beginning of the report period, Pu in the U was in the range of 7 - 10 parts per billion parts of U, compared to the specification of 10. On Sept. 17, following a rate increase started on the 16th, the 2D Column suddenly flooded, and the Pu-in-U increased to 20 ppb. Subsequently, adjustments were made aimed at correcting this condition, and an attempt was made to rework the out-of-specification material through the Final Uranium Cycle. Excessive contamination of the uranium product continued, however, throughout the report period. No apparent response resulted from

- (a) Increased ferrous sulfamate flow to the 2DF;
- (b) Pulse frequency changes in the 2D Column;
- (c) Increased 2DS flow.

Laboratory analysis showed the expected ferrous ion to be present in the 2DW. Other laboratory studies were being made to further investigate the chemistry of the system. At the end of the report period the HNO_3 concentration in the 2DF was being reduced in a further effort to correct the problem. Neptunium losses to the uranium product averaged about 25% of virgin HAF during this period.

A brief investigation by Chemical Research showed 50% to 100% more nitrite take-up by Soltrol-170 as compared to Shell E-2342 after about one hour of exposure to nitrite. In the plant, the nitrite to the HAX has been increased about 60% since start-up, until the ESP nitrite concentration has become about normal. Control of HAW Np losses has been excellent.

5. Plutonium Concentration

Ion Exchange DF has been excellent during the report period, ranging between 25 and 30.

On Sept. 15, an orifice was placed back in the push line as a means of preventing channeling during pushes. The orifice had been removed during the previous plant shutdown.

6. Solvent Treatment

The gamma activity in the IOO which had peaked at about 5000 uc/gal. shortly after plant start-up, has slowly decreased to about 1200 uc/gal. The rate of decrease was interrupted only slightly by the shutdown and start-up of the HA Column on Sept. 18 indicating that initial activity was probably from equipment flushes and solvent and diluent displacement during the shutdown period. The ZrNb activity has dropped from about 2500 uc/gal. to about 300 uc/gal. since start-up while the Ru has increased from 250 to ~ 600 uc/gal. The activity in the ICW decreased from about 20,000 uc/gal. to 15,000 uc/gal. during this period.

The effect of increasing the Turbomixer speed from 430 to 540 rpm during the report period was difficult to judge due to the short shutdown of HAF; however, from the gamma plots little change was noted in the decreasing trend in gamma activity.

7. Waste Treatment and Acid Recovery

Although a leak in the overflow from the Acid Concentrator E-F6 has precluded normal overflow, operation of the Waste Treatment equipment has been satisfactory. Build-up of corrosive products in F6 is being prevented by routine jetting of F6 to TK-F7, with the frequency maintained to provide as close as possible the flowsheet 2W rate. With a condenser vent vacuum of 45 in. of water, E-F11 pot vacuum has been greater than 20 in. of water, and F6 pot vacuum has been about 5 to 10 inches. The F7 dilution water, however, is about 15% below normal.

8. Fission Product Recovery

(a) Cesium

Loading of the fourth STT cask with 33.5 kilocuries of cesium-137 was completed on Aug. 15. Word was received from ORNL that the first two STT's had been eluted yielding 56 kilocuries. This does not compare well with the HAPC estimate of 71 kilocuries loaded. ORNL also transmitted the following information on the elution of the first cask loaded in April.

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Elution Concentrations - Curies/Liter

Ce-137	11
Ce-144	0.11
Sr-90	0.018
Ru-106	2×10^{-4}
ZrNb-95	2.5×10^{-4}

(b) Cerium - Promethium

On Sept. 16, a cerium - promethium test run was started. The purpose of this run was to demonstrate a cerium flowsheet (peroxyacetate process) and to provide a promethium rare earth cut for the high level cells. The processing scheme consisted of the following steps:

- (1) Two typical sulfate runs except lead carrier were omitted since strontium is considered a contaminant during Ce-Pm processing;
- (2) One oxalate run modified to include a metathesis with 1 M sodium hydroxide;
- (3) A peroxyacetate run on the rare earth fraction of the oxalate run;
- (4) A carbonate concentration run to obtain a volume practical for cask loading.

The peroxyacetate processing conditions were as follows:

Feed

Volume	- 740 Gallons
pH	- 1.6
Cerium Concentration	- 2140 Curies/Gallon

Precipitation Reagent

Volume	- 120 Gallons
pH	- 5.0
H ₂ O ₂	- 3.5 M
Na C ₂ H ₃ O ₂	- 1.47 M

Centrifuge Feed (Feed Plus Precipitation Reagent)

Volume	- 860 Gallons
pH	- 4.6
Digestion Time	- 60 Minutes
Temperature	- 50° C

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By the end of the report period the peroxyacetate run had been completed. A complete material balance for the various steps was as follows:

Stream	Curies				
	Ce	Pm	Sr	Ru	ZrNb
IW	1,300,000	300,000*	685,000	215,000	1,900,000
Sulfate Waste	330,000	68,000	374,000	290,000	1,700,000
Flush	--	Nil	--	--	--
Oxalate Waste	87,000	12,000	385,000	6,000	720,000
Cerium Product	670,000	93,000	15,000	7300	22,000
Promethium Product	290,000	163,000	54,000	7814	5,000

*Estimate - IW Analyses not consistent.

Stream	Percent of Fission Product Present				
	Ce	Pm	Sr	Ru	ZrNb
IW	100	100	100	100	100
Sulfate Waste	25	23	55	135	90
Flush	-	-	-	-	-
Oxalate Waste	7	4	56	3	38
Cerium Product	51	31	2	-	1
Promethium Product	22	54	8	-	-
Total	105	112	121	138	129

As seen in the above material balance, the separation of cerium and promethium from each other was poor. The high promethium loss to the cerium fraction was expected since the flowsheet was aimed at a maximum cerium DF at the expense of promethium recovery. The poor separation of cerium from promethium was not expected.

On September 11, a special long shaft, slow speed agitator was installed in TK-E3. This agitator reaches within a couple of inches of the tank bottom and can be operated when the tank is empty without damage to the shaft. Preliminary results indicate most of the precipitate is now transferred from TK-E3 to the centrifuge. Previously, about one third of a run was centrifuged without agitation. Tank flushes showed that large amounts of precipitate settled out and could not be transferred to the centrifuge.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 9/22/51 TO 2400 ON 9/28/51

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.6	3.9	-	-	0.003	0.03
Final	3.0	3.1	13.0	2.7	0.004	-
Ion Exchange	-	1.4	-	0.1	-	-
Overall	6.6	8.4	13.0	2.8	0.007	0.03

II. Production

A. HAF Processed: 188 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbm U</u>	<u>Tons</u>	<u>Shipped To</u>
41	5.2	10.6	7.0	P3
42	5.7	7.8	7.6	P1
43	5.6	6.2	7.1	P1
44	5.5	5.4	7.3	P1
45	5.5	4.4	5.2	P1
46	4.2	2.4	7.0	P1
47	3.7	2.5	6.5	P1
48	3.0	1.3	7.5	P1
49	2.6	1.4	7.6	P1
50	2.5	1.2	7.3	P1
51	3.1	1.2	7.5	P1
52	2.4	1.6	7.6	P1
53	2.2		7.5	P1
54	2.3	0.7	7.6	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
55	2.1		7.0	P1
56	2.3	0.5	7.5	P1
57	2.5		7.4	P1
58	2.4	0.1	7.4	P1
59	1.9		7.3	P1
60	2.2	0.2	7.3	P1
61	2.7		7.5	P1
62	2.4	0.1	7.2	P1
63	2.5		7.5	P1
64	2.7	0.1	7.1	P1
65	2.7		7.3	P1
66	2.5	0.1	7.3	P1

2. Silica Gel

Gamma Ratios: Feed = 3.1
Product = 1.0
Tonnage = 130 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Pu, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
18	0.9	0.024		5	2	234-5
19	1.1	0.004		5	2	234-5
20	1.1	0.036		5	2	234-5
21	1.0	0.002		5	2	234-5
22	0.9	0.018		4	2	234-5
23	1.0	0.011		5	2	234-5
24	0.9	0.002		5	2	234-5
25	0.9	0.030		5	1	234-5
26	15	0.020		4	2	234-5
27	1.1	0.019		6	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.05%
Pu = 0.12%

E. Rework - % of Total Production:

U = Negligible
Pu = 8.7% (Predominantly out-of-spec Pu Product via TK-L11 to HAF
Make-up TK-E6)

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III. Waste Treatment

A. Volumes to UCS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	477	104 & 106
Cell Drainage	60	106
FP Waste	17	106
Coating Waste	270	107C

B. IW Flows: 5.0 Flows

C. Tank Pans:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	11.0
102	7.0
103	0.3
104	10.2

Notes:

1. The HAF rate was maintained at 3.0 CF until 1330 on Sept. 25 at which time an increase to 3.3 CF was made. Subsequent rate checks showed the actual rate to be 3.4 CF.

2. Flowsheet

- (a) On Sept. 22 the 3AB-HNO₃ was increased from 2.9 to 4.1 flows. It was reduced to 2.9 flows after 12 hours and was stopped on Sept. 23.
- (b) On Sept. 22 the 2DF-HNO₃ was reduced from 4.5 to 2.7 flows. It was increased to 7.0 flows on Sept. 23.
- (c) From Sept. 22 through 26 the IUC uranium concentration was reduced in order to reduce the 2DF from 2.4 to 2.0 M U.
- (d) From Sept. 24 through 26 the 2DF-ferrous sulfamate was reduced from 0.69 to 0.27 flows.
- (e) On Sept. 23 the 2DS was reduced from 76 to 61 flows.
- (f) On Sept. 26 the IBXP-HNO₃ was increased from 6.2 to 6.4 flows. It was further increased to 7.2 flows on Sept. 28.

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3. Feed Preparation

On Sept. 23 operation of A3 Dissolver was resumed, following replacement of a failed thermohm (see p. 179).

4. Solvent Extraction

Process performance during the report period was excellent with respect to product quality and losses. A gradual increase in intercycle gamma levels did not significantly affect the products.

At the beginning of the report period the problem of excessive plutonium and neptunium in the uranium product was improving. On Sept. 23 a scheduled change of the 2D Column scrub-section cartridge was successfully effected, with only a temporary shutdown of the Final Uranium Cycle required. The replacement cartridge was again a model assembled with 6% free area nozzle plates compared to 10% free area in the cartridge replaced (see pp. 130, 166). However, in order to improve its operability, the plate spacing was graded. The bottom sections contains 22 plates at 2-in. spacing; the middle section contains 15 plates at 3-in. spacing; and the top section contains 12 plates at 3-in. spacing with a final upper plate at 2-1/2 inches. The plate sections are about 42 inches long, separated by louver plates.

As indicated above, the problem of Pu and Np in the U product was correcting itself when the cartridge change was made, and the Pu content of the 2DU stream at that time was estimated to be less than 4 parts Pu per billion parts of U (compared to the specification of 10). A study of process variables before and during this period indicates no positive explanation for this process problem. At the moment the most likely explanation involves an initial improvement in U product quality as a result of briefly lowering the 2DU HNO_3 concentration (by reducing 2DU HNO_3) from 0.95 to 0.48 M. Subsequent good operation at normal acidity may be explained by the change to a more efficient cartridge. During the period of excessive Pu in U, Process Chemistry indicated nothing chemically wrong with the system. Also, normal iron content in the uranium product indicated the probable absence of any entrainment problem. Additional laboratory study of a special 2DU sample is in progress.

The 2DU plutonium loss was decreased from 0.55 to 0.35% by increasing the 2DU Column pulse frequency from 57 to 61 cycles per minute. Based on instrument readings this change resulted in a slight cyclic operating pattern.

5. Solvent Treatment

The gamma activity in the ICO stream increased from ~ 1,000 uc/gal. to ~ 2,000 uc/gal. during the report period. Since plant start-up the ICO activity spectrum has gradually shifted from predominantly Zr-95 to about 2/3 Ru and 1/3 Zr-95.

Restrictions in the pumping rate from TK-01 to the IO Column has necessitated water flushing and carbonate flushing of this line. Pumping capacity is currently limited to about 75% of design capacity.

6. Water Demineralizer

On Sept. 22 the lead-lined sulfuric acid mix tank was replaced with a spare. The failed tank had only been in service seven weeks.

7. Fission Product Recovery

A concentration run to prepare a promethium crude suitable for cask load-out was started on Sept. 23. The promethium crude was adjusted to 0.2 M sodium carbonate, digested for an hour, centrifuged and the cake dissolved in 0.2 M nitric acid. Samples of the product and waste solutions failed to show any cerium or promethium. The waste fraction had been prematurely discarded and was not available for resampling. On the chance that the sampler in the product solution tank was inoperative, a cask was loaded and sent to HLO for analyses. The analyses confirmed that the product had been lost.

On Sept. 26, a second series of runs aimed at obtaining a promethium crude for the high level cells was initiated. By the end of the report period the sulfate runs had been completed and the oxalate run was underway.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 9/29/61 TO 2400 ON 10/12/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.4	3.7	-	-	0.003	0.05
Final	3.2	3.2	12.0	5.4	0.003	-
Ion Exchange	-	1.4	-	1.0	-	-
Overall	6.6	8.3	12.0	6.4	0.006	0.05

II. Production

A. HAF Processed: 238 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
67	2.8		7.3	P1
68	2.6	0.4	7.5	P3
69	2.6		7.4	P3
70	2.6	0.1	7.5	P3
71	2.1		7.5	P3
1	2.7		7.5	P3
2	3.1	0.3	7.5	P3
3	2.8		7.6	P3
4	2.3	0.3	7.5	P3
5	2.9		7.4	P3
6	2.9	0.3	7.5	P1
7	2.6		7.6	P1
8	2.9	0.5	7.5	P1
9	3.2		7.4	P1

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
10	3.2	0.4	7.2	P1
11	3.1		6.9	P1
12	3.3	0.6	7.5	P1
13	3.7		7.5	P1
14	4.2	0.6	7.5	P1
15	4.2		7.3	P1
16	4.3	0.3	7.6	P1
17	4.1		7.4	P1
18	4.0	4.5	7.4	P1
19	3.8	0.4	7.5	P1
20	3.6	0.8	7.4	P1
21	4.1		7.5	P1
22	4.5		7.4	P1
23	4.2		7.2	P3
24	4.1	0.3	7.5	P1
25	3.7		7.5	P1
26	4.2	0.2	7.5	P1
27	4.2		7.4	P1

2. Silica Gel

Gamma Ratios: Feed = 3.2
 Product = 1.2
 Tonnage = ~ 140 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNo./AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Wt./Hr.</u>	<u>Shipped To</u>
28*	1.1	0.009		5	2	234-5
29	1.0	0.030		4	2	234-5
30	1.3	0.020		6	2	234-5
1	1.7	0.031		6	2	234-5
2	1.5	0.020		5	2	234-5
3	1.6	0.030		5	2	234-5
4*	1.4	0.007		5	2	234-5
5	1.5	0.004	7890	5	2	234-5
6	1.4	0.025		5	3	234-5
7	2.1	0.001		5	3	234-5
8	1.8	0.022		5	3	234-5
9	1.5	0.033		4	2	234-5
10	1.6	0.068		5	2	234-5
11	1.5	0.004		6	2	234-5
12	1.9	0.030		5	3	234-5
13	2.1	0.038		6	2	234-5

*(1 Can from Batch 28 and 4 Recycled via L9 to XAF TK-H1 Thru Final Pu Anion Exchange Unit only)

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D. Overall Waste Loss - % of Total Production:

U = 0.06%

Pu = 0.09%

E. Rework - % of Total Production:

U = Negligible

Pu = 3.4% (Includes 2 cans reworked from TK-L9 to XAF Make-up TK-W1 Thru Pu Anion Exchange Unit, only, and Miscellaneous Pu Rework via TK-L11 to HAF Make-up TK-E6)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	446	104 TK
Cell Drainage	25	106 TK
FP Waste	46	106 TK
Coating Waste	270	107C TK

B. EW Flows - 5.4 Flows

C. Tank Farm

<u>Tank</u>	<u>Estimated Boil-Off Rate, Gal/Min</u>
101	10.9
102	3.6
103	0.2
104	8.1
106	8.0

Notes:

1. The HAF rate was maintained at a nominal 3.3 CF until 1240 Oct. 7, when the plant was shut down for correction of waste diversion box difficulties. Periodic checks indicated the actual instantaneous rate to be 3.4 CF during the period of operation.

2. Flowsheet

(a) On Sept. 29, the IBXP-HNO₃ was reduced from 6.9 to 6.7 flows.

(b) On Sept. 29, the 2DF ferrous sulfamate was reduced from 0.27 to 0.22 flows.

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- (c) On Sept. 29, the 2EX-HNO₃ concentration was reduced from ~ 0.01 M to ~ 0.005 M.
- (d) On Oct. 1, the 2DS was reduced from 6.7 to 6.2 flows. It was further reduced to 5.7 flows over the next two days.
- (e) On Oct. 3, the 2EX temperature was increased from 47 to 49.5° C.
- (f) On Oct. 4, the IBXP-HNO₃ was increased from 6.7 to 7.5 flows. It was further increased to 7.7 flows on Oct. 5 and to 8.0 flows on Oct. 6.
- (g) On Oct. 5, the IBS was increased from 1.02 to 1.26 flows. ^{18.2 19.6}
- (h) On Oct. 5, the 2EX temperature was reduced from 49.5° to 47° C. It was returned to 49° C on Oct. 6.
- (i) On Oct. 5, HNO₃ addition to the 2AF was started and continued for 20 hours.

3. Solvent Extraction

Solvent-extraction performance during the report period was generally excellent. A gradual increase --- say, 10% --- in the gamma activity throughout the plant did not significantly affect the product quality. Superimposed on this pattern, however, was a temporary, approximately 50% gamma increase resulting from an HSS flow outage on Oct. 3. The first cycle product activity was kept under control, but at the expense of a slight increase in the HAW losses near the end of the operating period. Likewise, the Final Plutonium Cycle DF also showed an improvement of nearly 50 per cent toward the end of the operating period, at the expense of a three-fold increase in the per cent recycled in the 2AW. The Final Uranium Cycle DF decreased approximately 30 per cent during the report period, but the activity was still well within silica-gel feed limits. Decontamination of the uranium product from plutonium and neptunium was excellent. The uranium and plutonium inventory in the backcycle system was slightly above normal, due to high losses in the 2DW and 2AW streams.

The plant was shut down at 1300, Oct. 7 according to the standard equilibrium shutdown procedure, with the following exceptions:

- (a) The HA-HS Columns were stripped for 30 minutes.
- (b) The 2A-2B Columns were stripped for four hours.

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4. Final Plutonium Concentration

Since the beginning of the current operating period, occasional samples of ion exchange resin have been analyzed for gamma activity. Analytical results have shown a gradual increase in the activity associated with the resin to a more-or-less constant value of about 500 uc./gal. of ZrNb. This compares with a reported activity of 2×10^3 uc./gal. of ZrNb at the end of the last run period. During the current run period, mechanical performance of the ion exchange equipment has been excellent.

Two cans of normal Pu product (~ 1800 grams per can) were reworked during the report period in order to further test valence adjustment using ferrous sulfamate. Sampling problems and other difficulties prevented evaluation of all facets of the test; however, the adjustment step was apparently successful as no noticeable increase in XAW losses were noted. Prior to adjustment the Pu to be reworked was about 30% PuVI and 70% PuIV.

During the report period the last of the out-of-specification cans generated during the June-July-August operating periods were thief-sampled and found to be suitable for shipment to Z Plant.

On Oct. 7, following the plant shutdown, the ion exchange facility was shut down after being stripped.

5. Solvent Treatment

The gamma activity in the IOO continued to increase slowly to about 2500 uc./gal. The increase is essentially all Ru, and the ratio of Ru to ZrNb in the IOO is currently about 4:1. Meanwhile, the total gamma activity in the ICW has increased from ~ 16,000 uc./gal. to ~ 23,000 uc./gal., due to increased Ru in this stream. Decreasing the change cycle from 6 hours to 5 hours on TK-G1 carbonate had no apparent effect on the gamma activity of the IOO.

6. Waste Treatment and Acid Recovery

(a) F6 Demister Test

On Sept. 28 and Oct. 5, the water to the F6 Demister was turned off for periods of 4 shifts and 6 shifts, respectively to check the effect on acid activity. At the time, the gross gamma activity in the AAA (Absorber acid in TK-F3) was about 1500 uc./gal., and the ratio of ZrNb to Ru ranged from about 2:1 to about 4:1. No effect was noted when the Demister water was turned off or on.

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(b) Diversion Box Troubles

Non-scheduled plant shutdown on Oct. 7 was necessitated by 241-A-151 diversion box difficulties. For several weeks leakage had been noted in this box, as indicated by increasing Catch Tank liquid levels. Attempts to jet the Catch Tank failed, so the diversion box was opened on Oct. 7 on the theory that the Catch Tank jet-out jumper (to underground storage) probably required replacement. However, any work was prevented by steam issuing from the box when the cover was removed. Even after adding water to the box, the steaming problem continued. A special line has been constructed which will permit emptying the Catch Tank and box back to TK-F18, after a pump is installed in the Catch Tank. Temperature readings in the Catch Tank are up to 105° C near the bottom of the tank.

7. Fission Product Recovery

The second series of runs to obtain a crude promethium cut for the HLO High Level Cells was completed on Oct. 3. The processing scheme for this series of runs was similar to the first series (see page 181) except for minor changes made in the peroxyacetate step. The precipitation pH was lowered from 4.5 to 4.1 to reduce promethium losses to the cerium fraction and the hydrogen peroxide concentration was increased two fold. The latter change was based on High Level Cell experience with plant oxalate product from the first run series. The plant solution was found to require a large initial peroxide butt to overcome some apparent reductant in the solution. After the initial large peroxide butt, the behavior of plant solution was normal.

Similar to the first run series, an attempt was made to prepare a promethium concentrate for cask loadout by precipitation with sodium carbonate. Precipitation was not successful and the promethium remained in the supernatant solution. The pH of the supernatant solution was checked and found to be greater than 9.0. The failure of this step in the first run series had been attributed to inadvertent acid addition to the centrifuge through incomplete header flushing or from acid left in the line between the centrifuge and centrifuge spray pump. Special flushes were performed during the second run series to insure that the lines were free of acid.

The waste and product fractions from the unsuccessful concentration run were recombined, acidified to a pH of 0.4 and precipitated with oxalic acid. The slurry was centrifuged, metathesized with 1.0 M caustic and dissolved in a minimum of 0.5-M nitric acid. The volume of dissolved cake (promethium crude) was insufficient to permit agitation for sampling. Rather than dilute this solution for sampling, it was loaded into 200 gallon casks and shipped to the High Level Cells. Cerium analyses were completed immediately to confirm the success of the oxalate concentration step. Promethium analyses are currently in process.

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A material balance for this series of runs is listed below:

Cerium-Promethium Test Run #2
Fission Product Material Balance

	Per Cent of Fission Product Present				
	Sr	Ce	Zr-Nb	Ru	Pm
I/W Feed	100	100	100	100	100
Sulfate Waste	80	20	90	102	40
Flush	5	2	1	1	2
Oxalate Waste	65	4	5	1	30
Peroxyacetate Ce	1	55	/1	/1	3
Peroxyacetate Pm	8	10	2	/1	50
Flush	4	15	/1	/1	20
Carbonate Conc. Run*					
Product	-	/1	/1	/1	2
Waste	8	15	/1	/1	95
Oxalate Conc. Run					
Product**	1	9	/1	/1	30
Waste	<u>6</u>	<u>1</u>	<u>/1</u>	<u>/1</u>	<u>15</u>
Total	170	116	98	104	190

*Not included in total.

**Sample from unagitated vessel.

The material balances for promethium and strontium were poor. Based on the carbonate product samples and the analyses of oxalate waste and product fractions, the sulfate waste strontium analyses was high by a factor of two. No firm conclusions about promethium behavior can be drawn until the analyses on the cask contents are complete.

Following loadout of the promethium crude on Oct. 31, strontium production was resumed. At the time of plant shutdown on Oct. 7, three sulfate runs had been completed.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 10/13/61 TO 2400 ON 11/2/61

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During the report period, the plant was shut down because of the diversion box difficulties described on p. 193 and below. Other process notes of interest are also reported below.

1. Iodine Emissions

The pattern of iodine emissions and control during the past operating period indicated an initial breakthrough via the condenser and vessel vent system, followed by an increase in emissions from the Head End. During September, radioiodine input to the plant was in the range of 100 to 400 curies per day. A trend for increased emissions in mid-September - peaking near 1.8 curies/day Sept. 15 - 18 - was controlled by addition of mercuric nitrate to the metal solution. This indicated the iodine from the F-Cell (vent) system to be controlling. Early in October the radioiodine input was greater - in the range of 600 - 1000 curies per day. Apparently this resulted in an emission increase to 1.6 curies/day on Oct. 3. Subsequently, plant shutdown on Oct. 7 was followed by a decline to 0.4 curies/day.

On Oct. 11, the F-Cell silver reactor was regenerated. The B-Cell reactor was regenerated on Oct. 14; it had processed the off gases from 761 tons of uranium since the last regeneration.

2. Waste Treatment and Acid Recovery

(a) Diversion Box Troubles

As previously indicated (p. 193), a special line was constructed from the catch tank in the 241-A-151 diversion box, and a pump was installed, to permit emptying of the box and tank via TK-F18 to underground storage. Large-volume water flushes, added both via the diversion box spray system and directly to the box, were then used to reduce temperatures and eliminate the steaming problem.

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Following initial water additions the steaming of the diversion box persisted, even though the flush-water from the box reduced the liquid temperature in the catch tank. Subsequently, the 202-A Building steam was shut off to eliminate this possible source. Further water flushes confirmed the same pattern of catch tank temperature decrease but ultimately resulted in elimination of box steaming. Fission-product leakage into the box was finally judged to be responsible for the original temperature and steaming problem.

After the steaming problem was eliminated, two leaking jumpers in the diversion box were replaced: (1) from the catch tank to underground storage, and (2) from TK-F16 to underground storage.

(b) E-F11 Jet-Out Problem

During the shutdown period, attempts to empty the E-F11 Concentrator were unsuccessful. In connection with these attempts, the concentrator contents were cooled and diluted; a new jet was installed; and backflush water was added. Although some transfer did finally take place, time did not permit resolution of this problem prior to plant start-up.

(c) Tube Bundle Replacement

Shutdown testing revealed the failure of the E-F6 east tube bundle. The bundle was replaced.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 10/23/61 TO 2400 ON 11/2/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.7	3.9	-	-	0.003	0.02
Final	2.5	2.7	10.0	10.4	0.004	-
Ion Exchange	-	1.3	-	0.1	-	-
Overall	6.2	7.9	10.0	10.5	0.007	0.02

II. Production

A. HAF Processed: 258 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
28	5.1	0.2	5.5	P1
29	7.4		6.3	P1
30	19.3	1.5	6.6	P3
31	32.1		5.7	E6
32	42.3	1.1	6.2	E6
33	-		7.2	E6
34	31.4	0.4	7.1	E6
35	20.5		7.3	E6
36	17.4	0.4	6.9	P3
37	7.4		6.9	P1
38	4.1	0.8	6.8	P1
39	2.9		7.0	P1
40	2.6	0.2	7.2	P1
41	2.5		7.0	P1
42	2.7	0.1	6.9	P1
43	3.0		7.0	P1

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
44	4.2	0.1	7.0	P1
45	3.9		7.3	P1
46	4.3	0.1	7.3	P1
47	5.1		7.4	P1
48	5.0	0.1	7.0	P1
49	5.5		7.1	P1
50	5.4	0.2	7.2	P1
51	4.7		7.6	P1
52	3.9	0.1	7.0	P1
53	5.5		6.9	P1
54	5.9	0.4	6.4	P1
55	3.9		7.2	P1
56	4.0	0.4	7.2	P1
57	3.5		7.3	P1
1	7.5	8.2	6.9	P1
2	12.4	9.9	7.0	P3
3	10.4	8.8	7.3	P3
4	8.2	6.0	6.9	P3
5	7.6	5.8	7.0	P1
6	5.0	2.1	7.3	P1
7	4.3		7.4	P1
8	3.8	0.2	7.2	P1

2. Silica Gel

Gamma Ratios: Feed = ~ 4.9
 Product = ~ 1.5
 Tonnage = 231 Tons

C. Plutonium Product:

<u>Batch</u>	<u>Zr/Nb AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Shipped Mr/Hr</u>	<u>Shipped To</u>
14	100	-		-	-	E6
15	3.4	0.028		4	10	Holding
16	8.4	0.004		4	15	E6
17	11.5	0.055		5	6	E6
18	6.9	0.020		4	6	Holding
19	4.5	0.020		5	6	Holding
20	3.5	0.013	2127	5	4	234-5
21	2.0	0.008		5	7	234-5
22	1.5	0.010		5	3	234-5
1	1.3	0.007		5	4	234-5
2	1.5	0.007		5	2	234-5
3	2.3	0.004		5	5	234-5
4	2.5	0.004		5	3	234-5

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D. Overall Waste Loss - % of Total Production:

U = 0.23%
Pu = 0.57%

E. Rework - % of Total Production:

U = 15.3% (Includes out-of-spec. U product from 2EU TK-K6 to HAF make-up Tank E6 and U Storage Tank P3 to 2DF TK-K1 through Final U Cycle, only)

Pu = 37.3% (Predominantly out-of-spec. Pu product via Tank L11 to HAF make-up Tank E6)

III. Waste Treatment

A. Volumes to UCS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	678	104
Cell Drainage	196	104-106
FP Waste	155	106
Coating Waste	270	107C

B. IWW Flows: 6.2

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	5.7
102	5.7
103	~ 0.1
104	7.6
106	13.4

Notes:

1. Normal processing was commenced at 3.0 CF with "hot" HAF on at 0415 on Oct. 23. Recycle of "cold" feed was started at 0500 on Oct. 24 because of high activity throughout the system. The activity was down sufficiently to permit return to "hot" feed at 2045 on Oct. 25. The HAF was maintained at a nominal 3.0 CF rate until 1200 on Nov. 2, when it was increased to a nominal 3.3 CF.

2. Flowsheet

The start-up flowsheet was essentially the same as that in effect at prior shutdown except the 2BP stream was initially routed to the L-Cell Package, instead of to the N-Cell Ion Exchange Facility. During the report period, flowsheet changes were made as follows:

- (a) On Oct. 24, the HSS-HNO₃ was increased from 11 to 12.3 flows.
- (b) On Oct. 24, the HSR-HNO₃ was increased from 7.2 to 7.3 flows. It was further increased to 8.1 flows on Oct. 29.
- (c) On Oct. 24, the IBXP-HNO₃ was decreased from 6.3 to 6.0 flows. It was increased back to 6.5 flows on Oct. 25 and to 6.9 flows on Oct. 28.
- (d) On Oct. 25, the 2DF-ferrous sulfamate was reduced from 0.44 to 0.29 flows. It was further reduced to 0.25 flows on Oct. 26.
- (e) On Oct. 25, nitric addition to the 2AF was discontinued.
- (f) On Oct. 25, the 2BP stream was diverted from the L Cell Package to the N-Cell Ion Exchange Facility. The 2BX composition and flow were changed accordingly.
- (g) On Oct. 25, the 2EX temperature was increased from 47 to 49° C. It was further increased to 56° C on Oct. 26.
- (h) On Oct. 27, the 2AX was increased from 6.0 to 6.5 flows.
- (i) On Oct. 28, the addition of waste rework from TK-P8 to the HAF was commenced at an approximate rate of one volume of waste to 40 volumes of HAF. Processing of this material in the HA Column was commenced on Oct. 29. The addition rate was increased to about one volume of waste to 27 parts of HAF on Oct. 29.
- (j) On Oct. 29, the HAO rate was increased from 87 to 91 flows.
- (k) On Oct. 29, the Final Uranium Cycle rates were increased to 3.3 CF in order to reprocess out-of-specification uranium product from TK-P3 storage to the 2DF Tank K1.
- (l) On Oct. 29, the IBXP was decreased from 23 to 22.2 flows. It was further decreased to 21.7 flows on Oct. 30.
- (m) On Oct. 30, the 2AS was increased from 1.9 to 2.1 flows.
- (n) From Oct. 30 to Nov. 1 the 2EX temperature was gradually reduced from 56° C to 50° C.
- (o) On Nov. 1, HNO₃ addition to the 2DS was commenced at 0.08 flows.
- (p) On Nov. 1, the addition of 0.008 M NaNO₂ to the IWF was commenced.
- (q) On Nov. 1, the addition of 0.016 M HNO₃ to the 2DS was commenced.

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3. Feed Preparation

Failure of a thermohm on the A3 Dissolver necessitated shutdown of this dissolver for an approximately 30-hour period commencing on Oct. 31.

4. Solvent Extraction

Startup of the solvent-extraction system on Oct. 23 was marked by instability of the HS Column and the 2A Column. The HS Column instability was attributed to too high a start-up pulse frequency, with a resulting increased HSR flow causing a severe gamma burst from the HA Column. The 2A Column start-up, under a revised procedure specifying reduced rates and pulse frequency, was considerably more stable than previously. It is hypothesized that the instability noted may have been caused by temporarily too low an acid concentration in the 2AF.

The consequences of the HS Column instability included temporarily excessive product losses to the HAW and a severe gamma burst. Total recycle of uranium product to the HAF was required for about 40 hours. Process recovery was effected in about 3 days.

Decontamination and product recovery performance of the First Cycle, subsequent to the start-up problems, has been as good or better than prior to shutdown. The performance of both final cycles, however, has been substandard with respect to decontamination. The Final Uranium Cycle and Final Plutonium Cycle decontamination factors have averaged only about 450 and 600, respectively, as compared to 1200 for both cycles prior to shutdown. In addition, the Final Uranium Cycle product activity was temporarily increased by a factor of two as a result of a flood in the 2D Column on Nov. 1. The sensitivity of the column was demonstrated by the excessive time required (~ 16 hours) before it was back on stream and operating smoothly. The addition of ~ 0.015 M HNO_3 to the 2DS commencing on Nov. 1 appeared to have no beneficial effect on the 2D Column stability.

A trend for high uranium losses to the 2EW during the report period required 2EX temperature increases to as high as 56° C, compared to the usual 47° C. Process Chemistry studies of the 2EX and 2EW streams indicated no unusual conditions of uranium distribution ratio and no condition of uranium retention in the organic phase.

Rework of waste from TK-F8 from Oct. 28 through the balance of the report period was uneventful. An initial slight "tonic" effect may have resulted.

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5. Final Plutonium Concentration

Following plant startup on Oct. 23, transfers from the L-Cell Package to the Product Sample Tank L9 and from TK-L9 to the Plutonium Vacuum Tank L11 became very difficult due to high radiation fields in the PR Room. Tank L9 radiation readings of 50 Rads were encountered. After normal N-Cell operation had once again been established repeated flushes, about thirty in number, have failed to completely restore the equipment to normal activity levels.

As has been experienced before, several presumably good batches of N-Cell Pu product contained excessively high Zr-Nb activity after being transferred to the Product Sampler Tank L10. Apparently some cross-contamination from the contaminated PR Room equipment was occurring, although the path could not be detected.

6. Solvent Treatment

As a result of start-up difficulties described above (Note 5), the IOO gamma activity increased sharply to ~ 67,000 uc./gal., mostly Zr-Nb. Coincidentally the ICW gamma activity was 2.1×10^5 uc./gal., about 10 fold above normal. Following the general plant recovery, solvent activity levels have returned to normal. IOO Zr-Nb activity has averaged about 300 uc./gal., with Ru at 2000 to 3000 uc./gal.

7. Fission Product Recovery

(a) Cerium-Promethium

Promethium analyses were completed by HLO on the promethium crude shipped to the high level cells last month. Only 60,000 curies of promethium were found in the two casks. The material balances on this run (see page 192) were poor. However, from the available analyses, it appears most of the promethium was lost to the sulfate waste and oxalate waste. A high loss to the sulfate waste was expected but the promethium loss to the oxalate supernatant solution is not consistent with cerium losses. Based on "cold" chemistry studies, cerium should be more soluble than the rare earths. Special large volume samples have since been obtained from strontium run series 105 - 108 and submitted to HLO to define the path of promethium in the sulfate and oxalate steps. These results are not yet available.

(b) Strontium

Two series of strontium runs were completed during the report period. The composition and decontamination factors for the product fractions sent to storage were as follows:

[REDACTED]

	Runs 98 - 104			Runs 105 - 111		
	Curies/Gal	DF	Lb/Gal	Curies/Gal	DF	Lb/Gal
Sr 89 + 90	920			3800		
Zr/Nb 95	430	6		2000	4	
Ce 141 + 144	85	14		550	9	
Ru-Rh 103 - 106	10	80			75	
Fe			1.002			0.08
SO ₄			-			0.20
HNO ₃			1.35*			0.17
Sr 89/90 ratio	5.44					

*Believed to be a flyer.

The material balances for the two series of runs are given below:

	Runs 98 - 104(1)			Runs 105 - 11		
	Curies Sr	Percent of IWW Input Output		Curies Sr	Percent of IWW Input Output	
<u>Sulfate Precipitations</u>						
IWW	2,800,000	100		2,600,000	100	
Sulfate Waste	460,000		16	260,000		10
Flush	56,000		2	55,000(2)		2
Carbonate Product	1,700,000		61	1,900,000		73
Total	2,200,000	100	79	2,200,000	100	85
<u>Oxalate Precipitations</u>						
Carbonate Product	1,700,000	61		1,900,000	73	
Oxalate Waste	230,000		8	480,000		18
Flush	31,000		1	110,000		4
Oxalate Product	1,200,000		44	1,300,000		50
Total	1,500,000	61	53	1,900,000	73	72
<u>Concentration Precipitations</u>						
Oxalate Product	1,230,000	44		1,300,000	50	
Concentration Waste	83,000		3	46,000		2
Concentration Product	940,000		34	1,300,000		50
Total	1,000,000	44	37	1,300,000	50	52
Overall Total	1,800,000	100	64	2,250,000	100	86

(1) Numbers corrected for Sr 89 decay during suspension of strontium processing 10/6 - 10/24.

(2) Bad sample - this value is an estimate.

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Runs 98 - 104 were started on Oct. 3 and three sulfate runs had been completed when all building processing was suspended because of diversion box work. During the building outage, the El Wt.Fac. instrument failed and was not repaired for several days. When the instrument was returned to service, the El vessel was found to be full. The origin of the material that diluted TK-El remains unknown.

Strontium processing was resumed on Oct. 23 with disposal of sulfate run 101 to underground storage because of TK-El volume limitations. The dilute carbonate product in TK-El was then successfully processed through the oxalate and concentration steps.

Material balances on this series of runs were poor with no particular step showing an abnormal loss.

Runs 105 through 111 were started on Oct. 26 and completed on Nov. 2. Two variables were tested in this run series. The sulfate butt to the INW was reduced 20% to test the effect of sulfate concentration on strontium losses. Recent laboratory studies indicate maximum strontium yields are obtained at a sulfate concentration of 0.4 to 0.6 M vice the current flowsheet value of 1.0 M. Sulfate waste losses vary enough to prevent any conclusions on the basis of one series of runs. The sulfate concentration will continue to be reduced stepwise to 0.6 M unless a definite increase in waste losses is noted.

The other variable tested was oxalate precipitation temperature. This variable had been tested before (page 123) with promising results. On the current series of runs the oxalate precipitation was carried out at 30° C on run 109 and 90° C on run 110. Contrary to the previous test, the best yield was obtained at 90° C. This variable will continue to be tested.

Considerable difficulty was experienced in both run series with sulfate cake removal. Centrifuge background Beckman readings built up progressively during the sulfate runs with considerable heat noted remaining in the centrifuge following the last (fourth) sulfate cake removal. Caustic tartrate flushing of the centrifuge failed to remove this heat. However, during the subsequent oxalate runs the centrifuge Beckman background would return to normal and the heat would disappear. This behavior has been noted in the past with special oxalate flushes of the centrifuge failing to remove the heat producing cake in spite of the fact that the oxalate process solution usually is successful.

(c) Other items of interest to fission product recovery were:

- (1) The analyses of flushes between different processing steps has shown the new E3 agitator to be efficient in getting the precipitate transferred from the E3 vessel to the centrifuge.
- (2) A 10-gallon-per-minute Penberthy jet was located in spare parts and sent to HLO for performance tests. The jet performance equals or exceeds the performance curves supplied by the company. This jet will be used to "cold"-load the cerium cask and will then be returned to CPD for incorporation in a new jumper to replace the malfunctioning load-out jet (see page 170).

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 11/3/61 TO 2400 ON 11/13/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.8	3.9	-	-	0.004	0.03
Final	2.7	2.8	7.0	6.7	0.003	-
Ion Exchange	-	1.3	-	1.0	-	-
Overall	6.5	8.0	7.0	7.7	0.007	0.03

II. Production

A. HAF Processed: 245

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
9	3.7		7.1	P1
10	5.2	1.2	7.4	P1
11	5.4		7.4	P1
12	6.3	0.5	7.4	P1
13	6.0		7.4	P1
14	5.7	0.8	7.4	P1
15	5.9		7.4	P1
16	5.0	1.8	7.5	P1
17	5.3		7.5	P1
18	4.9	1.4	7.5	P1
19	4.5		7.4	P1
20	4.7	1.7	7.3	P1
21	5.2		7.4	P1
22	5.6	1.5	7.2	P1

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
23	5.3		7.5	P1
24	5.0	1.2	7.5	P1
25	5.6		7.7	P1
26	6.4	1.0	7.0	P1
27	6.0		7.5	P1
28	5.7	1.3	6.9	P1
29	7.0	0.8	6.9	E6
30	6.2	0.6	7.0	E6
31	5.4		7.0	P1
32	4.3	0.7	7.2	P1
33	3.7		7.5	P1
34	3.7	0.3	7.4	P1
35	3.1	0.7	7.3	P1
36	3.7	0.8	7.3	P1
37	3.8	2.0	7.5	P1
38	3.9	1.3	7.4	P1
39	4.0	1.0	7.4	P1
40	5.6	0.9	7.5	P1
41	5.7	0.8	6.0	P1
42	6.8	1.0	6.7	E6
43	6.8		6.0	E6
44	8.3	0.9	4.1	E6
44a	8.3	0.9	2.7	P1
45	7.9	6.	6.7	P1
46	8.0	0.9	6.3	P1
47	5.9		2.3	P1
47a	5.9		4.4	E6

2. Silica Gel

Gamma Ratios: Feed = 5.3
 Product = 1.9
 Tonnage = 185 Tons

C. Plutonium Product:

<u>Batch</u>	<u>Zr/Nb AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Mr/Hr</u>	<u>Shipped To</u>
5	2.3	0.004		5	5	234-5
6	2.4	0.006		5	5	234-5
7	2.4	0.006		5	5	234-5
8	3.7	0.004		5	6	234-5
9	3.2	0.002		5	7	234-5
10	2.7	0.003	450	4	5	234-5
11	3.1	0.002		5	6	234-5

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C. Plutonium Product:

<u>Batch</u>	<u>Zr/Nb AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr/Hr</u>	<u>Shipped To</u>
12	2.3	0.005		5	6	234-5
13	2.8	0.006		6	3	234-5
14	3.0	0.011		3	5	234-5
15	3.0	0.010		5	7	234-5
16	3.1	0.004		4	4	234-5
17	2.8	0.004		5	4	234-5
18	2.6	0.002		5	4	234-5
19	2.5	0.001		5	3	234-5
20	2.6	0.003	500	5	4	234-5
21	3.2	0.001		5	5	234-5
22	3.4	0.005		5	4	234-5
23	9.9	0.283		5	10	Holding

D. Overall Waste Loss - % of Total Production:

U = 0.11%
Pu = 0.17%

E. Rework - % of Total Production:

U = 14.4% (2UC TK-K6 to HAF Make-up TK-E6)
Pu = 21.0% (Predominantly out-of-spec. Pu product via the TK-L11 to HAF Make-up TK-E6)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	532	104
Cell Drainage	30	106
FP Waste	87	106
Coating Waste	270	107-2

B. IWW Flows: 3.9

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	5.4
102	6.2
103	0.2
104	5.2
106	11.2

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Notes:

1. The HAF rate was continued at a nominal 3.3 CF until shutdown on Nov. 13. The solvent extraction columns were shut down for approximately 12 hours on Nov. 7 and ten hours on Nov. 8 for replacement of the 3WF jumper and 2AF pump, respectively. On Nov. 9, approximately 14 tons of "cold" uranium was recycled as HAF. On Nov. 12 at 2000, the processing of "cold" UNH was commenced in preparation for scheduled shutdown. The HAF flow was stopped at 0700 on Nov. 13.

2. Flowsheet

- (a) On Nov. 3 the rework of waste from TK-F8, by addition to the HAF, was completed. On the same day the rework of IWW by addition to the HAF was commenced at an approximate rate of one volume of IWW to 30 volumes of HAF.
- (b) On Nov. 3, a rate check showed the HAF and HAX rates to be at a 2.94 CF. The flows were subsequently adjusted to 3.3 CF rates.
- (c) The 2DX rate was adjusted during the period from Nov. 5 through Nov. 7 to bring the 2DW loss down to around 5 per cent.
- (d) On Nov. 7, the 2EX temperature was increased from 50 to 52° C.

3. Solvent Extraction

Solvent extraction performance during the report period was characterized by:

- (a) Some improvement in both the First and the Final Uranium Cycle decontamination performance, as compared to the previous report period.
- (b) A general decrease in the First Cycle plutonium decontamination performance, coupled with an increase in the Final Plutonium Cycle performance.
- (c) Near-normal HAW losses, except during the periods of "upset".

The loss of First Cycle Pu decontamination performance resulted from start-up upsets following the outages described under Note 1, above. The bulk of the excessive activity followed the plutonium stream. The final Pu product activity was maintained under reasonable control, however, by a two-fold improvement in the 2A Column performance over the run period, which was accomplished at the expense of a two to

five-fold increase in the plutonium recycled in the 2AW. Even so, the recycle of some high-gamma Pu product was necessary.

Rework of TK-F8 waste and of LW were accomplished during the report period without incident.

The scheduled shutdown of the plant was commenced at 2000, Nov. 12, and completed on Nov. 13. Highlights of the shutdown operations were as follows:

- (a) About 12 tons of "cold" uranium were recycled to reduce the intercycle plutonium and gamma levels.
- (b) The HA-HS system was stripped and the organic displaced from the HS Column in preparation for replacement of the HAP jumper.
- (c) The ICU Concentrator E-J8 and ZDF Tank K1 contents were processed before shutting down the Final Uranium Cycle for neptunium recovery.
- (d) The 2D and 2E Columns were stripped and emptied in preparation for flushing.

4. Final Plutonium Concentration

Two N-Cell resin samples taken during the week of Nov. 6 contained an average of 700 uc/gal of Zr-Nb. This compares favorably with the 150 uc/gal during the first portion of the run period and 2000 uc/gal prior to the August shutdown. A visual inspection indicated that the physical quality of the resin was good and that only the normal amount of fines was present.

5. Solvent Treatment

The IOO gamma activity remained in the 3000 - 4000 uc/gal range throughout the report period. The ratio of Ru to Zr-Nb was about ten to one.

The IOO pump failed on Nov. 10. Batch changes of carbonate in the Turbomixer TK-G7 were effective for control until a replacement pump was started up two shifts later.

6. Waste Treatment and Acid Recovery

The desire to save all possible LW for fission product recovery resulted in a tendency to overconcentrate in the Waste Concentrator E-F11. The E-F11 specific gravity reached a value greater than 1.40 compared to the normal control value of 1.32. Frequent dip-tube

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plugging was experienced at the higher specific gravity. The IWW flows and concentrator specific gravity were adjusted to normal prior to plant shutdown.

A ten-fold decrease in the ruthenium content of the absorber acid (TK-F3) apparently resulted from the introduction of 0.008 M NaNO_2 to the IWF (TK-F7) on Nov. 1.

7. Demineralized Water

A sample of Purex demineralized water taken on Nov. 9 was tested by "C" Column operation at the 321 Building and was found to be of good quality, comparable to 321 Building steam condensate. This result is of particular significance in view of recent tendencies for 2E Column instability and inefficiency.

8. Fission Product Recovery Thru 11/20/61

(a) Strontium

One short series of strontium runs was completed during the report period. The decontamination factors for the product fraction sent to storage are as follows:

Run 113 Through 118

	<u>Curies/Gal</u>	<u>DF*</u>	<u>Lb/Gal</u>
Sr 89 + 90	1380		
Zr-Nb 95	45	31	
Ce 141 + 144	7	110	
Ru 103 - Rh 106	1/2	1210	
Fe ⁺⁺			1.002
SO ₄ ⁼			1.0
HNO ₃			.29
Sr 89/90 Ratio	8.7		

*It is believed the high DF numbers are in error because of analytical difficulties. The DF's from Ce and ZrNb through the oxalate step were 8 and 4, respectively. Usually these DF's are slightly higher than final product DF's because of cross-contamination during the concentration run.

The material balance for this series of runs are given below:

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Runs 113 - 118

	<u>Curies</u> <u>Sr</u>	<u>Per Cent IWW</u> <u>Input</u>	<u>Output</u>
<u>Sulfate Precipitations</u>			
Raw IWW	1,430,000	100	
Sulfate Waste	104,000		7
Flush	36,500		3
Carbonate Prod.	318,000		22
Total	458,500	100	32
<u>Oxalate Precipitations</u>			
Carbonate Product	318,000	22	
Oxalate Waste	54,200		4
Flush	313,000		22
Oxalate Product	460,000		32
Total	827,200	22	58
<u>Concentration Precipitations</u>			
Oxalate Product	460,000	32	
Concentration Waste	75,600		5
Concentration Product	518,000		36
Total	593,600	32	41
Overall Total	1,430,000	100	79

Again, strontium material balances were poor. Equipment difficulties undoubtedly contributed to the poor material balances.

The sulfate concentration of the precipitation solution was again reduced by 20% from the previous runs to 1.2 M sodium sulfate - 1.17 M tartaric acid. With such poor material balances across the sulfate precipitation, it is impossible to determine the effect of lower sulfate concentration.

The temperature of the sulfate solution during centrifugation was raised from 60 to 65° C. Also in an effort to reduce sulfate waste losses. Again poor material balances made it impossible to predict the results of this process change. Evaluation of lower sulfate concentration and higher centrifugation temperature will be continued.

Sulfate runs 115 and 116 were deleted from this series of runs due to malfunction of the cake temperature thermohm, the centrifuge diverter valve, and the centrifuge skimmers. The diverter valve and the thermohm were replaced, and the skimmers were made to partially operate by use of an extended handle on the hydraulic skimmer bar.

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(b) Promethium

Promethium analyses on the large volume samples obtained from strontium runs 105 - 111 (see page 203) were completed by HLO. These analyses show only 35 per cent of the promethium is recovered in the sulfate step and essentially all of the promethium entering the oxalate step follows the cerium into the oxalate waste (peroxyacetate feed). These analyses are consistent with a bulk sample of the feed to the peroxyacetate step taken during the first promethium test run. The high promethium loss to the sulfate waste thus explains the poor overall promethium recovery in the two test runs.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 11/14/61 TO 2400 ON 12/1/61

During the report period, the plant was shut down with respect to normal uranium processing. A scheduled neptunium run was conducted and other shutdown processing and maintenance operations were performed. Process notes of interest are presented below.

1. Solvent Extraction

Shutdown items of interest included:

- (a) The 2D-2E Columns were flushed with hot 5% caustic because of their continued poor operating characteristics during the preceding run period.
- (b) The location of the 2A Column interface float was changed from the side chamber at the bottom of the column to the bottom beavertail. It is expected that the column operability will be improved by more rapid interface response.
- (c) On Nov. 30 and Dec. 1, operability testing of the Final Plutonium Cycle was conducted primarily to test flooding characteristics of the 2A Column, and secondarily to check the operating characteristics of the new interface float. Using synthetic 2AF (HNO_3 and water), tests were conducted as follows:
 - (1) At 3.0 CF and normal 2AS rate (1.85 flows), the 2A Column flooded at a pulse frequency of 100 cycles/min.
 - (2) At 3.0 CF and twice normal 2AS rate, the 2A Column flooded at 102 cycles/min.
 - (3) At 4.0 CF and normal 2AS rate, the 2A Column was operated satisfactorily at 75 cycles/min. Time did not permit determination of flooding frequency at this capacity factor.

Results of the flooding runs at 3.0 CF are tabulated below:

<u>CF</u>	<u>Volume Velocity, GPH/Ft²</u>		<u>2AS Rate, Flows</u>	<u>Flooding Frequency, Cycles/Min.</u>
	<u>Scrub</u>	<u>Ext'n.</u>		
3.0	200	1040	1.85	95
3.0	258	1150	3.7	100

A comparison of this data to HLO semiworks data indicates that flooding occurred in the extraction section. This is verified by the fact the flooding frequency actually showed an increase by a flow increase which, percentage-wise, was only significant in the scrub section.

2. Plutonium Concentration

(a) Pu Rework Operation

During the period Nov. 20 to Nov. 22, nearly 23 kg. of concentrated rework plutonium was charged to the N Cell ion exchange facility for rework processing. Of this total, about 18 kg. was processed during that period; processing of the balance was delayed until just prior to plant start-up. The 18 kg. total included about 3 kg. from Finished Products Operation. All of this material was processed without valence adjustment of the feed. According to laboratory analyses the Pu from Finished Products Operation was about 6.5% Pu(VI), with the balance Pu(IV).

Rework operation results were good with respect to recovery, but the product gamma content was excessive. In fact the gamma content of the product was up to about two fold higher than the feed. Cross-contamination between PR Room product vessels is the probable cause for this apparent discrepancy. Prior to the rework operation the ion exchange resin had been completely changed. According to previous experience, the rework flowsheet (without XAF fluoride) should provide adequate DF.

The FPO Pu was processed as a single batch which was sampled for solids content in TK-L9 after make up with HNO₃ and water to the rework flowsheet composition. Analyses showed approximately 1 wt.-% solids (weight of solution basis), which is approximately equivalent to 150 grams of solids per kg. of FPO rework processed.

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(b) Resin Change

During the shutdown period the ion exchange resin was changed. The replacement change was the same make-up as previously, viz., 75% 20 - 40 mesh and 25% 10 - 20 mesh.

3. Neptunium Recovery Operation

(a) Summary

A neptunium recovery run was started at 1000 on Nov. 14 and was completed at 1245 on Nov. 19. After product concentration 1.59 kg. of neptunium were loaded out, giving 95% recovery based on the 3WB processed.

Compared to "normal", the run was very good. Product losses were low and the run time was short. Compared to the August run (p. 169), gamma activity in the product was higher due to more activity in the 3WB feed.

(b) Material Balance*

	<u>Kg.</u>
Np in Plant	1.78
Np in 3WB	1.74
Np Processed as 3WB	1.67
Np Load-out	1.59
Np Recycle	0.19
Np Lost	0.00

(c) Typical Solvent-Extraction Losses*

<u>Stream</u>	<u>% of 3WB Processed</u>
2AW	0.4
2BW	1.5

(d) Product Analytical Results

	<u>Np</u>	<u>UO₃</u>	<u>% Contamination of Np</u>		<u>G/Np</u>
	<u>g/l</u>	<u>M</u>	<u>Pu</u>	<u>U</u>	<u>ATx10⁷</u>
Principal Load-out	37.0	11.1	1.1	2.7	12
First Flush	16.4	11.2	2.1	5.7	1.1
Second Flush	1.5	11.8	12	7.2	-
Third Flush	1.1	12.3	13	27	-
Fourth Flush	0.7	12.3	49	17	180

*Losses recycled for recovery during normal processing operation.

4. Solvent Treatment

During the shutdown period, flushes of the solvent treatment systems were conducted as follows:

- (a) The No. 2 Solvent Treatment System, except for the 200 Tanks R5 and R7, were flushed with oxalic-nitric and caustic-tartrate. Purpose of the flushes was to determine the radiation levels that would exist during the course of a proposed 20 Column change.
- (b) The G1 Tank (No. 1 Solvent Treatment System) was flushed with oxalic-nitric to reduce the MnO_2 hold-up. Subsequent calibration checks indicated negligible hold-up.

A preliminary comparison has been made of the use of Soltrol-170 as diluent vs. the use of Shell E-2342 as diluent, in terms of the effects on process performance. The September-October-November, 1961, run period, using Soltrol, was compared to the May-June, 1961, and to the July-August, 1961, run periods, using Shell E-2342. The comparison indicated general process performance using Soltrol was as good as the best Shell performance of 1961 (May-June run period). Solvent losses using Soltrol were at least 5% less than for the best period using Shell.

5. Waste Treatment and Acid Recovery

Attempts were made to restore the operability of the IWW Concentrator (F11) jet-out system, which has not been operable since at least Oct. 10.

- (a) Water flushes were made via the instrument dip tubes.
- (b) The F11 bottoms were diluted out with water and the diluted material was simmered while adding water via the tower and via the instrument dip tubes.
- (c) The F11 vessel was flushed twice with hot, dilute caustic.

During and following these treatments, emptying of the concentrator using the installed jet was attempted without success. Use of a temporarily installed portable jet during flushing operations was successful.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM COCO ON 12/2/61 TO 2400 ON 12/10/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.8	4.0	-	-	0.004	0.07
Final	2.6	3.1	11.0	4.9	0.007	-
Ion Exchange	-	1.1	-	1.0	-	-
Overall	6.4	8.2	11.0	5.8	0.011	0.07

II. Production

A. HAF Processed: 187 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
1	8.1	2.5	1.0	P1
1A	8.1	2.5	4.0	E6
2	5.8	2.1	4.8	P1
3	8.3		6.3	P1
4	8.5	6.5	7.2	P1
5	5.6		7.2	P1
6	3.6	2.4	6.3	P1
7	2.9		7.8	P1
8	2.4	1.0	6.9	P1
9	3.0		7.6	P1
10	2.7	0.5	7.5	P1
11	3.0		7.5	P1
12	2.9		6.9	P1
13	3.2		7.3	P1
14	3.6	0.2	7.4	P1

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B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
15	4.0		7.3	Pl
16	4.2	0.3	7.2	Pl
17	5.1		7.3	Pl
18	5.1	0.5	7.0	Pl
19	5.2		7.1	Pl
20	5.3	0.6	7.2	Pl
21	5.6		7.2	Pl
22	4.5	0.6	7.5	Pl
23	4.2		7.1	Pl
24	7.0	4.1	7.2	Pl
25	5.9	5.4	7.4	Pl
26	4.5	3.6	7.5	Pl
27	2.9	2.3	6.8	Pl

2. Silica Gel

Gamma Ratios: Feed = 4.7
 Product = 1.7
 Tonnage = 77 Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 1011</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Shipped Mr./Hr.</u>	<u>To</u>
24	7.3	0.057		5	8	Holding
25	4.2	0.100		4	6	234-5
1	3.8	0.050		4	4	234-5
2	3.1	0.012		5	3	234-5
3	1.7	0.028		5	4	234-5
4	1.3	0.035		5	2	234-5
5	1.7	0.003	7860	5	3	234-5
6	2.1	0.004		4	3	234-5
7	2.0	0.004		5	4	234-5
8	2.0	0.004		5	4	234-5
9	1.8	0.004		5	4	234-5
10	1.0	0.001	2590	5	4	234-5
11	0.7	0.003		4	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.26%
 Pu = 0.37%

E. Rework - % of Total Production:

U = 14.5% (Predominantly out-of-spec. "U" product from the P3 to 2DF TK-K1, through Final U Cycle, only)

Pu = 12.3% (Predominantly out-of-spec. "Pu" product, high sumps, and misc. flush material via TK-L11 to HAF Make-up TK-E6)

III. Waste TreatmentA. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	1209	104 & 106
Cell Drainage	267	106
FP Waste	208	106
Coating Waste	270	107C

B. INW Flows: 3.1C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	3.8
102	3.6
103	0.2
104	5.1
106	10.0

Notes:

1. Due to HA Column operational difficulties, plant start-up at 3.0 CF was attempted several times, as follows:

(a) Initial "cold" HAF flow commenced Dec. 2 at 1615. Flow was stopped at 1915.

(b) "Cold" HAF flow was again commenced Dec. 2 at 2250. Flow was stopped at 0215 on Dec. 3.

(c) "Cold" HAF flow was again commenced on Dec. 3 at 0415, but was stopped again at 0445.

(d) At 0800 on Dec. 3, "cold" HAF flow was again commenced. At 0100 on Dec. 4, processing of "hot" HAF was commenced.

At 1715 on Dec. 4, the HAF rate was increased to 3.3 CF.

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2. Flowsheet

- (a) The plant was started up at a nominal 3.0 CF on the same flow-sheet that existed at the end of the last operating period, except for the following:
 - (1) The ZDF-ferrous sulfamate flow was started at 3.3 CF.
 - (2) The ZBP was initially diverted to the L Cell Package.
 - (3) The ZEX temperature was 50° C.
 - (4) The Final Uranium Cycle was started up on recycled "cold" uranium.
 - (5) The First Decontamination Cycle was started up on "cold" dilute (~ 40% flowsheet uranium concentration) feed (HAF) at 59 flows.
- (b) On Dec. 3, the HAF was increased from 59 to 75 flows.
- (c) On Dec. 4, "hot" HAF at flowsheet uranium concentration was started at 75 flows.
- (d) On Dec. 4, the ZEX temperature was increased from 50 to 55° C.
- (e) On Dec. 3, the IBXP-HNO₃ was increased from 6.2 to 7.3 flows. It was further increased to 8.0 flows on Dec. 4.
- (f) On Dec. 4, the HSS-HNO₃ was increased from 13.9 to 14.7 flows.
- (g) On Dec. 5, the ZDF-HNO₃ was reduced from 9.3 to 7.3 flows.
- (h) On Dec. 6, the ZDF-ferrous sulfamate was reduced from 0.19 to 0.13 flows.
- (i) On Dec. 6, the HSS-HNO₃ was increased from 14.7 to 15.3 flows. It was further increased to 16 flows on Dec. 7.
- (j) On Dec. 7, the IBXP-HNO₃ was increased from 8.0 to 8.4 flows.
- (k) Commencing on Dec. 7, the N Cell wastes were routed to the JWF Tank F10 rather than to the 3MB Receiver TR-J1.
- (l) On Dec. 8, a change from the processing of normally irradiated uranium to processing of non-representative metal was made. At the time of commencing dissolution of non-representative uranium, the addition of 0.005 M Hg(NO₃)₂ to the metal solution was stopped. (See Note 3, below.)

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- (m) During the period Dec. 8 through Dec. 10, the 2EX temperature was gradually decreased from 55° C to 46° C.

3. Feed Preparation

Normal uranium dissolved for plant start-up and for the initial portion of the report period averaged approximately 684 MWD/T, with an average "cooling" time of 120 days. A switch to the processing of a special run of non-representative uranium, commencing on Dec. 8 in the solvent-extraction system, was preceded by complete cutting of normal metal heels. Close dissolver scheduling permitted the change to be made without processing interruption and with cross-contamination between metal types in the Feed Preparation section limited to vessel jet heels.

The non-representative uranium averaged about 860 MWD/T, 207 day "cooled". Because of this long "cooling" time, the use of mercuric nitrate for metal solution iodine suppression was stopped with the change to non-representative uranium processing.

The I-131 emission for the report period was 0.69 curies.

4. Solvent Extraction

At the beginning of the report period, start-up of the solvent-extraction system was finally accomplished after considerable difficulty with apparent HA Column flooding. During this time "cold" uranium, considerably diluted compared to the normal flowsheet value, was being processed, and the HSR stream appeared to be at least partly returning with the HAP to the bottom of the HS Column. After several false start-ups during the period Dec. 2 to Dec. 4, the condition was corrected by the introduction of "hot" HAP. Meanwhile slight instability was also experienced in the 2A Column as a result of erratic operation of the 2AF control valve.

Following start-up, process performance with respect to waste losses and fission-product decontamination was essentially normal. With the change to the processing of non-representative uranium on Dec. 8, gamma levels, in general, dropped two to three fold.

The change to non-representative uranium processing was made without process interruption. No attempt was made to make a sharp Pu segregation. However, the HAP Tank H1 level was reduced to a minimum --- about 300 - 400 gallons --- at the time of change. The process streams were then purged with the non-representative material for about 16 hours, at which time the ion exchange concentrator and receiver, E-N6 and TK-N6, were emptied in order to accomplish some degree of segregation.

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Final uranium cycle performance continued to be unsatisfactory during the report period. On Dec. 9, the 2D Column suddenly flooded with no warning. The flooding pattern was typical --- a rapid sharp decrease in extraction-section differential pressure accompanied by an equally rapid sharp increase in the scrub-section differential pressure. Operation was restored after a rapid pulse frequency reduction of 10 cycles/min.

5. Solvent Treatment

Processing of non-representative uranium was accompanied by a greater than two-fold decrease in ICW gamma activity, to about 9000 uc./gal. Similarly, the IOO ZrNb activity decreased from about 400 uc./gal. to about 250 uc./gal.

6. Plutonium Concentration

On Dec. 7, indications of plugging of the XAW line to the 3WB Receiver Tank J1 were observed, and the XAW flow was diverted to the 3WF TK-F10. On Dec. 8 attempts were made to flush the line to TK-J1 using XCX from the spare scrub pump. Low pump capacity limited the effectiveness of this flush, and flow to TK-J1 was not successfully re-established.

7. Fission Products

(a) Purex Strontium Recovery

One series of strontium runs was completed during the report period. The decontamination factors for the product fraction sent to 244 CR-003 Tank are as follows:

Run 119 Through 125

	<u>Curies/Gal.</u>	<u>DF</u>	<u>Lb./Gal.</u>
Sr ⁸⁹ + 90	1695		
Zr-Nb ⁹⁵		5	
Ca ¹⁴¹ + ¹⁴⁴		5	
Ru ¹⁰³ -Rh ¹⁰⁶		1200	
Fe ⁺⁺			.04
SO ₄ ⁼			/.2
HNO ₃			.118
Sr ⁸⁹ /Sr ⁹⁰ - ~ 8*			

*The Sr⁸⁹/Sr⁹⁰ was reported as 9.3 and 6.8 on 11/29. Since the feed material of this series of runs was similar to the previous series which had a reported ratio of 8.7 on 11/21, a ratio of ~ 8 is assumed.

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The material balance for this series of runs are given below:

Runs 119 - 125

	Curies Sr	Per Cent IWW	
		Input	Output
<u>Sulfate Precipitation</u>			
Raw IWW	2,240,000	100	
Sulfate Waste	177,000		8
Flush	94,500		4
Carbonate Product	615,000		27
	<u>886,500</u>	<u>100</u>	<u>39</u>
<u>Oxalate Precipitation</u>			
Carbonate Product	615,000	27	
Oxalate Waste	156,000		7
Flush	208,000		9
Oxalate Product	740,000		33
	<u>1,104,000</u>	<u>27</u>	<u>49</u>
<u>Concentration Precipitation</u>			
Oxalate Product	740,000	33	
Concentration Waste	76,000		3
Concentration Product	615,000		27
	<u>681,000</u>	<u>33</u>	<u>30</u>
Overall Totals		100	58

Poor strontium material balances continue; however, the cerium material balances across this series of runs was fair. A reasonable cerium material balance coupled with a poor strontium material balance indicates either analytical difficulties or strontium is being preferentially lost in the metathesis waste. The metathesis waste (1 M Na_2CO_3 - 5 M NaOH) is not routinely sampled because of the expense of acidifying each batch before a solution sample can be obtained.

No further reduction in sulfate concentration was made; however, advantage was taken of the previous reduction to reduce the volume of the butt solution from 900 gallons per batch to 450 gallons per batch by increasing the sulfate concentration of the butt solution. The butt solution is now 2 M Na_2SO_4 and 2.3 M tartaric acid.

(b) Hot Semiworks

The quantity of strontium stored at the Hot Semiworks has not been accurately established. The uncertainty of the quantity stored is caused by the large discrepancy between strontium values obtained by analytical and heat balance techniques. The data accumulated as of December 5 are as follows:

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<u>Vessel</u>	<u>Date</u>	KC Sr ⁹⁰ <u>By Heat Balance</u>	KC Sr ⁹⁰ <u>By Analysis</u>	<u>89/90 Ratio on Nov. 28</u>
TK-1	11-28	395*		0.23
Product	11-30		142	
	12-1		136	
	12-5	214		
TK-6	11-9		173 (ppt in sample)	0.74
Product	11-21	245		
	11-28	213		
	12-5	236		
TK-54	10-13		160	0.74
Rework	11-9		193	
	11-21	200		
	11-28	131		
	12-5	130		
TK-64	11-30		141	0.23
Product	12-1		135	
	12-5	205		

*Contained TK-64 solution at this time.

The large discrepancy between laboratory analyses and heat measurements on TK-64 and TK-1 for Sr-90 has not been explained. Sr-90 values of TK-1 and 64 obtained by heat measurements still exceed laboratory values by about 20% even when no corrections are applied for heating the vessel or for heat losses to the cell air (the specific heat of the solution used for the calculations was 0.90 - 0.95 BTU/# of solution/°F).

The volume of solution in TK-6 has been decreasing since the storage of strontium in this vessel began in early October. The strontium was initially stored in a media which was about 2.0 M citric acid. It is known that citric acid of this concentration is responsible for about 30% of the volume, and some reduction in TK-6 volume is expected from radiolysis of the citric acid. However, since water is one of the decomposition products, it is not possible to estimate the volume reduction from the above data. Strontium analyses of sump samples do not show TK-6 is leaking, but the volume reduction is small enough that puddling and evaporation on A Cell floor would probably prevent solution from reaching the sump. The rate of volume decrease gradually lessened and appeared to have stopped in mid-November.

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On November 16 TK-6 was made 0.5 M HNO_3 to eliminate the precipitate which had formed. Following the acid addition the rate of TK-6 volume decrease returned to about one liter/day. As of Dec. 1, the total volume lost from TK-6 is about 75 liters (~ 10% of the original volume).

Radiation Beckmans in the vicinity of TK's 6 and 64 showed variations when starting and stopping agitators during November. These observations indicated precipitates in these tanks. The presence of precipitate in TK-6 was confirmed by sampling on November 9. The precipitates in both tanks were redissolved by the addition of nitric acid (final solution 0.5 - 1.0 M HNO_3).

Laboratory tests show that SrCO_3 cannot be satisfactorily precipitated from a citrate solution for filtration in the shipping cask. A satisfactory precipitate, however, was achieved when the citrate was destroyed by radiolysis. A total irradiation dose of 2×10^9 R was required to completely destroy the citrate in a 2.0 M citric acid solution. This amounts to absorption of about 5200 watt-hours of beta energy per liter. This irradiation dose has been exceeded by product stored in TK's 64 and 1 and will be reached by TK-6 in mid-December.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 12/11/61 TO 2400 ON 12/21/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	4.0	4.1	-	-	0.002	0.05
Final	2.4	3.2	11.0	4.7	0.003	-
Ion Exchange	-	1.3	-	0.3	-	-
Overall	6.4	8.5	11.0	5.0	0.005	0.05

II. Production

A. NAF Processed: 290 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
28	3.6	1.8	7.5	P1
29	2.1		7.4	P1
30	2.9		7.2	P1
31	2.8		6.8	P1
32	2.7	1.7	7.3	P1
33	3.0		7.4	P1
34	2.9	2.1	7.2	P1
35	2.9		7.1	P1
36	2.8	1.9	7.6	P1
37	5.1	5.7	4.6	P1
38	8.2	16.5	7.2	P1
39	8.4	20.4	7.4	P3
40	6.8	16.8	7.3	P3
41	6.0	12.0	7.1	P3
42	6.0	12.0	7.4	P1
43	5.2	12.0	7.4	P3

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
44	4.4	9.0	7.0	P1
45	4.4	5.3	7.1	P1
46	3.8	5.3	5.6	P1
47	4.1	4.8	7.2	P1
48	4.6	4.9	7.0	P1
49	4.8	6.0	7.3	P1
50	5.0	4.6	7.4	P1
51	4.0	4.9	6.9	P1
52	3.7	4.5	6.9	P1
53	3.9	2.6	7.1	P1
54	4.3	4.3	7.0	P1
55	3.7		7.2	P1
56	3.8	4.6	7.1	P1
57	3.6	4.9	7.7	P1
58	3.6	3.5	7.2	P1
59	4.0		7.0	P1
60	4.3	4.3	7.1	P1
61	4.3	3.8	7.5	P1
62	4.2	4.2	6.7	P1
63	4.0		7.2	P1
64	4.1	3.4	6.7	P1
65	4.4		7.8	P1
66	4.6	3.0	7.1	P1
67	4.5		6.8	P1
68	4.6	2.0	6.8	P1

2. Silica Gel Gamma Ratios

Feed = 4.0
 Product = 1.5
 Tonnage = 23¹/₄ Tons

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Shipped Mr./Hr.</u>	<u>To</u>
12	0.9	0.001		5	3	234-5
13	0.7	0.008		5	3	234-5
14	0.6	0.010		4	2	234-5
15	0.7	0.006	1330	5	2	234-5
16	0.7	0.002		5	1	234-5
17	0.7	0.004		5	2	234-5
18	0.8	0.004		6	1	234-5
19	0.7	0.001		5	3	234-5

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C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Shipped Mr./Hr.</u>	<u>To</u>
20	0.7	0.003		5	1	234-5
21	0.6	0.004		5	2	234-5
22	0.8	0.007		5	2	234-5
23	0.9	0.004		5	1	234-5
24	0.8	0.004		5	2	234-5
25	0.8	0.002		5	2	234-5
26	0.8	0.008		5	2	234-5
27	0.8	0.014		5	2	234-5
28	0.8	0.023		5	2	234-5
29	0.6	0.023		4	2	234-5
30	0.8	0.036	890	5	1	234-5
31	0.8	0.025		5	2	234-5
32	0.9	0.030		4	2	234-5
33	1.0	0.032		5	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.06%

Pu = 0.12%

E. Rework - % of Total Production:

U = 5.4% (Out-of-spec "U" product from U Storage TK-P3 to 2DF TK-K1 through final U Cycle only)

Pu = 0.9% (Misc. sump material via TK-L11 to HAF Make-up TK-E6)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	469	104 & 106
Cell Drainage	8	106
FP Waste	99	106
Coating Waste	270	107C

B. IWW Flows: 5.4 Flows

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	5.2
102	4.2
103	0.1
104	5.6
106	12.0

Notes:

1. The HAF rate was maintained at a nominal 3.3 CF throughout the report period. The actual rate averaged about 3.2 CF.

2. Flowsheet

- (a) On Dec. 12, rework of IWW was commenced at a rate of 250 gallons per 4000-gallon batch of HAF.
- (b) On Dec. 12, the HCS-nitrite flow was reduced from 0.71 to 0.63 flows. It was further reduced to 0.60 flows on Dec. 13.
- (c) On Dec. 15, the ZDF-ferrous sulfamate was increased from 0.28 to 0.37 flows.
- (d) On Dec. 15, the ZEX temperature was increased to 46° C.
- (e) On Dec. 17, the rework of IWW via the HAF was terminated.
- (f) On Dec. 19, the ZEX-HNO₃ was increased from 0.18 to 0.36 flows. It was decreased to 0.09 flows on Dec. 21.
- (g) On Dec. 19, the ZDS was increased from 58 to 67 flows.
- (h) On Dec. 20, the ZDF-HNO₃ was decreased from 7.3 to 5.9 flows.
- (i) On Dec. 20, a change from the processing of HAF from non-representative uranium dissolution to the processing of HAF from normally irradiated uranium dissolution was made.

3. Feed Preparation

Following the processing of a special run (about 300 tons) of non-representative uranium, a return to the dissolution of normal uranium was commenced on Dec. 19. Similarly to the original change to non-representative metal (see p. 222), the change was made with a minimum of cross-contamination between the metal types.

The iodine emission from the non-representative uranium (average 860 MWD/T, 207 day "cooled") was 0.39 curies.

4. Solvent Extraction

Solvent-extraction performance continued without significant change. The principal problem area continued to be the Final Uranium Cycle where performance was substandard with respect to decontamination and with respect to uranium losses to the 2EW. In the case of the

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2D Column, efficiency was not significantly improved by 2DS flow increases nor by 2DE acidity reduction (Note 2 (g) and 2 (h), above). In the case of the 2E Column, 2EX temperature and flow increases were only partially effective in controlling 2EW uranium losses.

On Dec. 13 the 2D Column suddenly flooded following a one cycle/minute pulse frequency increase. Several hours were required to break the flood, and about 45 tons of product was produced which was out-of-specification with respect to Pu. On Dec. 14, slight 2D Column instability was corrected by rapidly reducing the pulse frequency five cycles/minute.

Laboratory investigation of possible causes of the Final Uranium Cycle performance problem has thus far revealed no abnormal conditions.

The excessive 2EX rate required to curb 2EW uranium losses has resulted in an excessive concentrating load in the 2EU Concentrator K4. Occasionally, the concentrator has pressurized; inspection from the crane shows that UMH has leaked out around a tube bundle flange.

Rework of IWW from Dec. 12 to Dec. 17 was completed with no noticeable process effects.

The change from non-representative uranium processing to normal uranium processing was made on Dec. 20 without process interruption and without process effect. The method of accomplishing the change was the same as described on page 222.

5. Solvent Treatment

A process test designed principally to reduce solvent losses in the No. 2 Solvent Treatment System and to permit eventual reduction in the number of solvent treatment processing steps was commenced on Dec. 12. According to the conditions of this test, TK-R1 was used as a batch carbonate contactor, similar to TK-G1 in the No. 1 System except no KMnO₄ was used. Carbonate changes from TK-R1 were routed to TK-G1 for re-use, and to avoid solvent losses from R Cell. An ultimate change to use of acid in the 2D Column, and elimination of the R Cell centrifuges was planned, if the tests were successful. Unfortunately, the carbonate changes from TK-R1 to TK-G1 were accompanied by transfers of unusual volumes of solvent. The R-Cell inventory was depleted too rapidly, and the No. 1 Solvent Treatment System was somewhat upset. The test was discontinued pending investigation of this problem.

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6. Fission Product Recovery - Strontium Recovery

Two series of strontium runs were completed during the report period. The decontamination factors for the product fraction sent to 244 CR 003 Tank are as follows:

	<u>Runs 126 Through 132</u>			<u>Runs 133 Through 139</u>		
	<u>Curies/Gal</u>	<u>DF</u>	<u>Lb/Gal</u>	<u>Curies/Gal</u>	<u>DF</u>	<u>Lb/Gal</u>
Sr 89+90	1100			878		
ZrNb95	843	3.7		802	4.2	
Ce 141+144	64	23.7		242	7.0	
Ru103-Rh106	1	>940		1	>590	
Fe++						
SO ₄ ²⁻			1.2			.107
HNO ₃			pH 5.6			1.2
Sr89/Sr90		7.3 on 12/15/61			5.7 on 12/18/61	.665

The material balances for these two series of runs are given below:

	<u>Runs 126 Through 132</u>			<u>Runs 133 Through 139</u>		
	<u>Curies Sr</u>	<u>Percent Input</u>	<u>IWW Output</u>	<u>Curies Sr</u>	<u>Percent Input</u>	<u>IWW Output</u>
<u>Sulfate Precipitation</u>						
Raw IWW	1,646,000	100		1,450,000	100	
Sulfate Waste	265,000		16	127,200		9
Carbonate Product	1,085,000		66	1,068,000		74
	1,350,000	100	82	1,195,200	100	83
<u>Oxalate Precipitation</u>						
Carbonate Product	1,085,000	66		1,068,000	74	
Oxalate Waste	185,200		11	119,300		8
Flush	26,300		2	129,000		9
Oxalate Product	1,060,000		64	520,000		36
	1,271,500	66	77	768,300	74	53
<u>Concentration Precipitation</u>						
Oxalate Product	1,060,000	64		520,000	36	
Concentration Waste	52,000		3	47,300		3
Concentration Product	475,000		29	609,000		42
	527,000	64	32	1,176,300	36	45
<u>Overall Totals</u>		100	61		100	71

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Poor strontium material balances are still prevalent. No sample of an acidified carbonate waste has been obtained, but will be accomplished on the next series of runs.

The caustic-tartaric flush of the tanks after the carbonate metathesis was deleted from both series of runs. The following oxalate waste appeared to increase from 3 or 4 per cent to 6 or 7 per cent. However, this deletion will permit increasing the number of sulfate runs per series from four to five.

The E4 Centrifuge failed on Dec. 4 during the fourth sulfate precipitation. A rebuilt centrifuge was placed in the E2 position and strontium production was resumed. With the rebuilt centrifuge in the E2 position the fourth sulfate run and the oxalate and concentration steps were completed on the first series of runs and the second series were completed with two procedural changes. The first batch of raw DW was centrifuged prior to sulfate precipitation. The solid material removed during this centrifugation appeared to be easily removed. The dissolved sulfate cake solution was removed to E1 Tank with slight difficulty. The three following sulfate cake solutions without prior removal of solids from the DW were removed with great difficulty. The E2 Beckman readings became high and the centrifuge began making a loud knock at high speeds. As a result the E3 Agitator was left motionless during the centrifugation of the oxalate run to keep the precipitated cerium in the E3 Tank. Oxalate waste analysis increased slightly, but further testing is required to completely evaluate this process change.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 12/22/61 TO 2400 ON 12/31/61

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	3.8	3.5	-	-	0.004	0.02
Final	2.6	3.3	8.0	6.8	0.005	-
Ion Exchange	-	1.5	-	0.3	-	-
Overall	6.4	8.3	8.0	7.1	0.009	0.02

II. Production

A. HAF Processed: 263 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
69	5.0		7.1	P1
70	5.8	3.3	7.2	P1
71	6.0	5.3	6.8	P1
72	6.6	4.3	7.0	P1
73	6.2	-	6.8	P1
74	5.9	3.3	6.7	P1
75	5.8	2.6	6.8	P1
76	6.0	4.0	7.3	P1
77	6.3	4.0	7.0	P1
78	6.1	3.9	6.9	P1
79	6.6	4.6	7.2	P1
80a	5.8	4.1	7.1	P1
80b	5.8	4.1	0.3	P3
81	5.8	3.1	6.6	P1
82	5.1	3.1	7.0	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
83a	6.2	3.8	5.5	P1
83b	6.2	3.8	1.7	P3
84	4.9	2.8	6.7	P1
85	4.2	2.2	6.3	P1
86	4.2	2.9	7.1	P1
87	3.9	4.1	7.2	P1
88	3.7	4.0	6.3	P1
89	4.0	-	7.1	P1
90	4.5	5.0	6.7	P1
91	8.3	19.0	7.1	P1
92	7.7	16.1	7.3	P3
93	7.2	12.8	7.4	P3
94	6.4	6.9	7.1	P1
95	6.1	8.0	7.3	P1
96	6.8	9.5	6.8	P1
97	6.3	11.6	7.3	P1
98	7.2	6.4	7.2	P1
99	6.3	5.9	7.2	P1
100	7.2	4.9	7.0	P1
101	6.9	4.0	6.3	E6
102	7.2	3.9	6.1	E6
103	8.0	4.3	6.7	E6
104a	8.9	5.6	4.0	E6
104b	8.9	5.6	3.6	P1
105	8.0	5.0	7.5	P1
106	8.1	4.1	4.0	P1

2. Silica Gel

Gamma Ratios: Feed = 5.7
 Product = 1.7
 Tonnage = ~ 135

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./Mr.</u>	<u>Shipped To</u>
34	1.2	0.017	897	5	0	234-5
35	1.4	0.034		5	1	234-5
36	0.6	0.112		5	2	234-5
37	2.5	0.059		4	3	234-5
38	1.9	0.022	1420	5	3	234-5
39	3.2	0.022		5	3	234-5
40	1.9	0.033		5	2	234-5
41	1.0	0.017		5	2	234-5

C. Plutonium Product:

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Shipped Mr./Hr.</u>	<u>To</u>
42	0.7	0.021		5	2	234-5
43	0.9	0.035		5	2	234-5
44	1.3	0.027		5	3	234-5
45	1.4	0.030		4	3	234-5
46	1.4	0.007		5	3	234-5
47	0.9	0.003		4	3	234-5
48	0.9	0.005		5	2	234-5
49	2.0	0.003		4	2	234-5
50	10.9	0.016		6		Holding

D. Overall Waste Loss - % of Total Production:

U = 0.08%

Pu = 0.08%

E. Rework - % of Total Production:U = 9.7% (Predominantly U recycle from 2UC TK-K6 to HAF Make-up
TK-E6 at shutdown)Pu = 6.8% (Predominantly out-of-spec Pu product via TK-L11 to HAF
Make-up TK-E6)**III. Waste Treatment****A. Volumes to UGS:**

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Solvent Washes	452	104 & 106
Cell Drainage	-	106
FP Waste	152	106
Coating Waste	270	107C

B. IWW Flows: 5.6**C. Tank Farm:**

<u>Tank</u>	<u>Est. Boil-Off Rate, Gal/Min</u>
101	5.4
102	4.2
103	0.1
104	5.2
106	12.1

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Notes:

1. The HAF rate was maintained at a nominal 3.3 CF until scheduled plant shutdown, beginning Dec. 30. At 2200 on Dec. 30, processing of "hot" HAF was completed.

2. Flowsheet

- (a) On Dec. 22, the 2EX-HNO₃ was increased from 0.09 to 0.18 flows. It was further increased to 0.36 flows on Dec. 24.
- (b) On Dec. 22, the addition of 0.0005 M Hg(NO₃)₂ to the metal solution was commenced.
- (c) On Dec. 22, rework of waste from TK-F8 was commenced at a rate of 150 gallons per 4000-gallon batch of HAF.
- (d) On Dec. 23, the 2EX temperature was increased from 49 to 51° C. It was further increased to 55° C on Dec. 24 and to 57° C on Dec. 28.
- (e) On Dec. 23, the IBXP-HNO₃ was increased from 8.5 to 9.0 flows.
- (f) On Dec. 29, the ZDS was decreased from 67 to 58 flows. It was further decreased to 48 flows on Dec. 28.
- (g) On Dec. 29, the ZDS was increased from 48 to 57 flows.

3. Feed Preparation

Because of increased iodine emissions late during the previous report period, the addition of 0.0005 M mercuric nitrate to the metal solution was resumed on Dec. 22. The total emission for the current report period was 0.87 curies. Normally irradiated uranium processed since the recent non-representative uranium campaign has averaged 693 M/D/T, 135-day "cooled".

In preparation for plant shutdown, all dissolver heels were removed.

4. Solvent Extraction

Process performance was satisfactory except for continued poor performance of the Final Uranium Cycle. The return to "normal" uranium processing combined with apparent reduction of HA Column saturation resulted in temporary two and four-fold increases in the gamma activity in the 2DF and 2AF, respectively. Meanwhile the HAW Pu losses were about three fold lower than for the previous report period.

- (a) On Dec. 24, the 2E Column became unstable as a result of low acidity in the 2EX (0.002 to 0.005 M). Gross entrainment of organic into the 2DU occurred, and appeared in the concentrated UH product. An attempt was made to segregate the solvent phase into the UH rework storage tank, TK-P3. Rework from this tank back to the solvent-extraction system was suspended pending laboratory investigation of the process characteristics of this material.
- (b) On Dec. 27, a 2D Column flood resulted in the production of three batches of uranium product which were excessively high in gamma and plutonium content.

5. Plutonium Concentration

Continued routing of N Cell wastes to the 3WF Tank F10 (see p. 223) has been necessary due to an apparent flow restriction in the normal routing to TK-J1. More thorough flushing of this line is planned for the shutdown period.

On Dec. 27, pressures in the XA Column reached as high as 100 psig, and it appeared that either the waste routings were both becoming plugged or that the XAW screen was becoming plugged.

During the course of the ion exchange mechanical problems, Pu levels in the XC Column and the XSW losses became excessive, apparently because of a carryover of high acid from the XA Column. This resulted in a stripping problem which reached its peak on Dec. 28 and 29, when about 4 or 5 kg. of Pu were lost to TK-F10 via the XSW. At the same time the TK-F10 sampler was out of service because of a leaking jumper, and the 3WB concentration in E-H4 was being increased in preparation for a neptunium recovery run. These factors combined to cause a Pu inventory in the 3WB Concentrator E-H4 above the normal critical mass control limit of 7 kg.

6. Silica Gel

During the December run period, silica gel operation was excellent. During the period Dec. 14 through Dec. 31, approximately 350 tons of uranium were processed. This is exceptional performance relative to past runs.

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