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PROCESS SUMMARY OF PUREX PLANT OPERATION
JANUARY 1962 THRU DECEMBER 1962

AUTHOR

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PROCESS SUMMARY OF PHEAS PLANT OPERATION
FROM 0000 ON 1/1/62 TO 2400 ON 1/15/62

During the report period the plant was shut down with respect to normal plutonium and uranium processing. A neptunium recovery run was conducted and necessary maintenance was performed. Process notes of interest are summarized below.

1. Feed Preparation

On Jan. 8 the lower bed of the C2 silver reactor was regenerated after having processed the off gases from the dissolution of 2249 tons of uranium.

2. Solvent Extraction

Shutdown of the solvent-extraction system following the December 1961 operating period was aimed primarily at preparing the plant for the subsequent neptunium recovery run.

(a) Exceeding the Critical Mass Control Limit in E-14

During the operations of shutting down the plant, excessive plutonium losses in the waste from the E-Cell Pu ion exchange unit resulted in the accidental accumulation of up to an estimated 11.6 kg. of Pu in the J-3 just prior to the beginning of the neptunium recovery run on Jan. 2. This amount of Pu is about 166 per cent of the critical mass control limit of 7 kg. The temporary loss of control was caused primarily by the following:

- (1) The ion exchange loss was principally in the XSW stream, a stream of normally low Pu content which is not routinely sampled.
- (2) The combined ion exchange wastes have recently been routed to the J-7 Tank F10, which feeds the J-3 Concentrator R4, rather than to the J-3 Tank J1, which receives from the R4 Concentrator. This abnormal routing has been necessary because of recent inability to maintain flow from E Cell to TK-J1.

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- (3) The sampler for TK-F10 was out of service because of a faulty in-cell sampler jumper.

This problem was also aggravated by the fact that at the time of the unplanned E Cell loss, the 3-B concentration was being increased, as is normal procedure in preparation for neptunium recovery. Also, plutonium levels in the plant were already artificially high by virtue of Pu rework via the HAF Tank 80. The peak inventory of Pu in E4 was probably maintained for only about 30 to 40 minutes, after which E4 overflowed to TK-J1 reduced the value to an estimated 10.1 kg. About 6 hours later, when E4 had been cooled following shutdown, the excessive inventory was transferred to TK-J1, in which this amount of Pu is permissible. It was at this time that the excessive backcycle inventory was discovered, based on samples of TK-J1 contents.

(b) Water Flush of 2EU Concentrator E-14

Recent evidence of UHM loss from the 2EU Concentrator E4 at the tube bundle flanges, and continued tendencies for the concentrator to pressurize during operation resulted in a decision to waterflush this vessel during the current shutdown period. Hot water was added via the concentrator tower and was boiled in the concentrator. Loss of UHM in the flushing operation was prevented by ultimate return of the flush water via the 20F Tank K1 and the 2D Column to Tank F10 and the 3-B system.

3. Neptunium Recovery Run

(a) Summary

A neptunium recovery run was started at 1040 on Jan. 2 and was completed at 1915 on Jan. 9 (2EP flow stopped). After product concentration, 0.78 kg. of neptunium was loaded out, equivalent to a recovery of only 30% of the neptunium in the 3-B processed.

On Jan. 11 and Jan. 12 L-Cell sump samples showed high Hp content. Subsequently, a leak check indicated an appreciable leak in either L3 or L4, the stripper and concentrator, respectively, used in the Hp concentration operation. Ultimately, the loss of neptunium via this leak was confirmed by transfer and analysis of the sump volume in TK-F13. An estimated 490 grams of Hp received in this vessel were transferred to TK-F8 for eventual rework. On Jan. 13, an L-Cell entry and inspection revealed a leak at the upper tube bundle flange of the E-14 Concentrator.

In addition to the gross "loss" of product described above, this particular run was interrupted by apparent plugging of the 2EP-to-2AF recycle jet. The first flow failure and shutdown of operations occurred at about 0400 on Jan. 5. Operation was resumed at about

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2300 on the same date, but the flow again failed about four hours later. At this time an entire new jet assembly was installed, and the recovery operation was completed without further trouble. The original jet assembly had been built with a flow control valve in the jet suction which had previously been used in the control of ZBP during the transition to the Phase III concentration portion of these operations. Since this valve constituted a relatively severe restriction for solids hold-up, it was omitted from the new design. The procedure during the following Phase III transition was successfully altered to compensate for this change.

Discovery of solids in ZBV samples during the recovery run lent credence to the theory of plugging of the ZBP-to-ZAV jet routing. Two types of solids were observed and examined in the laboratory. A whitish, waxy-appearing material was insoluble in all ordinary reagents and analyzed 10,000 ppm of titanium, with no other metallic constituents. Similar material has been previously encountered in Purax process streams and most likely originates as Amercont in waste rework. Another solid material found in the ZBV had all the appearance of foam rubber, even burning with a smoky flame.

(b) Material Balance*

	<u>Kg</u>
Kp in Plant	1.62
Kp In 3-B	1.61
Kp Processed as 3-B	1.55
Kp Load-out	0.73
Kp Recycle	0.88**
Kp Lost	0.00

(c) Typical Solvent-Extraction Losses*

<u>Stream</u>	<u>% of 3-B Processed</u>
ZAV	1.3
ZBV	2.6

*Losses recycled for recovery during normal processing operation.

**Estimate of recycle indicates 0.35 kg in 3-B system due to ZBP jet failure and overflow to L Cell package and 0.49 kg in Tank F8 as result of 1A Concentrator flange leak.

(d) Product Analytical Results

	<u>Ep</u> <u>g/l</u>	<u>HNO₃</u> <u>M</u>	<u>% Contamination of Ep</u> <u>Pu</u>	<u>U</u>	<u>G/Ep</u> <u>ATx107</u>
Principal Load-out	17.1	10.4	1.7	0.2	15.0
First Flush	7.2	12.0	2.4	0.4	15.0
Second Flush	2.5	11.4	5.0	0.1	15.0
Third Flush	0.5	11.0	5.0	2.0	170.0

4. Plutonium Concentration

During plant shutdown, the ion exchange resin was stripped of plutonium in preparation for a resin change. The removal of resin from the columns was effected largely by the use of the new system for jet-transfer of the resin to the 204 Tank R3. The replacement resin again included 25 per cent 10 - 20 mesh and 75 per cent 20 - 40 mesh.

A shutdown flush of the R1 Tank and the XAF flow system revealed the accumulation of considerable amounts of resin. It is suspected that resin most likely enters the R1 Tank by leakage through the resin receiver top-hat screen and via the vent system. Inspection of the top-hat screen, as well as the XAV and XSW screens revealed no unusual holes or tears. At the extremely high operating pressures of the recent run period, it is still conceivable that small resin beads and fines were forced through the screen openings.

In an effort to free up the normal waste routing from H Cell to TK-31 (see EW-67800, p. 238), a relatively high-volume ECX flush was pumped through this system, using a temporary hook-up of the U1-to-E6 pump. The routing to TK-310 was likewise flushed. These routings both appeared to be unrestricted and some question now exists as to whether the previous trouble stemmed from a line restriction or from substandard performance of the XAV jet, itself.

5. Solvent Treatment

Following the neptunium recovery run, TK-01 (carbonate-permanganate solvent washer) was flushed routinely with oxalic and nitric acids for K_2O_2 removal. Subsequent water calibration indicated the normal volume was restored.

Shutdown operability checks of jet routings from TK-R1 to TK-01 and from TK-R1 to TK-R3 showed that the former has a shortened 3/2 dip-tube in TK-R1. This accounts for the poor operability (high solvent losses to TK-01) of the recent test of re-use of R1 carbonate in 01 (see EW-67800, p. 231). Transfer via this jet stopped at a liquid level of 38 inches, about 6 inches above the intake of the 207 (solvent) pump.

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6. Waste Treatment and Acid Recovery

(a) E-F11 Flushes

Following plant shutdown, the normal E-F11 empty-out jet continued to be inoperative, and a temporary portable jet was installed for this duty. In a vain attempt to restore the operation of the normal jet, a series of caustic-tartrate flushes of F11 were conducted. In addition, attempts were made to back-flush with caustic-tartrate into the jet chamber, and pressure was applied via the jet dip-leg. All of these techniques were unsuccessful.

On Jan. 15 design peculiarities of this particular F11 vessel were discovered which accounted for the jetting problem. The design had been modified with respect to the original design in that the jet-out chamber is a vertical, heavy-walled 3-in. pipe, into which the jet dip-leg is inserted. The jet-out chamber then elbows into the circular-section concentrator side chamber. Pipe sizes and clearances would cause this system to be prone to plug and extremely difficult to unplug.

(b) Formaldehyde Prototype

Operability testing of the EW formaldehyde denitration prototype during the last operating period was hampered by contamination in the Pipe and Operating Gallery, near the prototype piping, and by high liquid levels in TK-F15 due to Fission-Product Recovery operations and equipment failure.

Tests to date have checked the equipment operation except actual formaldehyde flow to the reactor and continuous EW flow through the reactor. These tests have indicated:

- (1) Good preheater temperature control;
- (2) Questionable reactor temperature control due mainly to heat loss from reactor to TK-F15. While running water at ~ 0.3 gal/min, the maximum reactor temperature was 98° C at a boil-off rate of ~ 0.4 gal/min. No major difficulties are anticipated from the low boil-off rate as the formaldehyde reaction is exothermic.
- (3) No pressure indications have been evident in the tests.
- (4) Weight factor measurements in the reactor are dependent upon TK-F15 liquid level, the condenser vent system vacuum, and the F11 differential pressure. It may be necessary to operate with the TK-F15 liquid level below 2000 gallons for good

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reactor control. At high F15 liquid levels, IAW can back up into the reactor via the overflow line from Tank F15, apparently from the vacuum imposed by the vent system.

- (5) Equipment failures have necessitated replacement of the F12 pump, F12 electrical jumper, and the F12 pump process jumper (DOV failure).
- (6) The final operability test will be in operation for an extended period of time with IAW flow but no formaldehyde.

(a) Flush of TK-P8

Following recent completion of waste rework from TK-P8, the vessel was flushed with water and with hot 5% caustic-2% tartaric acid.

7. Fission Product Processing

(a) Cesium

Decontamination of E Cell equipment in preparation for cesium processing was initiated on Dec. 23. Following decontamination, the malfunctioning in-canyon loadout jet (see HA-G7500, p. 205) was replaced and loading of the first STT commenced on Dec. 27, 1961. By Jan. 15, 1962 the four casks had been loaded with a total of 200 kilocuries of cesium. The casks were purged with an inert gas shortly before being released for shipment on Jan. 19, 1962.

Cask loading proceeded very smoothly with the new in-canyon load-out jet performing satisfactorily. This jet is a factory built Penbarthy jet that was operability tested by Process Equipment Development Operation before installation in the jumper. A Benford built jet was also tested and found to not meet the hydraulic requirements of the E Cell location.

To take advantage of improved Decalzo loading at reduced temperatures, an unused cooler was relocated from the Hot Semiworks 276-C Bldg. to the Purex loadout station. The cooler was placed in a wooden box, shielded with two feet of sand, and connected in series with the inlet line to the STT. To compensate for the pressure drop introduced by the cooler, the two valve assemblies in the fill line Snap-Tite were removed.

Initial attempts to get flow through the cooler were unsuccessful, apparently because of air locking. Testing of the cooler with raw water showed the coil to be free of obstructions. The cooler was bypassed and loading of the first cask initiated. Satisfactory operation of the in-canyon loadout jet permitted cooling the feed

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to the jet to 10 - 15° C. This resulted in a cask feed temperature of 30° C vice previous loading temperatures of 50° C. This 20° C reduction in feed temperature was sufficient to allow loading the casks to the 50,000 curies limit imposed by the OREL hazard review.

During loading of the first STT cask the exit line TP readings were noted to gradually increase from four-fold below inlet line reading to three-fold above inlet line reading. Straight undiluted waste samples were obtained, and analyzed for cesium. No cesium breakthrough was detected. On the second, third, and fourth casks a portable gamma spectrometer was used to monitor the exit line in addition to the routine waste samples. No cesium breakthrough was detected. Gamma scans on the exit line indicated the reading to be mostly cerium, however, the exit line did not exhibit the high readings noted with the first cask.

Preparation of the loaded STT casks for shipment included the following operations:

- (1) The cask void volume was adjusted with the liquid level probe to 100 gallons. The void was then confirmed by inserting a dip-tube of fixed length through a spare instrument nozzle.
- (2) The casks were pressurized to 20 psig through a spare instrument nozzle. The process nozzle closures were then flooded and observed for leaks for a minimum of 60 minutes.
- (3) The casks were vented through a spare instrument nozzle, sealed and observed for pressure and temperature behavior for a minimum of 24 hours.
- (4) Just before shipment, the casks were vented through a spare instrument nozzle and purged with 50 cubic feet of argon to sweep out the hydrogen.

(b) Hot Semiworks

TK-6 volume stopped decreasing about Dec. 25, 1961 indicating that all of the citrate in this solution has decomposed after 80 days storage. TK-6 volume appeared to have stopped decreasing in the middle of November, but started decreasing again following the addition of nitric acid. This phenomenon suggested a leak in the side of the tank. The volume presently stored in TK-6, however, is greater now than in the middle of November and the existence of a leak in the side of the vessel is disproved.

The decomposition of nitric acid from radiolysis is apparently very slow. Analyses of TK-6 samples taken 40 days apart showed that TK-6 had decreased from 1.32 to only 1.27 μ HNO₃. From this it is

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concluded the long term storage of strontium will not pose problems when stored in the nitrate form. Previous data had shown a rapid loss of nitric acid when citric acid was present. Apparently the loss of nitric acid results from the reaction with carbonate which is formed by the radiolysis of citric acid rather than direct radiolysis of nitric acid.

Laboratory work with TX-4 samples show that a satisfactory Sr CO₃ precipitate for cask filtration is obtained by the addition of KI CO₃.

Preparations for loading the HAFU II-1 cask for the second time commenced on Jan. 8, 1962. All equipment was checked for operability and instrument calibrations were completed. During cask coil testing, a leak was found in one of the new welds made when converting the process nozzles from Gamp-Tite closures to Hanford connector closures. The weld leak was repaired and the cask coil leak test repeated; no additional weld leaks were found.

8. Miscellaneous Notes

(a) Tube Bundle Failures

During the report period, three concentrator tube bundle failures were detected, and the tube bundles were replaced as follows:

- (1) E-F6, west, after 5.3 months of service;
- (2) E-F11, west, after 9.3 months of service; and
- (3) E-F4, west, after 19.3 months of service.

(b) Vent System Maintenance

On Jan. 4, steam leakage was noted at the vapor flange of the Heater E-F2 discharge to the Silver Reactor T-F2. A suspected heater tube leak was confirmed by shutting off the steam flow to the heater. Consequently, on Jan. 9, the failed heater was removed and a spare was installed.

During the course of the heater investigation and replacement, about 72 gallons of liquid was jettied from the bottom of the silver reactor. The silver reactor was given a normal two-shot regeneration on Jan. 12 and 13.

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(c) Canyon Cell Water Flood

On Jan. 3, during the start-up of E₄ Concentrator for the neptunium recovery run, a raw water line which was open to H Cell was inadvertently valved open. Several hours later the error was discovered, but by this time water which had backed up via the wind tunnel to L Cell had entered into the FR Room. By means of cell sweep jets a total of about 125,000 gallons of water was transferred via TK-713 to underground storage (241-A TX-106).

BF Judson/EC Rathvon

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 1/16/62 TO 2400 ON 1/25/62

I. Typical Solvent-Extraction Performances 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	3.5	-	-	0.003	0.04
Final	2.7	3.3	9.0	2.5	0.027	-
Ion Exchange	-	1.5	-	0.4	-	-
Overall	6.3	8.3	9.0	2.9	0.030	0.04

II. Production

A. UAF P processed: 204 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>
1	6.1	4.6	3.1	PI
2	-	-	5.6	MC
3	4.1	3.6	4.2	PI
4	4.3	1.8	2.4	PI
5	3.9	-	6.5	PI
6	6.8	2.0	6.0	PI
7	2.9	-	6.7	PI
8	3.4	2.2	6.0	PI
9	3.1	-	6.5	PI
10	3.2	3.4	7.3	PI
11	3.7	-	6.0	PI
12	4.1	3.9	7.2	PI
13	4.7	-	7.8	PI
14	-	-	7.1	PI
15	5.7	-	6.9	PI
16	6.2	5.0	7.8	PI

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1. Solvent Extraction

<u>Batch</u>	<u>Gamm Ratio</u>	<u>Pu, gms U</u>	<u>Tons</u>	<u>Shipped To</u>
17	6.7		7.4	P1
18	7.1	8.5	7.1	P1
19	8.4	7.7	7.3	P1
20	8.6	8.5	7.4	P1
21	8.9	11.6	7.5	P3
22	9.5	13.8	7.9	P3
23	9.1	13.2	7.9	P3
24	7.3	6.7	7.5	P1
25	8.6	6.9	7.0	P1
26	8.2	4.8	7.9	P1
27	7.3	4.6	7.7	P1
28	8.6	4.3	6.9	P1
29	8.5	3.7	7.1	P1
30	9.0	4.7	7.6	P3
31	9.2	4.3	7.0	P1

2. Silica Gel

Gamm Ratios: Feed = 6.4
Product = 1.5

Tonnage: ~ 350

C. Plutonium Product:

<u>Batch</u>	<u>ZrO₂/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Hr./Hr.</u>	<u>Shipped To</u>
3	1.4	0.132		5	4	234-5
4	2.0	0.016		5	3	234-5
5	1.3	0.004		5	3	234-5
6	1.3	0.014		5	3	234-5
7	1.2	0.018		5	2	234-5
8	1.2	0.014		5	2	234-5
9	1.0	0.024		5	3	234-5
10	1.2	0.008	✓600	5	3	234-5
11	1.3	0.008		5	2	234-5
12	1.5	0.023		5	2	234-5
13	1.4	0.076		5	3	234-5
14	1.5	0.130		4	4	234-5
15	1.7	0.077	✓600	6	4	234-5
16	1.4	0.055		5	3	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.33%
Pu = 0.5%

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E. Rework - % of Total Production:

R = 2.7%

Pu = 15.7% (Predominantly scraps and out-of-specification Pu product via TK-L11 to HAF Make-up TK-26)

III. Waste Treatment

A. Volumes to WWT:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	833	104 & 106
Cell Drainage	819	106
FP Waste	175	106
Coating Waste	270	107C

B. Ind Flows: 3.5 Flows

C. Tank Pans:

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	4.8
102	4.1
103	0.2
104	5.2
106	10.4

Notes:

1. The plant was started up (HAF on) at 0545 on Jan. 16. Processing of "hot" feed commenced at 1515 on Jan. 16. The operating rate was a nominal 3.0 CF throughout the report period, except for a period of operation at a nominal 3.3 CF from Jan. 19 to Jan. 22. The actual rate averaged about 2.8 CF for the report period.

2. Flowsheet

- (a) The start-up flowsheet was the same as that in effect at the end of previous operating period, except as follows:
  - (1) The IBX, IBSP, IBU, L-Cell, and H-Cell flows were started at 3.3 CF.
  - (2) The ZDF-ferrous sulfamate was started at 0.39, rather than 0.2, flows.

- (3) The 3EX temperature was 48° C, rather than 56° C.
- (4) The 2EP was routed to L-Cell Package, rather than to the H-Cell Ion Exchange facility.
- (b) On Jan. 16, the HAX-nitrite was increased from 0.66 to 0.84 flows. It was returned to 0.66 flows on Jan. 17.
- (c) On Jan. 17, the 2EP stream was routed to the H Cell Ion Exchange facility. The H Cell wastes were initially routed to the 3-B Tank J1, but were rerouted to the 3-B Tank F10 on the same day.
- (d) On Jan. 17 the water flow to the Demister was shut off because of a leak in the drain line to the 2M Concentrator E-76.
- (e) On Jan. 17 the 2EX-HNO₃ was increased from 0.15 to 0.39 flows. It was further increased to 0.78 flows on Jan. 25.
- (f) On Jan. 17 the 2EX temperature was increased from 53° to 54° C. It was further increased to 56° C on Jan. 18.
- (g) On Jan. 20 nitric acid addition to the 2DS was commenced at a rate of 0.23 flows of 12.2 M HNO₃.

3. Feed Preparation

Irradiated uranium "cooling" times varied from 122 to 181 days and averaged 140 days, during the report period. Use of mercuric nitrate in the metal solution, for iodine suppression, was continued. The total iodine emission for the report period was 0.37 curies, with a maximum one-day emission of 0.06 curies.

Late on Jan. 25, it was necessary to shut down the B3 Dissolver because of a thermocouple failure.

4. Solvent Extraction

Start-up of the plant was accomplished smoothly and "hot" feed was started to the solvent-extraction columns in minimum time. Particular attention was directed toward maintaining the initial "cold" feed at sufficiently high concentration to prevent the EA Column instability experienced at the start of the last run period.

Performance of the First Cycle with respect to both product recovery and fission product decontamination has been as good as prior to shutdown. The Final Plutonium Cycle also continued to show excellent performance. The decontamination performance showed a 30 to 50-fold improvement over the last run period, while the product losses to the Backcycle Waste System averaged a factor of two less.

Below-normal performance by the Final Uranium Cycle continued, both with respect to fission product decontamination in the 2D Column and product recovery in the 2E Column. Operation of the 2D Column was maintained under control (no floods occurred), but at the expense of loss of efficiency in the scrub section. Consequently, it was necessary to segregate three batches of uranium product which were slightly above silica-gel limits for rework.

In an attempt to stabilize the 2E Column, thereby permitting higher frequencies and improved efficiency, a program to increase the acidity in the 2E Column was commenced on Jan. 17 and was continued throughout the report period. The HNO_3 acid increase was aimed at a tentative maximum of 0.03 M (compared to the 0.01 M flow-sheet value). Also, on Jan. 20, addition of HNO_3 was commenced at a rate sufficient to theoretically increase the HNO_3 concentration by about 0.005 M. At the end of the report period this program was still underway. The 2E Column pulse frequency had been increased from 33 to 35 cycles/minute.

On Jan. 24 a floating organic phase of approximately 1700 gallons was discovered in the 3AF Tank F10. The manner in which this material had accumulated was not known.

5. Plutonium Concentration

The XAF was again routed to F10 for the bulk of the report period in spite of the heavy flooding of the J1 routing during the recent shutdown.

Resin was again found in the XAF screens indicating that resin or resin fines are continually escaping via the top-hot screen. Push pressures remained above average during the report period.

From a standpoint of product quality, operation was excellent, with the DP ranging between 25 and 30.

6. Waste Treatment and Acid Recovery

Formaldehyde Treatment Prototype

Operability testing of the D4 Formaldehyde Treatment Prototype continued during the report period. Reaction in the vessel occurs about 1 to 2 minutes after introducing formaldehyde to the system; the reaction is easily detected by means of the TK-F15-2 Reactor Tower differential pressure instrument and the weight factor and specific gravity instruments. The reaction is vigorous resulting positive pressures of 1 to 4 inches of water. Efforts to operate at lower pressures were not successful during the short time the equipment was operating. Further testing has been suspended due to a leak at the flange of the Reactor and the Reactor Tower.

7. Plasmon Products

Test Run

A special test run to demonstrate recovery of promethium from sulfate waste was attempted. The test run started with centrifuged LW and a standard sulfate precipitation (pH 1.0) except for centrifugation at 55° C instead of 65° C to favor leaving promethium in the supernatant solution. The sulfate waste was returned to TK-E3, butted to 0.5 M oxalate with 1 M oxalic acid and the pH adjusted to 1.6. After digesting at 20° C for 60 minutes, the solution was centrifuged, the cake neutralized with 1.0 M sodium hydroxide, and the cake dissolved with 1.0 M nitric acid. This solution was loaded into two 200-gallon casks and shipped to 300 Area.

The results of the run were disappointing. The E5 sampler was inoperative during the entire test run and no analyses were obtained on either the sulfate waste or waste from the oxalate strike. The solution in the casks was found to have a pH of 4 and be about 0.5 M in oxalic acid, apparently through cross-contamination of chemicals in the aqueous make-up headers. In the presence of oxalic acid at this pH, any promethium slurried from the centrifuge would have precipitated and remained in TK-E1. Analyses on the LW and carbonate product indicated that 60 kilocuries of promethium were in the sulfate waste. Due to lack of E5 samples, it is not known whether the oxalate precipitation failed or succeeded.

On January 23, normal strontium processing was resumed. By the end of the report period, the third sulfate run was underway.

Hot Strontium

Following completion of the operability checks on the RAPO II-1 Cask in D-Cell last week, the strontium nitrate solution segregated for loadout was neutralized from 1.3 M to 0.4 M nitric acid with sodium hydroxide. Neutralization and precipitation of the strontium was then completed with potassium bicarbonate. The KHCO_3 solution was about 5 M and was added slowly (estimated 1 l/min - no control system) to the strontium nitrate solution. The reacting vessel temperature was 30° C at the start, but climbed rapidly to 60° C before being controlled and returned to 40° C. In addition to the brief loss of temperature control, the solution foamed-out of the vessel (TK-67) into TK-64. After the completion of the KHCO_3 addition TK-64 solution was returned to TK-67 and the solution was simmered for 2 hours at 30° C. The solution was cooled to 15° C and the filtration operation was started.

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During the filtration operation the filter dp increased rapidly and the flow of solution decreased sharply. After about two hours the radiation recorder near TK-67 indicated little or no strontium was being moved through the slurry line. In addition small quantities of air were noted in the slurry inlet line. During the early portion of the filtration operation, the slurry inlet valve was periodically closed to allow slumping of the cask filter cake. This was terminated when no benefit was noted. After about 4 hours of filtration and since little product was being removed from TK-67, the cake in the cask was slumped by stopping the slurry jet, closing the slurry inlet valve, and then opening the slurry inlet valve when the cask vacuum was essentially zero. The filter dp decreased substantially but little or no precipitate was removed when the filtration operation was resumed. Also observation of the slurry inlet line showed large quantities of air in the system. The cask was allowed to rest about 10 - 12 hours. When the filtration operation was resumed, the solution and precipitate moved rapidly through the slurry inlet line. TK-67 was emptied and about 300 liters of filtrate solution from TK-64 was processed through TK-67 and the cask in about 3 hours. The filtration and flushing operation was ended when the radiation Beckman near TK-67 had returned to its normal reading.

Preliminary calorimeter tests and analytical data were evaluated during the interval from January 19 to 23 to determine a satisfactory cask load had been obtained. When it was confirmed that in excess of 100 kilocuries of Sr 90 had been loaded, the process lines and connectors were removed, except for the vent line on the Jamesbury valve and a pressure gauge on the solvent inlet process nozzle. Just prior to removal of the process connectors, the cask vent line froze and pressurized the cask to 20 psig. This caused contamination of D Cell, but the contamination spread was not serious.

After the process lines and connectors were removed, the cask was dried at about 17 inches of Hg absolute. The cask was heated slowly from about 60° C to 145° C over a 4 hour period and maintained at 145° C for 16 hours.

After drying the cask the cask coil was leak-tested and all cask connections were also checked for leaks. No leaks were found so the test to measure the rate of pressure build-up was started. While the cask was cooling, the pressure decreased from 12 to 2 psig. This was greater than expected so another leak-test was made. It was found that a leak had developed around the stem of the cask vent valve, apparently during cooling. Efforts to stop the leak by tightening the valve were unsuccessful.

At the end of the report period, preliminary decontamination of D Cell and the cask was underway. It is planned to place the secondary closure over the failed vent valve and complete the cask stability tests. The cask will then be moved to Parent for repair of the failed valve.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 1/20/62 TO 2400 ON 2/1/62

I. Typical Solvent-Extraction Performance by Cycle

	Gross GF		% Recycle		% Loss	
	U	Pl	U	Pl	U	Pl
First	3.7	3.7	-	-	0.004	0.02
Final	2.5	3.0	11.0	3.4	0.007	-
Ion Exchange	-	1.5	-	0.1	-	-
Overall	6.2	8.7	11.0	3.5	0.013	0.02

II. Production

A. RAF Processed: 135 Tons

B. Uranium Products:

1. Solvent Extraction

<u>Batch</u>	<u>Gross Ratio</u>	<u>Pl, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
32	6.3	5.5	7.4	Pl
33	7.3	6.3	7.6	Pl
34	7.5	6.4	7.7	Pl
35	7.5	7.1	7.0	Pl
36	6.3	4.8	7.3	Pl
37	7.1	6.0	7.0	Pl
38	6.6	-	7.0	Pl
39	6.4	4.4	7.4	Pl
40	6.0	3.9	5.4	Pl
41	6.1	3.6	7.4	Pl
42	7.1	2.6	7.5	Pl
43	7.0	-	7.7	Pl
44	7.0	4.2	7.2	Pl

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1. Solvent Extraction

<u>Batch</u>	<u>Gama Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
43	5.1		7.5	P1
45	8.6	2.7	7.6	P1
47	8.6		7.4	P1
1	11.4	5.9	7.3	P3
2	10.4	3.0	7.6	P3

2. Silica Gel

Gama Ratios: Feed = 7.4
Product = 1.3

Tonnage: ~ 53

C. Plutonium Product

<u>Batch</u>	<u>2r5b/AT x 10¹¹</u>	<u>% U in Pu</u>	<u>Pu, ppbp Pu</u>	<u>No. Cans</u>	<u>Average Hr./Hr.</u>	<u>Shipped To</u>
17	1.7	0.003		5	1	234-5
18	0.7	0.060		5	1	234-5
19	0.6	0.067		5	2	234-5
20	0.5	0.003	885	5	2	234-5
21	0.5	0.294		4	2	234-5
22	0.5	0.001		4	1	234-5
23	0.6	0.007		5	2	234-5
24	0.8	0.019		5	2	234-5
1	4.9	0.773		5	10	Holding
2	5.1	0.720		5	10	Holding

D. Overall Waste Loss - % of Total Production:

U = 0.20%
Pu = 0.24%

E. Revert - % of Total Production:

U = Negligible

Pu = 6.3% (Out-of-spec "Pu" product and miscellaneous sweep material via L11 to HAF Make-up TK-25)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gel/Ton</u>	<u>Waste Tank</u>
Wash Solution	560	104
Cell Drainage	515	106
FP Waste	377	106
Coating Waste	270	107C

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B. INF Flows: 3.4 Flows

C. Tank Farn:

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	3.9
102	4.2
103	0.2
104	4.9
106	11.8

Notes:

1. The HAF rate was maintained at 3.0 CF until 2120 on Jan. 30 at which time a shutdown was necessary because of a leak in the JWF jumper. After starting up at 3.0 CF at 1830 on Jan. 31, another emergency shutdown occurred at 1030 on Feb. 1 as a result of failure of the INF Concentrator 3-F11.

2. Flowsheet

- (a) On Jan. 26, the addition of 0.003 M NaNO_2 to the INF was commenced.
- (b) On Jan. 29, rework commenced to the INF Feed Tank, of organic (28%) which had collected in the Backcycle Waste (JWF) tank. After no adverse effects on the solvent-extraction columns were detected from a 90-gallon batch, the rework was commenced at a nominal rate of 100 gallons every four hours.

3. Feed Preparation

On Jan. 26, the failed J3 Dissolver thermocouple (p. 14) was replaced and operation of this dissolver was resumed.

4. Solvent Extraction

Except for the Final Uranium Cycle, solvent-extraction performance was good throughout the report period, with some improvement in the First Cycle decontamination performance. Final Uranium Cycle performance continued to be substandard.

The accumulation of organic phase in the JWF Tank F10 previously reported (see p. 15) continued until Jan. 29. At this time the continuous discharge of the recirculating 28% sampler was found to be the source of this material. The rate of accumulation had been estimated to be about 0.3 gal/min., resulting in the accumulation of 3600 gal. in the TK-F10. On Jan. 29, the rework of this solvent by introduction to the INF Tank J3 was tried with the initial transfer of one 90-gallon increment. Subsequently, rework was successfully accomplished at a nominal rate of 100 gallons every 4 hours.

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Failure of the JWF jumper necessitated an equilibrium shutdown of the entire plant on Jan. 30. The plant was started up again without trouble on the following day, but failure of the IWF Concentrator necessitated another complete shutdown on Feb. 1. Moderate bursts of activity occurred during the startup as a result of temporary low saturation in the HA and 2D Columns.

4. Plutonium Concentration

During the report period N-Cell decontamination performance was exceptionally good; a peak IF of about 40 was reached.

Mechanical operation of N-Cell continued to be poor, however, with frequent push troubles accompanied by heavy runbacks of liquid into the XAF Tank 31. This problem was believed to have been caused by improper positioning of the three-way Durco valve located above the resin reservoir. Faulty operation of the XAF system may also have been a contributing cause. The N-Cell wastes (XAW) continued to be routed to TX-F10 during the report period.

5. Waste Treatment and Acid Recovery

On Jan. 28, a leak was discovered in the drain jumper from the Demister to 2WF Concentrator E-F6. It was necessary to stop the Demister water flow.

The Formaldehyde Prototype was operated for only a few hours during the report period. Operation to date has been characterized by slight pressurization of the Tower, and a leak at the Tower flange has been evident. On Jan. 28, an attempt to stop this leak by tightening the flange nuts was not successful.

Instrument diptubes in the IWF Concentrator E-F11 plugged during the report period and were out of service at the time of concentrator failure on Feb. 1. Concentrator failure was apparently a leak in the shell of the vessel.

The addition of 0.008 M $\text{Na}_2\text{S}_2\text{O}_3$ to the IWF was commenced on Jan. 25 because of reported Savannah River experience indicating a reduction in recovered acid Pu concentration by this technique. The concentration was limited at 0.008 M (vice SRP's 0.01 M) by test flow control equipment. By the end of the report period a possible downward trend in the Absorber Acid (F3) Pu concentration was partially observed by the plant emergency shutdowns.

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7. Fission Products

Lead End

Strontium run series 140 through 147 was completed during the report period. Run 140 was the special promethium run described last report period. The strontium product from run 140 was saved and combined with the strontium carbonate product from sulfate runs 141, 2, 3 and 4. The series was then completed with two oxalate runs and a concentration run.

This was the first series of strontium runs since the cesium campaign. In an attempt to extend centrifuge life, changes were made in the operating procedures. Briefly these were:

- 1) A jet out route was installed from the E2 centrifuge to E5 and all IW centrifuged prior to sulfate precipitation,
- 2) The centrifugation speed was reduced from 1500 to 1200 RPM, and
- 3) The carbonate metathesized sulfate cake is slurried from the centrifuge with water followed by acid rinses instead of cake dissolution within the centrifuge with acid.

Precentrifugation of all IW was initiated to reduce the weight and heat in the centrifuge bowl during the sulfate step. It was estimated that 75 percent of the weight and 40 percent of the heat in the bowl during the sulfate step is associated with the siliceous solids from raw IW. The speed reduction was aimed at reducing the load on the bearings. The change in cake removal procedure was to minimize foaming and the heat of neutralization caused by the reaction of dissolution acid with the carbonate solution heel and carbonate product cake. It has been postulated that foaming in the centrifuge moves fission product precipitates to points inaccessible to the spray system. The precipitates then may accumulate, unbalance the machine, cause overheating, and contribute to machine failure.

The procedure of centrifugation of IW prior to sulfate precipitation worked very well. The IW solids were easily slurried from the centrifuge with water. The subsequent sulfate product cake was metathesized and also found to be easily removed from the centrifuge. This is in sharp contrast to the difficulty experienced with cake removal when the two cakes are combined.

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During this series of runs the sulfate cake wash and metathesis wash solutions were recycled and combined with the next batch of raw IWF feed. This change was made to recover any product lost during the wash steps. However, analyses of raw IWF versus centrifuged IWF indicated the lead recycled with the metathesis wash precipitated in raw IWF and carried strontium. This strontium was then lost with the IWF solids.

The ES sampler was inoperative during the run series so no material balances could be made. The product solution sent to the 244 CR vault contained 66 kilocuries of Sr 90 and had the following composition and decontamination factors.

	<u>Curies/gal</u>	<u>DF</u>	<u>lb/gal</u>
Sr 89 & 90	1370		
Zr90	240	20	
RuRh 103 & 106	<1	>100	
Ce 141 & 144	390	10	
Fe			0.06
HNO ₃			0.20
SO ₄			<0.5
Sr 89/90 ratio		7.35	

Special analyses were run for americium and curium during runs 133 through 136. It was found that about 80 percent of the Am-Cm is recovered with the strontium product in the sulfate step and essentially all of the Am-Cm follows the cerium in the oxalate step. The americium content of IWF when processing uranium with an exposure of 830 MSD/T and cooled 175 days was 0.07 g/ton.

Promethium analyses on this run series indicated the promethium split equally between the sulfate waste and carbonate product.

Hot Semiworks

Preliminary decontamination of D Cell and the cask was completed and the secondary vent line closure installed to check cake stability. While the stability check was underway, final calorimeter measurements were completed. The cake was found to be stable and calorimeter measurements indicated the cask contained between 100 and 114 kilocuries of Sr 90. The cask was then moved to Purex for valve repair and final testing.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 2/2/62 TO 2400 ON 2/13/62

During the report period the plant was shut down with respect to normal uranium and plutonium processing because of failure of the 1W Concentrator P11 previously reported (p. 21). Process notes of interest for the report period are summarized below.

1. Feed Preparation

Chemical flushing of the Coating Waste Tank D2 was commenced on Feb. 5 because of a plugged jumper, which was apparently caused by a build of coating waste solids. TX-D2 was flushed three times with hot 12% HNO_3 - 2% tartaric acid, digesting for 24 hours, and finally with hot dilute caustic. Finally, on Feb. 14, the D2 sumpier was unplugged following the application of high pressure water to the diptubes.

2. Solvent Extraction

On Feb. 14 an unsuccessful attempt was made to fix the failed 2B Column float. The float was removed and the float rod was straightened. Upon re-installation, it was still not operable.

In preparation for plant start-up, the Final Uranium Cycle was started up on Feb. 16 in order to work off a large volume of off-standard 2DF and to purge the processing system. Instability of both the 2D and 2E Columns resulted in excessive 2EW losses... to 0.25 percent...early during the operation. Subsequently, stability was restored, the 2EX rate was increased 15 percent, and the 2EW losses decreased 10 to 20 fold. During the last 4 to 6 hours of the 2-day operation, sanitary water was substituted for demineralized water for 2EX make-up, to scout any possible effect on the 2EW losses. Due to the short duration of this variation, the process response could not be judged with certainty. An apparent further decrease in 2EW losses could have been further response to the 2EX increase.

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3. Final Plutonium Concentration

During the February shutdown the H-Cell resin was stripped and replaced with the same mixture of 75% 20-40 mesh and 25% 10-20 mesh.

The condition of the XAW routing to TK-J1 was checked using full steam supply pressure of 40 psig to the XAW jet. Normal full flow to TK-J1 was obtained. Recent problems in maintaining this flow were apparently caused by the closing of a steam throttling valve which should have been open.

The 3-way Durco valve was also overhauled and a new teflon sleeve installed. The replaced sleeve was deeply scarred and conceivably had been the cause for a great deal of the push problems and liquid loss troubles which have plagued the ion exchange equipment during the past few run periods.

4. Solvent Treatment

On Feb. 9, the addition of about 3000 gallons of "used" solvent from TK-P2 storage to the No. 1 Solvent Treatment System was commenced. The TK-P2 solvent is that material which was removed from the process equipment in preparation for the test of Soltral-170 diluent starting in September of 1961 (see NY-67000, p. 167). An approximately 5000-gallon aqueous heel containing about five hundred pounds of uranium was initially transferred into the building and was discarded to underground storage. The organic volume was then transferred to the G Cell equipment over a period of about 24 hours. During this period and for about a shift afterward, the G Cell solvent treatment equipment was operated to continuously reprocess the solvent inventory. At shutdown, the solvent quality was excellent...negligible $ZrNb$ activity, less than 300 $\mu\text{c/gal}$ of Ru , and low Pu retention (4.6×10^{-7}). During the transfer operation from TK-P2, flow control was good, demonstrating the practicability of routine measured solvent additions by this routing during normal processing operations.

Following the solvent addition operation, the IOO in TK-G5 was batted to 0.003 M nitrite in order to check for the possibility of any unusual nitrite "kill" caused by the P2 solvent addition. No such "kill" was noted. The nitrite in the IOO was removed by a short period of G Cell solvent reprocessing in preparation for plant start-up.

5. Waste Treatment

(a) Replacement of E-F11 Concentrator

Failure of the E-F11 Concentrator on Feb. 1 caused plant shutdown primarily for the purpose of replacing this equipment piece. The failure was a leak in some part of the concentrator body, apparently on the west side. According to vessel drop-out measurements taken Feb. 2, the leakage rate was about 3 gal/min. and the liquid level in the concentrator was about a foot and a half above the vessel bottom when drainage stopped.

In preparation for its removal, the failed concentrator was chemically flushed with the following series of standard flushes, during the period Feb. 2 through Feb. 6.

- (1) Caustic - Tartrate
- (2) Oxalic - Nitric
- (3) Caustic - Permanganate
- (4) Oxalic - Nitric
- (5) Caustic - Tartrate
- (6) Oxalic - Nitric
- (7) Caustic - Tartrate

On Feb. 7, the interior of the failed E-F11 was inspected by television camera through the west tube bundle flange. Visibility was limited, and evaluation of the condition of the metal and welds was difficult. A couple large chunks of irregularly shaped solids were observed, estimated to measure up to a foot across and roughly 3 inches in thickness. According to the crane operator, these solids were grayish and rusty colored.

On Feb. 8, the failed concentrator was removed and buried in the Railroad Tunnel Extension. Burial was accompanied by considerable contamination of the railroad cars and tunnel. This was postulated to have been caused by leakage of highly contaminated liquid from the concentrator seal pot. The concentrator seal pots cannot be emptied by any normal method.

The replacement E-F11 Concentrator was installed on Feb. 11.

(b) Single Concentrator Test

On Feb. 9 a single concentrator test was begun to determine the feasibility of operating the acid recovery system with only one evaporation step. The test used the E-F6 Concentrator and Denister system with TX-F10 serving as the feed tank. The

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absorbed acid product collected in TX-F3 was returned to F10 and butted with water to constitute a synthetic feed stream of normal LW acid. The activity level in the recovered acid was used to analyze the decontamination capability of the system. During the 5-day test period, the activity was increased in the E-F6 bottoms by stepwise additions of LW to the synthetic feed, as follows:

- (1) At start-up on Feb. 9 the normal F6 bottoms gamma activity level was increased about 10 fold, to 3×10^4 uc/gal.
- (2) On Feb. 10, the level was increased 5 fold, to 4×10^5 uc/gal; and
- (3) On Feb. 13, the level was increased 4 fold to 1.6×10^6 uc/gal, approximately 75 percent of full level LW.

On two occasions, Feb. 11 and Feb. 12, the test was temporarily shut down to facilitate the installation of the new X-F11 Concentrator. The test was finally terminated on Feb. 14. Maximum LW feed rates of ~35 gpm (equivalent to ~2.25 CF) were all that could be reached due to vacuum limitations. Several steps were taken in an effort to increase the concentrator's decontamination efficiency. These included adding water to the Denister pad, adding water to one tray of the concentrator, adding nitrite continuously to the feed, and periodically adding trace amounts of organic to the feed.

Through the first four days, test results were very favorable. ZrTb activity generally remained below 1000 uc/gal. and except for one surge the Ru activity remained below 10,000 uc/gal. During this period the F6 activity concentration was ~20% of LW and 30 times normal ZrTb. The low ratio of ZrTb to Ru activity was wholly unexpected as it was felt that the main loss in DF during the test would be due to increased entrainment which presumably would sharply increase the ZrTb activity. One factor which may have contributed heavily to the high Ru activity was that the acidity in the F6 bottoms, which in all probability was between 6 to 7 molar rather than the normal 4 to 5 molar.

Following the F6 activity increase on Feb. 13 the activity levels of the recovered acid samples began to increase sharply. At the time of test termination on the 14th, the Ru had leveled off at ~40,000 uc/gal and the ZrTb had peaked at ~4,000 uc/gal. The

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cerium levels, which beforehand have been almost insignificant, also jumped sharply to over 10,000 $\mu\text{c/gal}$. The termination of the test, dictated by the plant startup schedules, made it impossible to determine if the sudden increase in activity represented the equilibrium activity or merely a surge due to the sudden change in Z-76. Other factors which may have influenced the rise in activity were an increase in both the Z-F6 specific gravity (and the accompanying increase in boiling temperature) and the F6 liquid level.

The activity of the acid at the conclusion of the test was higher than has generally been considered acceptable, but the fact that the ZrF6 was effectively controlled during the first two-thirds of the test indicates that the basic system of a concentrator plus Denister may have the basic decontaminant capability to permit removal of one of the P-Cell Concentrators. In general, the duration of the test was too short to permit investigation of effects...activity level fluctuations...which were observed. The fluctuations suggested, however, that control by operating or other techniques is likely to be feasible. The development of a method to control the ruthenium appears to be the most critical problem, but because this chiefly involves a valence and a volatilization mechanism, a chemical means of control may be successfully applied.

(c) Miscellaneous

On Feb. 13, hydrostatic testing of the TX-F6 coil indicated a leak. TX-F6 is the vessel which has been used for the reflux of miscellaneous wastes in preparation for their rework by addition to the HAF.

6. Fission Products

Head End

Run series 143 through 155 consisting of five sulfate runs, two caustic runs and one concentration run was completed. A total of 73 kilocuries of Sr 90 were recovered with a DF of 40, 560, and 70 for cerium, ruthenium and zirconium-niobium, respectively. Recycle of the sulfate wash, caustic-carbonate metathesis solution and carbonate cake wash into the next raw LSW feed batch was deleted because of premature precipitation of the strontium described on page 23. Clarification of all LSW prior to sulfate precipitation has eliminated the sticking cake problem and has improved decontamination from ZrF6 and Ru.

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Strontium Semiverts

The HAPO II cask leaking vent valve was repaired and final testing of the cask completed. The cask has a pressure buildup rate of 0.5 psig per day and the temperature differential between the cask interior and ambient air is 27°C.

On Feb. 13 the cask was loaded into the shipping buffer and the system allowed to come to thermal equilibrium. A 6°C temperature gradient between ambient air and the outer buffer annulus was measured. Additional insulation of the buffer will be required before shipment.

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PROCESS SUMMARY OF FARMER PLANT OPERATION FROM 0000 ON 2/15/52 TO 2400 ON 2/17/52

I. Typical Solvent-Extraction Performance by Cycles

	<u>Concn of</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>P₁</u>	<u>U</u>	<u>P₁</u>	<u>U</u>	<u>P₁</u>
First	4.4	4.0			0.002	0.05
Final	2.3	3.0	11.0	3.0	0.055	
Ion Exchange		0.7		1.0		
Overall	6.7	6.5	11.0	4.5	0.057	0.05

II. Production

A. RAF Processed: 198 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Concn Ratio</u>	<u>P₁, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
4	6.3	11.5	4.0	P1
5	4.4	13.4	3.5	EC
6	27.0	11.8	6.0	EC
7	57.7	6.0	2.0	EC
8	37.0	1.5	6.42	EC
9	70.0	2.7	4.17	EC
10	60.0	2.1	7.5	EC
11	37.0	1.4	6.2	P3
12	22.1	1.5	5.45	P3
13	12.3	1.0	6.55	P1
14	6.5	1.4	7.45	P1
15	4.0	2.2	7.4	P1
16	3.5	1.1	7.3	P1
17	3.55	1.0	7.55	P1
18	4.0	1.7	7.4	P1
19	4.77	2.45	7.4	P1
20	3.7	4.7	7.4	P1

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
21	4.45	4.2	6.65	P1
22	2.37	4.7	7.33	P1
23	2.7	5.3	7.4	P3
24	3.2	2.0	6.6	P3
25	2.3	1.7	7.2	P3
26	2.4	2.5	7.6	P1
27	2.2	1.6	7.2	P1
1	2.2	1.0	7.3	P1
2	2.0	1.4	7.2	P4
3	2.3		7.0	P1
4	2.6	1.0	7.2	P1
5	1.8	0.9	7.2	P4

2. Silica Gel

Gamma Ratios: Feed = 4.0
Product = 1.0

Tonnage: ~ 222

C. Plutonium Products:

<u>Batch</u>	<u>GrU/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>
3	38.0	0.17		5	50	C11 → B6
4	3.4	0.018		5		234-5
5	3.7	0.02	11.00	4	5	234-5
6	3.2	0.02		5	5	234-5
7	1.6	0.02		4	2	234-5
8	1.0	0.003		5	2	234-5
9	0.6	0.01		5	3	234-5
1	0.6	0.01		5	2	234-5
2	0.4	0.06		4	2	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.37%
Pu = 0.46%

E. Rework - % of Total Production

U = 24.2% ("Cold" UNH product recycle to HAF Make-Up Tank E6)
Pu = 50% (Principally out-of-spec. Pu product from TK-L11 to HAF
WD 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	444 & 265	104 & 106
Cell Drainage	50	106
FP Waste	140	106
Coating Waste	270	107

B. IMP Flows: 5.4 Flows

C. Tank Pans:

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	4.9
102	4.2
103	0.2
104	5.1
106	10.1

Notes:

1. The plant was started up "hot" (HAF on) at 1500 on Feb. 19. During the period Feb. 20 to Feb. 22, approximately 38 tons of "cold" uranium was recycled as HAF. The HAF flow was off from 0140 to 0305 on Feb. 22 because of failure of the HSH jet steam jumper. The HAF flow was off from 1540 to 2230 on Feb. 22 because of failure of the IMP pump.

2. Flowsheet

(a) The plant was started up on the same flowsheet that was in use at the end of the January run period, with the following exceptions:

- (1) The HAX-HaNO₂ was started at 0.77, rather than 0.66 flows.
- (2) The 2DF ferrous sulfamate was started at 0.98, rather than 0.39 flows.
- (3) The 2RX temperature was 50°, rather than 58°C.
- (4) The 2BP was initially diverted to the L-Cell Package, and the 2BX make-up was adjusted accordingly.
- (5) No nitric acid was added to the 223 stream.

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- (b) On Feb. 20, recycle of "cold" EAF was commenced. Processing of "hot" feed was commenced again on Feb. 22.
- (c) On Feb. 20, the 2EX temperature was increased from 50 to 57°C. It was further increased to 60° on Feb. 22. On Feb. 21, the 200 temperature was increased from 35°C to 40°C.
- (d) On Feb. 22, the 2BP stream was routed to H-Cell ion exchange unit, and the 2BX make-up was adjusted accordingly.
- (e) On Feb. 23, the 3WB was increased from 39 to 54 flows. It was gradually increased to 62 flows by Feb. 26.
- (f) On Feb. 24, rework of IAW which contained excessive plutonium and neptunium was commenced and increased to a nominal rate of 45 gal./T.U.
- (g) On Feb. 25, a 24-hour test of the use of sanitary water, rather than demineralized water, was commenced. The 2EX temperature was reduced to 50°C during the test.
- (h) On Feb. 25, a test of the use of sanitary water for the 2DB was commenced. The test was halted on Feb. 26.
- (i) On Feb. 26, rework of IAW was completed.
- (j) On Feb. 26, batch washing with water of the 2BX in TK-R1 was commenced. Approximately 13 flows of water is used.
- (k) On Mar. 1, rework of P8 material was commenced at a nominal rate of 20 gal./T. The rate was increased to 35 gal./T. after the initial batch.
- (l) On Mar. 1, the uranium concentration in the EAF was reduced from 3.57 to ~3.4 lb/gal.

3. Feed Preparation

The C3 Dissolver was shut down on Feb. 24 because of a failed thermometer. On Feb. 26 the thermometer was replaced and the dissolver was returned to routine service.

4. Solvent Extraction

Start-up of the plant on Feb. 19 was characterized by instability in the BA, 35, and 2D Columns, resulting in excessive waste losses and resulting in gamma levels 10 to 100 fold above normal. The instability problem was at least partly caused by organic degradation

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products formed during the prior 18-day shutdown period, during which the organic was in contact with the "hot" aqueous phase in the columns. Instability of the columns and the resultant activity burst necessitated switching to "cold" uranium for about 43 hours to allow the activity levels in the plant to return to normal.

The start-up was also plagued by mechanical problems on Feb. 22, as follows:

- (a) A temporary shutdown of the IX and ES Columns and the IXF flow was required to replace a defective HSR jet steam jumper.
- (b) A seven-hour shutdown of the First and Final Uranium Cycles and the Waste Treatment System was required to replace a failed IXF pump. Also during this time, the start-up batch of IXF L1 E-Fill Concentrator was segregated for rework because of suspected high product content.

Decontamination performance of the First Cycle following the start-up difficulties was excellent. Product recovery, however, has been somewhat lower than during the two preceding run periods (with losses averaging about 0.07 percent), but a sharp downward trend was noted at the end of the report period. The Final Plutonium Cycle has continued to show excellent decontamination performance, in spite of the unusually low losses to the Backcycle Waste System (1 to 2 percent).

The Final Uranium Cycle continued to exhibit below-normal performance - both with respect to waste loss and fission product decontamination. A BP of 100 to 200 was typical and the activity of the uranium product was maintained at acceptable levels (two to four gamma ratio) only because of the excellent performance of the First Cycle. Uranium losses via the 2EW stream continued high (a factor of 50 to 100 above normal). The uranium losses to underground storage were minimized by batch-contacting the 2EW with water in the 20V tank (commencing on Feb. 26) and by storing for future recovery as much of the 20W as space permitted. In addition to the high waste losses, the 2E Column was limiting in capacity, necessitating continued operation at biased-low 3.0 CP.

Recent semiworks studies have indicated the Purex water to be a possible cause for the Final Uranium Cycle problem. C-Column capacity studies were made in which samples of Purex water were compared to the use of steam condensate for IXF make-up, with the effect measured in terms of column capacity. Purex fully desmineralized water, Purex cation exchange water, and Purex sanitary water all caused 15 to 40 percent loss of capacity, with the sanitary water being perhaps the "best" quality of the 3 Purex samples.

In an effort to evaluate the water as a variable in the plant, the normal demineralized water in the 2EX was replaced by sanitary water for 26-hour period on Feb. 25-26. No significant change in performance was noted. Likewise the subsequent use of sanitary water for the 2DS stream resulted in no significant change.

A test was initiated at the end of the run period to evaluate the effect of a more dilute HAF (3.4, rather than 3.57 lb./gal. uranium) on HA Column performance. The objective of the test was to demonstrate the feasibility of making the transfer from the HAF Make-Up Tank 26 to the HAF Tank III by jet rather than pump. Transfer by jet is desirable in order to permit use of the pump space for a second tank agitator, thereby improving critical mass control during transfers of plutonium rework from TK-LII to TK-26.

5. Final Plutonium Concentration

During the current operating period the Z-Cell ion exchange unit has operated exceptionally good relative to past run periods. Push pressures remained normal throughout the report period and liquid runbacks to XAF Tank XI have essentially ceased. The improvements are attributed to the sleeve replacement in the 3-way Dureco valve (p. 29). The XAW stream has also been successfully routed to TK-JI for the entire run period.

The slight decline in W-Cell DP during the report period is chiefly a result of the abnormally low activity in the 2BP stream.

6. Solvent Treatment

Processing longer "cooled" metal and the generally good plant decontamination in the extraction section has allowed reduction of the volumes of wash wastes in the No. 1 Solvent Treatment System. Streams varying from normal flowsheet are:

<u>Stream</u>	<u>Normal Flowsheet</u>	<u>Current Flowsheet</u>
10B - Na_2CO_3	19.2 flows	10.0 flows
10B - KNO_3	1.7 flows	1.5 flows
10C - Na_2CO_3	11.9 flows	7.6 flows
10D - Na_2CO_3	11.9 flows	7.6 flows

High losses of uranium from the 2SE stream to the No. 2 Solvent Treatment System washes have resulted in heavy uranium precipitation in the 2CS Tank R2. Part of this high-loss material has been stored in TK-R3 and R5 for future rework. In an attempt to reduce the uranium loss to

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the carbonate, water-washing of the 224 in TK-R1 was commenced on Feb. 26. The water extract is periodically letted to the JWB Back-cycle system via the JWF Tank F10 for recovery. Variance in 224, TK-R1 and TK-R2 samples have made it difficult to evaluate the effectiveness of this contact. The volume of the water extracts amounts to approximately 13 flows.

7. Waste Treatment and Acid Recovery

High product losses occurring during plant start-up were subsequently segregated, by emptying the IAW Concentrator 8-P11, and were successfully reworked during the report period.

Operation of the IAW Formaldehyde Prototype was commenced on Feb. 26. Start-up of the unit was relatively smooth with good control of the IAW flow. Control of the formaldehyde flow was good only if maintained about 50% higher than the flowset value. The reactor tower occasionally pressurized, showing continued leaks at the flanges.

8. Fission Products

Head End

Run series 156 through 160, three sulfate runs, one oxalate run, and one concentration run was completed. A total of 46 kilocuries of Sr 90 was recovered with decontamination factors of 26, > 1000, and 10 for cerium, ruthenium, and zirconium-niobium, respectively. This series was limited to three sulfate runs due to lack of IAW feed material.

Another run series was started when the IAW used for the P6 test became available. Three sulfate precipitations were made, runs 161, 162, and 163 but the cake was lost from the centrifuge during the zeta-thesis step. The reasons for the abnormal cake behavior are not known, but are believed to be ascribable to the off-standard feed resulting from the P6 test.

Strontium Semiworks

An additional layer of insulation was added to the HAPD II-1 buffer. This increased the temperature gradient between ambient air and the outer buffer annulus to 20°C which is adequate for the ambient air temperature expected during shipment this time of the year. At the end of the report period the cask was scheduled for shipment on March 2.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 1/2/62 TO 2400 ON 1/19/62

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma RF</u>		<u>% Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pl</u>	<u>U</u>	<u>Pl</u>	<u>U</u>	<u>Pl</u>
First	4.4	4.2			0.002	0.025
Final	2.3	3.1	3.2	4.4	0.017	
Ion Exchange		1.3		1.2		
Overall	6.7	3.7	3.2	5.6	0.069	0.025

II. Production

A. UAF Processed: 148.5 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pl, ppb U</u>	<u>Time</u>	<u>Shipped To</u>
6	2.0	0.6	7.0	Pl
7	2.7		6.9	Pl
8	1.9	1.2	7.0	Pl
9	2.4	1.3	7.3	Pl
10	3.1	1.1	7.0	Pl
11	2.3		7.2	Pl
12	2.9	1.6	6.7	Pl
13	2.7		7.1	Pl
14	2.3	2.3	7.2	Pl
15	2.3		7.2	Pl
16	3.6	1.4	7.7	Pl
17	2.9		7.0	Pl
18	2.5	1.0	7.3	Pl
19	2.7		7.0	Pl
20	2.6	2.1	7.2	Pl

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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>
21	2.1		7.1	Pl
22	2.4	0.3	6.7	Pl
23	2.3		7.1	Pl
24	2.6	2.4	6.0	Pl
25	3.3		7.3	Pl
26	4.7	5.7	2.7	Pl
27	4.2	4.9	6.7	Pl
28	3.0	4.2	7.4	Pl
29	2.2	3.4	7.9	Pl

2. Silica Gel

Gamma Ratios: Feed = 2.3
Product = 0.35

Tonnage: 160.85

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>
3	0.6	.09		5	1	234-5
4	0.4	.03		5	1	234-5
5	0.5	.04		5	2	234-5
6	0.4	.02		6	3	234-5
7	0.6	.05		6	5	234-5
8	1.6	.007		4	7	234-5
9	0.3	.03		6	5	234-5
10	0.7	.02		5	5	234-5
11	0.7	.02		3	5	234-5
12	0.7	.04		6	1	234-5
13	0.3	.06		3	3	234-5
14	0.1	.06		5	6	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.27%
Pu = 0.35%

E. Rework - % of Total Production:

U = 12.5 ("Cold" UHM product recycled to HAF MU Tank EG for plant shutdown.)

Pu = 5.3 (Principally Pu product from TK-141 to HAF MU Tank EG; also waste rework Pu from TK-P3 to HAF same-up.)

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III. Waste Treatment

A. Volumes to DCH:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	532	104
Cell Drainage	130	106
YP Waste	149	106
Coating Waste	270	107C

B. I&M Flows: 7.5 Flows

C. Tank Farm:

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	4.4
102	3.9
103	0.3
104	4.4
106	9.2

Notes:

- The HAF rate was maintained at a nominal 3.0 CF until 0745 on March 2 at which time scheduled shutdown of the solvent-extraction system was commenced. The actual processing rate averaged about 2.9 CF. The processing of "hot" HAF was completed at about 0300 on March 3, and was followed by "cold" uranium recycle in preparation for shutdown.
- Flowsheet
  - Commencing on Mar. 4, the 2AW and 2DW losses to the Backcycle system were gradually reduced in preparation for the shutdown and subsequent neptunium recovery run.
  - Commencing on March 5, the nitric acid concentration in the 3WB was gradually reduced from 7 to 6 M (4 to 3.3 g/gal) in preparation for shutdown.
  - On Mar. 6, the 3WB was increased from 62 to 69 flows to reduce the inventory in the Backcycle System in preparation for the shutdown.
  - On Mar. 6, rework of F3 material was finished and acid dilutions of the remaining "heel" commenced. Processing of the acid rinses continued.

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- (e) On Mar. 6, the addition of 16 flows of water to the tower of the 22W Concentrator 2-Ft was commenced.

3. Feed Preparation

In preparation for the processing of about 175 tons of special non-representative irradiated uranium, cutting of the A and C Dissolver heels was commenced on Mar. 6. Following heel removal, the metal solution was processed as HAF and the high-uranium heels in the D-Cell metal solution tanks were acid flushed. The acid flushes were combined in a "cold" uranium HAF batch in the HAF Make-Up Tank E6. No attempt was made to process the rinse water from TK-D1.

4. Solvent Extraction

Solvent-extraction performance continued at about the same level as during the latter portion of the last report period. The First Cycle continued to give excellent decontamination and low plutonium waste losses (<0.03%, average). The Final Plutonium Cycle DF continued good (averaging from 1 to 2 x 10³), in spite of below-normal losses to the Waste Backcycle System (average ~1%). The Final Uranium Cycle continued to be the big problem area, with marginal decontamination performance and excessive losses via the 22W stream. Also, the 22 Column flooded on Mar. 3, and the 2D Column flooded on Mar. 8 and Mar. 10. In spite of the problems in the Final Uranium Cycle, all of the uranium product produced met silica-gel processing specifications.

Rework of TK-F8 waste and H₂O₂ dilution of the TK-F8 heel was completed during the report period without incident. At the end of the report period, the 34F Tank F10 contained about 1300 gallons of solvent as a result of continued transfer of the water waste from TK-R1 to TK-F10.

The scheduled shutdown of the plant was commenced early on Mar. 8 and completed early on Mar. 10. Highlights of the shutdown operations were as follows.

- (1) About 15 tons of "cold" uranium were recycled to the HAF to reduce the intercycle plutonium levels and the volume in the Backcycle System. This was done both for the scheduled 3p recovery run, and in preparation for a change to processing of non-representative Pu at the next normal start up.

- (2) The HA and HS Columns were stripped, the organic displaced, and the columns emptied in preparation for replacement of the HA Column interface float and the installation of a new HAP jumper for use with a new gamma monitor.
- (3) The ICU Concentrator, E-J8, and the ZDF Tank, K1, contents were processed before shutting down the Final Uranium Cycle for neptunium recovery.
- (4) The 2D and 2E Columns were stripped and emptied in preparation replacement of the 2D Scrub Cartridge and inspection of the 2E Column Cartridges.

According to data received from UO₃ Plant, the calcium content of the UO₃ powder reached a maximum of only 50 parts per million parts of U, as a result of the recent test use of sanitary water in the ZEX at Purex (p. 35).

5. Waste Treatment and Acid Recovery

Plans are being made to replace the Waste Rework Tank F8 because of recent coil failure. Contamination of the No. 2 trap pit has resulted from this coil failure and/or failure of the right hand (east) tube bundle of the IDW Concentrator E-F11.

On March 7 the water addition to the fourth tray of the F6 concentrator was started. A flow rate of 2 gal./min. (~ 16 flows) was used. The succeeding four samples of TK-F3 indicated that the 3rB activity of the recovered acid had decreased by almost a factor of ten. The amount of Ru present did not change appreciably. Shutdown prevented obtaining more data but further testing is planned for the next run period.

Operation of the prototype unit for the formaldehyde treatment of IDW was suspended due to difficulties in controlling the formaldehyde flow. Although the unit was operated at only about half the design rate, the differential pressure across the reaction tower exceeded 50 inches of water during most of the run resulting in slightly positive reaction pot pressures. The gamma activity in the recovered acid (concentration of about 1% to acid) ranged from 1×10^7 to 1×10^8 , indicating a DF of about 300.

6. Fission Products

Head End

Run series 14 through 16) was completed during the report period. It consisted of four sulfate runs, one emulsate run and one concentration run. A total of 57 kilocuries of strontium-90 were recovered with decontamination factors of 6, >300 and 50 for cerium, ruthenium, and zirconium-niobium, respectively.

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No strontium was recovered in Run 104 because the cake was lost from the centrifuge during the metathesis step. The cause of cake loss was determined by HLO hot cell experiments to be due to an increase in the iron concentration of IW. Iron tartrate was precipitated during the pH adjustment step and later when contacted with caustic would solubilize the cake. A reduction in precipitation pH from the range 1.5 to 2.5 to 0.7 to 1.5 prevented the iron tartrate precipitation and resulted in normal cake behavior.

Strontium Separation

Preparations for another cask loadout was underway at the end of the report period.

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PROCESS SUMMARY OF URANIUM PLANT OPERATION
PERIOD OF 1/11/62 TO 2400 ON 3/2/62

During the report period the plant was shut down with respect to normal plutonium and uranium processing. A neptunium recovery run was conducted and necessary maintenance was performed. Process notes of interest are summarized below.

1. Solvent Extraction

Aside from the neptunium recovery operation (Note 2, below), shutdown solvent-extraction operations were involved principally in (a) inspection and change-out of Final Uranium Cycle cartridges, and (b) the flushing of Final Uranium Cycle equipment and the UCN Storage Tanks P1 and P4.

(a) Column Cartridges

On March 13, the 2E Column cartridge was inspected visually at close range for evidence of any unusual condition which might be responsible for its record of poor recovery performance. The cartridge appeared to be very clean and in excellent condition. Nevertheless, following its re-installation, the 2E Column was flushed with warm, dilute HNO_3 prior to plant start-up.

On Mar. 16, the graded, 85 free-area, stainless steel scrub-section cartridge in the 2D Column was replaced with a "standard" nebra scrub-section cartridge. This was done to determine a possible beneficial effect on the performance of the Final Uranium Cycle. The replacement nebra cartridge is composed of alternate pairs of 21 percent free area, stainless steel sieve plates (0.005-inch holes) and 23 percent free area, linear polyethylene plates (0.0075-inch holes), all on 1-inch spacing. Prior to plant start-up, the new cartridge was conditioned by flushing with hot dilute sodium carbonate. Inspection of the stainless steel cartridge which was removed showed that the upper support spider outer ring had fractured in four places - the four points where the cross, which supported the tie rods, had been attached to the ring. The ring broke so that about half

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of its segments remained with the cartridge and the other half was attached to the top flange. Only one of the eight support rods connecting the top flange to the spider had broken loose from the spider ring. The most plausible explanation for the fracture centered around metal fatigue caused by bending which occurred whenever the column was pulsed.

(b) Vessel Flushing

Water flushing of UHJ product handling vessels was commenced on March 17 for the purpose of purging possible organic phase buildup resulting from recent instances of 22 Column flooding. Vessels flushed included the 22J Concentrator 2-K4, the 22C Receiver TX-K5, the 22C Sampler TX-K6, and the P1 and P4 UHJ Storage Tanks. The fluxes containing product UHJ were accumulated in an E-Fill waste batch which was concurrently being prepared for eventual waste rework. (See Note 3, below.) The 22 Column acid flush (Note (a) above) was also combined in E-Fill.

(c) Processing of Solvent from the 3MF Tank F10

On March 13, approximately 1400 gallons of solvent accumulated in the 3MF Tank F10 during transfer of TX-A1 water washes were transferred to TX-J3 (IBXF) for recovery. This solvent was combined with neptunium recovery solvent wastes and reprocessed in the No. 1 Solvent Treatment System.

2. Neptunium Recovery Run

(a) Summary

A neptunium recovery run started at 1400 on March 10 (cold start up) and was completed, after three forced shutdowns, at 0945 on March 22 (2BP flow stopped). Following product concentration 1.03 kg of neptunium was loaded out, equivalent to a recovery of only 6% of the neptunium processed as JN3. The lower-than-normal recovery efficiency was the result of a progression of process leaks, plugs, and upsets.

Initially during the "cold" run start-up, a leak at a manual valve on the 2BP jumper delayed the "hot" start up until 1450 on March 12. After repairing the leak, by removing and then replacing the 2BP jumper, a series of plugs occurred in the 2BP to TX-J3 recycle jet line forcing another shutdown from 1045 on March 13 to 0615 on March 15. During the continuation of the Phase I portion of the run several upsets of the 2A Column occurred because of loss of 2A3 flow. This flow problem resulted from overheating of the demineralized water tank 30 to $> 70^{\circ}\text{C}$ which in turn caused the

hydrogen^{engine} and ferrous sulfamate in the 2A3 to react and form gas which trapped the line. Finally, during the concentration portion of the run (Phase III) four separate leaks in the L Cell Package (E-13 thermowall, upper flange on E-14 reboiler section, E-14 thermowall and flange on E-13 stripping section) forced another shutdown from 0140 on March 19 until 2200 on March 20.

The voluminous losses which occurred during the many upsets were handled in several unusual ways which greatly increased the overall recovery of neptunium for the run.

Contaminated overflows to the L Cell Package during Phase I and Phase II portions of the run were recovered by routing the material back to TK-35 via TK-19 to XAW to TK-31 line in E Cell hood and pumping TK-31 to TK-35. Leaks to the SIA sump during the Phase II and the early part of Phase III portion of the run were returned to TK-35 by connecting the SIA sump sample line to the ZBP sample return line with plastic tubing in the Sample Gallery and then getting the material entering the ZBP sample pot to TK-35 with the recycle jet. During the latter part of Phase III the leakage was returned to the ZBP sample pot and allowed to overflow into the L3 stripper.

A material balance of the system after the run showed 13% recycle loss via the 2AW, 6% recycle loss via the 2BW and 7% loss unaccounted for and presumed lost to the various waste tanks. Analysis of the run data indicates that the majority of the 2AW loss occurred while returning the sump material to the 2AF TK-35. The excessive 2BW loss occurred during the start-up portions of the run when the ZBP interface position was questionable (the ZBP interface control was out of service).

After the concentration (Phase III) portion of the run was started the relative dilute product in the L Cell Package was loaded out rather than recycling when the package had to be emptied to repair the leaks. As a result three dilute load outs were made before the main concentrated load out. Then excessive flushing was carried out in order to clean out the package for further maintenance after the run.

(b) Material Balance*

	Kg
Np in Plant	1.80
Np in 3WB	1.79
Np Processed as 3WB	1.66
Np Load-out	1.11
Np Recycle	≥ 0.57**
Np Lost	≤ 0.12***

* Losses recycled for recovery during normal processing operation.

** An estimated 0.30 kg was recycled via the 2AW during shutdowns and startup operations caused by ZBP jet failures.

*** An unknown amount of product was recovered from the cell floor as a result of 3 different leaks in the L Cell Package during the final concentration portion of the run. The loss represents the amount unaccounted for after the run.

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(c) Typical Solvent-Extraction Losses*

<u>Stream</u>	<u>% of Pu Processed</u>
2AW	0.9**
2BW	2.7

(d) Product Analytical Results

	<u>Sp g/l</u>	<u>HNO₃ M</u>	<u>% Contamination of Sp</u>		<u>G/Sp ATx10⁷</u>
			<u>Pu</u>	<u>U</u>	
First Load-Out***	4.6	6.7	2.0	3.0	19
Second Load-Out	2.3	6.5	9.0	4.7	50
Third Load-Out	1.2	11.4	11.8	2.9	90
Principal Load-Out	17.4	12.0	5.7	0.4	15
First Flush	7.6	8.4	13.0	1.7	28
Second Flush	3.9	7.3	25	0.3	30
Third Flush	0.9	12.0	50	0.7	40
Fourth Flush	0.8	12.2	50	25	50
Fifth Flush	0.6	11.6	50	0.4	120
Sixth Flush	0.2	10.6	52	0.7	200
Seventh Flush	0.1	10	70	1.0	300

3. Final Plutonium Concentration

In preparation for the production run of non-representative uranium, special E-Cell and P2 Room procedures were used to strip the ion exchange columns and to flush the H6, H7, L10 and L11 vessels of as much Pu as possible. After loading out as product all Pu acceptable to 234-5 Building, slightly under one kilogram of Pu was flushed from the system and loaded into cans for future recycle or rework at Porex. The final flush material contained about 10 g/gal of Pu.

No major maintenance or resin change was scheduled for the shutdown.

* Losses recycled for recovery during normal processing operation.

** An estimated 0.30 kg was recycled via the 2AW during shutdown and startup operations caused by ZEP jet failures.

*** Partial unconcentrated loadouts were made in order to repair the three leaks in the L Cell Package.

4. Solvent Treatment

During the shutdown period the No. 2 Solvent System was given a complete water and acid flush to remove accumulated uranium from recent high 2SW losses. These wastes, containing about 800 pounds of uranium, combined and concentrated with plant waste in the I/W Concentrator Z-F11, for future rework.

5. Waste Treatment

During the shutdown period, available waste from F Cell, E Cell, and the Final Uranium Cycle were combined into one batch by concentration in the I/W Concentrator (Z-F11). Approximately 100,000 gallons of waste were combined into a single 4,000 gallon batch and stored in TK-F13 for future rework. Source of these wastes were:

- (1) Cell drainage wastes (sumps) collected during February and March.
- (2) Tanks Pland P4 in the 203-A Uranium Storage area were flushed after transferring the uranium to TK-P1. These tanks had contained solvent due to maloperation of the K3 column during the previous run period.
- (3) E Cell water and acid flushes containing approximately 800 pounds of uranium due to high 2SW losses.
- (4) Flush of the 2E Column, the 2EW Concentrator K4, and Tanks K5 and K6.

The final waste batch contained about 5000 pounds of uranium and about 375 grams of plutonium.

On March 17, a failed right-hand (east) tube bundle in the Z-F11 Concentrator was replaced.

6. Demineralizers

Because of a possible correlation between water quality and recent poor performance of the Final Uranium Cycle (p. 34), the demineralizer resin was completely replaced during the period March 13 through March 16. The appearance of the cation resin was excellent. The anion resin was reported to be discolored. The replacement resin was the same brand, viz., ^{Dowex} Meltite HCR-W (Darver 50d) and Meltite WBR (Darver 3) for the cation and anion beds, respectively. The Anthracite (coal) support beds were not replaced.

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7. Fission Products

Head End

Run series 170 through 177, five sulfate runs, two oxalate runs and one concentration run was completed. Approximately 105 kilocuries of strontium were recovered with decontamination factors of 6.4, >2600, and 90 for cesium, ruthenium and zirconium-niobium, respectively. Centrifugation of D.W prior to the sulfate precipitation continued to provide cakes that were easily removed from the centrifuge in the subsequent sulfate precipitation.

Strontium Semiprocess

The 170 kilocuries of Sr^{90} in Tanks 1, 64, and 67 remaining from the first CPU loadout were split between TK-67 and TK-1 and diluted with water. Dilution was required to prevent precipitation of sodium nitrate. To minimize the amount of sodium contamination in the loaded product, the strontium in both TK-1 and TK-67 was precipitated, allowed to settle and the sodium-rich supernatant solution decanted through the slurry filter cask. Very little of the strontium precipitate was transferred to the cask and the filtrate losses were less than 0.1 percent. After disposal of the sodium solution, the precipitate in Tanks 1 and 67 was slurried to the cask with 0.3 M potassium bicarbonate and the filtrate returned to TK-64 for boildown.

The cask was dried in the usual manner and removed to Purox for final decontamination and testing. On the basis of heat balances, the cask load was estimated as 140 kilocuries of Sr^{90} .

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PROCESS SUMMARY OF FUMEX PLANT OPERATION FROM 0000 ON 4/24/52 TO 2400 ON 4/4/52

I. Typical Solvent-Extraction Performance by Cycles

	Gamma RF		% Recycle		% Loss	
	U	P ₂	U	P ₂	U	P ₂
First	4.0	4.7			0.032	0.04
Final	2.5	3.0	12	1.2	0.10	
Ion Exchange		1.2		1.12		
Overall	6.0	6.7	12	1.32	0.102	0.04

II. Production

A. NAF Processed: 170 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>P₂, ppb U</u>	<u>Tons</u>	<u>Shipped To</u>
30	1.3	1.3	3.3	P4
31	6.3		3.2	P3
32	4.2	20	3.3	P3
33	13.6	12.4	7.0	P3
34	7.3	9.3	6.9	P1
35	3.7	6.2	7.2	P1
36	2.7	9.3	7.1	P1
37	2.2	11.1	7.2	P3
38	2.2	10.2	7.3	P1
39	2.2	9.3	6.9	P1
40	2.5	8.1	6.9	P1
41	2.2	6.6	7.2	P1
42	2.3	4.2	7.2	P1
43	3.3	5.6	7.0	P1
44	4.2	6.6	7.0	P1
45	4.3	4.2	7.3	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	2.3	5.5	7.2	P1
47	3.0	5.98	7.2	P1
48	4.2	2.7	7.0	P1
49	4.4	5.5	7.1	P1
50	3.7	3.5	7.7	P1
1	3.6	2.0	7.2	P1
2	3.7	2.6	7.2	P1
3	3.8	2.3	7.7	P1
4	3.6	2.0	6.8	P1
5	4.7	1.2	5.6	P1
6	4.0	2.8	5.9	E6
7			6.2	P1
8			4.9	E6
9	4.4		7.2	P1
10	3.1		6.8	P1
11			5.8	E6
12	2.2	2.6	7.6	P1
13	1.2		5.2	P4
14	1.7	1.7	4.4	P4

2. Silica Gel

Gamma Ratios: Feed = 4.0
Product = 1.1

Tonnage: 244

C. Plutonium Product:

<u>Batch</u>	<u>Grav/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>
15	1.3	0.009	~~800~~ ~~0.002~~	4	4	234-5
16				2	3	E-6
17				3	2	E-6
18				2	2	E-6
19				2	2	E-6
20	1.3	0.12		4	2	234-5
21	0.2	0.16		7	7	234-5
22	0.3	0.37		5	3	234-5
23	0.7	0.61		5	2	234-5
24	0.3	0.02	1100 ← 0.002	5	2	234-5
25	1.0	0.01		5	1	234-5
26	1.7	0.32		5	3	234-5
27	1.7	0.61		5	3	234-5
28	0.7	0.01		5	2	234-5

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C. Plutonium Products

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>\$ U in Pu</u>	<u>Pu, pyro Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
1	0.6	0.01		5	4	234-5
2	0.5	0.01		5	1	234-5
3	0.4	0.01		5	3	234-5
4	0.5	0.01	2500	5	1	234-5
5	1.2	0.23	627	5	1	234-5
6	1.4	0.63		4	1	234-5
7	30.0	0.03		5	1	234-5
8	3.7	0.8		5	1	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.32%

Pu = 0.39%

E. Rework - % of Total Production

U = 17% (Start-up - Shutdown URM from TK-86 to HAF Make-Up TK-86)

Pu = 0.3% (FR Room sumps and flushes from TK-111 to HAF Make-Up TK-86)

III. Waste Treatment

A. Values to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	693	104
Cell Drainage	104	106
FP Waste	373	106
Coating Waste	270	107C

B. INW Flows: 0.2 Flows

C. Tank Fares:

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	4.4
102	3.9
103	0.3
104	4.4
106	9.2

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Notes:

1. Normal processing was commenced (HAF on) at 3.0 CF at 0130 on March 27, with processing of "hot" feed commencing at 0300. The rate was maintained at a nominal 3.0 CF (approximately 2.9 CF, actual) until scheduled shutdown at 1100 on April 2 (HAF off). Processing of "hot" feed was completed at 1100 on April 1.
2. Flowsheet
  - (a) The start-up flowsheet was the same as that in use at the end of the last operating period, with the following exception:
    - (1) The HAF-H₂O₂ was started at 0.62, rather than 0.77, flows.
    - (2) The addition of water to the tower of the 2nd Concentrator FC was stopped.
    - (3) The 2EX-HNO₃ was started at 0.36, rather than 0.70 flows.
    - (4) Fresh HNO₃ was added to the 2D3 Stream at a rate of 0.21 flows.
    - (5) The 2D3 flow was started at 43 flows.
  - (b) On March 27, the 2DF-ferrous sulfate was increased from 0.35 to 0.48 flows. It was reduced back to 0.35 flows on March 28.
  - (c) On March 27, the use of fresh HNO₃ (12.2 M) in the 2DF was started in place of UO₂ Recovered HNO₃ (~10.4 M). The flows were changed proportionally to the change in concentrations.
  - (d) On March 28, the 2D3 was increased from 43 to 49 flows. It was further increased to 53 flows on March 29 and to 57 flows on March 30.
  - (e) On March 30, the addition of 16 flows of water to the tower (fourth tray) of the 2nd Concentrator E-FC was commenced.
  - (f) Commencing on March 30, the 3WB flow was increased and its concentration reduced in preparation for shutdown and return to processing of regular metal.
  - (g) On April 1, "cold" uranyl was recycled to the HAF stream to purge the plutonium from the system, preparatory to shutdown. Also, "cold" uranyl was added to the 2DF Tank to increase the 2DF inventory for shutdown preparations.

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3. Feed Preparation

On March 24-25, the B-Cell Silver Reactor was regenerated. The off-gases from 366 tons of uranium had been processed through this reactor since the last regeneration.

During the report period, non-representative uranium containing abnormally high Pu-240 content was processed. This material was typically 675 MBQ/T, and generally in the range of 140 to 150 days "cooked". The completion of the dissolution of this metal in A and C Dissolvers was followed by complete seal cutting and vessel flushing as previously described on page 40.

On April 2, iodine emission to the Purex stack suddenly increased from about 0.1 curies/day, or less, to nearly 3 curies/day. After peaking at about 23 curies/day on April 3, the emissions had declined to about 7 curies/day at the end of the report period on April 4. During this time, there was no evidence to indicate an emission from any of the dissolvers. Since an emission from the process vent was most likely, the addition of 0.0003 M mercuric nitrate to the metal solution (D-Cell vessels) was commenced on April 3.

4. Solvent Extraction

Start-up of the solvent extraction system on March 25 was characterized by:

- (a) Smooth operation of the First Cycle and Final Plutonium Cycle, with no significant bursts of activity. The plutonium product was routed directly to M-Cell at start-up without adverse effect.
- (b) Extremely unstable operation of the Final Uranium Cycle, with severe flooding in the 2D Column and high losses continuing in the 2E Column.

Decantation performance of the First Cycle continued to be very good, with total product activity about the same as prior to the March shutdown. However, a greater portion of the activity remaining appeared to be following the uranium stream, with less in the plutonium stream. HAW losses have averaged about 50 percent higher (.04 - .05%) than during the last run period.

Performance of the Final Plutonium Cycle has continued to be excellent, but with dP's averaging slightly lower than the previous run period (1000 vs. 1500). The lower decantation was undoubtedly due to the lower pulse frequency during the current report period (36 on dial) compared to the previous run period (90 on dial). The 2AN losses remained generally less than two percent.

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Performance of the Final Uranium Cycle continued to be very poor, in spite of the March shutdown change to the zebra cartridge. Uranium losses to the 2EX stream continued to be about 100-fold high, in spite of excessive 2EX flow ($\sim 15\%$ above flowsheet) and high 2EX temperature (60°C). The maximum operable frequency with the "zebra" cartridge scrub section appeared to be 20 cycles per minute lower than with the all-stainless steel cartridge. An important feature of the zebra cartridge, however, is that a flood is much more easily dissipated than in the all-stainless steel cartridge and consequences of upsets are not so severe, in terms of gross increases in activity and plutonium contamination of the product stream.

The use of TK-R1 for water-washing 2EX, for uranium recovery, continued during the report period. Transfer of the water extract to the 3NF Tank F10 was accompanied by the entrained loss of organic to TK-F10 at a rate of about 300 gal/day.

Investigation of the Final Uranium Cycle problem during the report period included operation of a glass semiworks 2E column, using Purex process solutions. Operating this column with Purex 2DU resulted in an instability threshold at 3.0 CF in the range of 3-10 cycles/min. below normal. The problem appeared to be aggravated, also, by simultaneous operation with Purex-Plant 2EX.

Laboratory investigation of the Final Uranium Cycle problem has shown reduced uranium transfer rates using Purex Plant solvent streams (2EX, 2DU). Carbonate-permanganate washing of the 2EX is reported to restore normal uranium transfer rates.

On March 30, a plant test made to determine the possible effect of fresh solvent on the Final Uranium Cycle operation. Operation with wholly fresh solvent alone was not possible. However, over a period of less than two shifts, about 5500 gallons of fresh solvent was added to about 8500 gallons of normal inventory in the No. 2 Solvent Treatment system. Losses to the 2EX stream were closely monitored during this time, but no particular effect was noted.

Plutonium isotopic analysis of two samples of product during the current non-representative processing campaign indicated a Pu-240 content of about 5%. These results indicate good segregation during the prior shutdown period.

On April 3, following plant shutdown, a water hold-up test was conducted on the ICU Concentrator to determine the possibility of failure of the stripping tower internals. Such failure and resultant loss of

solvent stripping efficiency could account for poor performance of the Final Uranium Cycle. However, the test showed no evidence of failure.

5. Final Plutonium Processing

Following the processing of the last non-representative plutonium, the H Cell ion exchange columns were stripped and the product vessels were acid-flashed. About 1000 grams of Pu were flushed and carried out for processing at 234-5 Building. It is estimated that not more than 500 grams of non-representative plutonium remained in the H-Cell - PA-Room complex following the flush.

Prior to start-up, the stripped E Cell resin was replaced with the normal blend of new resin.

6. Solvent Treatment

Attempts to make a material balance for 2EW uranium losses to the Final Uranium Cycle have indicated sampling and/or analytical problems. Typically, analyses of the 2EW water strip in TK-R1 (being recovered by transfer to the 3WF Tank F10) indicate a high percentage recovery (say, 80%) of the 2EW losses measured at the 2E Column (K3). At the same time, measurements of uranium in the carbonate wash wastes indicate losses two to three fold higher than 2EW sample results.

During plant shutdown at the end of the report period, the Shell E-2342 solvent stored in TK-P2 since the Soltrol test in September, 1961, was transferred for storage to the Diluent Storage Tank-40. TK-40 was prepared by transferring the Soltrol-170 diluent inventory to a railroad tank car (previously used for UO_3 acid storage). Tank B5, in the No. 2 Solvent Treatment System, was then routed to TK-P2, so that carbonate wash wastes can now be accumulated in TK-P2 for eventual rework, if desired.

7. Waste Treatment and Acid Recovery

Review of acid recovery data showed that the addition of 16 flows (~ 2 gal/min) of water to the tower of the 2WW Concentrator F6 was followed by an immediate, nearly 10-fold, drop in acid Cr-Sb content. Consequently, on March 30, this water addition was again started, in an attempt to repeat the effect. The results of the previous test were duplicated with the 2WW level decreasing ten fold.

During the report period the Waste Rework Tank F8 was acid-flushed to the process for complete product removal, after which chemical flushing to waste was started in preparation for removal and re-placement of the vessel. As previously reported (p. 28), the TK-F8

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coil had failed. Chemical flushes were standard and included hot caustic-tertrate, hot caustic-nitric, caustic-permanganate, and nitric-ferrous sulfamate.

8. Fission Products

Head End

Run series 178 through 184, four sulfate runs, two oxalate runs and one concentration run was completed during the report period. About 93 kilocuries of strontium-90 were recovered with decontamination factors of 8.9, >2300, and 120 for cerium, ruthenium and zirconium-niobium, respectively. Over-all recoveries for strontium-90 continue to be around 60 - 65 percent with 25 - 30 percent of the loss occurring in the sulfate precipitation step.

Strontium Scriveworks

The solution in TK-6 containing 230 kilocuries of strontium-90 was sampled and found to have a pH of 9 and was black in color. Acid additions to lower the pH resulted in pressurizations and foaming of the material into the vent header. A sample sent to Process Chemistry indicated one mole of acid per liter was required to reduce the pH to 1.6 and foaming was severe. Large quantities of solids were observed in the sample and the solids were insoluble in dilute or concentrated nitric acid. Addition of hydrogen peroxide to boiling solutions at least 2.5 M in nitric acid dissolved the solids and resulted in a clear, pale yellow solution.

Most of the report period was spent removing plugs from the vent header, which resulted from foam excursions in both Tanks 1 and 6, and concentrating solutions used to remove the plugs. At the end of the report period, peroxide treatment of TK-1 (part of TK-5) was under way and proceeding smoothly. Concentration of TK-1 without foaming difficulties was possible for the first time since starting to work on Tank 6 material.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 4/5/62 TO 2400 ON 4/12/62

I. Typical Solvent-Extraction Performance by Cycles

	Gamma RF		% Recycle		% Loss	
	U	Pu	U	Pu	U	Pu
First	4.6	4.0			0.502	0.10
Final	2.1	3.2	5.0	0.75	0.25	
Ion Exchange		*		0.22		
Overall	0.7	*	5.0	0.97	0.252	0.10

II. Production

A. HAF Processed: 64 Tons

B. Uranium Product:

1. Solvent Extraction

Batch	Gamma Ratio	Pu, ppbp U	Tons	Shipped To
14	1.7	1.7	4.4	P4
15	1.5	1.8	6.5	P4
16	4.1	1.4	7.1	P1
17	5.0		7.1	P1
18	5.2	3.4	7.2	P1
19	5.0	3.3	7.1	P1
20			4.3	P1
P3 K6 E6			5.0	E6
21	3.5	4.75	6.2	P1
22	2.2		7.0	P4
23	1.7	2.0	7.2	P4
24	1.4	1.9	7.7	P4
25	1.7	3.2	6.4	P4
K6 E6			5.6	E6
26	1.7	3.9	6.5	P4
27	1.3		6.5	E6

*Insufficient data because of several "startups" during report period.

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2. Silica Gel

Charge Ratios: Feed   = 3.5
                          Product = 2.5

Tonnage: 45

C. Plutonium Product:

Batch	TrkB/AT x 10 ⁻¹¹	% U in Pu	Fe, ppm Pu	No. Cans	Average Mr./Hr.	Shipped To
9	3.66	0.3		5	4	L7
10	1.5	0.07		4	2	Hold
11	0.53	1.35				L10 L11 B6
12	0.26	2.96				L10 L11 B6
13	0.21	0.3				Hold

D. Overall Waste Loss - % of Total Production

U = 0.31%
 Pu = 1.5%

E. Rework - % of Total Production

U = 23.2% (Uranium product recycles from TK-E6 to HAF Make-Up TK-E6)

Pu = 104% (Primarily out-of-spec. Pu product via TK-L11 to HAF Make-Up TK-E6)

III. Waste Treatment

A. Volumes to UCS:

Source	Gal/Ton	Waste Tank
Wash Solution	346	104
Cell Drainage	210	106
FP Waste	201	106
Coating Waste	270	107C
Chemical Flashes	313	105

B. DW Flows: 3.9 Flows

C. Tank From:

Tank	Est. Boil-off Rate, Gal/Mia
101	3.9
102	3.8
103	0.3
104	4.4
106	7.2

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Notes:

1. The plant was started up at 1600 on April 5 (HAF on) with the processing of "cold" UMH as HAF. Processing of "hot" HAF commenced at 2000 on April 5. Between 1915 on April 7 and 0300 on April 9 the HAF flow was off because of failure of the HAF pump. At about 1130 on April 9, the recycle of "cold" UMH as HAF was commenced. At about 1900 on the same date, the processing of "hot" HAF was resumed. At about 1930 on April 10, the recycle of "cold" UMH was again commenced, in preparation for shutdown of the plant. The plant was shut down (HAF off) at 0130 on April 12. During the entire report period the nominal processing rate was 3.0 CF. However, the actual processing rate was only about 2.3 CF.

2. Flow sheet

- (a) At start-up, the IREU flow was routed to the IREU Tank J3 instead of to the HAD Tank J2. IEO (HCO) was routed direct to the HA Column as HAO. This was done to make the J2-J3 spot available for installation of the J-Cell Neptunium Recovery Package during plant operation.
- (b) Due to a faulty flow recorder-controller, no nitric acid was added to the HAF stream from startup on April 5 until April 9.
- (c) On April 10, the 2DF-ferrous sulfate was increased from 0.39 to 0.49 flows.

3. Feed Preparation

Further analysis of the iodine emission previously reported (p.) confirmed the suspicion that the emission was via the vent off-gas system. The origin of the emission was traced to a routine dissolution in C3 Dissolver on April 2, with the actual emission to the atmosphere resulting from the transfer of a cut from C3 to the Metal Solution Tank J4. From the peak emission of about 23 curies on April 3, the levels gradually declined to near normal by the end of the report period. The total emission for the 10-day period was nearly 50 curies. The maximum emission from the C Dissolver was only a fraction of a curie.

By analysis of process samples, it was concluded that the C Dissolver charge contained "green" metal amounting to one or two buckets "cooled" to only 20 to 30 days. Strontium ratios, iodine levels, and the UMH product also ratios were used in this analysis.

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The C3 Dissolver was taken out of service pending further "cooling" of the particular charge.

4. Solvent Extraction

Performance of the solvent-extraction system continued at about the level of the last report period. However, performance characteristics were quite variable due to start-up and shutdown conditions, coupled with frequent recycle of "cold" uranium as HAF. Also, limitations of the Turbomixer G7 solvent rate reduced the maximum plant uranium processing rate to about 2.3 CF, while other process streams were at 3.0 CF levels. Meanwhile, the uranium loss to the 2EW stream continued on an upward trend, ultimately to about 0.3% at the end of the report period.

Lack of HNO₃ in the HES during the initial portion of the report period (due to instrument failure) plus heavy leakage through the IBB valve during First Cycle shutdowns resulted in excessive uranium in the plutonium product during the report period.

On April 12, the First Cycle, Final Plutonium Cycle, and No. 1 Solvent System were shut down as a special test in the Final Uranium Cycle was initiated in an attempt to determine the effect of feed (2DF) history on performance of the 2D and 2E Columns. The general plan was to recycle "cold" uranium (2DC) back to the 2DF Tank and, after sufficient time to flush the plutonium from the system, shut off the ferrous sulfate flow. Depending on the performance of the columns after several throughputs of "cold" uranium, it was planned to test the effects of various operating and process variables.

5. Final Plutonium Processing

On April 4, it was necessary to suspend operation of the plutonium ion exchange facility for about a shift in order to replace a leaking jumper between the XCP line and the XCP Concentrator H6.

On April 10 and April 11 the uranium specification of the Pu product was exceeded by a factor of six. The increased uranium originated in the partitioning cycle from a lack of acid in the IDCP stream.

6. Solvent Treatment

The 10 (T-G2) and 20 (T-R2) column pulse frequencies were reduced from 60 to 35 frequencies per minute to check effect of pulse on solvent losses.

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Solvent pumps from G7 Turbomixer Tank to the 100 Tank G5 occasionally lost capacity and were limiting the plant production capacity during the report period.

High uranium carbonate wastes from the No. 2 Solvent Treatment System were routinely transferred for storage to TK-P2 during the report period.

I. Waste Treatment and Acid Recovery

Failure of the IMF pump on April 7 caused a plant shutdown which was prolonged to about 14 days because of difficulties in jumper re-installation.

On April 11 inadvertent transfer of recovered acid product from the Formaldehyde Prototype Unit direct to the Absorber Acid Tank F3 caused the plant recovered acid activity to increase by a factor of about 1 ± 10^4 to $\sim 10^6$ u/gal. Sample gallery radiation readings near the Recovered Acid Reader increased 50 to 100 fold; a reading of 25 R/hr. was measured at one point on the piping. At the end of the report period, reprocessing of the recovered acid via the F Cell system was in progress in an attempt to purge the lines and vessels.

As a result of the introduction of water to the tray of the E-F6 tower on March 30 (see p.), the activity in the F3 absorber product decreased 10 fold up to the time of the April 1 shutdown. It is planned to continue this water flow for several days during operation, then shut it off to see if the opposite effect is noted.

The Formaldehyde Prototype was operated commencing on April 10, but the operation was plagued with poor formaldehyde flow control.

Chemical flushing of the Waste Hework Tank F8 continued during the report period.

B. Fission Products

Head End

The numbering system for identifying the strontium runs has been revised. A single number is used to identify a standard run series followed by a letter to designate the type of run (S-sulfate; O-oxalate, and C-concentration) and a final number to designate the number of consecutive runs of that type. For example, the third sulfate run of the current series is 35-S-3.

Approximately 137 kilocuries of strontium-90 were recovered in run series 35 which consisted of five sulfate runs, two oxalate runs and one concentration run. Decontamination factors were 9, > 950, and 140 for cerium, ruthenium, and zirconium-niobium, respectively.

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Cesium-137 analyses are being used to estimate DW concentration (gpl/ton) and the ratio of strontium to cesium-to-estimate age (cooling time). The heat content of DW can be estimated from the concentration and age. Batch size is now set by estimated heat content rather than volume. A stepwise increase in heat load to the centrifuge is under way.

Strontium Semisworks

Peroxide treatment of solutions containing citrate decomposition products was completed. The material that foamed into tank 5 was found to meet specifications and was combined in TK-1 with the rest of the original TK-6 material. Material foamed into TK-2 and TK-44 sump were combined in TK-55. At the end of the report period, 300 kilocuries of specification strontium-90 were available for loadout. An additional 200 kilocuries of out-of-specification strontium was stored in Tanks 55 and 54.

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PROCESS SUMMARY OF FUELS PLANT OPERATION FROM 0000 ON 4/12/62 TO 2400 ON 4/21/62

I. Typical Solvent-Extracting Performance by Cycles

	<u>Gamma AP</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	4.3	4.5	4.5		0.002	0.03
Final	2.4	2.5	9.0	1.05	0.0	
Ion Exchange		1.3		1.2		
Overall	6.7	8.3	9.0	2.25	0.002	0.05

II. Production:

A. HAF Processed: 145 Tons

B. Uranium Product:

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, % of U</u>	<u>Tons</u>	<u>Shipped To</u>
26			5.0	P4
27	1.1	26	5.0	P4
28	2.0	67	7.2	P3
31	3.5	70.5	7.1	P3
32	4.2	67	6.4	P3
33	3.7	37	7.1	P3
34	2.1	3.7	5.0	P4
35	1.6	5.1	7.1	P4
36	1.7	6.0	7.1	P4
37	2.7	5.7	7.1	P1
38	2.9	10.2	7.2	P1
39	3.0	2.6	8.5	P1
40	2.6	9.4	7.3	P1
41	3.0	10.0	7.3	P1
42	2.6	7.7	7.3	P1

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1. Solvent Extraction

<u>Batch</u>	<u>Grain Ratio</u>	<u>Pu, prep U</u>	<u>Tons</u>	<u>Shipped To</u>
43	2.3	6.8	7.1	Pl
44	2.5	7.5	7.1	Pl
45	2.4	7.5	7.2	Pl
46	2.7	5.9	7.3	Pl
47	2.4	4.2	7.2	Pl
48	2.4	7.0	7.4	Pl
49	2.7	3.1	7.6	Pl

2. Silica Gel

Grain Ratios: Feed = 2.3
Product = 1.02

Tonnage: 143

C. Plutonium Product

<u>Batch</u>	<u>ZrNb/AT x 10¹¹</u>	<u>\$ U in Pu</u>	<u>Fa, prep Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
14	0.1			5	1	Hold
15	0.1	0.43	930	2	2	Hold
16	2.6	0.04	0.002	5	4	Hold
17	4.2	0.19		5	5	Hold
18	6.3	0.07		5	5	Hold
19	4.8	0.10		5		Hold
20	2.9	0.12		5	3	Hold
21	1.7	0.047		5	2	Hold
22	1.1	0.3		6	1	Hold
23	0.7	0.07		5	10	Hold
24	0.3	0.00	930	5	11	Hold
25	0.5	0.10	0.002	5	10	Hold
26	0.1	0.09		6	12	Hold

D. Overall Waste Loss - % of Total Production:

U = 0.25%
Pu = 0.50%

E. Rework - % of Total Production:

U = 5.2%
Pu = 7.5%

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III. Waste Treatment

A. Volumes to UOS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	129	104
Cell Drainage	124	106
FP Waste	153	106
ISW	17	106
Chemical Flushes	323	106
Coating Waste	270	107C

B. ISW Flows: 5.5 Flows

C. Tank Pans:

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	3.0
102	3.8
103	0.3
104	4.4
106	9.2

Notes:

1. The plant was started up at 0110 on April 18 at a nominal 3.0 CF. Processing of "hot" HAF was started at 1900 on the same date. On April 20, from 0640 to 1930 the HAF flow was off because of ISF pump failure. The actual HAF processing rate averaged about 2.2 CF.

2. Flowsheet

(a) The start-up flowsheet on April 18 was the same as that in effect at the end of the previous operating period except that:

- (1) The 12.2 M fresh acid to the 20F was replaced with 10.4 M UO_3 Recovered Acid.
- (2) The HAF- HNO_3 was started at 0.75, rather than 0.69 flows.

(b) On April 23, the changeout schedule for the 20F was changed from once every 16 hours to once every 24 hours.

- (c) On April 26, the changeout schedule for the LDW was changed from once every 10 hours to once every 16 hours. At the same time, the LDJ and 203 - Na_2CO_3 concentration was increased from 2.5% to 3%.

3. Feed Preparation

On April 13 a second agitator was installed in the HAF Make-up Tank 26. This agitator is for critical mass control and will reduce the chances of no agitation, through agitator failure, when making transfers of rework in solutions from TK-L11 to TK-26.

Late during the report period the B Cell Silver Reactor differential was becoming excessive.

Uranium metal charged during the report period varied from 150 to 225 dry "cooled", and averaged 170 days. Iodine emissions for the report period totaled 1.34 curies.

4. Solvent Extraction

Solvent-extraction performance continued to be very good in the First Cycle and Final Plutonium Cycle. The problem of uranium contamination in the plutonium stream still existed after startup on April 13, but rapidly dissipated during the report period and was down to a tolerable level by the end of the report period.

The Final Uranium Cycle continued to give poor performance. The decontamination performance of the 2D Column was much lower than normal (DF's averaged ~ 200). The 2E Column continued to give very high waste losses (0.5%, on the average) in spite of high 2EX flows (30 to 50% above flowsheet) and high 2EX temperature (60°C).

At the end of the report period, the decision was made to increase the carbonate concentration to 3% and commence washing the 2EX with carbonate in the 2DF Tank, in addition to the 2D Column in an attempt to remove all of the uranium from the solvent prior to its "clean up" in the column. The 2D Column would then be able to perform its normal function of removing organic degradation products from the solvent.

The process test operation of the Final Uranium Cycle, prior to the April 18 start-up, showed no gain in process performance associated with recycle of 2UD as 2DF and operation without the usual essential materials (ferrous sulfamate and UO_2 Recovered HNO_3). The test operation was conducted for about 60 hours.

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5. Final Plutonium Processing

Following shutdown of normal processing on April 13, rework of out-of-specification plutonium product was commenced with E-Cell ion exchange system. A total of 12 cans was processed, with an overall DP of only 1.5 to 2.0. The apparent low DP probably resulted from contamination of the TK-19 (Pu rework) to TK-11 (XAF Tank) occurring during the recent repetition recovery run.

Startup of the plant on April 15, including the transition from "cold" to "hot" feed, was carried out with the 1BP stream routed directly to E-Cell rather than initially to the L-Cell Package as has been recent normal practice. E-Cell CP readings during the start-up stayed well below operable limits.

On April 23 the first loadout using the new HAPU PA cans was completed. These cans were built on-site because of the sudden need for increase product storage capability. They are not shielded.

Throughout the report period extensive acid flushing of the L-Cell Package was carried on in preparation for inspection and maintenance.

6. Solvent Treatment

Carbonate wastes from the No. 2 Solvent Treatment System were routinely acidified in TK-15 and transferred to TK-F2 for storage during the report period. Changes in the 2DW change-out frequency during the report period were ineffective in changing the level of 2EW losses. Also, operation of both No. 2 system centrifuges in parallel, commencing on April 21, made no apparent change in solvent quality.

7. Waste Treatment and Acid Recovery

During the first part of the report period, the high-activity recovered acid (see p.) was being continuously reworked at a maximum rate of about 3 gal/min, pumped to the IAF Tank F7. By the time of plant start-up on April 15, only slight improvement in Sample Gallery and Vacuum Fractionator radiation readings was noted. Typically, readings were down about a factor of three. Recovered acid samples from Tanks U1 and U2 were difficult to procure because of the excessive sample activity. On samples taken April 15, the fission product content was essentially all ^{239}Pu , at 1×10^6 and 1×10^5 uc/gal for U1 and U2, respectively.

On April 20, failure of the IAF pump in TK-F7 necessitated a 13-hour plant shutdown for replacement. On April 25 a failed IAF pump discharge jumper was replaced without plant shutdown.

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A failed east tube bundle in the 2nd Concentrator FC was replaced on April 17. This bundle had been in service for 5.9 months.

Chemical flushing of the Waste Rework Tank FS continued through the report period. Approximately 18 feet of sludge was observed on the tank bottom, and installation of an agitator was required for slurring this material.

On April 12, 4 - 10 ft. sections of 2-inch thin wall electrical conduit with 2-inch unions welded onto each end were assembled and dropped into Tank 2H-C-103. The opening in the bottom section was restricted to 3/16-in. dia. by welding an orifice over the end of the pipe. The total pipe assembly weighed 63 lbs. which represented a loading of 22.4 lbs/sq. in. on the end surface of the pipe. A check of the dimensions indicated that the end of the pipe dropped directly to the bottom of the tank. The length of the probe in the tank measured 35 ft. 6-1/4 inches. From the behavior of the probe it was concluded that there is no hard sludge in this coating waste tank.

As a double check, hot water $> 80^{\circ}\text{C}$ was pumped through the probe at pressures ranging up to > 60 psig, but no change occurred in the length of pipe inserted in the tank.

The formaldehyde prototype unit was test operated during the report period as follows:

- (a) The unit was operated for a short time using steam sparging in the reactor vessel, with no indication of change in reaction behavior.
- (b) Air flow tests using the sparger air to the reaction pot indicated the tower to be capable of handling greater than 70 cu. ft./min. (The design capacity is 150 to 250 cu. ft./min.)
- (c) Varying the reactor coil pressure from 39 to 60 lb./sq. in. pressure had no apparent effect on the reaction rate.

An apparent transfer of some unreacted formaldehyde was noted during sludge-product processing (neutralization) of a batch of formaldehyde-treated waste. A higher-than-normal temperature increase occurred. Subsequently, operating procedures were changed to specify continuous digestion of formaldehyde-treated LW at 70° to 80°C to insure complete decomposition of any unreacted formaldehyde.

8. Fission Products

Head End

Run series 35, five sulfate runs, two oxalate runs, and one concentration run, was completed. A total of 156 kilocuries of strontium-90 were recovered with decontamination factor of 3.5,

2000, and 130 for cerium, ruthenium, and zirconium-niobium, respectively. The behavior of the centrifuge while processing the largest batch sizes to date was normal in all respects. Efforts to define the maximum head load compatible with the equipment will continue.

Strontium Semivorks

Another HAPD-II Cask was loaded during the report period from the material stored in TK-6 and TK-64. The loading technique was similar to the last loadout (see page) where the strontium was precipitated, decanted through the cask to waste and then slurried to the cask with 0.1 M potassium bicarbonate. Based on ORNL analyses of the previous two casks, the method reduces the sodium content of the strontium cake to less than 1.0 weight percent.

The first filtrate passing through the cask during the decant operation was high in strontium and the pH was 6.9. Additional potassium bicarbonate was added and the filtrate passed through the cask a second time. Strontium losses continued to be excessive and the pH was 7.8. Approximately 7.2 kilocuries of Sr⁹⁰ were reacted with the filtrate to TK-69. A sodium hydroxide butt followed by a potassium bicarbonate butt was made to the solution not yet decanted. The pH increased to around 9 and the strontium losses were reduced to less than 0.5 percent.

The filtrate containing 7.2 kilocuries of strontium was precipitated with bicarbonate in TK-69 and decanted to TK-55. The precipitate was then dissolved in dilute acid and returned to Tank 3 for rework. About 75 percent of the filtrate loss was recovered by this technique. Out of a starting inventory of 310 kilocuries, 170 were loaded into the cask, 2.5 lost to underground storage, 5.5 returned to rework, and 132 left in inventory.

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PROCESS PERFORMANCE OF URANIUM PLANT OPERATION FROM 0000 ON 7/27/52 TO 2400 ON 7/28/52

I. Typical Solvent-Extraction Performance by Cycles

	Gross OF		% Recycle		% Loss	
	U	PI	U	PI	U	PI
First	4.4	4.4			0.002	0.015
Final	2.3	3.4	11	0.3	3.35	
Ion Exchange		1.4		0.2		
Overall	6.7	9.2	11	1.0	0.352	0.015

II. Production

A. NAF Processed: 135 Tons

B. Uranium Product:

1. Solvent Extraction

Batch	Gross Ratio	PI, only U	Time	Shipped To
50	2.3	3.4	7.3	PI
51	2.3	3.5	7.1	PI
52	2.7	3.50	7.0	PI
53	2.3	3.0	7.2	PI
54	2.7	3.3	7.3	PI
55	3.2	2.7	7.2	PI
56	3.2	2.0	7.1	PI
57	2.5	2.0	7.3	PI
58	2.5	1.9	7.2	PI
59	2.5	2.2	7.1	PI
1	2.4	2.45	7.3	PI
2	2.5	1.1	7.2	PI
3	2.2	3.0	7.3	PI
4	2.4	3.9	7.4	PI
5	2.5	4.4	7.4	PI
6	3.1	5.8	7.3	PI
7	3.0	6.3	7.3	PI
8	4.1	6.3	7.4	PI
9	4.1	5.3	7.0	PI

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2. Silica Gel

Gamma Ratio: Feed = 2.8
Product = 1.3

Tonnage: 123

C. Plutonium Product:

Batch	Time/AT x 10 ⁻¹	% U in Pu	Pu, pump Pu	No. Cans	Average Hr./Hr.	Shipped To
27	0.3	0.015		8	10	Hold
28	0.3	0.05		8	9	Hold
29	0.3	0.02	880	8	8	Hold
30	0.7	0.01	882	8	11	Hold
31	0.3	0.11		8	9	Hold
1	0.3	0.01		8	9	Hold
2	0.3	0.01		8	2/11	Hold
3	0.3	0.01		8	12	Hold
4	0.3	0.1		8	9	Hold
5	0.3	0.01		8	8	Hold

D. Overall Waste Loss - % of Total Production:

U = 0.23%
Pu = 0.27%

E. Revers - % of Total Production

U = 1/2
Pu = 6.3% (Pu product revers from TK-L11 to HAF 22 TK-26.)

III. Waste Treatment

A. Volumes to UGS:

Source	Gal/Yr	Waste Tank
Wash Solution	494	104
Cell Drainage	69	106
FP Waste	107	106
Coating Waste	270	101C

B. Int Flows: 6.5 Flows

C. Test Facts:

<u>Task</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	3.7
102	3.0
103	0.3
104	4.1
105	11.1

Notes:

1. The HAP rate was maintained at a nominal 3.0 CF throughout the report period, except for a 4-hour outage from 0130 to 0530 on April 26. The actual processing rate averaged about 2.3 CF.
2. Flowsheet
  - (a) On April 27 water-washing of the 2E1 in the 20F Tank was discontinued and washing with 5% Na_2CO_3 commenced with a partial change-out of the carbonate every four hours, of 270 gallons.
  - (b) On April 28, the IBA, IBS, and IBSP- H_2SO_4 streams were reduced to half-rate, during the short shutdown. Also, during this time, the 2AF flow was shut off and the 2DF flow was maintained by recycling "cold" 2BC from Tank-K6.
  - (c) On April 28, the 2DF Tank temperature recorder was checked and found to be recording 5°C high. The problem was corrected and the 2DF temperature was increased 5° to 60°C .
  - (d) On April 27, the 3AB flow was reduced by about 20 percent. On April 29, it was increased by about 10 percent.
  - (e) On April 30, a portion of the No. 1 Solvent System solvent from the 10 Column was routed to bypass the Turbocriver G7, because of the G7-to-G8 flow restriction. The fraction bypassed varied depending on rate and G7 performance.
  - (f) On April 30, the 20F- Na_2CO_3 change-out schedule was changed from 270 gallons every four hours to 270 gallons every six hours. The schedule was changed back to once every four hours on May 1.
  - (g) On May 2, the HCR- H_2SO_4 was increased from 7.3 to 7.9 flows.
  - (h) On May 2, the 2E1 change schedule was changed from 24 to 30 hours.

3. Feed Preparation

Uranium metal charged during the report period varied from 144 to 297 days "soaked", and averaged 150 days. The total iodine emission for the report period was 0.25 curies.

On May 1, metal solution in TK-14, containing a small fraction of the "green" metal dissolved early in April (see p.), was processed without incident.

4. Solvent Extraction

Solvent-extraction performance showed a continuing, steady improvement in all aspects, except the plutonium and neptunium contamination in the final uranium product. The problem of uranium contamination in the final plutonium product, which plagued the process during the previous two report periods, was overcome and the uranium contamination was down to the normal (< 0.05 percent) level.

Performance of the First Cycle was excellent with respect to waste losses and decontamination. Waste losses averaged less than 0.02%. Use of excessive HAY appeared to be the rule, however. While the product activity was not affected, it raised a possible problem of 3.3 CF operation, if the trend continued.

Performance of the Final Plutonium Cycle was excellent, with "losses" to the Waste Backcycle System averaging less than one percent and DF's showing a steady rise from $\sim 1 \times 10^3$ at the start to 2.7×10^3 at the end of the report period.

The Final Uranium Cycle performance improved somewhat during the report period. The DF increased from about 200 to about 300 and the plutonium and neptunium contamination in the uranium product decreased slightly. The uranium loss via the 2EW Stream improved about two and a half-fold during the report period, decreasing from $\sim 0.8\%$ to $\sim 0.3\%$. The improved performance was attributed to the gradually improved "clean up" in the No. 2 Solvent System.

5. Solvent Treatment

On April 27, the use of a 5% carbonate wash was commenced in TK-R1 for the purpose of removing the uranium, so that the 2D Column could then effectively clean up the solvent. As a result of this change, 2EW losses had decreased about three fold by the end of the report period.

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An attempt was made to eliminate the apparent flow restriction in the Turbomixer G7 pumps to the IO Tank G5 by replacing the pump discharge pumps on April 27. No flow improvement was noted. On April 30, the solvent flow from the IO Column was jumpered to permit partial bypass of the Turbomixer, thereby eliminating this rate restriction.

6. Waste Treatment and Acid Recovery

On April 27, the water flow to the tower trays of the 2M Concentrator F3 was stopped. At the end of the report period, no trend toward increased fission-product activity in the Absorber and product was noted.

On May 1, the original Waste Rework Tank F8 was removed. Replacement was necessary because of coil failure. On May 2, the original HAO Tank J2 was installed as a replacement for the failed F8 tank. The HAO Tank was previously removed from the J2 spot, and will be replaced by the new neptunium recovery package.

7. Fission Products

Feed End

Run series 37 was in progress at the end of the report period. Batch sizes were slightly larger than run series 36, and the centrifuge cake performance continued to be satisfactory. Run results will be reported when completed next week.

Strontium Semitanks

Efforts were under way during the week to rework miscellaneous wastes stored in Tank 54. This waste contains 200 kilocuries of Sr⁹⁰; 140 kilocuries from HLO operations, 55 kilocuries from product which foamed out of product tanks (see page) and 5 kilocuries recovered from a batch of filtrate from cask loading. The composite rework is grossly contaminated with calcium, barium, and manganese and contains moderate amounts of cerium, zirconium-nickel, ruthenium and iron.

An attempt was made to precipitate the strontium with sodium bicarbonate in Tank 54 to obtain a separation from sodium (approximately 4 M) prior to solvent extraction. The precipitation was not successful and the solution was re-acidified.

Organic movements from C-Cell to A-Cell were under way at the end of the report period in preparation for batch contacting in Tank 54.

Two other items of interest that were accomplished during the report period were:

- 1) The HA, HC, and IX Columns were flushed with acid peroxide; however, the IX Column outlet remains plugged.
- 2) The strontium cask was removed from D-Cell to Purex for final decontamination and testing.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 1/4/62 TO 2400 ON 5/10/62

I. Typical Solvent-Extraction Performance by Cycles

	<u>Gamma 4F</u>		<u>5 Recycle</u>		<u>% Loss</u>	
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>
First	4.2	4.2			.002	0.025
Final	2.4	3.0	12.0	1.2	0.04	
Ion Exchange		1.2		0.1		
Overall	6.6	8.6	12.0	1.3	0.042	0.025

II. Production

A. HAZ Processed: 134 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>
10	3.6	3.5	7.3	P1
11	3.7	6.0	7.3	P1
12	3.5	6.9	7.7	P1
13	4.0	7.0	7.3	P1
14	4.2	6.8	7.2	P1
15	4.2	6.7	7.3	P1
16	4.4	6.8	6.9	P1
17	4.0	7.0	7.3	P1
18	3.7	5.3	7.7	P1
19	4.0	3.2	7.4	P1
20	4.1	3.3	7.3	P1
21	4.3	3.7	7.7	P1
22	5.3	4.2	7.3	P1
23	5.7	4.5	7.2	P1
24	4.7	4.5	7.4	P1
25	4.4	1.6	7.2	P1
26	6.7	3.5	7.4	P1
27	3.7	3.5	7.3	P1
28	3.3	3.2	7.3	P1
29	3.4	3.4	7.3	P1

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2. Silica Gel

Gamma Ratios: Feed = 4.2
Product = 1.8

Tonnage: 204

C. Plutonium Product

Batch	Gr/b/AT x 10 ¹¹	g U to Pu	Fe, prep Pu	No. Cases	Average Mr./Mr.	Shipped To
6	0.4	.01		4	10	Hold
7	0.5	.01		5	10	Hold
8	0.8	.01		5	12	Hold
9	1.5	.01		5	15	Hold
10	2.2	.007		4	16	Hold
11	1.4	.005		5	20	Hold
12	1.6	.01		5	17	234-5 (Ret'd)
13	1.0	.005		5	13	Hold
14	1.2	.005		5	12	Hold
15	0.8	.003	900	6	12	Hold

D. Overall Waste Loss - % of Total Production

U = .027%

Pu = .073%

E. Rework - % of Total Production:

U = 4.2 (UWH product recycled from TK-K6 to the HAF Make-Up Tank E6)

Pu = 13.1 (Out-of-spec. Pu product recycle from TK-L11 to the HAF Make-Up Tank E6)

III. Waste Treatment

A. Volumes to WGS:

Source	Gal/Ton	Waste Tank
Wash Solution	483	104
Cell Drainage	43	106
FP Waste	151	106
Coating Waste	270	101C

B. INW Flows: 5.0 Flows

C. Tank Data

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Mia</u>
101	3.7
102	3.6
103	0.3
104	4.1
105	11.1

Notes:

1. The rate was maintained at a nominal 3.0 CF throughout the report period. The actual rate averaged only 2.4 CF, however. Processing of "hot" HAP was interrupted by the recycle of about 5.5 tons of "cold" UMI product on May 5.

2. Flowsheet:

- (a) On May 5, the change schedule for the MgCO_3 in TX-81 was changed from 250 gal. every six hours to 250 gal. every seven hours. Also, the change schedule for the 2DS was increased from 30 hours to 32 hours.
- (b) On May 5, small volume (~100 Gal) of organic was transferred from the 3NF Tank to the IBXF Tank. Another "shot" of ~400 gallons was transferred on May 6.
- (c) On May 10, the 2DF- HNO_3 was increased from 6.3 to 7.3 flows.
- (d) On May 10, the HSH- HNO_3 was increased from 8.5 to 9.0 flows.
- (e) On May 10 increase in the 3/D- HNO_3 concentration was commenced. The concentration had been about 35% low.

3. Feed Preparation

During the report period the plant operating rate was limited by the dissolving operations. The C Dissolver has been out of service due to the "green" metal heel which had previously caused an excessive iodine emission (see p.). The B Dissolver processing rate has been limited by high pressure drop through its silver reactor (see p.). On May 7, the C Dissolver was started up, however, without excessive emission of iodine. Total iodine emitted for May 9 and 10 was 0.31 curies, compared to 0.4 curies for the entire report period.

Average "cooling" time for the metal charged during the report period was 165 days, varying from 132 to 195 days.

4. Solvent Extraction

Solvent-extraction performance continued to be very good with respect to the First Cycle and Final Plutonium Cycle. The uranium loss picture in the Final Uranium Cycle continued to show improvement, with 2EX losses dropping to 0.0%, average, from about 0.2%, average, at the beginning of the report period.

The 1XW losses averaged less than 0.02 percent during the report period. Decontamination performance remained essentially the same as it has been for the last two report periods, except for a brief activity burst which resulted from the transfer of solvent from the 3WF Tank to the 1BWF Tank.

Performance of the Final Plutonium Cycle, continued to be excellent, with "losses" averaging less than 1.0% and product activity averaging about 1×10^{-11} G/AT. The higher activity during this report period was caused by the solvent transfer mentioned above. All product met shipment spec's after an ion-exchange treatment and the activity level was on a downward trend at the end of the report period.

The Final Uranium Cycle continued to exhibit mediocre decontamination performance. All uranium product met silica-gel processing limits, but the plutonium and neptunium contamination remained above nominal.

The plant processing rate remained low during the report period. Since the nominal rate was a CF of 3.0 (all streams, but the feed stream and the 2DX were on 3.0 CF rate) the saturation throughout the Solvent-Extraction System was abnormally low. Also the acidity was generally low and difficult to control. Consequently, at the end of the report period, an effort was made to increase the acidity back to flowsheet values and raise the processing rate, as feed supply allows. While the low saturation and acidity has not resulted in any out-of-specification product, it has made it more difficult to control the process and has allowed little leeway for minor upsets such as the activity burst experienced as a result of the solvent transfer from the 3WF Tank to the 1BWF Tank.

5. Final Plutonium Processing

During the report period, mechanical operation of the E-Cell ion exchange unit has been erratic with push control difficult to maintain, and resultant difficulties in the use of the programmer. In spite of these problems, the final plutonium product has remained excellent.

6. Solvent Treatment

On May 7, the practice of storing carbonate wastes, from the No. 2 Solvent Treatment System, in TK-P2 was terminated. Uranium losses from the 2SW at this time were about 0.02%, compared to losses in the 0.1 to 1.0% range during most of April. The improvement in 2SW losses resulted from the use of a carbonate wash in TK-P1 which commenced on April 27. Approximately 4 tons of uranium in about 10,000 gallons of acidified 2SW was transferred to TK-P2 during the period April 5 to May 7.

On May 4, the addition of "used" Shell S-2342 solvent from TK-40 storage to the No. 2 Solvent Treatment System was commenced. This material represents the entire building solvent inventory which had originally been transferred to TK-P2 in September of 1961 to permit process testing of Solvrol-170 diluent. During the P2 storage period, to March of 1962, the fission-product activity had declined to levels sufficiently low for its use in the No. 2 Solvent Treatment System.

Recent gradual loss of the IDF flow rate indicates probable plugging of the IDF line. During the report period, hot water flushing has been necessary in order to maintain flow.

7. Fission Products

Head-End

Run series 37, five sulfate runs, two oxalate runs and one concentration run was completed. A total of 165 kilocuries of strontium-90 were recovered with decontamination factors of 9.7, 21, and 68 for cerium, ruthenium, and zirconium-niobium, respectively.

Strontium Reworks

The strontium rework solution in TK-54 was processed by batch extraction as follows:

- (1) The aqueous rework was added in Tank 54 to 0.06 M DEPA, 0.35 M acetate, 0.0025 M sulfate and adjusted to a pH of 4.0 with sodium hydroxide. The rework feed was then transferred to Tanks 3 and 5 for temporary storage.
- (2) About 2200 liters of 3SW solvent were transferred from C-Cell to Tank 54.
- (3) Rework feed was added to Tank 54 in 550 liter batches. The two phases were mixed with the pH adjusted by small sodium hydroxide additions until the pH remained constant at 4.2.

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- (4) The phases were separated and the aqueous phase jettied to TK's 73, 55, and 21. This procedure was repeated until all the feed in Tanks 3 and 5 had been processed.
- (5) The organic phase was washed with three 500 liter batches of 0.06 M DTPA, 0.2 M acetate, and 0.001 M sulfate. The pH of each wash solution was adjusted as necessary until the pH remained constant at 4.3.
- (6) The organic phase was stripped with 550 liters of dilute nitric acid, adjusted with incremental acid additions until the pH remained constant at 1.9 to 2.9. Strontium analyses on the strip solution indicated inadequate recovery so the pH was lowered to 1.7. The phases were separated and the aqueous stored in TK-6.
- (7) The organic phase was stripped a second time at a pH of 2.1 and the strip solution stored in Tank 5.
- (8) The organic phase was then washed in TK-54 with 550 liters of 3.0 M nitric acid and the wash discarded to waste.

The process performance of the batch contacting procedure is summarized as follows:

<u>Material Balance</u>		<u>Decontamination Factors</u>
Starting Inventory	300 Kilocuries	Cs 3.4
Losses		Ba 30
A Contacts	19	Mn 45
Organic Phase Washes	2	
Tank Flushes	4	
Return to Rework		
A Contacts	27	
Spill	15	
Strips		
First	130	
Second	3	
Unaccounted	4	

The strontium losses across the extraction contacts were higher than anticipated and surprisingly constant at 20 per cent. Most of the waste losses were stored in Tanks 73, 55 and 21 for further contacting.

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The product quality was excellent except the calcium exceeded specifications by a factor of two. This apparently resulted from too low a pH in the first strip. Analysis of the two strip solutions indicated the first strip solution probably had a pH near 1.4 instead of 1.7.

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FLUORIDE SEPARATION OF URANIUM PLAST OPERATIONS
FROM 0000 ON 3/11/52 TO 2400 ON 3/17/52

I. Typical Solvent-Extraction Performance by Cycles

	Gamma SF		% Recycle		% Loss	
	U	Pl	U	Pl	U	Pl
Fluoride	1.2	1.3			0.002	0.03
Fluoride	2.3	1.3	12.5	1.4	.025	
Ion Exchange		1.3		0.15		
Overall	0.5	0.5	12.5	1.55	.0252	0.03

II. Production

A. RAF Processed: 120 Tons

B. Uranium Product:

1. Solvent Extraction

Batch	Gamma Ratio	Pl, ppm U	Tons	Shipped To
30	3.4	2.7	7.3	Pl
31	3.4	3.2	7.1	Pl
32	3.3	3.9	7.5	Pl
33	3.5	4.1	7.5	Pl
34	3.9	7.1	7.5	Pl
35	4.1	7.5	7.3	Pl
36	4.1	3.5	7.3	Pl
37	4.4	3.0	7.5	Pl
38	4.5	7.0	7.7	Pl
39	4.3	5.3	7.4	Pl
40	3.5	4.5	7.3	Pl
41	3.5	4.5	7.3	Pl
42	3.9	3.4	7.2	Pl
43	3.2	3.4	7.5	Pl
44	2.5	3.4	7.0	Pl
45	3.3	2.4	7.5	Pl
46	3.3	2.5	7.4	Pl
47	3.4	2.5	7.2	Pl
48			7.7	Pl

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2. Silica Gel

Gross Ratios: Fuel   =   3.6
                          Product =   1.6

Formage: 113

C. Plutonium Product

Batch	APXU/AT x 10 ⁻¹	% U in Pu	Pu, prep Pu	No. Cans	Average No./Hr.	Shipped To
16	0.9			4	12	Hold
17	0.9	0.25		4	12	Hold
18	0.7	0.10		6	10	Hold
19	0.9	0.16		4	12	Hold
20	0.4	0.07		6	10	Hold
21	0.4	0.02		5		Hold
22	0.4	0.01		3	14	Hold
23	0.3	0.005		5	13	Hold
24	0.5	0.07		5	8	234-5(2 cans)
25	0.4	0.005		5	10	Hold
26	1.9	0.002		5	1	234-5

D. Overall Waste Loss - % of Total Production:

U = 0.41%

Pu = 0.24%

E. Rework - % of Total Production

U = 3.2% (UHM product from TK-86 to HAF Make-Up TK-26)

Pu = 4.0% (Mostly Pu product via TK-111 to HAF Make-Up Tank 26)

III. Waste Treatment

A. Volumes to UCS:

Source	GAL/Ton	Waste Tank
Wash Solution	397	104
Cell Drainage	102	106
FP Waste	106	106
Conting. Waste	270	101C

B. TX Flows: 4.3 Flows

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C. Tank Turns:

<u>Tank</u>	<u>Est. Roll-off Rate, Gal/Min</u>
101	3.7
102	3.5
103	0.3
104	4.1
105	11.1

Notes:

1. The plant rate was a nominal 3.0 CF throughout the report period, except for an outage from 0915 to 1330 on May 11 due to electrical failure. The actual processing rate averaged only about 2.3 CF due to limitations caused by restricted HOF flow and unavailability of B Dissolver. On May 17, about 4 tons of "cold" UNH was recycled as HAF because of a temporary lack of normal "hot" HAF.
2. Flowsheet
  - (a) The effort to increase the 34B HNO₃ to flowsheet concentration, commenced on May 10, was continued. By May 17 the concentration had been increased from 2.5 to 3.1 lb/gal.
  - (b) Commencing on May 11, the 25X was gradually reduced from 310 to 472 flows. The effort was halted on May 14.
  - (c) On May 12 the HDS change schedule was changed from 24 hours to a half batch every 12 hours.
  - (d) From May 13 to May 16 the HNO₃-HNO₂ was increased.
  - (e) On May 16, the HAD was increased from 64 to 67 flows. It was further increased to 71 flows on May 17.

3. Feed Preparation

During the report period, the B Cell Dissolver was out of service because of high pressure drop through the B silver reactor. Thio-sulfate flushing of the reactor was commenced, to correct this problem. Up to the time it was sent down, the B Cell reactor had processed the off-gases from 147 tons of uranium since its last regeneration on March 24.

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4. Solvent Extraction

Solvent-extraction performance in the First and Final Plutonium Cycles continued to be very good. A slight activity increase in the Final Plutonium Cycle was experienced after the startup from the "crash" shutdown on May 11, but product activity was well within ion-exchange treatment limits. Four catches of product contained higher-than-normal uranium contamination, as a result of the IIS Column leakage to the 2AP Tank during the brief shutdown. However, the four batches were within shipment specifications. After a brief increase following the startup, the HAW losses dropped to less than 0.02% for a couple of days, then commenced an upward trend to an average of 0.03 percent, followed by a downward trend at the end of the report period. The fluctuations were due partly to high entrapment and partly to the continuing problem of maintaining sufficient HNO_3 concentration in the HA Column. Only negligible effect on HSP gamma activity was noted during this period.

The Final Uranium Cycle continued to exhibit mediocre performance, both with respect to decontamination and waste (23W) loss. The decrease in the 22X rate had no apparent effect on the 22W loss.

Toward the end of the report period, neptunium recovery was unusually poor, both because of continued high losses to the uranium product and because of about a five-fold increase in HAW Sp losses. The HAW losses resulted from a decline in HNO_3 concentration in the HAW. Acid control throughout the First Cycle has been poor apparently because of the accumulation of solids in the Recovered Acid Heater system, and consequent poor performance of the flow control systems. In addition the acid addition to the 3XB Tank J1 has been unavailable because high radiation readings have hampered valving this system into service. To correct the low HAW HNO_3 concentrations, the HSE- HNO_3 valve was being opened, toward the end of the report period. No response had yet been noted.

5. Final Plutonium Processing

I-cell operating conditions remained poor during the report period. Pushes were very difficult, requiring pressures of 35 psig and lasting ~30 seconds. For the majority of the report period cycle times have been operated manually rather than by the programmer. The last resin change was made on April 5, and it is considered possible that the resin is the primary source of operating problems.

6. Z Plant Supernate Recycle

During the report period, plans were being made for the routine processing of supernate waste solutions from Z Plant. It was proposed to reprocess supernates containing up to a maximum total of 1000 grams of Pu per HAF batch, and to simplify operational control it was proposed that the number of supernate (SN) cans emptied to each HAF batch be limited to 20. With two recycle stations at Turax -- one in the Sample Gallery and one in the PR Room -- 10 cans containing up to 100 grams of Pu could be processed from each station to each HAF batch in TK-24. In the PR Room, control of the amount of Pu is by sampling each 5-can batch in the 19 Sample Tank. In the Sample Gallery, control of the amount of Pu recycle is specified to be by neutron monitor check of each can. During the report period, a supernate can standard, containing less than 10 g. of Pu, was prepared to be used for routine standardization of the neutron monitor.

7. Solvent Treatment

Decreasing ICF flow, apparently caused by solids in the ICF line, was a serious problem during the report period. On May 15, temporary by-passing of solvent treatment in both TK-G1 (carbonate-permanganate) and in the ID Column (dilute acid) was necessary until hot water flushing of the ICF line could be accomplished.

8. Demineralized Water

Recent chromatographic and infrared studies of used demineralized water filters have shown the presence of unusual organic materials. The compositions of these materials have not been defined; a partial relationship to demineralizer action resin is possible. On the basis of these findings, a more complete laboratory investigation of the Turax process water system will be undertaken.

9. Silica Gel

The silica gel run made during the current report period was terminated after only 6 days of operation because of Br⁸³ breakthrough. This extremely short run period was also unique in that CP readings of the silica-gel drums remained relatively low even after the Br⁸³ breakthrough. Apparently the levels of silica gel in the drums have been reduced by loss of gel, or some channelling is occurring due to other reasons.

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10. Fission ProductsRead-End

Run series 38 was being completed at the end of the report period. Final analytical numbers are not yet available.

Strontium Reworking

Strontium returned to rework last week was combined with miscellaneous tank flushes and concentrated in TK-55. Concentrated waste was prepared for recontacting by making it an additional 0.02 M in DTPA to compensate for any loss to radiolysis; the acetate and sulfate concentrations were believed adequate. The waste was then batch-contacted in TK-54 in exactly the same manner as described last week, except the strontium product solutions, high in calcium, stored in Tank 6 and Tank 5, were used as the strip solutions. This technique permitted decontamination of last week's rework from calcium while stripping this week's TK-55 rework. The first strip was made with Tank 6 solution and the second with Tank 5 solution both at a pH of 2.1. The resulting strontium solutions met all specifications and were combined in Tank 6 for future loadouts. The solvent was washed with 1.0 M nitric acid - 0.5 M oxalic acid solution and again with 1.0 M nitric acid before being returned to solvent storage for reuse.

The process performance of the batch contacting procedure is summarized as follows:

<u>Material Balance</u>		<u>Decontamination Factors</u>	
Starting Inventory	59 kilacuries	Ca	7.6
First Strip Input	100	Ba	20
Second Strip Input	3	Mn	95
		Ce	> 200
 <u>Losses</u>			
A Contacts	2		
Solvent Cleanup	1		
 <u>Strips</u>			
First	102		
Second	26		
Unaccounted	31		

Concurrent with batch contacting operations, a cask was loaded with strontium for The Martin Company. To insure a satisfactory load could be obtained, 10 kilocuries of strontium were transferred from Tank 6 to TK-64. This transfer was made before the Tank 6 material was brought into specifications by the batch contacting procedure described above; however, it blended well within specifications with Tank 64 material. At the end of the report period, the cask was being dried.

Estimated composition of the cask contents was as follows:

<u>Curie Ratios - Isotope/⁹⁰Sr</u>		<u>Gram Ratios - Cation/Total Strontium</u>	
⁹⁰ Sr	0.040 (7/6/62)	Ca	0.19
Ce	1.0×10^{-3}	Ba	< 0.01
Ra	$< 6.0 \times 10^{-4}$	Mn	0.005
ZrO ₂	$< 7.0 \times 10^{-4}$	Pu	< 0.07

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 5/18/62 TO 2400 ON 5/24/62

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	4.3	4.4			0.002	0.02
Final	2.3	3.3	11.5	1.4	0.045	
Ion Exchange		1.1		0.4		
Overall	6.6	8.8	11.5	1.8	0.047	0.02

II. Production

A. HAF Processed: 134 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>
49	4.05		7.10	P1
50	3.42	2.95	7.35	P1
51	3.38	3.1	7.45	P1
52	2.76	1.8	7.45	P1
53	3.0	2.5	7.70	P1
54	3.45	3.3	7.20	P1
55	3.08	2.7	7.35	P1
56	3.59	4.0	7.65	P1
57	3.56	2.2	7.05	P1
58	3.90	3.4	7.40	P1
59	3.81	4.4	7.65	P1
60	4.31	5.7	6.90	P1
61	4.01	6.3	7.70	P1
62	3.68	6.36	5.70	P1
63	4.05	4.5	7.40	P1
64	3.05	5.9	7.60	P1
65	3.1	3.8	7.45	P1
66	3.05	3.9	6.80	P1
67	2.23	4.6	7.80	P1

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<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
68	3.95	4.0	7.55	Pl
69	3.92	3.5	7.40	Pl
70	3.83	2.2	7.75	Pl
71	4.07	3.1	7.20	Pl
72	4.16	2.9	7.15	Pl

2. Silica Gel

Gamma Ratios: Feed = 3.5
Product = 1.7

Tonnage: 134.7

C. Plutonium Product

<u>Batch</u>	<u>ZR-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
27	0.8	0.003		6	1	234-5
28				5	1	234-5
29	0.5	0.003		5	1	Hold
30	2.3	0.002	1160	5	1	234-5(2 Cans)
31	0.7	0.002		5	2	Hold
32	0.6	0.002		5	2	234-5
33	0.4	0.002		5	2	234-5
34	0.6	0.008		6	2	234-5(3 Cans)

D. Overall Waste Loss - % of Total Production

U = 0.2%
Pu = 0.11%

E. Rework - % of Total Production

U = 7.5% (UNH product from TK-K6 to HAF Make-Up TK-E6)

Pu = 10.3% (Z Plant supernate to HAF-MU TK-E6)
0.1% (Sumps to HAF MU TK-E6)

III. Waste Treatment

A. Volumes to UGS:

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
Wash Solution	556	104
Cell Drainage	95	106
FP Waste	169	106
Coating Waste	270	101-C

B. IWW Flows: 5.3

C. Tank Farm: Boil-off Rates

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	3.7
102	3.6
103	0.3
104	4.1
106	11.1

Notes:

1. Plant Operating Information

The processing rate was maintained at a nominal 3.0 CF throughout the report period. Two batches of "cold" UNH product, totalling approximately 10 tons of U, were recycled as HAF, one each on May 19 and May 21, to maintain operating continuity. The actual HAF processing rate averaged about 2.5 CF.

2. Flowsheet

- On May 18, the 2DF-HNO₃ was increased from 7.7 to 8.5 flows.
- On May 20, the 20 Pulse Frequency was increased from 64 to 66 cycles per minute.
- On May 20, the 2BP was reduced from 1.86 to 1.62 flows. It was increased back to 1.86 flows on May 21.
- On May 22, the HAX-NO₂ was increased from 0.63 to 0.69 flows.
- On May 22, the 2DS was increased from 63 to 69 flows. The 2DX rate was also increased to compensate for the increased 2DS flow.
- On May 23, the addition of 0.0005 M mercuric nitrate the initial solution was stopped.

3. Feed Preparation

Thiosulfate flushing of the B-Cell silver reactor was completed during the report period, and regeneration was commenced on May 22. On May 24, regeneration was complete and charging with uranium was commenced.

4. Solvent Extraction

Solvent-extraction performance continued at essentially the same level as during the previous report period. Both HAW Pu and Np losses improved, although the Np losses remained about two fold above normal. The improvement attained resulted from improved control of HAW HNO₃ to the flowsheet value.

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5. Final Pu Concentration

Operation of the N-Cell ion exchange unit during the report period was very erratic, with periods of satisfactory operation followed by periods of essentially complete collapse when the unit would not push for extended intervals. While the resin appeared normal upon visual inspection, the operating characteristics pointed to a possible breakdown in the physical properties of the resin beads.

Considerable difficulty was also experienced in obtaining sufficient steam through the N6 steam supply system. On May 21 the N6 steam trip was replaced. This relieved the immediate problem.

6. Supernate Recycle

On May 19, routine rework of supernate solutions from Z Plant was commenced. Additions of recycle to the HAF Tank E6 were made direct from the Sample Gallery recycle station and via tanks L9 and L11 from the PR Room. The control method was the same as previously outlined (p. .)

A total of 300 cans containing about 6700 grams of Pu was processed during the report period.

7. Waste Treatment and Acid Recovery

Since installation of a smaller formaldehyde flow rotometer, operation of the formaldehyde denitration equipment at low formaldehyde -to-lWW ratios has been under better control. A formaldehyde flow of 50% of the flowsheet values has been maintained.

Fission Product Recovery

Head End

Run series 38, five sulfate run, two oxalate runs and one concentration run was completed. A total of 145 kilocuries of strontium 90 were recovered with decontamination factors of 5.7, 97, and 91 for cerium, nithinium and zirconium-niobium, respectively. Run series 39 is in progress.

Strontium Semiworks

Most of the week was spent preparing for and making training runs. The organic in TK 54 was returned to C-Cell and all A-Cell tanks flushed to rid them of solvent. C-Cell solvent inventory was washed and a sample sent to Process Chemistry for evaluation.

Training runs were started on May 21. All equipment was operated except the HA column feed pumps. They were primed and run only for brief periods to conserve their life.

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Strontium Semiworks (Continued)

Near the end of the report period, preparations for the first hot run were underway including the transfer of the first batch of hot fuel from TK002 in the vault to TK54 in A-Cell.

On May 22 the cask loaded last week was transferred from D-Cell to the Purex loadout building for final tests and decontamination.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 5/25/62 TO 2400 ON 5/31/62

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	4.3	4.3			.002	0.03
Final	2.2	3.4	11.0	2.1	.07	
Ion Exchange		1.2		7		
Overall	6.5	8.9	11.0		0.072	0.03

II. Production

A. HAF Processed: 134 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>
73	4.1	3.4	7.55	Pl
74	4.41	3.5	7.15	Pl
75	4.01	8.3	6.80	Pl
76	3.75	4.5	7.65	Pl
77	3.42	4.4	7.30	Pl
78	3.40	3.9	7.25	Pl
79	2.41	2.8	7.40	Pl
80	2.76	3.9	7.20	Pl
81	3.54	6.5	7.15	Pl
82	4.01	8.0	7.25	Pl
83	3.83	7.9	7.40	Pl
84	4.08	11.2	7.45	Pl
85	4.12	4.8	7.40	Pl
86	3.85	3.5	7.40	Pl
87	3.70	3.1	7.15	Pl
88	4.53		7.30	Pl

2. Silica Gel

Gamma Ratios: Feed = 3.8
Product = 1.7

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Tonnage: 77 Tons

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mz/hr.</u>	<u>Shipped to</u>
35	0.39			5	1	234-5
36	0.79			5	1	234-5
37	0.95	0.003		6	1	234-5
38	0.57	0.001		5	1	234-5
39	0.46	0.002		6	2	234-5
40	0.138	0.005	1330	5	1	234-5
41	0.43	0.005		6	2	234-5
42	0.38	0.003		6	2	234-5
43	0.66	0.002		5	2	234-5
44	0.68	0.003		6	2	234-5

D. Overall Waste Loss - % of Total Production

U = 0.48
Pu = 0.36

E. Rework - % of Total Production

U = 4.15 (Product UNH from TK-K6 to HAF MU Tank E6)
Pu = 13.6% (Z Plant supernate to HAF MU TK-E6)
0.2% (Sumps to HAF MU TK-E6)

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash Solution	641	104
Cell Drainage	38	106
FP Waste	213	106
Coating Waste	270	101-C

B. IWW Flows: 8.2

C. Tank Farm Boil-off Rate

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	3.7
102	3.6
103	0.3
104	4.1
106	11.1

DECLASSIFIEDNotes:1. Plant Operating Information

The processing rate was maintained at a nominal 3.0 CF throughout the report period. On May 26-27, about 5.5 tons of "cold" UNH was recycled as HAF for the purpose of lowering plutonium levels in the plant. The actual processing rate averaged about 2.4 CF.

2. Flowsheet

- a) During the period May 28 through May 30, approximately 1000 gallons of solvent was transferred in small increments from the 3WF Tank F10 to the 1BXF Tank J3. This material had accumulated in TK-F10 during the period of processing water washes from TK-R1 (p.) to TK-F10.
- b) On May 29, carbonate washing of the 2EW in TK-R1 was stopped.
- c) On May 30, the 2EX flow was reduced from 472 to 433 flows.
- d) On May 31, the 2EX temperature was reduced from 60 to 53 degrees C.

3. Solvent Extraction

Solvent extraction performance continued to be generally good during the report period. Both decontamination performance and waste losses continued to be excellent in the First and Final Plutonium Cycles. Decontamination performance of the Final Uranium Cycle continued to be mediocre with respect to neptunium, fission products, and plutonium. However, all uranium product met shipment specifications after processing through the Silica Gel Facility.

On May 28, a new cartridge was installed in the 2E Column, without plant shutdown and during a four hour Final Uranium Cycle outage. The new cartridge contains both stainless steel and plastic plates arranged in three sections of different configurations.

- a) The bottom approximately 6-foot section contains successive groupings of two nozzle plates spaced about three inches apart with a 3/4-inch thick linear polyethylene plate 1/2 inch below. Groupings are spaced at 7-1/2 inches.
- b) The middle approximately 6-foot section contains successive groupings of three nozzle plates spaced about three inches apart with a 3/4-inch thick linear polyethylene plate 1/2 inch below. Groupings are spaced at about 10-1/2 inches.
- c) The upper approximately 5-1/2-foot section contains successive groupings of six nozzle plates spaced about two inches apart with a 3/4-inch thick linear polyethylene plate 1/2 inch below. Groupings are spaced at about 13-1/2 inches.

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The stainless steel nozzle plates have 3/16-inch holes, 0.04 inch minimum nozzle depth pointing up, with 23% free area. The plastic plates are 23% free area with 3/16-inch holes.

Following startup of the Final Uranium Cycle with the new 2E column cartridge, the pulse frequency of the 2E column was increased from 40 to 54 (actual) cycles/min. over the next ten hours and to 58 cycles/min. over the following 12 hours. With the frequency increases, 2EW losses decreased in about a day to nondetectable values. No evidence of column instability was noted during the relatively rapid frequency adjustments. Furthermore, no increases in waste loss were noted to result from a nearly 10 percent 2EX flow reduction on May 30, or from the 2EX temperature reduction from 60 deg. C to 53 deg. C on May 31.

On May 26-27, a single batch (about 5.5 tons) of "cold" UNH from P3 Tank storage was recycled as HAF because of higher-than-normal Pu levels caused by poor operation of the N-Cell ion exchange unit.

4. Final Plutonium Processing

The operation of the N-Cell ion exchange unit continued to be dismal the first five days of the report period. Periods of up to four hours without resin pushing were experienced along with the accompanying higher losses.

On May 31, the entire operating resin inventory of N-Cell was changed without shutdown using a special procedure. Approximately 24 gallons of resin were removed from the resin reservoir and replaced with new resin of the standard mix. The change was made without incident and the resin removed was essentially free of plutonium, allowing its disposal without washing. It is estimated that the actual changeout was only 75% effective due to resin back-mixing and nonplug flow of the resin through the unit.

5. Supernate Recycle

During the report period, the contents of 300 SN cans containing about 11-1/2 kg. of plutonium was reprocessed by addition to the HAF make-up tank E6.

On May 30, a new standard SN can, for standardizing the sample gallery neutron monitor, was prepared. The original standard was determined to contain only about 35 g of Pu, compared to the desired 50 g. The new standard was an actual supernate recycle (SN) can with a verified Pu content of 50 grams.

6. Solvent Treatment

As a result of the decrease in 2EW losses to negligible values, the practice of carbonate washing 2EW in TK-R1 was stopped on May 29.

6. Solvent Treatment (continued)

Continued occasional water flushing of the 20F jumper system was necessary because of loss of 20F flow, apparently due to plugging of the system.

7. Waste Treatment and Acid Recovery

Operation of the Formaldehyde denitration prototype at low formaldehyde-to-IWW flow ratio continued during the report period.

8. Fission Product Recovery

Head End

Run series 39, five sulfate runs, two oxalate runs, and one concentration run was completed. A total of 195 kilocuries of strontium was recovered with decontamination factors of 7, 100, and 60 for cerium, ruthenium, and zirconium-niobium, respectively.

Some difficulty was experienced in removing the oxalate waste cake from the second oxalate run. Several extra cake removal attempts were made but the Beckman reading remained above normal.

During the routine acid flush of TK E3, G-E2, and TK E1, between oxalate and concentration runs, the Beckman readings returned to normal. The centrifuge is overflowed during this flush, indicating the activity was probably in the case instead of the bowl.

Strontium Semiworks

Hot processing was started on May 27 at 0830. Startup was smooth except for flooding problems in the HA column scrub section and high HCW losses. The HA column was stabilized by reducing flows 20 percent and lowering the HA column pulse frequency. The HCW losses were reduced from the range of 30 percent to 5 percent by a 10 percent increase in the HC column L/V and increases in HC column pulse frequency. By May 29 the plant had leveled out and was running smoothly. Decontamination performance was excellent with the product meeting all radiochemical specifications. Calcium and barium DF's cannot be determined until the run is over. Average instantaneous waste losses were 2.0 percent and 5 percent for the HAW and HCW, respectively.

On May 30, the HCP line became partially plugged and most of the flow was routed through the HCP-A line. Since the HCP-A line does not contain a sampler, HCP samples were no longer available. Also, the HCP-A line routes the HCP to TK-6 instead of T-6 so the product was concentrated without steam stripping.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 6/1/62 TO 2400 ON 6/7/62

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	4.2	4.0			.002	0.02
Final	2.4	2.8	10.5	2.3	.002	
Ion Exchange		1.7		0.15		
Overall	6.6	8.5	10.5	2.45	0.004	0.02

II. Production

A. HAF Processed: 151Tons

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	3.2	8.9	7.3	P1
2	3.1		7.5	P1
3	2.9	3.0	7.3	P1
4	3.2	4.0	7.2	P1
5	4.7	5.9	7.2	P1
6	6.4	9.7	8.0	P1
7	7.3	10.9	7.8	P1
8	6.2	7.7	7.0	P1
9	5.6	7.3	7.4	P1
10	5.4	7.0	7.3	P1
11	4.9	8.2	7.1	P1
12	4.5	9.9	6.7	P1
13	4.9	8.1	7.5	P1
14	4.6	10.5	7.1	P1
15	4.7	7.8	7.2	P1
16	4.6	7.4	7.7	P1
17	4.7	6.5	7.5	P1
18	4.3	5.7	7.1	P1
19	3.9	3.0	7.3	P1
20	4.4	5.7	7.4	P1
21	3.9	7.1	7.5	P1

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2. Silica Gel

Gamma Ratios: Feed   = 4.0
                          Product   = 1.8

Tonnage: 87

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr/hr.</u>	<u>Shipped to</u>
1	1.60	0.004		5	1	234-5
2	1.66	0.007		6	2	234-5
3	1.84	.008		5	1	234-5
4	1.47	.007		6	1	234-5
5	1.72	.005		5	1	234-5
6	1.57	.02		5	1	234-5
7	2.1	.003		5	2	234-5
8	2.04	.01		6	3	234-5
9	2.6	.01		6	3	234-5
10	2.42	0.01	1300	5	3	234-5
11	1.82	0.01		6	1	234-5

D. Overall Waste Loss - % of Total Production

Pu = 0.25%
 U = 0.24%

E. Rework - % of Total Production

Pu = 7.9 (Supernate recycle* from 234-5 Bldg. to HAF make-up TK-E6)
 U = Nil

* 352 Cans containing 8099 grams.

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash Solution	537	104
Cell Drainage	45	106
FP Waste	153	106
Coating Waste	270	101-C

B. IWW Flows: 5.6

C. Tank Farm Boil-off Rates

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	3.4
102	3.3
103	0.3
104	3.8
106	11.0

DECLASSIFIEDNotes:1. Plant Operating Information

The processing rate was maintained at a nominal 3.0 CF throughout the report period. The actual HAF processing rate averaged about 2.6 CF, however.

2. Flowsheet:

- a) On June 1, the 2EX was increased from 433 to 510 flows.
- b) On June 1, the 2EX temperature was decreased from 53 to 45 degrees C over a ten-hour period.
- c) On June 1, the HSS-H₂O was reduced from 57 to 49 flows.
- d) On June 2, the 1BXP was increased from 21 to 23 flows. It was reduced back to 21 flows on June 4.
- e) On June 2, the 1BS was increased from 21.7 to 23 flows. It was reduced back to 22 flows on June 6.
- f) On June 2, the 2DF-ferrous sulfamate was increased from 0.39 to 0.93 flows. It was reduced back to 0.39 flows on June 3.
- g) On June 2, the carbonate concentration in the 2OS stream and in the Turbomixer G7 was changed from 5% to 2.5%.
- h) On June 4, the Demister spray water (24 flows) was stopped.
- i) On June 5, the 2DS was increased from 70 to 74 flows.
- j) On June 6, the HA-NaNO₂ was increased from 0.68 to 0.74 flows. It was further increased to 0.80 flows on June 7.
- k) On June 7, the Demister spray water was started at 24 flows.

3. Feed Preparation

On June 2, the regeneration of the A3 Silver Reactor was started. This reactor had processed the off-gases from 757 tons of uranium since the last previous regeneration.

4. Solvent Extraction

Solvent-extraction performance followed recent normal patterns except for a brief activity burst early in the report period which resulted from loss of HA Column saturation during an attempted increase in the processing rate. Prior to the end of the report period, activity levels were again near normal.

Control of the nitric acid concentration in the HA and 2A Columns

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continued to be a problem, due to the malfunction of the flow metering systems on the 3WB and HSR-HNO₃ streams. However, losses were maintained under control (averaging less than 0.03% in the HAW stream and less than 2.0% in the 2AW stream).

A problem of excessive plutonium in the Final Uranium Cycle, caused by reduction of partitioning efficiency, grew steadily worse during the latter part of the report period. As a result, the plutonium contamination in the uranium product stream approached the upper limits for subsequent processing.

Final Uranium Cycle performance was essentially unchanged from the previous report period. The 2E Column continued to exhibit excellent performance, while the performance of the 2D Column continued to be marginal.

Increased Np losses to the HAW commencing on about June 4 continued during the balance of the report period. To correct this trend, the HA-NaNO₂ was increased 15-20% on June 6-7, and the HA Column Uranium saturation was reduced. Recent high losses to both the HAW and 2EU have resulted in negligible Np accumulation in the back cycle system.

5. Final Plutonium Processing

Operation of the N-Cell ion exchange unit following the resin changeout has been excellent with no apparent push problems. A push pressure of 50 lbs. is being used (which is ~ 15 lbs. above the desired value) to insure that the resin keeps moving.

The final product activity has increased slightly reflecting the higher 2BP activity levels. Ion exchange DF has remained excellent, ranging between 25 and 40.

6. Waste Treatment and Acid Recovery

Commencing with the continuous operation of formaldehyde on May 19 the Ru in the F3 recovered acid has shown much greater fluctuations with the overall affect being an increase by a factor of about three. The ZrNb numbers have also increased but these have generally stayed below 200 µc/gal. The Pu activity has reached levels as high as 6×10^3 µc/gal.

While the increased activity is certainly the result of jetting TK-F24 to the 2 WW concentrator F6, no clear-cut correlation between the jetting times and sample results can be determined.

From June 4 to 7, the Demister spray water was not flowing because of a leak in the drain jumper from the Demister thru the 2WW concentrator F6. No effect on the acid product (TK-F3) was noted.

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7. Fission Product Recovery

Head End

Strontium run series 40 was completed, recovering a total of 215 kilocuries of strontium 90 with a decontamination factor of 16 from cerium. This number is based on oxalate product sample because the TK-E1 sampler was out of service and concentration product was not sampled. Ruthenium and zirconium-niobium analyses are not normally run on oxalate product so decontamination factors for these fission products are not available.

The E2 centrifuge thermohm jumper failed during the first oxalates run and the run series was completed without temperature surveillance. A new thermohm jumper was installed before the start of run series 41.

Strontium Semiworks

Run HSW-1 was completed at 1900 on June 1, 1962 without incident. The flowsheet and column operating conditions that evolved during the run are shown in Table I and process performance is summarized in Table II.

TABLE I

Strontium Semiworks Flowsheet Run SSW-1

		<u>Flows*</u>	<u>Liters/Min.</u>
Vault	Crude	80	.
HAF	Butt	20	
HAF		100	3.30
HAS		10.6	0.35
HAX		27	0.89
HGX		8.2	0.27

* Stream compositions are shown in HW-73761

Column Operating Conditions

	<u>Pulse</u>	<u>Amplitude</u>	<u>Pulse Frequency</u>
HA		0.80 inches	48
HC		1.0 inches	43

TABLE II

Process Performance RUN SSW-1

Material Balance

Feed	620	Kilocuries	Sr-90
HAW (waste)	13	"	"

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TABLE II (Continued)

Process Performance RUN SSW-1

Material Balance

HCW (to rework)	57	Kilocuries	Sr-90
Product	550	"	"
Total	620	620	" "
Unaccounted	0	"	"

Waste Losses - Percent

	<u>Instantaneous</u>	<u>Average</u>
HAW	0.4 - 3.0	2.0
HCW	4.0 - 30.0	9.0

Decontamination Factors

	<u>HA Column</u>	<u>HC Column</u>	<u>Overall</u>
ZrNb	> 3000	>30	>100,000
Ru	>90	>50	>4,500
Ce	390	50	20,000
Ba	-	-	330
Ca	-	-	25
Fe	-	-	>1,000

Product Quality

<u>Curie Ratio</u> <u>Isotope to Sr 90</u>	<u>Run SSW-1</u>	<u>Specifications</u>
Ce	5.8×10^{-5}	$< 3.0 \times 10^{-3}$
Ru	$< 5.8 \times 10^{-5}$	$< 6.0 \times 10^{-4}$
ZrNb	$< 3.5 \times 10^{-5}$	$< 8.0 \times 10^{-4}$
<u>Percent Inerts</u> Ca+Ba+Fe/Sr+Ca+Ba+Fe	2.2	<25.0

The HCW loss (57 KC) was recovered in TK-21 by batch washing with 2.0 M HNO₃. The strontium will be reworked at a later date.

After completing the solvent extraction portion of the run TK-6 was made 2.0 M HNO₃. During the HNO₃ acid addition moderate gassing of the concentrated HCP stream was noted. This was unexpected since the solution had been made 1.0 M HNO₃ before the run was started. After the addition of the nitric acid 3.5 moles of H₂O₂ was added per mole of citrate while boiling the solution in order to destroy the citrate acid. During this operation the specific gravity of the solution decreased from 1.270 to 1.025.

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Laboratory work with similar solutions (cold) indicated that 10 moles of H_2O_2 per mole of citrate was required to destroy the citrate, and that nitric acid was not destroyed in the process. The plant solution, however, apparently required only 3.5 moles of H_2O_2 per mole of citrate for destruction of the citrate ion. In addition, about 90% of the nitric acid was also destroyed.

An excellent $SrCO_3$ precipitate on a laboratory scale was obtained from the plant solution, but the supernatant solution appeared milky. Analytical results, however, showed that virtually no strontium was present in the supernatant solution. Since this solution is to be stored in TK-1 which does not have a sampler, the pH of the solution was reduced from 1.1 to 0 with nitric acid and sufficient H_2O_2 added to the solution to increase the H_2O_2 to citrate ratio from 3.5 to 5.0. During this last nitric acid addition, it was noted that the solution was still moderately buffered.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 6/8/62 TO 2400 ON 6/14/62

1. 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	4.4	4.1			4.002	0.03
Final	2.3	2.8	12.5	*2.8	4.002	
Ion Exchange		1.6		.0.07		
Overall	6.7	8.5	12.5	*2.9		0.03

II. Production

A. HAF Processed: 166 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>
22	2.84	9.0	7.20	P1
23	2.73		7.20	P1
24	3.04	15.7	7.50	P3
25	3.08		6.05	P3
26	2.90	8.6	6.95	P1
27	3.15	9.2	7.35	P1
28	2.70	5.5	7.45	P1
29	2.60	5.6	7.50	P1
30	2.27	4	7.00	P1
31	2.19	2	7.60	P4
32	2.09	3.0	7.60	P4
33	2.14	3.5	7.15	P4
34	2.60	3.2	7.65	P1
35	3.59	3.6	7.50	P1
36	3.95	3.4	7.25	P1
37	4.48	3.5	7.55	P1
38	3.61	3.7	6.85	P1
39	3.36	3.4	7.40	P1
40	3.28	2.4	7.75	P1

*Insufficient data

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
41	2.95		7.90	P1
42	2.45	3.6	7.30	P1
43	2.94		7.70	P1
44	2.67		7.90	P1

2. Silica Gel

Gamma Ratios: Feed = 3.0
Product = 1.75

Tonnage: 121 T

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
11 (cont.)	1.82	0.01		1	1	234-5
12	1.22	0.01		7	3	234-5
13	1.45	0.01		6	2	234-5
14	1.25	0.01		6	1	234-5
15	1.39	0.01		3	2	234-5
16	1.39	0.01		5	1	234-5
17	1.80	0.02		6	2	234-5
18		0.01		6	1	Hold
19				6	2	Hold
20	2.36	0.005		5	2	234-5
21	2.15			5	3	234-5

D. Overall Waste Loss - % of Total Production

U = 0.23

Pu = 0.33

E. Rework - % of Total Production

U = Nil

Pu = 1.9% - L and N Cell sump to HAF Make-up TK-E6.

= 4.9% - 118 SN cans containing ~4,400 g.

III. Waste TreatmentA. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash Solution	457	104
Cell Drainage	71	106
FP Waste	100	106
Coating Waste		101-C

B. 1WW Flows: 4.8

C. Tank Farm: Boil-off Rates

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	3.4
102	3.3
103	0.3
104	3.8
106	11.0

Notes:

1. Plant Operating Information

The HAF rate was maintained at a nominal 3.0 (actual, 2.8) CF. rate during the report period.

2. Flowsheet

- (a) On June 10, the 20F - ferrous sulfamate was decreased from 0.58 to 0.39 flows.
- (b) On June 10 and 11, the 20S was increased from 74 to 78 flows.
- (c) On June 14, the IOS flow was changed from normal "once-through" to 100% recycle, with a batch change frequency of 24 hours.

3. Solvent Extraction

Overall solvent extraction performance continued at recent normal levels. Control of recovered nitric acid flows continued to be a problem, resulting in fluctuating 2AW "losses" and occasional erratic DF performance. Two batches of Pu product with excessive gamma activity content resulted from this control problem. Occasions of moderate instability in the IBX and 1C Columns may also have been caused by fluctuating acidities, although the process water quality was also suspect.

Early during the report period, high IBU losses previously reported (see p.) were found to have been caused by an undetected downward drift of the IBX Column pulse frequency. The frequency had drifted from a normal 55 cycles/min. to 38 cycles/min. with a return to 55 cycles/min., the IBU losses declined.

Performance of the 2F Column continued to be excellent.

4. Final Plutonium Processing

Operation of the N-cell ion exchange unit during the report period was generally good, with the gamma DF remaining at ~30 during the report period.

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An increase in the plugging frequency of the XAF filters coupled with a slowly falling resin level in the unit indicated that resin is slowly being lost from the system with at least a portion of it going to the XAF Tank N1, (via the vent system).

5. Solvent Treatment

Solvent treatment wash wastes were reduced on a temporary basis to allow high-level waste storage tanks to self-boil to a lower liquid level. Changes made were:

- (a) The IOBW (carbonate) in the TK-G1 contactor was reduced by 20%.
- (b) The IOSW (acid) in the IOS Tank G2 was reduced 75% by recycling the stream.
- (c) The 20SW (carbonate) in the 20S Tank R2 was reduced about 30%.

6. Silica Gel

Recent silica gel problems of obtaining good DF at normal throughputs between regenerations continued during the report period. Process chemistry studies showed the same poor DF response in the laboratory, with moderate improvement resulting from acidity increase. On the other hand, Purex UNH was processed through the Redox silica gel unit with no unusual behavior whatsoever.

During the report period the TK-P1 silica gel feed UNH was butted with HNO_3 to raise the nitric concentration from 0.18 M to ~ 0.35 M. Subsequent TK-K6 nitric butts were also increased by 70% to raise the acid concentration to 0.35 M.

In an effort to obtain better DF, an attempt was made to run the Purex silica gel units in series (normal downflow through B-line and then upflow through A-line). This attempt was halted when the effluent actually increased in gamma ratio, apparently from crud loosened by the backwashing action through the A-line.

7. Waste Treatment

Operation of the LWW denitration equipment at 50% normal formaldehyde flow continued with no significant pressure surges.

Due to high liquid levels in the 241-A tank farm underground storage tanks, solvent treatment flowsheet changes were made as described under Note 5, above.

During the report period the ZrNb activity in the Acid Absorber product (TK-F3) increased from ~ 100 $\mu\text{c/gal}$ to ~ 1000 $\mu\text{c/gal}$ while the Ru remained at a moderately high level of between 2000 to 5000 $\mu\text{c/gal}$. The effects of formaldehyde operation and the jetting of TK-F24 to E-F6 through 2WW concentrator has not been clearly defined, but overall F3 activity levels have shown much more erratic behavior since the prototype formaldehyde unit has been operating.

8. Fission Product Recovery

Run series 41 was in progress during the report period. Some difficulty was experienced with the centrifuge during run 41-S-5. The machine would not restart for about an hour after braking from slow speed. The subsequent cake removal was difficult. Special chemical flushes with 5M NaOH-1M Na₂CO₃, 57 per cent nitric acid and 5M NaOH were required to reduce the centrifuge Beckman reading to background.

Strontium Semiworks

The second hot processing run (SSW-2) was started on June 9 at 0930. Startup was very smooth with all equipment operating normally. The startup flowsheet was the same as used during run SSW-1 except for minor changes to accommodate the new vault crude, and hydrogen peroxide was added continuously to TK6 after the first day of operation.

Process waste losses were good but decontamination performance was poor. HCP cerium values were 3 to 4 fold above specifications and HAW pH values were around 4.3 while HAF pH values were 4.9. The low HAW pH was at first believed due to high acid in the HAX. Solvent samples, however, showed the HAX acidity was normal.

In an effort to improve the cerium DF, the pH of the HAF was gradually increased from 4.8 to 5.5 which increased the HAW pH to 4.9. Cerium values in the HCP gradually decreased and by June 12 the HCP met specifications.

Other items of interest were:

- (1) The HCP line became partially plugged and most of the flow was routed via the HCP-A line, however, contrary to the experience in run SSW-1, the HCP samples continue to work.
- (2) TK6 continuous citrate kill with peroxide is apparently successful. TK6 specific gravity stopped increasing when the peroxide flow was started.
- (3) No change in process performance was noted when the HAX was switched to recycled solvent.

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Series A.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 6/15/62 TO 2400 ON 6/21/62

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	4.5	3.9			<0.002	0.03
Final	2.3	3.0	10.0	1.7	<0.002	
Ion Exchange		1.6		0.07		
Overall	6.8	8.5	10.0	1.8	<0.004	0.03

II. Production

A. HAF Processed: 157 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>
45	2.50			P1
46	2.81	5.4	7.20	P1
47	2.36		7.30	P1
48	2.23	3.6	7.35	P1
49			7.85	P1
50	2.52	2.1	7.75	P1
51	2.75		7.25	P1
52	2.79	1.4	7.75	P1
53	2.67		7.60	P1
54	2.26	1.7	7.85	P1
55	2.57		7.75	P1
56	2.49	1.7	7.50	P1
57	2.11		7.20	P1
58	2.51	1.4	7.80	P1
59	2.41		7.40	P1
60	2.15	1.4	7.40	P1

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1. Solvent Extraction (Continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
61	2.41		7.80	P1
62	2.84	0.95	7.70	P1
63	1.92		7.35	P1
64	1.91	1.80	7.30	P4
65	2.11		7.70	P4
66	2.70	1.70	0.70	P1
67	2.40		6.55	P1
68	2.68	6.0	7.50	P1

2. Silica Gel

Gamma Ratios: Feed   =   2.3
                               Product =   1.7

Tonnage: 129

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
22	1.40	0.01		6	2	234-5
23	1.20			6	1	234-5
24	1.50	0.015		6		234-5
25	1.55	0.01		6	1	234-5
26				5	5	234-5
27	1.34	0.005		5	3	234-5 (4 Cans)
28	1.77			6	2	234-5 (2 Cans)
29	1.76	<0.01		6	3	234-5
30	1.55	<0.01		6	2	234-5
31	1.67	0.01		5	3	234-5

D. Overall Waste Loss - % of Total Production

U - 0.22   Pu - 0.25

E. Rework - % of Total Production

U - 3.7% - "cold" UNH product to HAF Make-Up E6 in preparation for shutdown.

Pu - 1.8% - 197 SN Cans containing 1685 g.

1.1% - I-Cell sumps 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 TK</u>
	203	104
Wash Solution	21	106

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III. Waste Treatment (Continued)

A. Volumes of UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Cell Drainage	17	106
FP Waste	53	106
Coating Waste	270	101-C
IWW	25	106

B. IWW Flows: 3.6

C. Tank Farm Boil-off Rates

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	3.4
102	3.3
103	0.3
104	3.8
106	11.0

Notes:

1. Plant Operating Information

The HAF rate was maintained at a nominal 3.0 CF throughout the report period. The actual processing rate averaged 2.7 CF. On June 21, at 2215, the recycle of "cold" UNH as HAF was commenced in preparation for plant shutdown..

2. Flowsheet

- (a) During a 2-day period beginning June 17, the 1CX was reduced from 450 to 400 flows.
- (b) The addition of 1 ppm Na_2SO_3 to the demineralizer feed water was stopped on June 19.
- (c) On June 21, the XAW-XSW flow was re-routed from the 3WB Tank J1 to the 3WF Tank F10 in preparation for plant shutdown.

3. Solvent Extraction

Solvent extraction performance was generally good during the report period, in spite of the continuing problems of: a) maloperation and/or plugging of the flow and stream systems to the HA and HS Columns; b) poor decontamination performance of the Final Uranium Cycle; and c) a rapidly increasing plugging problem in the First Organic Treatment System.

Good decontamination performance coupled with low losses ($\sim 0.02\%$, average) continued to be exhibited in the First Cycle, but the flow

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control problems demanded much more than normal attention to maintain control. Decontamination performance of the Final Plutonium Cycle was somewhat lower than has been characteristic of recent months (~1000 v.s. ~2000 DF.), but the product continued to meet shipping specifications after treatment by anion exchange. The brief burst of activity which occurred at the end of the last report period was brought rapidly under control. The 2AW "loss" to the Backcycle System decreased considerably during the report period, from ~4 percent at the start to ~1.5 percent at shutdown.

4. Final Plutonium Processing

Operation of the N-cell ion exchange system continued good with the DF increasing to the 35 to 40 range. The slow loss of resin from the system continued with slightly over 2 gallons of resin being added during the report period to maintain operating level. Repeated cleaning of the XAF filters was also required.

5. Solvent Treatment

The solvent pumps from the G7 Turbomixer to the I00 Tank G5 continued to deliver low flow, necessitating diversion of a portion of the I0 Column solvent flow directly to TK-G5. Flow delivery measurements showed 25.5 gal/min. and 9.6 gal/min. for the G7-1 pump and the G7-2 pump, respectively. The two pumps together delivered only 35 gallons per minute, only about 60% of the total I00 flow.

Flow restriction in the I0F system required water flushing of this system during the report period in order to maintain operation.

On June 18, a series of 5 samples were taken of the acidified solvent wastes (from No. 2 System) stored in TK-P2. The samples were taken at different tank levels varying from 6 inches above the tank bottom up to the liquid surface. The liquid depth is 289.6 inches, equivalent to about 86,128 gallons.

No organic phase was found in any of the samples, and analytical results were consistent, as follows:

Uranium - 0.11 lb./gal.
HNO₃ - 0.29 lb./gal.
Sp. G. - 1.049

6. Silica Gel Processing

During the report period some improvement was noted in performance of the silica-gel unit.

A trend for improved decontamination performance permitted an approximately 20 percent increase in uranium tonnage processed between regenerations. The improved performance resulted from the increase in feed acidity previously reported (see p.). Meanwhile, Redox Plant reported routine

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silica gel performance while processing the same vintage Purex UNH. For the period June 14-16, Redox data for the silica-gelled product were:

GR	- 0.7 to 1.25
Zr-Nb, uc/gal	- 3 to 7
Pu, uc/gal	- ~25

Apparently the Purex units have been fouled (for example, during regeneration) and/or have been channelling. Radiation readings of the Purex silica gel drums reflect this likelihood, also, since they have recently been abnormally low at the times that regeneration has been necessary.

7. Demineralized Water

Beginning in May, the filters (Fulflo fiberglass) in the demineralized water supply line to 202-A Building have shown an increased tendency for plugging. Past experience has shown that a normal change time for the filter bank is about every four weeks, but the past few changes have been made about every two weeks. The criterion for changing filters is the pressure drop across the entire filter assembly.

As previously reported (p.), Chemical Research analyses of the filtered material has shown the presence of organics. More recently, Process Chemistry has discovered the presence of impurities in the Purex process water by evaporating large volume samples. The impurities are revealed by a distinct brown color in the concentrate, a characteristic odor, and presence of "suds" when the sample is shaken. In addition, contacting a relatively large volume of the process water through a stagnant solvent phase (30% TBP in Soltrol-170) results in gradual accumulation of distinct grayish, filing interface cruds. More work is underway aimed at characterizing and identifying the substances causing this behavior.

Samples of both anion units taken on May 28 reveal that the anion bed efficiencies are very poor. After 128,000 gallons (~one-half of a run) approximately 60% of the influent sulfate is being passed and about 5% of the chloride. At 273,000 gallons (nearly one full run) 75% of the sulfate is leaking through and 10% of the chloride.

The amount of total dissolved solids found in the anion effluents is also abnormally high. Past results indicate less than 1 ppm while two current anion water samples averaged 20 ppm.

On June 19, the addition of Na_2SO_3 to the demineralized feed water was stopped on the theory that this chemical may be contributing to the Purex water problem by degradation of demineralizer resin.

8. Fission Product RecoveryHead End

Strontium run series 41, five sulfate runs, two oxalate runs, and one concentration run was completed. A total of 220 kilocuries of strontium 90 were recovered with decontamination factors of 6.5, 130, and 170 for cerium, ruthenium, and zirconium-niobium, respectively. Run series 42 is in progress.

Strontium Semiworks

Run SSW-2 was completed and shutdown on June 16 without incident. The HA column was emptied and flushed with HAS and water. Because of the product in TK6, the HC column was left full. All organic washes were concentrated in TK55 and held for rework.

Process performance for this run are shown in the following tables:

Process Performance Run SSW-2Material Balance - Kilocuries Sr90

Feed	975	
HAW (waste)		20
HCW (to Rework)		70
Spills		4
Product		<u>905</u>
Total	975	1,000
Overaccounted		25

Waste Losses - PercentInstantaneous   Average

HAW	1.75 - 2.25	2.0
HCW	5.0 - 10.0	7.5

Decontamination FactorsOverall

ZrNb	8000
Ru	>750
Ce	1650
Ba	20
Ca	20
Fe	>1000

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Product Quality

Curie Ratio
Isotope to Sr90

Ce
Ru
ZrNb

Run SSW-2

2.0×10^{-3}
 $<6.2 \times 10^{-5}$
 $<8.0 \times 10^{-4}$

Specification

3.0×10^{-3}
 6.0×10^{-4}
 8.0×10^{-4}

Percent Inerts

Ca + Ba + Fe/Sr + Ca + Ba + Fe 2.0

<25

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 6/22/62 TO 2400 ON 6/28/62

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						
Final						
Ion Exchange						
Overall						

Insufficient Data

II. Production

A. HAF Processed: 68 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>
68	2.7	6	7.50	P-1
69	2.8	-	6.90	P-1
70	3.1	7.07	5.45	P-1
71	4.4	13.6	3.90	P-3
72	4.0	19.9	5.65	P-3
73	2.9	16.9	6.35	P-3
74	2.4	10.6	7.35	P-1
75	1.9	5.6	7.10	P-1
76	1.5	-	7.80	P-1
77	1.4	3.7	7.60	P-4
78	1.0	2.2	3.50	P-4
79	1.0	1.8	7.40	P-4
80	1.3	-	7.0	P-4

*Insufficient typical data during report period.

2. Silica Gel

Gamma Ratios: Feed = 2.1
Product = 1.2

Tonnage: 144

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>%U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
32	1.5	< .001		6	3	234-5
33	10.6	1.14	1.27	5	12/4	Hold
34	12.1	0.90		4	19/13	Hold
35	8.5	0.64		6	3	Hold
36	7.5	0.43		5	9/5	Hold

D. Overall Waste Loss -% of Total Production

U = 0.68%   Pu = 0.58%

E. Rework -% of Total Production

U = 33.9% (Out-of-spec. UNH product to HAF Make-up TK-E6)
Pu = 15.6% (Supernate recycle from 234-5 Building to HAF Make-up TK-E6.)
0.5% (Sumps 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	391/1200	104/106
Cell Drainage	270	106
FP Waste	244	106
Coating Waste	270	101-C

B. LWW Flows: 3.2

C. Tank Farm Boil-off Rates

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min.</u>
101	3.4
102	3.3
103	0.3
104	3.8
106	11.0

Notes:

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1. Plant Operating Information

The plant was shut down during the first part of the report period, principally for flushing of the No. 1 Solvent Treatment vessels and in preparation for flowsheet changes in the No. 2 Solvent Treatment System, as described under Note 2, below:

The plant was started up (HAF on) at 3.0 CF at 0900 on June 25, but was shut down at 1400 on the same day because of inadvertent flooding of K Cell via the cell spray-down system. The plant was again started up (HAF on) at a normal 3.0 CF at 2120 on June 25. Processing of "hot" HAF was commenced at 2315 on the same date and was continued throughout the report period. The actual processing rate averaged about 2.7 CF.

2. Flowsheet

- (a) The plant was started up under essentially the same flowsheet conditions that existed at the prior shutdown, except for changes in the Solvent Treatment Systems resulting from a change to permanganate-carbonate washing in the No. 2 System. The changes are summarized in the following table. (Also, see Note 5, "Solvent Treatment", below.)

<u>Solvent Treatment System</u>	<u>Stream</u>	<u>Description</u>	<u>At June 22 Shutdown</u>		<u>At June 25 Start-up</u>	
			<u>Composition</u>	<u>Flows</u>	<u>Composition</u>	<u>Flows</u>
No. 1	10B	Batch to TK-G1	2.5% Na ₂ CO ₃	7.2	0.77% KMnO ₄ 2.5% Na ₂ CO ₃	10.5
No. 1	10B-KMnO ₄	Continuous to TK-G1	4% KMnO ₄	1.6	-	None
No. 1	10S-H ₂ O	Continuous to TK-G2	H ₂ O	34.1		
No. 1	10S-HNO ₃	Continuous to TK-G2	57% HNO ₃	0.5		
No. 1	10C-Na ₂ CO ₃	Continuous to Turbomixer G7	2.5% Na ₂ CO ₃	7.6	1% NaOH	7.6
No. 2	20B	Batch to TK-R1	-	None	2.5% Na ₂ CO ₃ 0.77% KMnO ₄	5.9
No. 2	20S		2.5% Na ₂ CO ₃	7.0	5% Na ₂ CO ₃	4.5

(b) On June 28, the 1CX rate was reduced from 402 to 386 flows.

(c) On June 28, the 2EX rate was reduced from 510 to 490 flows.

3. Solvent Extraction

Start-up of the solvent extraction system was complicated by (a) a severe burst of activity in the Final Plutonium Cycle caused by leaking of the LBP valve during the prior shutdown period, and (b) temporary shutdown of the HA - HS Column system nearly immediately following initial start-up due to inadvertent flooding of K Cell with the cell sprays. Initial First Cycle performance was substandard, particularly with respect to HAW waste losses. Plutonium losses were about 4 fold above normal; Np losses averaged about 50 percent of virgin Np feed.

Immediately following start-up, performance of the Final Uranium Cycle showed marked improvement, resulting from the use of permanganate washing of solvent in the No. 2 Solvent Treatment System. In particular the gamma and Np content of the uranium produce declined sharply. Also, stability of the 2D Column was improved and the 2D Column pulse frequency was increased from 43 to 65 cycles/min. by the end of the report period. The Np losses to the uranium product had declined from approximately 35-40% to less than 5%. The uranium product was within specifications with respect to gamma activity without benefit of silica-gel processing.

4. Final Plutonium Processing

Following the plant shutdown on June 22, the N-cell ion exchange columns were stripped and a standard resin change was made. The resin had given no hint of trouble during its 25 days of service, but the resin change was considered advisable in view of plans for an extended operating period.

During the shutdown, the XAF TK-N1 was flushed clean, and an estimated 5 gallons of resin heavily loaded with Pu was recovered. Inspection of the top hat screen of the resin receiver showed no breaks, and unless resin was being pushed through the screen by high operating pressure (50 psig push pressure) an easy explanation for the resin in TK-N1 is not available.

Operation of the N-cell ion exchange unit was good following start-up, but the Pu produce exceeded specifications due to 2BP gamma levels of about five times normal.

5. Solvent Treatment

Prior to plant start-up, equipment pieces in both solvent treatment systems were chemically flushed. In the No. 1 Solvent Treatment System, standard oxalic-HNO₃ flushes were used to clear TK-G1 and the IOF line of solids. As a result of these flushes, the TK-G1 solids hold-up was reduced from greater than 500 gallons to about 75 gallons, and the IOF flow problem (see p.) was corrected. In the No. 2 Solvent Treatment System,

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carbonate-permanganate flushing was used to prepare the vessels for routine use of this system for solvent washing in TK-R1.

Start-up flowsheet changes (see 2. Flowsheet, above) featured the use of 2.5% Na_2CO_3 -0.77% KMnO_4 as a solvent wash in TK-R1. For convenience, the combined make-up is batched to both TK-R1 and TK-G1, (No. 1 System) from Aqueous Make-up facilities. This means that a permanganate-free carbonate stream is no longer available from Aqueous Make-up; thus, the Turbomixer G7 aqueous phase has been changed from 2.5% carbonate to 1% NaOH . Initially the use of carbonate has been retained in the 20 Column; again for convenience, a 5% make-up is being used.

Initial operation of the TK-R1 washes was plagued with excessive carry-over of the R1 aqueous phase to the 20 Column. On June 27, reversal of the electrical leads to the R1 recirculation pump apparently reduced the degree of agitation in the vessel, thereby, stopping the excessive carryover. On June 28, the G1 recirculation pump leads were also reversed because of excessive carryover from that vessel to the IO Column.

6. Silica Gel

Operation of silica-gel following the June 19-20 regeneration has been much improved over recent runs. After eight days operation (~200 Tons) the unit was still producing uranium with gamma ratio under 1.5, but because of lack of feed, operation was stopped and the units were again regenerated.

7. Fission Products Recovery

Head End

Strontium run series 42 consisting of five sulfate runs, two oxalate runs, and one concentration run was completed. Approximately 220 kilocuries of strontium 90 were recovered with a decontamination factor of 9.0 from cerium. Ruthenium and zirconium-niobium decontamination factors are not available because the TK-E1 sampler has been removed from service to permit installation of an incline gamma spectrometer.

A 200 gallon HLO cask was loaded with 1WW on June 27 and shipped to the 325A Building. This material is to be used for waste calcination studies.

Strontium Semiworks

The 127 kilocuries of strontium lost via the HCW during runs SSW-1 and SSW-2 were concentrated in TK55 and reworked by batch contacting in TK21. The recovered product consisted of three equal volume strips of the organic. All three strips met the radiochemical specifications but only the first strip met the inert specification. The large inventory in TK6 permitted blending the first two strips with TK6 and the third strip was set aside for blending with HAF during run SSW-3.

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Strontium Semiworks (continued)

The disposition of the 127 kilocuries of rework was as follows:

Starting Rework Inventory	127 Kilocuries
Recovered and combined with TK6 product	113
Returned to rework	11
Sent to waste	3

TK6 product composition after adding the rework is estimated to be:

Cation to Strontium
Ratio

Ca	0.049
Ba	< 0.004
Fe	0.003

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 6/29/62 TO 2400 ON 7/5/62

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	4.3	3.9			.002	.03
Final	2.8	2.9	12.0	5.4	.002	
Ion Exchange		1.2		0.10		
Overall	7.1	8.0	12.0	5.5	.004	.03

II. Production

A. HAF Processed: 143 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>
80 A	1.3	---	0.96	P4
81	2.1	1.7	6.60	P4
82	2.1	1.6	6.65	P4
83	1.6	0.9	7.75	P4
84	1.0	0.6	7.00	P4
85	0.6		6.75	P4
86	0.4	1.7	7.35	P4
1	0.6	3.7	7.20	P4
2	< 1.0	4.1	7.50	P4
3	< 1.0	4.5	7.35	P1
4	1.1	0.7	7.65	P1
5	1.1	4.0	7.55	P1
6	1.0	5.0	7.70	P4
7	1.0	4.8	7.75	P4
8	1.4	4.5	7.25	P4
9	1.2	5.2	7.75	P4
10	1.2	1.9	7.60	P4
11	1.1		7.30	P4
12	1.3	5.0	7.25	P4
13	0.9		7.60	P4

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
14	1.0	3.7	7.60	P1
15	1.7	3.8	7.60	P1

2. Silica Gel*

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>%U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
37	6.9	0.2		6	12/5	Hold
38	2.9	0.2		3	10/5	234-5
1	12.5	0.18		6	8/4	Hold
2	10.1	0.03		6	10/8	Hold
3	7.15	0.01		5	8/4	Hold
4	5.2	0.04		5	47/26	Hold
5	4.3	0.03		6	5/4	234-5
6	4.35	0.06		4	8/4	234-5
7	23.8	0.08		5	27/10	Hold

D. Overall Waste Loss - % of Total Production

U - 0.37   Pu - 0.33

E. Rework - % of Total Production

U - 11.4 - (UNH product recycle to HAF Make-up TK-E6)

Pu - 7.8 - (Out-of-spec. product to HAF Make-up TK-E6)

6.1 - (Supernate from 234-5 Building to TK-E6)

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash Solution	390	104
Cell Drainage	213	106
FP Waste	136	106
Coating Waste	270	101-C

B. 1WW Flows: 4.0

* Silica Gel units did not operate during report period.

C. Tank Farm Boil-off Rates

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min.</u>
101	3.4
102	3.3
103	0.3
104	3.8
106	11.0

Notes:1. Plant Operating Information

At the beginning of the report period, the recycle of "cold" UNH at a nominal 3.0 CF (2.7 CF, actual) was starting. At 0445 on June 29, shut-down of the solvent extraction system was commenced (HAF off) in preparation for an HAF rotometer-jumper change. The plant was started up (HAF on) at 0500 on June 30 at a nominal 3.0 CF, with "cold" UNH as HAF. Processing of "hot" HAF was commenced at about 1200 on June 30. The actual processing rate averaged about 3.1 CF throughout the remainder of the report period.

2. Flowsheet

- (a) On July 2, the 1CX was reduced from 387 to 370 flows. It was further reduced to 360 flows on July 3.
- (b) On July 2, the 2EX was reduced from 480 to 464 flows. It was further reduced to 450 flows on July 3, but was increased back to 464 flows on July 5.
- (c) On July 3, the 2OS was changed from 4.5 flows of 5% Na_2CO_3 to 21.7 flows of 0.05 M HNO_3 .
- (d) On July 5, the rework of off-standard UNH product was commenced at a rate of about 4 tons per day by batchwise addition to the 2DF.
- (e) On July 5, the HAX - NaNO_2 was reduced from 0.80 to 0.70 flows.

3. Feed Preparation

At the beginning of the report period, plugging of the coating waste line from TK-D2 to the waste storage tank TK-101-C (C Tank Farm) was threatening a plant shutdown. On June 30, however, the line was successfully cleared by use of water pressure applied to the line in D Cell.

4. Solvent Extraction

Process performance of the First and Final Uranium Cycles was generally good during the report period. Final Plutonium Cycle performance was erratic, however, resulting in out-of-specification product batches.

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The patterns of excellent performance of the Final Uranium Cycle, established during the last report period, continued.

Process control in the First Cycle continued to be difficult to maintain, due to the continuing difficulty with a number of flow controllers. In addition to the trouble with HAF Controller, it was necessary to operate the 3WB, HSR-HNO₃, and HSS-HNO₃ Controllers on manual operation. This resulted in considerable fluctuation in the HNO₃ concentration in the columns and, consequently, fluctuating HAW losses and difficulty in maintaining the 2AF nitric concentration within control limits. In spite of the mechanical problems, the decontamination performance remained good and the HAW Pu losses were maintained at an average of less than 0.05 percent.

The chief problem area was the Final Plutonium Cycle. Subsequent to startup on June 25, the average DF dropped about a factor of two. Moreover, the usual improvement did not occur, and after the second startup on June 30, another gamma burst occurred in this cycle. On July 4, the second agitator in the 2AF Tank, which had been off for about a month (see p.) was turned on. An extreme gamma burst resulted, which increased the 2BP activity about fifty-fold. Apparently gross quantities of "crud" had been held up in the 2AF Tank as a result of a loss of the IBS Interface during the June 22-25 shutdown. The single agitator apparently did not cause sufficient mixing to purge the tank and when the second agitator was turned on, the gamma burst resulted. However, at the end of the report period the 2BP activity was dropping rapidly and the DF was also showing improvement.

Continued high neptunium losses to the HAW prompted the decision to reduce the amount of nitrite added to the bottom of the HA Column, on July 5. In recent months, this addition rate has been gradually increased to maintain a particular "normal" control concentration in the HSP stream. It is possible that the apparent need for these increases correlates with the change from Shell E-2342 diluent to Soltrol - 170 diluent in the solvent. The point of close nitrite concentration control is at the bottom of the HA Column, however, rather than in the HSP stream. On the basis of this reasoning, the reduction in nitrite flow was made.

5. Final Plutonium Processing

The N-cell ion exchange system experienced a sharp gamma burst on June 29, correlating with the loss of Pu DF in the solvent-extraction system (Note 4, above). Radiation readings (CP) at the XAF Tank N1 reached a maximum of 500 mr/hr. and then fell to the 100 to 200 mr/hr. range where they remained for the remainder of the report period. The ion exchange DF remained at about 30, but all final product produced still exceeded specifications.

Mechanical operation of the N-Cell equipment was good, with consistent pushes at under 40 psig push pressure. No resin loss from the system has been noted and the XAF filters have not shown a high plugging frequency.

6. Solvent Treatment

Following the change from 2.5% carbonate to 1% NaOH in the Turbomixer (G7) an improvement was noted in the G7 to G7 pump rate. Flows are currently at normal rates.

Excessive aqueous carryover from both R1 and G1 has been noted when these tanks are at high liquid levels; hence, procedures have been revised to maintain liquid levels below the packing anulus to prevent aqueous from being pumped to the 10 and 20 columns.

Recent piping changes in R Cell (No. 2 Solvent Treatment) now permits 200 flow to be recycled to TK-R1 at controlled rates during normal operation or during plant shutdown. Also, revised piping allows use of the decanter pumps to transfer solvent from TK-R5 and/or TK-R7 (200 Tanks) directly to TK-R1 without affecting the 200 flow.

On July 3, overloading of the R-Cell centrifuges led to the discovery that a leaking control valve in this system was permitting excessive solvent flow through the 20 Column and the centrifuges. A solvent flow equivalent to ~ 5.0 CF was reduced to normal by closing a manual valve in the new piping.

The use of 0.05 M HNO_3 as 20S, in place of 5% carbonate, was commenced on July 3. This resulted in improved 20 Column stability.

7. Waste Treatment and Acid Recovery

Analytical results for a special 2WW sample thieved from the E-F6 concentrator on June 19 were submitted, as follows:

Fe	-	0.08 - 0.1 M
ZrNb	-	$2.8 \times 10^6 \mu\text{c/gal.}$
Ru	-	$7.8 \times 10^6 \mu\text{c/gal.}$
Ce	-	$9.3 \times 10^5 \mu\text{c/gal.}$

8. Fission Product Recovery

Head End

Strontium run series 43 was in the final concentration step at the end of the report period. Results will be reported next week.

Strontium Semiworks

Washing of the solvent with nitric acid and/or oxalic acid is underway in preparation for cribbing the solvent inventory. Disposal of the solvent is necessary to permit maintenance activities in C-Cell.

Nitric flushes of the 10 column in B-cell were unsuccessful in unplugging the 10W line, however B-Cell and hot shop close rates were significantly reduced.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 7/6/62 TO 2400 ON 7/12/62

I. Typical Solvent-Extraction Performance by Cycles

	Gamma dF		% Recycle		% Loss	
	U	Pu	U	Pu	U	Pu
First	4.1	3.7			< .002	0.03
Final	3.0	2.8	9.8	7.5	< .002	
Ion Exchange		1.8		1.2		
Overall	7.1	8.3	9.8	8.7	< .004	0.03

II. Production

A. HAF Processed: 176 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>
16	1.21	4.3	7.70	P4
17	1.32	-	7.45	P4
18	1.00	9.3	7.60	P4
19	0.97	8.6	7.70	P4
20	0.89	9.0	7.55	P4
21	0.91	8.9	7.70	P4
22	1.50	7.0	7.70	P4
23	1.18	1.3	7.80	P4
24	1.35	8.9	7.80	P4
25	0.92	7.9	7.80	P4
26	0.97		7.65	P1
27	1.12	7.5	7.60	P1
28	0.77	6.8	7.80	P1
29	1.17	7.0	7.50	P1
30	1.04	6.7	7.65	P4
31	0.91	6.5	7.45	P4
32	0.95	6.5	7.75	P1
33	1.09	6.0	7.70	P1
34	1.48	6.5	7.60	P1

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
35	1.86	7.0	8.00	P1
36	1.50	8.3	7.80	P1
37	1.25	13.6	7.65	P1
38	1.43	19.0	7.65	P1
39	1.62	18.0	7.20	P3
40	1.80	14.5	7.30	P1
41	1.70	17.8	7.60	P1
42	2.07	14.7	7.60	P1
43	1.89	12.3	7.70	P1

2. Silica Gel *

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mz/hr.</u>	<u>Shipped to</u>
8	18.2	.04		6	15/11	Hold
9	9.3	.035		6	15/7	Hold
10	5.8	.032		5	45/26	Hold
11	3.2	.01		3	30/13	234-5
12	2.2	<.01		5	31/7	234-5
13	1.8	.03		6	2	5 Cans 234-5
14	2.3	.04		5	3	2 Cans 234-5
15	2.2	.01		4	3	Hold
16	2.0	.01		5	3	234-5
17	1.7	<.01		5	3	234-5
18	2.0	0.015		5	4	234-5
19	2.5	<.01		5	2	234-5
20	3.0	.02		6	4	5 Cans 234-5

D. Overall Waste Loss - % of Total Production

Pu = 0.27   U = 0.25

E. Rework - % of Total Production

Pu - 13.0% (Out of Spec. product via HAF TK-E6 and via XAF TK-N1)
       3.18%
       3.2% (Supernate from 234-5 to TK-E6)

U - 22.05% (UNH from TK-P3 storage to 2DF TK-K1)

III. Waste Treatment

A. Volumes to UGS

* Silica Gel units did not operate during report period.

A. Volumes to UGS (continued)

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash Solution	358	104
Cell Drainage	61	106
FP Waste	80	106
Coating Waste	270	101-C

B. IWW Flows: 5.4C. Tank Farm Boil-off Rates

<u>Tank</u>	<u>Est. Boil-off Rate, Gal/Min</u>
101	3.2
102	3.0
103	0.3
104	2.0
106	12.0

Notes:1. Plant Operating Information

The HAF rate was maintained at a 3.0 CF throughout the report period except for a very short outage on July 10 caused by a power outage.

2. Flowsheet

- a. A close check of flow rates on July 6 indicated the LBXF (and 1BU) rate to be 590 flows (~25% high) and the LCX rate to be 365 flows (~9% low).
- b. On July 7, the 2DF ferrous sulfamate was increased from 4.0 to 4.9 flows.
- c. On July 7, the LCX temperature was reduced from 60 to 57 deg. C.
- d. On July 10, the HA Column nitrite was reduced from 0.70 to 0.63 flows.

3. Solvent Extraction

Solvent extraction performance during the report period was somewhat erratic. First cycle performance was good. Final Plutonium Cycle performance was improving somewhat; the Final Uranium cycle performance was good except for excessive plutonium content in the UNH product. At the end of the report period the specification for Pu in the UNH product was exceeded.

The cause for the higher-than-normal Pu in the UNH product has not yet been determined with certainty. Several factors no doubt caused

short term bursts; however, underlying these bursts has been an apparent general tendency for increased Pu contents since shortly after plant start-up in late June. Significant increases in the Pu content of the UNH correlated with the beginning of uranium rework (or the corresponding rate increase in the Final Uranium Cycle) on July 5 and with flooding of the 2D Column on July 10. The 2D Column flood occurred as a result of the July 10 power outage. Reclamation of off-standard solvent from TK-R5 into the No. 2 Solvent Treatment System on July 8, 9 and 11 may also have contributed to Final Uranium Cycle difficulties.

Still another possible contributor to the Pu-in-U problem is the possibility of introduction of an oxidizing potential to the 2D Column via the 2DX as a result of the recent change to permanganate treating of the No. 2 System solvent.

First Cycle waste losses were excellent during the report period. Reductions in nitrite flow to the HA Column on July 5 and 10 have been responsible for a sharp reduction in neptunium losses to the HAW -- of the order of five fold.

The pulse frequency of the 2D Column has been gradually increased since plant start-up to 70 cycles/min. on July 7.

4. Final Plutonium Processing

Early in the report period gamma levels in the ion exchange system had returned to about normal and the Pu product was meeting specifications.

On July 11 rework, via the N-Cell ion exchange system of out-of-specifications Pu was begun, using a ferrous sulfamate valence adjustment step prior to feeding the Pu into the XAF Tank N1. A total of six cans of Pu product (~10 Kg) was successfully processed with N2 losses only slightly above normal. An estimated 5% of the rework material was lost to the XAW. For a two-day period the average N-Cell Pu processing rate reached ~4.6 CF.

During the N-Cell rework operation, Pu rework was also being conducted via the L11-to-E6 route at the rate of 1000 grams per feed batch.

5. Solvent Treatment

Reclamation of solvent from TK-R5 back to the 20B Tank R1 was initiated during the report period. Solvent "loss" during aqueous phase changes in TK-R1 and TK-R2 have averaged about 480 gallons per day of which about 350 gallons per day is recovered from the 20W Tank R8 via TK-R5 to TK-R1.

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6. Waste Treatment and Acid Recovery

The 1WW flow has been difficult to control because of plugging specific gravity dip tubes in the 1WW concentrator E-F11. Control is maintained by close observation of the concentrator weight factor and the overflow rate into the TK-F12 receiver.

The formaldehyde flow pot and/or tower is apparently plugged, as repeated attempts to purge the line with water fail to indicate sustained flow.

During the report period the Pu content of the recovered acid increased roughly five fold, with several samples of the F5 Absorber Acid analyzing over 7000 $\mu\text{c/gal}$. This correlated with increases in the specific gravity of the 2WW in the E-F6 Concentrator and corresponding higher 2WW acid concentration.

7. Fission Product Recovery

Head End

Strontium run series 43 consisting of five sulfate runs, two oxalate runs and one concentration run was completed. A total of 208 kilocuries of strontium 90 were recovered with a decontamination factor of 10 from cerium.

Run series 44 was started. In run 44-S-1 an attempt was made to determine the amounts of fission products associated with the solids. The solids were washed with, water, 4 M HNO_3 and 5 M NaOH -0.5M tartaric acid. Additional back studies are planned for runs 45-S-1 and 46-S-1. Results will be reported at the completion of the third test.

Strontium Semiworks

The C-Cell organic inventory was sent to crib. Efforts to decontaminate B and C Cells continue.

Gas samples were pulled from TKs 64, 1, and 6 to monitor the hydrogen content. Analyses are not yet available.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 7/13/62 TO 2400 ON 7/19/62

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	3.9			<.002	0.06
Final	3.0	3.2	10.2	5.2	<.002	
Ion Exchange		1.3		0.7		
Overall	7.0	8.4	10.2	5.9	<.004	0.06

II. Production

A. HAF Processed: 178 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	1.87	10.8	7.5	P1
45	1.48	9.4	7.5	P1
46	1.55	9.9	8.0	P1
47	1.87	9.5	7.5	P1
48	1.80	9.6	7.8	P1
49	0.94	14.0	7.8	P1
50	1.42	14.0	7.5	P3
51	1.61	12.7	7.3	P1
52	1.62	14.0	2.1	P1
52A			5.6	P4
53	1.37	9.1	7.7	P4
54	1.42	7.7	7.5	P1
55	1.32	6.2	7.5	P4
56	1.15	7.5	7.4	P1
57	0.90	0.05	7.6	P1
58	1.13	6.2	7.6	P1
59	1.46	13.2	7.5	P3
60	0.90	23.2	7.5	P3

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<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
61	1.20	2.1	7.4	P3
62	1.20	22.8	7.5	P3
63	1.32	15.1	7.4	P3
64	1.04	12.3	7.7	P4
65	1.70	8.89	7.4	P1
66	2.11	9.7	7.5	P1
67	1.90	8.5	7.7	P1
68	2.40	7.7	7.4	P1
69	2.04	7.0	7.5	P1

2. Silica Gel - Not operated.

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped To</u>
21	2.42	0.02	500	6	3	234-5
22	1.92	0.03		5	3	234-5
23	2.00	0.015		6	3	234-5
24	1.48	0.02		5	3	234-5
25	1.36	0.035		6	2	Hold 3
26	1.56	0.028		5	2	234-5
27	1.55	0.038		6	15*	234-5
28	1.84	0.006		5	2	234-5
29	1.79			5	1	Hold 1 can
30	1.8	0.031		6	-	234-5
31	1.8	0.037		5	2	Hold 4
32	1.67	0.03		5	2	Hold 2
33	1.34	0.015		5	20*	234-5
34	1.47			5	15*	234-5
35	1.04			5	2	234-5

* Unshielded cans

D. Overall Waste Loss - % of Total Production

U = 0.27%

Pu = 0.24%

E. Rework - % of Total Production

U = 7.8% (P3 material to 2DF Tank-K1)

Pu = 14.2% (Out of spec product to HAF Make-up TK-E6 and to XAF TK-N1)

III. Waste Treatment

A. Underground Storage

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash Solution	335	106-A
	31	104-A
Cell Drainage	57	106-A

A. Underground Storage (continued)

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
FP Waste	127	106-A
1WW	24	106-A
Coating Waste	270	101-C

B. IWW Flows: 4.2

C. Tank Farm Boil-off Rates

<u>Waste TK</u>	<u>Est. Boil-off, Gal/Min</u>
101	3.0
102	2.8
103	0.3
104	1.4
106	16.1

Notes:

1. Plant Operating Information

The HAF rate was maintained at a nominal 3.0 CF throughout the report period, except for an emergency 5-hour outage on July 15-16. The actual HAF rate was gradually increased to 3.2 CF.

2. Flowsheet

- (a) On July 13, a gradual increase of 50 percent in the 2AS flow (to 1.3 flows) was commenced. The rate of 1.3 flows was attained on July 15.
- (b) On July 15, rework via the Final Uranium Cycle of "cold" uranium from P3 Tank was suspended.
- (c) On July 15, the permanganate concentration in the 20B (TK-R1) wash solution was decreased from 0.77 to 0.62 percent. The flowsheet permanganate concentration in the 10B Tank G1, was maintained by direct addition of 0.34 flows of 4% KMnO_4 .
- (d) On July 15, the 2DF-ferrous sulfamate addition was decreased from 0.67 to 0.4 flows. It was increased back to 0.67 flows on July 17.
- (e) On July 17, the 2DF- HNO_3 addition was decreased from 7.4 to 5.4 flows. It was further decreased to 4.6 flows on July 19.
- (f) On July 17, addition of recovered acid (50%) to the 3WB Tank was commenced at ~ 8 flows. The rate was increased to ~ 12 flows on July 18.

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- (g) On July 17 the source of 2DF-HNO₃ was changed from UO₃ recovered acid to fresh HNO₃ (because of a shortage of UO₃ acid).

3. Solvent Extraction

Solvent-extraction performance continued at an excellent level during the report period. The only negative aspect continued to be the problem of above-normal plutonium contamination in the uranium product and this condition showed continuing improvement until the recycle of "cold" uranium on July 16, or the associated rate increase caused another "burst".

The First Cycle exhibited a gradual improvement in decontamination performance (~15 and 50 percent decrease in activity of the uranium and plutonium streams, respectively). The HAW loss exhibited a sharp increase from <0.02 percent to ~0.08 percent at the beginning of the report period, but, had declined to an average of 0.03 percent by the end of the report period.

The Final Plutonium Cycle performance was excellent. A steady improvement in the decontamination performance (DF's increased from about 1000 at the beginning to about 3000 at the end of the report period) coincident with a two-to three-fold decrease in the average "loss" to the backcycle system was noted. The improved performance of the 2A Column was attributed partly to a purging from the system of crud, but primarily to the higher 2AS flow (50 percent higher than the normal rate for the past several months).

Late on July 15, it was necessary to shutdown the HA-HS Column due to a PRV failure in the HAX control system.

4. Final Plutonium Processing

During the report period 11 more cans of Pu product (~19.5Kg) were reworked by addition via the XAF system. Valence adjustment with ferrous sulfamate was made on all cans reworked. N-Cell operation continued good during the rework period with only slightly increased losses. A great deal of trouble has been encountered in controlling the "C" column acid split with the automatic CRC. The operating symptoms are similar to previous occasions when it was thought that resin plus push solution was channeling through the "C" column, and therefore not allowing the CRC to see a true picture.

5. Solvent Treatment

On July 15 KMnO₄ concentration in Tank 208 (carbonate-permanganate make-up) reduced 20% in an attempt to affect the excessive Pu in the solvent extraction UNH product. The KMnO₄ concentration in the No. 1 Solvent System was maintained at the flowsheet value by adding KMnO₄ directly to Tk-G1.

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5. Solvent Treatment (continued)

The Pu retention of the 200 has increased from a normal range of about 1×10^7 up to about 5×10^7 since July 1. Changes which could have affected the Pu retention were introduction of Na_2CO_3 - KMnO_4 to TK-R1, conversion to HNO_3 in the 20S vs Na_2CO_3 , and/or rework of uranium from TK-P3.

Fission Product Recovery

Strontium run series 44 consisting of five sulfate runs, two oxalate runs and one concentration run was completed. A total of 188 kilocuries of strontium 90 was recovered with a decontamination factor of 15 from cerium.

Run series 45 was started. The first run 45-S-1 included leaching of the solids as reported last week.

Decontamination efforts were started on the failed E4 centrifuge.

Strontium Semiworks

Decontamination efforts in B and C Cells continue. The 10W line has opened up and flushes of the 10 Column are continuing.

The composition of the gas sampler obtained last week is as follows:

Gas Analyses - Volume percent

	TK-1		TK-6		TK-64	
	#1	#2	#1	#2	#1	#2
CO ₂	<0.01	0.03	0.05	0.09	0.02	<0.01
A	0.93	0.93	0.92	0.91	0.92	0.93
O ₂	20.9	21.0	20.9	20.8	21.1	21.0
N ₂	78.2	78.0	77.8	77.0	78.0	78.1
CO	< 0.01	<0.01	<0.01	<0.01	<0.01	<0.01
He	< 0.01	<0.01	<0.01	<0.01	<0.01	<0.01
H ₂	< 0.01	<0.01	0.34	1.21	<0.01	<0.01

The above samples were taken with 27, 9, and 11 inches of water vacuum on TK's 1, 6 and 64 respectively. All analyses are well below the lower hydrogen explosive limit of 4 percent.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 7/20/62 TO 2400 ON 7/26/62

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	4.2	4.1			<.002	0.02
Final	2.8	3.4	11.5	2.45	<.002	
Ion Exchange		1.2		0.10		
Overall	7.0	8.7	11.5	2.55	<.004	0.02

II. Production

A. HAF Processed: 189 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>
70	2.10	7.7	7.5	P1
71	2.45	5.3	7.2	P1
72	2.20	3.7	7.5	P1
73	2.30	3.5	7.2	P1
74	2.0	2.9	7.5	P1
75	1.90	2.37	7.5	P1
76	1.66	3.5	7.6	P1
77	1.20	3.5	7.3	P1
78	1.35	3.1	7.6	P1
79	1.55	2.5	7.7	P1
80	1.39	2.1	7.4	P1
81	1.89	2.3	7.2	P1
82	1.54	2.45	7.3	P1
83	1.42	3.0	7.2	P1
84	1.32	3.5	7.4	P1
85	1.04	3.6	7.8	P1
86	1.37	3.9	6.8	P1
87	1.08	4.2	6.9	P1
88	1.81	4.2	7.4	P1
89	2.02	3.7	7.4	P1
90	1.54	3.2	7.4	P1

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
91	1.39	1.0	7.6	Pl
92	1.36	2.4	7.5	Pl
93	1.07	2.2	7.4	Pl
94	1.14	2.6	7.6	Pl

2. Silica Gel: Not Operating.

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
36	0.94			5	2	234-5
37	0.81	0.01		5	1	"
38	0.77			5	1	"
39	0.79	0.01		5	2	"
40	1.13			4	2	"
41	0.80	0.05		4	3	Hold 1
42	1.52			5	2	234-5
43	0.90			5	1	Hold All
44	0.57	0.015		5	2	Hold 3
45	0.56			6	2	Hold All
46	0.49	0.01		5	2	Hold 4
47	0.42			5	1	Hold 3

D. Overall Waste Loss - % of Total Production

U = 0.21%   Pu = 0.29%

E. Rework - % of Total Production

U = Nil
 Pu = 8.9% - Supernate from 234-5 to HAF Make-up TK-E6.
 7.5% - Out-of-spec. product to HAF Make-up TK-E6.
 4.4% - Sample Tank L10 pump leak to HAF Make-up Tank E6.

III. Waste Treatment

A. Volumes to Underground Storage

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash Solution	260/16	106/104-D
Cell Drainage	15	106-A
FP Waste	115	106-A
Coating Waste	270	101-C

B. 1WW Flows: 4.3

C. Tank Farm Boil-off Rates

<u>Waste TK</u>	<u>Estimated Boil-off, Gal/Min.</u>
101	3.0
102	2.8
103	0.3
104	1.4
106	16.1

Notes:1. Plant Operating Information

The plant was maintained at a nominal 3.0 CF during the report period. The actual rate averaged about 3.2 CF.

2. Flowsheet

- (a) On July 21, the 2AS was increased from 2.9 to 3.1 flows.
- (b) Commencing on July 21, the 2EX was reduced from 435 to 390 flows, over a three-day period.
- (c) On July 24, the addition of 0.005 M NaNO_2 to the 1WF was commenced.
- (d) Commencing on July 26, the 2DF- HNO_3 was slowly increased from 1.3 to 2.0 M over a four-day period.

3. Solvent Extraction

Solvent-extraction performance was excellent during the report period. Both the plutonium and uranium products were consistently within specifications and treatment of the uranium by silica-gel was not necessary. Waste losses and internal reflux remained near normal, with the HAW loss averaging 0.03 percent.

An approximately two-fold increase in the plutonium contamination in the uranium product (from ~2 to ~4 ppb) was noted after the rework of recovered solvent in the No. 2 Solvent System was commenced, but the uranium remained well within specifications.

A gradual decrease in the 2E Column aqueous/organic flow ratio from 1.0 to 0.85 did not increase the 2EW uranium loss significantly. (The losses increased from nondetectable to barely detectable.) The flowsheet ratio is 1.09.

4. Final Plutonium Processing

N-Cell ion exchange operation began to show signs of erratic behavior with the CRC control still not working properly and resin movement beginning to become difficult. This report period marks the fifth week.

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of operation since the last resin change and the unit may be repeating past patterns. The Sample Tank L10 recirculation pump began to leak noticeably during the report period, causing some PR room contamination and some loss of product to the SLO sump. All rework of Purex Pu product via TK-L9 to N-Cell was discontinued because of the need to use TK-L9 to rework Z-Plant supernates to the HAF Make-up Tank E6. Also, work was begun during the report period on installation of the flowmeter on the L9-to-N1 rework system.

5. Waste Treatment and Acid Recovery

On July 24, addition of 0.005 M-NaNO₂ to the LWF was begun to again test the effect on Pu content of the F3 recovered acid. The immediate result was a ten fold reduction in the F3 Ru activity level from 2×10^4 μ c/gal to 2×10^3 μ c/gal. Following samples indicated a gradual increase in Ru with the final Ru level being about a factor of five below the pre-test activity. These Ru suppression results check closely with previous test data.

6. Demineralized Water

On July 25 a new sulfuric mix tank for cation regenerations was put in service. The new tank is a lead-bonded-to-steel tank with about twice the old tank capacity and a much improved valving system for controlling the acid addition to the cation bed. Weight factor and specific gravity dip tube instrumentation are also incorporated into the new tank, replacing the sight glasses used on the old tank.

7. Fission Product Recovery

Head End

Strontium run series 45 consisting of five sulfate runs, two oxalate runs, and one concentration run was completed. A total of 158 kilocuries of strontium 90 with a decontamination factor of 18 from cerium was recovered and sent to storage.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 7/27/62 TO 2400 ON 8/2/62

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			<.002	0.05
Final	2.9	3.3	9.9	5.1	<.002	
Ion Exchange		1.1		3.0		
Overall	6.9	8.5	9.9	8.1	<.004	0.05

II. Production

A. HAF Processed: 185 Tons

B. Uranium Product

1. Solvent Extraction

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Tons</u>	<u>Pu,ppbp U</u>	<u>Shipped To</u>
95	1.22	7.7	2.8	P1
96	1.89	7.6	2.9	P1
97	1.20	7.0	4.3	P1
98	0.94	7.4	4.8	P1
99	1.20	7.2	4.75	P1
100	1.14	7.2	8.1	P1
101	1.17		10.1	P1
102	.943	7.3	8.6	P1
103	.979	7.8	7.0	P1
104		7.3	6.4	P1
105	1.12	7.4	4.9	P1
106	1.72	7.6	6.4	P1
107	1.46	7.4	3.9	P1
108	1.67	7.4	4.5	P1
109	1.58	7.2	4.8	P1
110	1.72	7.3	3.6	P1
111	1.51	7.3	4.9	P1
112	1.14	8.2	5.2	P1
112A	1.32	4.2	6.0	P1

1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Tons</u>	<u>Pu, ppbp U</u>	<u>Shipped To</u>
113	1.46	3.9	4.6	P1
1	1.54	7.1	6.9	P1
2	1.63	7.3	6.9	P1
3	1.79	7.4	5.9	P1
4	1.87	7.3	5.6	P1
5	2.03	7.4	5.3	P1
6	1.84	7.2	4.9	P1

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
48	0.44			5	10*	234-5
49	0.6			4	2	Hold
50	2.2	0.01		5	2	234-5
51	1.39			5	17*	234-5
52	1.30	<.01		5	2	1 Can 234-5
53	0.73	<.01		5		Hold
54	1.40	<.01		6	2	2 Cans 234-5
55	1.63	<.01		5	2	234-5
56	1.31	0.002		5	2	1 Can 234-5
57	2.04	<.01		5	2	234-5
58	2.02	.02		4		234-5
1	2.12	<.01		5	2	2 Cans 234-5
2	2.61	0.005		5	15*	234-5
3	1.9	0.002		5	15*	Hold
4	2.0	0.004		4		Hold

* Unshielded Cans

D. Overall Waste Loss - % of Total Production

U = 0.24% Pu = 0.23%

E. Rework - % of Total Production

U = Nil

Pu = Total: 8.86%
 SN-0.52% Out of Spec. Cans - 6.6%
 Sumps - 1.7%

III. Waste Treatment

A. Volumes to Underground Storage

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash Solution	88/176	106/104

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<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Cell Drainage	43	106
FP Waste	133	106
Coating Waste	270	101-C

B. 1WW Flow: 5.0 Flows

C. Tank Farm Boil-off Rates

<u>Waste TK</u>	<u>Est. Boil-off Rates, Gal/Min</u>
101	3.0
102	2.8
103	0.3
104	2.4
106	15.1

Notes:

1. Plant Operating Information

The plant was maintained at a nominal 3.0 (actual 3.17) C.F. during the report period.

2. Flowsheet

- On July 28, the 2DF-HNO₃ was reduced from 8.6 to 7.3 flows. It was increased back to 8.0 flows on July 29 and to 8.6 flows on July 30.
- On August 2, the 2DF temperature was reduced from 60 to 50 deg. C and the 2DX temperature was reduced from 42 to 39 degrees C.
- On August 2, the 2A pulse frequency was reduced from 87 to 83 cycles per minute. After about eight hours, it was increased back to 85 cycles per minute.
- On August 2, the 2 AS was reduced from 3.1 to 2.9 flows.

3. Feed Preparation

Mercuric nitrate (0.0005M) was added to metal solution tanks throughout the period. An average of 180 curies of I₁₃₁ was charged to dissolvers daily in metal with a minimum cooling time of 121 days. The daily I₁₃₁ emission averaged 0.35 curies.

4. Solvent Extraction

Solvent-extraction performance was generally excellent during the report period. The decontamination performance of all cycles was slightly lower at the end of the report period, with the activity

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4. Solvent Extraction (continued)

of both product streams showing an upward trend. Both products met specification; the uranium without silica gel treatment. Plutonium losses via the First Cycle Waste, (HAW), were quite erratic with a sharply increasing trend near the end of the report period. As in the past, the problem was due to the fluctuating acidity in the HA Column. The losses were maintained at tolerable levels ($\sim 0.05\%$, average) by adjustment of the organic phase saturation in the HA Column. The 2AW recycle to the Backcycle Waste (3WB) was higher than normal for a brief period, (8-10%), but was quickly reduced by adjustment of the 2AF acidity.

The plutonium contamination in the First Cycle Uranium (1BU) Stream showed a steady, two-fold increase over the first half of the report period, then a fairly sharp decrease to about 40 per cent of the peak. The reason for the fluctuations was suspected to be the fluctuating acidity of the HSP (organic) Stream, which caused minor "upsets" in the internal reflux in the 1BX Column.

The plutonium contamination in the uranium product from the Final Uranium Cycle was maintained under control during the report period. The acidity in the 2D Column appeared to have a negligible effect on the plutonium DF, as shown by operation at both 0.8 and 1.0 M HNO_3 in the 2DW. Rework of solvent in the Second Solvent System may affect the plutonium DF to a greater extent, but the cause-effect relationship was not clear-cut.

5. Final Plutonium Processing

On July 28 the L-10 recirculation pump was replaced. The old pump leaked and significant amounts of high-concentration plutonium solution were being lost to the SLD sump.

The operation of N-cell became increasingly erratic during the report period. Periods of little or no pushing were experienced and the XAW and XSW waste losses increased correspondingly.

The unit also was experiencing some loss of resin, but the route by which the resin was leaving could not be determined.

On August 2, approximately 23 gallons of resin were successfully changed on the fly. The resin had been in service since May 30. All resin removed was extremely low in plutonium content as determined by neutron monitor checks. Immediately following the change, resin pushing was much improved.

During the report period a magnetic flow meter and DOV were installed on the L9 to N1 rework line. Operating conditions in N-cell, however, prevented operational testing of the flow control system.

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6. Solvent Treatment

The Ru activity in the No. 1 solvent treatment stream remained at 1000 to 2000 uc/gal throughout the run period while the Zr-Nb activity generally remained between 200 to 1000 uc/gal. During the same period the LCW activity was 3000 to 5000 uc/gal for Ru and 2000 to 3000 for Zr-Nb. The normal DF across the turbomixer was unaffected by the conversion from 2.5% Na_2CO_3 to 1% NaOH. As before, essentially no DF for Ru was experienced.

The 200 temperature was reduced for 42°C to 39°C to check the effect on Pu carry-over to the final U loadout in K Cell.

7. Waste Treatment and Acid Recovery

The nitrite addition to F7 continued during the report period but the ruthenium activity of the F3 acid rose slowly to approximately 9×10^3 uc/gal. This still represents a factor of about three in improvement of Ru DF over the operating period, during which no nitrite was added to the concentrator feed. The Zr-Nb activity again remained virtually unchanged from previous values.

8. Waste Management

On July 27 the supernatant level in TK-241-A-103 was pumped from 116" to 48" (186,000 gal.) by the sluice pump at a rate of about 600 gpm.

The solution was pumped into TK-241-A-105. During and previous pumpings, a total of 485,000 gallons of supernatant was transferred from 103 to 105.

9. Fission Product Recovery

Strontium recovery run series 45 was completed during the report period. A total of 158 kilocuries of S-90 were recovered with a decontamination factor of 9.4 from cerium.

Efforts to define the amounts of fission products associated with LWV solids were continued on run series 46. The raw LWV centrifuge cake from run 46-S-1 was washed with water, 4 M nitric acid, and 5 M NaOH-0.5 M tartaric acid which is the same treatment given run series 44 and 45. On the basis of the solids studies to date, it appears 35, 25, 20, 10, and 10 per cent of the cerium, strontium, zirconium-niobium, cesium and ruthenium, respectively, in the LWV are associated with the solids.

About 90 per cent of the cesium and ruthenium, 80 per cent of the cerium and 50 per cent of the strontium and zirconium-niobium on the solids are removed with the water wash. The nitric wash removes the remainder of the cerium, cesium and ruthenium on the solids and another 25 per cent of the strontium.

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9. Fission Product Recovery(continued)

The remaining strontium and zirconium-niobium are removed with the caustic tartrate wash. The quantity of fission product that remained on the solids after the three washes was small as indicated by the centrifuge Beckman.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 8/3/62 TO 2400 ON 8/13/62

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	4.1	3.8			.004	.03
Final	2.9	3.2	6.5	2.15	.002	
Ion Exchange		1.5		1.56		
Overall	7.0	8.5	6.5	3.7	.006	.03

II. Production

A. HAF Processed: 182 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	2.18	3.6	7.1	P1
8	2.64	3.9	7.1	P1
9	1.28	2.8	7.2	P1
10	1.58	1.5	7.3	P1
11	1.29	1.4	7.3	P1
12	1.12	1.2	7.4	P1
13	1.34	-	7.3	P1
14	1.70	0.5	7.3	P1
15	1.20	1.3	7.4	P1
16	1.62	1.4	7.4	P1
17	1.37	1.2	7.6	P1
18	1.61	1.5	7.4	P1
19	1.52	2.0	7.1	P4
20	1.76	1.8	7.6	P1
21	1.34	2.52	7.4	P1
22	1.22	3.6	7.3	P1
23	1.72	3.9	7.3	P1
24	1.36	4.7	7.4	P1
25	1.67	5.1	7.3	P1
26	1.24	6.26	7.4	P1

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
27	1.22	5.5	7.2	P1
28	1.60	6.0	7.3	P1
29	1.82	4.5	7.0	P1
30	1.60	2.8	7.3	P1
31	1.34	1.9	7.2	P1
32	1.56	1.2	7.7	E6
33	1.52	2.7	7.1	P1
34	7.39	46.3	6.4	E6
35	7.93	45.3	6.4	E6
36	4.66	23.	6.4	E6
37	2.82	10.8	6.7	E6
38	1.36	6.3	6.7	E6
39	0.82	2.9	6.2	E6
40	2.41	1.8	6.2	E6
41	< 1	1.3	7.0	E6
42	0.03	0.8	5.4	P-1
43	0.709	0.37	6.4	E6
44	< 1	0.98	7.2	P1
45	< 1.28	----	6.3	P1

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
5	6.3	.019		5	5	Hold
6	5.31	.004		5	*35	Hold
7	3.05			6	2	5 Cans 234-5
8	1.74	.01		5		234-5
9	1.41	.02		5	2	234-5
10	1.97	.03		5	2	234-5
11	1.25	.05		5	2	1 Can 234-5
12	1.09			5	2	4 Cans 234-5
13	1.52			5		Hold
14	1.64			5	2	Hold
15	1.65	.01		5	2	234-5
16	1.39			5	2	234-5
17	3.5	.01		6	2	1 Can 234-5
				4	*22	Hold
19	28.16	0.14		5		Hold
20	14.4	0.18	571	6	8	Hold

D. Overall Waste Loss - % of Total Production

U = 0.18%

Pu = 0.26

E. Rework - % of Total Production

U - 36.0% (K6 → E6 in preparation for shutdown)

Pu - 10.4% Total

* SN Cans (Unshielded)

E. Rework - % of Total Production (continued)

8.7% Out-of-Spec material and sumps via L11 to E6
1.7% SN material via RC to E6

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III. A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash Solution	83/317	106/104
Cell Drainage	64	106
FP Waste	262	106
1WW	50	106
Coating Waste	270	101C
Formaldehyde Overheads	45	106

B. 1WW Flows: 3.9 FlowsC. Tank Farm Boil-off Rates

<u>Waste TK:</u>	<u>Est. Boil-off, Gal/Min</u>
101	3.0
102	2.8
103	0.3
104	2.4
106	15.1

Notes:1. Plant Operating Information

The plant was maintained at a nominal 3.0 (3.15) CF until an equilibrium shutdown on August 9 due to failure of the HAF pump. The pump replacement proceeded without trouble and the startup was commenced about 14 hours after shutdown. After running about nine hours on "hot" feed, the scheduled shutdown was commenced with recycle of "cold" uranium from the Final Uranium Cycle (K6 Tank) to the HAF Tank. Seven batches (~45 tons) of "cold" uranium were recycled prior to shutdown of the Solvent Extraction System to reduce the plutonium inventory in the Back-cycle Waste System preparatory to the neptunium recovery run. The HAF was shut off at 2300 on August 12. Operational trouble in plutonium anion exchange facility necessitated shutting down the Final Plutonium Cycle before it was stripped. Shutdown operations in the Final Plutonium Cycle were concluded at 0400 on August 14.

2. Flowsheet

- (a) On August 3, the nitric concentration in the Recovered Acid System was increased from 50 to 57%. The affected streams (HSR-HNO₃, HSS-HNO₃, 2DF-HNO₃) were adjusted accordingly.

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- (b) On August 4, the 2DW "loss" to the Backcycle Waste System was reduced from 11% to ~7% by increasing the 2DX flow. It was further reduced to 5% on August 7.
- (c) On August 4, the 2AW "loss" to the Backcycle Waste System was reduced from an average of three percent to an average of 1.5 percent.
- (d) On August 5, the 2DF Temperature was increased from 50 to 55°C. It was reduced back to 50°C on August 8.
- (e) On August 6, the HA- NaNO_2 was reduced from 0.63 to 0.57 flows.
- (f) On August 9, the Solvent-Extraction System was shutdown at equilibrium.
- (g) On August 10, the Solvent-Extraction System was started up on the same flowsheet that was in effect at shutdown, except for the following:
  - (1) The 2A Pulse Frequency was 80, rather than 85 cycles per minute.
- (h) On August 10, the 2A Pulse frequency was increased from 80 to 85 cycles per minute.
- (i) On August 10, "cold" uranium was recycled from the Final Uranium Cycle (K6-Tank) to the HAF Tank.
- (j) On August 11, the 3WB HNO_3 concentration was reduced from 6.5 to 5.9 molar. It was further reduced to 5.3 molar on August 12. The 3WB flow was increased, concurrently with the decrease in concentration to minimize the Backcycle Waste inventory.

3. Feed Preparation

0.0005 M Mercuric nitrate was added to metal solution tanks throughout the period for iodine suppression.

A3 thermohm failed on August 3 and the dissolver was out of service until the thermohm was replaced on August 6.

The last charge prior to shutdown on August 7, but I_{131} emission continued to 8/10 before it started to decline. Average emission for 11 day period was 0.24 curies/day. Average of 186 curies/day was charged for the 5 days this period there was charging.

The 1000 gallon (vice 400) fresh water rinse prior to charging, was used on 8 dissolver charges with no significant reduction in coating waste losses. Utilization of the extra rinse water was made possible by making 57% vs. 50% nitric acid in the fractionator and diluting the 57% acid to 50% acid in the dissolver with rinse water.

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4. Solvent Extraction

The generally excellent level of performance experienced during recent weeks continued, until the emergency shutdown on August 9. The decontamination performance of the First Cycle continued excellent, while the losses via the HAW Stream dropped from an average of 0.08% to 0.02%.

Performance of the Final Uranium Cycle continued very good, with all product meeting specifications without silica-gel treatment and "waste" losses to the Backcycle Waste System remaining normal.

Performance of the Final Plutonium Cycle was generally excellent. A brief upset on August 2 resulted in a "burst" of activity in the product and temporary high "losses" to recycle. The "burst" of activity was short-lived, however, and only two batches of product exceeded specifications. They will probably be blended after plant operation is resumed.

Startup of the Solvent-Extraction System after the brief shutdown on August 9 was characterized by above-normal activity in the product streams from all three cycles. The activity was well-purged from the system by the time the plant was shut down, but two additional batches of plutonium required segregation for eventual rework.

The "in-plant" program to investigate the effect of a number of process variables on the plutonium decontamination performance of the Final Uranium Cycle (Ref: Purex Process Summary, pages) was concluded with shutdown of the plant. The principle conclusions are summarized below:

- (a) The effect of the acidity in the 2D Column was negligible over the range from 0.7 to 1.0 M on the 2DW Stream.
- (b) Rework of recovered solvent (in the No. 2 Solvent System) resulted in generally poorer plutonium DF's in the 2D Column. The effect was not measured quantitatively. However, at minimum instantaneous recycle rates, the plutonium levels in the final uranium product increased by about two-fold.
- (c) Reducing the 2D Column temperature by about ten percent (2DF temperature was reduced from 60 to 50°C and 2D temperature was reduced from 42 to 39°C) reduced the plutonium level about five-fold.
- (d) Rework of off-standard uranium product from Tank-P3 increased the plutonium level by two-fold or more, the magnitude of the increase depending on the percent rework and method of introduction of the rework material.

Laboratory investigation of the plutonium decontamination problem in the 2D Column continuing. Preliminary observations indicate the oxidation potential of the KMnO_4 -washed 2DX to be about 50 percent higher than 2DX that has been washed with Na_2CO_3 , only. Furthermore, the potential of the carbonate-washed 2DX was two-fold higher than carbonate-washed laboratory solvent.

5. Final Plutonium Processing

N-Cell operation following the August 2 resin change (on-the-fly) was satisfactory for about five days, although resin was apparently being lost from the unit. Beginning on August 8th push control became extremely difficult and resin loss from the unit seemed to increase.

On August 10 the first indications of plugging in the XAF line between N1 and the filters was noted and by August 11 XAF flow from TK-N1 to the pump had ceased. N-Cell was essentially down the two final days prior to shut-down as pushes became infrequent and the XAF flow was nil.

On August 7th the installation of the magnetic flowmeter system on the L9 to N1 line for Pu rework was completed and flow tested with nitric acid.

6. Demineralized Water

On August 9, charcoal filters on the sanitary water inlet, number one cation effluent, and number one anion effluent were valved in following regeneration of the number one unit. On August 10, the filters were valved out. However, the test will have to be repeated because the plant shutdown changed the demineralizer flow rate and the anion rotometer leaked to the point it could not be run. The equipment was supplied by J. M. Skarpelos who was also to make the studies on the used charcoal filters.

7. Waste Treatment and Acid Recovery

Attempts to unplug the 1WW F12 to the formaldehyde prototype line were successful for the T-F95→T-F40 section of line; however, extremely high radiation levels from apparent blow back on the steam line in the P & O Gallery has stopped further efforts at freeing the remainder of line (T-F94→T-F80).

8. Fission Product Recovery

Strontium recovery run series 46 was completed and recovered 226 kilocuries of strontium 90 with a decontamination factor of 4.4 for cerium.

Because of the solids observed by HLO in a 200 gallon cask of centrifuged 1WW, run 46-S-5 was centrifuged twice prior to the sulfate precipitation. No additional rise on the centrifuge Beckman was observed on the second centrifugation. It is unlikely the solids observed by HLO passed through the Purex centrifuge.

Thorough decontamination flushes of the E-Cell vessels was initiated in preparation for cesium load out into the ORNL STT casks.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 8/14/62 TO 2400 ON 8/31/62

I. Production

A. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
21	25.9			5	1	Hold
22	24.2			4	3	Hold
23	1.47			5	8	Hold
24				5	1	Hold
25	9.8			4	1	Hold

B. Overall Waste Loss - % of Total Production

U = 1101# → 106A - Primarily From F18
Pu = 9.8% " " " "

C. Rework - % of Total Production

U - Nil
Pu - 26.3% - N Cell Sump Rework

II. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Volume</u>	<u>Waste TK</u>
R. G. Cell Flushes	23,214	106-A
	63,537	104-A
Cell Dr., 'F' Cell Flushes	100,730	106-A
1WW	7,464	106-A
FP (Cask Loadout)	39,525	106-A
CW	0	

During the report period the plant was shutdown with respect to normal plutonium and uranium processing. A neptunium recovery run was conducted and necessary maintenance was performed. Process notes of interest are summarized below.

1. Feed Preparation

- (a) Dissolver heels were carefully cut and the Pu holdup throughout the plant was reduced to a minimum.
- (b) A high differential pressure had been measured across C-Cell silver reactor lower bed during the last run period. During the shutdown the lower bed was water and sodium thiosulfate flushed, then regenerated. The upper bed was given a single shot of AgNO_3 to replace any washed away during the steaming of the lower bed. "C" reactor had processed 1009 tons since the last regeneration.
- (c) "B" Cell silver reactor was regenerated on a routine basis after processing 599 tons.
- (d) The back pressure valves which serviced the dissolver coils were removed from Trap Pit #1 and replaced by straight thru pipes to steam condensate header. A new jumper was installed on each dissolver containing a yawway trap, bypass valve and a throttling valve. This system also has an air pressurization system for the coil.

2. Solvent Extraction

- (a) A sampler jumper was installed to permit routine sampling of the LCU stream.

3. Neptunium Recovery

A normal neptunium recovery run was conducted during the shutdown. The run commenced at 1500 on August 14 and terminated at 0600 on August 20. Time intervals for the various phases were:

Phase I (Accumulation)	76 hours
Phase II (Decontamination)	18 hours
Phase III (Concentration)	34 hours

Phase I operation was completed successfully with negligible losses and the normal uranium and plutonium decontamination effectiveness in spite of numerous minor upsets due to equipment and service interruptions and the usual gassing of the 2AS Stream. Immediately preceeding the transition to Phase II operation, moderately-severe gassing resulted in a gradual decrease in the 2AS flow for about 60 minutes and total loss of flow for about 30 minutes. This condition should not have increased the neptunium loss, however. The effect should be limited to a temporary decrease in the plutonium decontamination.

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3. Neptunium Recovery (continued)

After transition to Phase II was completed, the 2AW loss commenced to increase rapidly, reaching 200-fold above normal nine hours after the start of Phase II and 300-fold above normal 18 hours after the start. At this time, the transition to phase III was commenced.

During Phase III operation, the loss (expressed as a percentage of the 2AF concentration) remained high, decreasing a quantity only as the 2AF concentration (and internal reflux in the column) decreased.

Scrutiny of the laboratory analyses of both the "cold" scrub streams and the "hot" streams and the operating data has not provided any clue to the reason for the high losses. The analyses indicated all "cold" streams to be within flowsheet specifications. Furthermore, the nitric acid concentration in the 2A Column was normal, as shown by the 2AF and 2AW nitric analyses. Ferrous ion at concentrations equal to or greater than flowsheet was detected in the 2AW during the entire run. Solvent quality appeared to be very good; the analyses of the solvent used for 2A are shown below:

Percent TBP	30.3%
Plutonium Retention	1.77×10^8 c/min/gal
Zr-Nb Activity	150 μ c/gal

The operating conditions in effect during the Phase II portion of the recovery run are listed below:

2A Pulse Frequency	80 cpm _o
2AF Temperature	$60 \pm 2^\circ$ C
2AX Temperature	$44 \pm 2^\circ$ C
2AF Tank Agitators	One Operating
2A Column L/V	
Extraction Section	1.7
Scrub Section	0.18

The only process conditions which were not within flowsheet (or normal operating) limits were the use of a single agitator in the 2AF Tank and the flow ratios in the 2A Column. The flow ratios were actually more favorable for low losses than the flowsheet values of 2.1 and 0.23 for the Extraction and Scrub Sections, respectively.

A review of the laboratory and process data indicates the high 2AW loss during Phase II to be most probably due to a portion of the Neptunium in the 2AF being in the improper valence form. This resulted from a combination of re-oxidation of a portion of the Neptunium in the 2BP Stream during its transfer by jet to the 2AF tank and inadequate mixing in the 2AF Tank. The relatively high temperature attained by the 2BP stream during its transfer by steam jet very likely causes partial oxidation of the 2BP stream. The use of a single agitator in the 2AF Tank results in relatively poor mixing of the 2BP and the ferrous ion in the 2AF Butt solution.

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A second possible reason for the high 2AW loss is that an undetected oxidizing agent was present in one of the "cold" streams, causing a partial re-oxidation of the neptunium in the 2A Column.

The loss picture, together with the overall material balance is summarized below:

a) Total Accumulation	By 3WB Stream Analysis	2850
	By 2DF Stream Analysis	2750
b) 3WB Tank "heel" at end of accumulation run		~ 200
c) Accumulation in the Final Plutonium Cycle:		
	By difference	2600
	By 2BP analysis	2500
d) "Losses" to backcycle:	Units	Percent*
Phase I (accumulation) 2AW	15	<1
Phase II (decontamination) 2AW	595	24
Phase III (concentration) 2AW	50	2
All Phases 2BW	40	1.6
Total losses:	700	28
e) Total recovery:	1760	70
f) Total accounted for:	2660	
g) Inventory difference (estimated):	140	

* Expressed as percent of the accumulation in the Final Plutonium Cycle.

4. Final Plutonium Processing

Following completion of normal plutonium processing on August 13, N-Cell was shut down and work begun on unplugging the XAF line which was filled with resin. Approximately seven or eight gallons of resin were removed from the N1 Tank and returned to the unit.

Several days were required to strip the unit because of mechanical difficulties with the push system. Following the resin strip and removal, the unit was almost completely dismantled and all screens inspected. The resin receiver top-hat and XAF distributor were both found to have large amounts of resin packed on the wrong side of the screens. Because no holes or tears were observed in the screens, the most reasonable explanation for the presence of the resin is that it leaked through the screen hold-down plates rather than having been forced through the screen by high push pressures. However, these two screens were replaced. All other screens appeared in good condition. No screens were found

4. Final Plutonium Processing (continued)

in vent lines from the XA and XC Column and subsequently screens were installed.

Also during the shutdown valves were installed which will permit the XAF pumps to be rate checked and the N6 mud leg to be rodded out. Considerable time was also spent on miscellaneous maintenance on valves and air cylinders. Prior to recharging fresh resin to the system the N1 tank was given a series of XCX flushes with the tank directly open to the floor to insure the removal of all resin.

Boilup flushes to clean out N6, N7 and L10 for the upcoming non-representative metal were completed following the maintenance work. A total of 28 cans containing ~6 Kg were loaded out to be saved for future rework. The estimated N-Cell inventory of regular Pu following the flushes was about 0.75 to 1.0 Kg.

5. Uranium Handling

Early in August, samples taken from TK-P3 were analyzed and found to be of acceptable shipping quality if blended to meet plutonium shipping specifications. Laboratory tests revealed the presence of no organic contamination, but Pu retention numbers for solvent contacted with P3 material were a factor of 200 greater than for organic contacted with normal uranium solution.

Based on the laboratory results, approval was obtained from U-Plant to blend about 40 tons of P3 UNH with the UNH in P1 and P4. Sample results following the blending indicated the P1 and P4 Pu levels to be 7 ppb and the material was shipped to U plant.

6. Demineralized Water

During the shutdown, installation of the new anion unit was completed and new Amberlite IR45, a weak base resin, was installed. However, because the manifold tie-in between the cation and anion units did not permit flexibility during regeneration, the new unit will not be operated until additional piping can be installed during the next shutdown.

7. Solvent Treatment

- (a) During the shutdown the 20 Column (T-R2) fluorothene cartridge was replaced with the spare fluoroethene cartridge. The cartridge consisted of 1/8 inch thick fluoroethene sieve plates with 1/8" holes on 1/4" centers, 23% free area on 2 inch spacing. The top disengaging section of the column was filled with a yellow tan sludge with some bright orange material all of which was washed down into the column. As a result the

7. Solvent Treatment (continued)

plates were coated with the sludge up to 1/8" thick samples of the sludge showed the orange material to be UO_2CO_3 and the other was sodium diuranate. This sludge apparently remained in the column as a result of processing organic with a high uranium content and was not removed by the flushes during shutdown.

Samples of the fluorothene snipped from the used cartridge were tested by flexing and the results indicated no significant loss of flexibility either by chemical or radiation attack.

(b) TK's G1 and G7 were given standard flushes to remove solids.

8. Waste Treatment and Acid Recovery

The recovered acid header system was flushed to facilitate installation of new flow meters in column acid streams. The vacuum fractionator and the acid storage tanks (U1 & 2) were also flushed. The acid pumps were inspected and repaired. The nitric-oxalic flushing appeared to be the most effective in reducing line radiation readings, however, the caustic-tartrate may have been required to condition the system. Radiation readings on various equipment pieces during decontamination are listed below:

Recovered Acid System
Radiation Readings at Different
Locations - Shutdown Aug. 1962

CP Readings on Acid System

<u>Check Pt.</u>	<u>Starting Rdg.</u>	<u>After Caustic Tart.</u>	<u>After Oxalic Nitric</u>	<u>After H₂O Flush</u>
On U1 TK	1500	15	-	-
On U1 TK	460	200	-	-
On Cuno Filter	4500	1000	-	250
On Pipe - A3	40	10	-	3
On Pipe - B3	80	10	-	3
On Pipe - C3	80	10	-	3
On Pipe - E6	100	50	-	4
On Lead Around Pipe J1	350	10	-	100
On Pipe Behind Lead LBXP	500	10	-	5
Thru Lead Plate HSS	450	5	-	5
A Round Edge at Shield HSS	900	450	-	100
On Pipe O K1	5	-	-	3
Flt. Side of Tank U5	110	-	32	20
Vacuum Fractionator Reboiler	900	900	330	300

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<u>Check Pt.</u>	<u>Starting Rdg.</u>	<u>After Caustic Tart.</u>	<u>After Oxalic Nitric</u>	<u>After H₂O Flush</u>
Fractionator Tower T-U6	4600	4000	3200	4000

9. Waste Management

- (a) The solids in the bottom of TK-101C were sampled and sent to Process Chemistry for analysis.
- (b) On 8/10/62 after 10 days of pumping the liquid level in TK 241-A-103 reached 121 inches as a result of pumping hot water from the cooling water hot well into the tank. The temperature of the water was approximately 60 degrees C.

10. Fission Product Recovery

The fourth cesium campaign was completed during the report period with the loading of 177,000 curies of cesium in 4 ORNL STT casks. The loading was routine except that cesium break through occurred at about 45,000 curies on each cask instead of the desired 50,000 curies. The reduced capacity is believed due to a higher loading temperature. Cooling water temperatures for this time of year limited Tank E1 minimum temperatures to 30-35 deg. C while during the third cesium campaign where 50,000 curie cask loads were obtained typical Tank E1 temperatures were 15-20 deg. C. Laboratory data indicate a 15 deg. C increase in temperature will reduce Duals loading capacity by about 25 percent.

Following completion of the cesium loadouts, preparations were underway for a plant test of the nickel-ferrocyanide precipitation process for the recovery of cesium.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 on 8/31/62 TO 2400 ON 9/6/62

I. Production

A. HAF Processed: 88 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	0.59	4.5	3.9	P4
2	88.9	2930	5.8	P3
3	78.3	1840	7.2	P3
4	37.4	1160	6.6	P3
5	19.7		7.5	P3
6	9.93	190	7.2	P3
7	5.4	100	7.3	P3
8	4.3	50	7.6	P3
9	2.4	12.6	7.7	P1

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
1	23.3			5	4	E6
2	11.7	0.33		6	30	E6
3				6	50	L9, L11
4	5.2	.006		5	40	2 Cans L9
5	6.0	.005		6		2 Cans 234-5
6	3.34	.002		6		4 Cans 234-5

D. Overall Waste Loss - % of Total Production

U = 0.25   Pu = 0.63

E. Rework - % of Total Production

U = Nil
Pu = 43.3% Primarily out of Spec. Material → E6/N1

II. Waste TreatmentA. Volumes to UGS

<u>Source</u>	<u>Volume</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	7222	82	106A
	26008	296	104A
Cell Dr.	6598	75	106A
FP	5981	68	106A
1WW	10190	116	106A
CW	23760	270	

B. 1WW Flows: 9.4C. Tank Farm - Boil-off Rates

<u>Waste TK</u>	<u>Estimated Boil-off, Gal/Min.</u>
101	3.0
102	2.8
103	0.3
104	2.4
106	15.1

Notes:1. Plant Operating Information

Normal processing was resumed on September 3, with "cold" HAF on at 0505 at a nominal CF of 3.0. Processing of "hot" feed commenced at 1630 on Sept. 3. Two batches (~ 12 tons) of "cold" uranium were recycled to the HAF at startup to provide receiver space for large volumes of plutonium-bearing flush and sump solutions from the Anion-Exchange Facility. Feed material containing higher-than-normal Pu-240 content was processed during the report period.

2. Flowsheet:

- (a) The plant was started up on the same flowsheet that was in use at the end of the last operating period, with the following exceptions:
- (1) The HAF-uranium concentration was 1.3, rather than 1.71 M. The HAF rate was 104, rather than 92 flows during processing of "cold" feed.
  - (2) The HAX rate was 385, rather than 460 flows, during processing of "cold" feed.
  - (3) The ICX-Temperature was 55, rather than 57°C.
  - (4) The HAW was routed directly to the 2WW Concentrator, and the 1WW Concentrator was removed from service during the special Single Cycle Concentrator Test.

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- (5) The 2AS was started at 3.6 flows.
- (6) The 2A Pulse Frequency was 82, rather than 87 cycles per minute.
- (7) The acidity in the Solvent Extraction System at startup was about 50% above flowsheet.
- (b) On September 4, the 2A Pulse Frequency was increased from 82 to 86 cycles per minute.
- (c) On September 4, the HS Pulse Frequency was increased from 75 to 80 cycles per minute.
- (d) During the period of September 4 through September 6, numerous adjustments were made in the rates of acid-addition streams to reduce the acidity to flowsheet values.
- (e) On September 5, the HA-NaNO₂ was reduced from 0.90 to 0.81 flows.
- (f) On September 4, the HAX was increased from 410 to 475 flows. It was reduced to 440 flows on September 4 and to 420 flows on September 5. The HAF rate was adjusted accordingly to maintain the proper saturation of organic in the HA Column.

3. Feed Preparation

Metal dissolution at startup continued to use 57% HNO₃ diluted to 50% in the dissolver with the extra rinse water. The extra 600 gal. rinse volume obtained from the use of 57% acid has failed to decrease in the U & Pu coating waste loss.

"Nonrepresentative" metal dissolution was accomplished in dissolvers A3 and C3. The metal age has been 203 days to 297 days old. No mercuric nitrate was used for iodine suppression. Even so I₁₃₁ emission was at the lower detectable limit.

4. Solvent-Extraction

Solvent-extraction performance immediately following startup of the plant was characterized by gamma activity levels throughout the system which were considerably above normal. This resulted in seven batches of uranium and five batches of plutonium product which exceeded specifications and required segregation for future rework. The high activity was due to a combination of the dilute HAF and 2DF, and high acid concentration throughout the system at startup. The dilute feed streams made it impossible to maintain the saturation in the HA and 2D Columns at levels high enough to provide good decontamination coincidental with tolerable waste losses. The excessive acidity, which decreased the Zr-Nb decontamination effectiveness, resulted from unfamiliarity, with numerous new flow-control systems which were installed during the Shutdown Period. As soon as "normal" HAF and 2DF concentrations were established and the acidity was reduced,

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the activity quickly returned to tolerable levels. At the end of the report period, both the uranium and plutonium products were within shipping specifications.

Temporary high First Cycle losses were experienced during the period of high activity and approximately 2000 gallons of LWW were segregated for eventual rework. As the system stabilized, the losses rapidly decreased to the limits of analytical detection.

Plutonium contamination in the uranium product - 3000 ppb immediately after startup - decreased to less than ten ppb after six additional batches had been processed, indication of excellent recovery of the Final Uranium Cycle. The poor plutonium decontamination at startup was caused by startup on dilute, 2DF which was accumulated from the neptunium recovery run.

5. Solvent Treatment

Failure of the 20F jumper solenoid closed the 20F valve and required a temporary shutdown of R-Cell and K-Cell on 9/17/62.

6. Final Plutonium Processing

Following startup on September 3, the operation of N-Cell has been excellent. No signs of pushing problems nor resin loss have been evident. Decontamination factors have ranged from 15 to 30 depending on the activity level of the 2BP stream.

7. Waste Concentration and Acid Recovery

Beginning with plant startup on September 3, the F-Cell single cycle acid recovery scheme has been in operation. Measures taken to keep the Zr-Nb entrainment and Ru volatilization to a minimum include adding NaNO_2 to make the LWF .005M NO_2 , adding .2gpm of water to E-F6 tower tray, adding 3gpm of water to the E-G6 demister pad, and overflowing E-F6 at approximately 125% of flowsheet.

Following startup the activity in the AAA quickly climbed, with the Zr-Nb leveling out at about 4×10^3 $\mu\text{c/gal}$ and the Ru fluctuating widely, but not exceeding 5×10^3 $\mu\text{c/gal}$. On September 5, F6 was partially jetted in order to save a LWW batch potentially high in plutonium and this resulted in upsetting the F-Cell equilibrium.

8. Fission Product Recovery

See report period 9/7/62 - 9/14/62 page 166.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 9/7/62 TO 2400 ON 9/13/62

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	3.7			0.003	0.03
Final	3.2	3.2	9.5	4.0	0.002	
Ion Exchange		1.1		0.5		
Overall	7.1	8.0	9.5	4.5	0.005	0.03

II. Production

A. HAF Processed: 144 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>
10	1.72	5.8	7.3	P1
11	1.67	5.4	7.7	P1
12	0.98	0.28	7.1	P4
13	0.94	2.1	7.6	P4
14	0.72		7.3	P4
15	0.58	1.4	7.1	P4
16	0.68	1.1	7.5	P4
17	1.18	2.5	7.2	P4
18	1.54	2.4	7.1	P4
19	1.32	2.8	7.5	P4
20	1.24	1.5	7.4	P4
21	1.11		7.1	P4
22	0.72	1.2	7.1	P4
23	0.80		7.3	P4
24	0.50	1.0	7.7	P4
25	0.66	1.6	7.6	P4
26	0.62	1.9	7.3	P4
27	0.60		7.5	P4
28	0.80	1.5	7.5	P4

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<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
29	0.87	1.8	7.7	P4
30	0.86	1.9	7.6	P4
31	1.16		7.8	P4

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
7	2.88	.008		5	20*	234-5
8	1.07	.009		6	10*	5 Cans 234-5
9	0.78	.005		5	2	Hold
10	2.31	.01	1000	5	2	234-5
11				5	25*	234-5
12	2.89	.003		6	2	4 Cans 234-5
13	1.98			5	4	4 Cans 234-5
14	1.38	.001		5	16*	Hold
15	1.63			6	15*	Hold
16	1.17	.001	1000	5	20*	Hold
17	1.30			6	20*	Hold
18	4.23	<.01		5	40*	Hold

* Unshielded Cans

D. Overall Waste Loss - % of Total Production

U = 0.24%   Pu = 0.45%

E. Rework - % of Total Production

U = 18.1% (P3 → K6)
 Pu = 3.15% Total
 SN - 2.01% Recy. Hd → E6
 Out of Spec. - 1.14% L11 → E6
 Material

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Vol.</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	11,384	79	106A
	52,465	364	104A
Cell Dr	13,062	91	106A
FP	9,506	66	106A
IWW	4,359	30	106A
CW	38,880	270	

B. IWW Flows: 8.4

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C. Tank Farm Boil-off Rates

<u>Waste TK</u>	<u>Est. Boil-off, Gal/Min</u>
101	3.0
102	2.8
103	0.3
104	2.4
106	15.1

Notes

1. Plant Operating Information

Failure of the 2AF Jumper on Sept. 7 necessitated a shutdown of the Final Plutonium Cycle on September 8 for replacement. Subsequent difficulty in obtaining electrical continuity from the 2AF Flowmeter in the new jumper necessitated an equilibrium shutdown of the entire Solvent Extraction System at 0700 on Sept. 8. The Final Uranium Cycle was started up at 0900 using "cold" uranium from the Rework Tank (P3) as feed material. The First Cycle and Final Plutonium Cycle were started up at 1700 on Sept. 8, after the difficulty was corrected, and maintained at a nominal 3.0 (actual 2.75) CF rate until Sept. 12. On Sept. 12, the actual processing rate was increased to 3.17 CF.

Two batches (~12 tons) of "cold" uranium were recycled from the Uranium Rework Tank (P3) to the HAF Tank on Sept. 7 to supplement the short supply of "hot" feed and prevent a rate reduction. Commencing on Sept. 11, "cold" uranium from Tank P3 was reworked through the Final Uranium Cycle at an average rate three tons per day.

2. Flowsheet

- a) The First and Final Plutonium Cycles were started up on Sept. 8 under the same flowsheet conditions that existed at shutdown with the following exceptions:
  - (1) The HA and HS Columns were partially stripped during startup of the Final Plutonium Cycle, due to a faulty HAX valve.
  - (2) The HSS-HNO₃ rate was temporarily in excess of 23 flows, due to a faulty Flow Controller.
- b) On Sept. 9, the HA-NaNO₂ was reduced from 1.11 to 0.96 flows. It was further reduced to 0.63 flows on Sept. 11.
- c) On Sept. 9, the 2AS flow was reduced from 3.6 to 3.3 flows, but was increased to 3.6 flows on Sept. 10.
- d) On Sept. 11, the Final Uranium Cycle processing rate was increased ~12 percent to rework material from P3 Tank.

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- e) On Sept. 13, the 2E Pulser was accidentally turned off for about 10 minutes.

3. Feed Preparation

Charging of all non-representative metal to the dissolvers was completed during this period. The average MWD/T and G/T for the 306 buckets processed 891 and 734, respectively. Five buckets contained 91% nickel coated slugs. Only one bucket of these slugs was added per dissolver charge because the nickel coating remained intact during coating removal and was not removed until contacted with acid in the dissolution step. The coating was then removed during the subsequent coating removal step. Coating waste samples have shown no significant reduction in uranium and plutonium since starting the 1000 gallon rinse test.

4. Solvent-Extraction

Solvent-extraction performance was generally very good during the report period, in spite of the forced shutdown. All uranium product was well within shipment limits. The plutonium contamination was generally lower than has been experienced in the past several months and the gamma ratio has averaged less than 1.0. Considerable fluctuation in the activity of the plutonium product was experienced during the report period. This was due to a combination of variable 3WB and HAF flow rates (caused by periodic partial pluggage of the systems) and a buildup of activity in the 2AF Tank (due to the use of a single agitator). However, the average plutonium product activity was within shipment specifications.

Decontamination performance of the First Cycle was slightly lower than has been typical of recent months, due to the unscheduled shutdown. Losses were generally very good during the report period. After a temporary high loss incident with startup, the HAW losses, rapidly decreased to non-detectable levels and remained low for the balance of the report period.

The apparent decontamination performance of the Final Plutonium Cycle was two to three-fold lower than normal. The problem here was attributed to a combination of high acidity in the 2AF and inadequate agitation in the 2AF Tank. The use of a single agitator in the 2AF Tank apparently permits activity to accumulate in the tank during "bursts" from the First Cycle. It then bleeds off slowly into the 2AF Stream, so that the 2AF activity is considerably higher than the 1BP activity, which is used as a measure of the decontamination performance of the Final Plutonium Cycle. Also, this gradual "bleeding off" of activity increases the time required for the product activity to return to normal level.

Performance of the Final Uranium Cycle continued to be excellent - with respect to both waste loss (via the 2EU Stream) and decontamination performance. No adverse effect was noted when the

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4. Solvent Extraction (continued)

2E Pulser was turned off. Neither high losses nor organic in the uranium product was noted. Rework of the material in P3 Tank proceeded without any noticeable effect.

5. Solvent Treatment

All checks indicate the 10 Column float has failed. Increasing the 10S acidity from 0.015 M to 0.05 M did not help.

6. Final Plutonium Processing

During the report period N-Cell operated smoothly and five cans of out-of-specification non-representative Pu were reworked via N-Cell using a FeS valence adjustment step. The new magnetic flowmeter was used for the first time to meter the rework flow from L9 to N1 and the results were highly successful. Virtually no increase in XAW losses were detected nor no problems encountered in flow control.

7. Waste Concentration and Acid Recovery

The single stage waste concentration test continued throughout the report period with mostly erratic results because of non-equilibrium operation of the equipment. Concentrator upsets occurred on Sept. 8 during a 16 hour plant shutdown and on Sept. 11 and Sept. 12 when F-Cell was shutdown for replacement of the F5 to F3 overflow jumper.

By the end of the report period it appeared F-Cell was operating at equilibrium conditions with the ZrNb remaining steady at about $6 \times 10^3 \mu\text{c/gal}$ and the Ru climbing slowly reaching about $1 \times 10^4 \mu\text{c/gal}$ by swing shift on Sept. 13.

8. Fission Product Recovery

Head End

A full scale plant test of the cesium nickel ferrocyanide process was completed during the report period. Cesium recoveries of 99 percent with a sodium decontamination factor of 900 were demonstrated. About 30 kilocuries of purified cesium product were shipped to Hanford Laboratories operation for future packaging studies. Complete details of this run are reported in HW-75051.

Following completion of the cesium run, strontium recovery run series 47 was started.

Strontium Semiworks

Loading of the HAPO II-2 cask (strontium shipment #9) was completed.

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8. Fission Product Recovery

Strontium Semiworks (continued)

The loadout proceeded smoothly except filtrate losses were higher than normal and the cask filter plugged before the desired loading was achieved. At weeks end measurements were being made to determine the quantity of strontium in the cask.

The cask loadout procedure was identical to previous loadouts with two exceptions:

- 1) The cask waste filtrate was routed to Tank 55 instead of Tank 64, and
- 2) The strontium product solution was made acid deficient before the potassium bicarbonate was added.

The first charge was necessitated by the high salt content of the product solution and the volume limitations of tank 67. To avoid sodium nitrate concentration in tank 67 near the solubility limit, the product solution was split between tank 64 and tank 67 and precipitated in tank 67 in two successive batches. The use of tank 64 for product storage during the first precipitation required the use of tank 55 for receiving waste filtrates.

The second change was made as a result of waste loss experience with the April cask loadout. In April, unsatisfactory filtrate losses occurred at pH 8. Chemical adjustment with sodium hydroxide and potassium bicarbonate to a pH of 9 corrected the loss problem. This prompted a laboratory investigation which indicated making the solution acid deficient before adding the potassium bicarbonate resulted in improved precipitation characteristics.

When flow was started through the cask, the rate was observed to be 6-fold below normal indicating a restriction in the loadout equipment or a partially plugged cask. Also, the dose rates on the lines indicated strontium was transferred to the cask even though the precipitates had been settled to allow decanting the high salt supernatant solution. During decanting of the second precipitation the cask plugged completely. A small volume of wash was passed through the cask with difficulty and loading efforts terminated. Efforts to explain the loading difficulty with cold synthetic solution in the laboratory were unsuccessful.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 9/14/62 TO 2400 ON 9/20/62

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	3.97	3.69			.003	.09
Final	3.04	3.0	9.1	3.0	.002	
Ion Exchange		1.6		.9		
Overall	8.0	8.3	9.1	3.9	.005	.09

II. Production

A. HAF Processed: 153.6 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>
32	1.19	2.4	7.7	P4
33	1.34		7.7	P4
34	1.35	2.5	7.5	P4
35	2.28		6.9	P4
36	1.84	2.0	7.5	P4
37	1.09		8.5	P4
38	1.33		6.9	P4
39	1.23		6.9	P1
40	1.89	12.6	7.2	P4
41	0.94	1.42	7.6	P4
42	0.88	2.8	7.5	P4
43	0.96	3.2	7.5	P4
44	0.84	2.3	7.6	P4
45	1.08		7.5	P1
46	0.91	2.2	7.7	P1
47	1.94		7.7	P1
48	0.94	1.8	7.5	P1
49	1.35		7.5	P1
50	1.70	1.5	8.3	P1

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
51	1.49	0.98	7.7	P1
52	2.01		7.8	P1
53	1.73		7.7	P1
54	2.06		7.7	P1
55	1.77	1.6	7.7	P1
56	2.08		8.2	P1
57	1.75		7.5	P1
58	1.73		7.0	P1
59	1.46		7.1	P3

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
19	4.84	<.01		6	*30	234-5
20	1.90	<.01		6	*22	234-5
21	1.4	.001		6	- 2	234-5
22	1.3			6	*20	234-5
23	1.7	<.01		5	*20	234-5
24	2.39			6	*20	234-5
25	1.9	.0037		4	*25	234-5
26	1.81	0.013		4	- 2	Hold
27	2.66	<.01		5	*25	Hold
28	3.68	.003		6	*30	Hold
29	5.48	.004		6	- 7	Hold

D. Overall Waste Loss - % of Total Production

U = 0.16%   Pu = 0.34%

E. Rework - % of Total Production

U = 5.2% - P3 → K1, K6
 Pu = 15.4% Total
 Sn - 0.67%
 Sumps - 7.4
 N. Cans - 7.0
 Flush - 0.3

III. Waste Treatment:

A. Volumes to UGS

<u>Source</u>	<u>Volume</u>	<u>G/T</u>	<u>Waste TK</u>
Wash	52,679	188.7	104-A
	10,422	37.3	106-A

A. Volumes to UGS(continued)

<u>Source</u>	<u>Volume</u>	<u>G/T</u>	<u>Waste TK</u>
Cell Dr.	15,419	55.2	106-A
FP	19,878	71.2	106-A
1WW	4,192	15.0	106-A
CW	75,357	270	102C → 103BX

B. 1WW Flows: 7.7Notes:1. Plant Operating Information

The plant was maintained at a nominal 3.0 (actual 3.0) CF during the report period. Reprocessing of off-standard uranium product through the Final Uranium Cycle was continued throughout the report period at an average rate of 3 tons per day.

Processing of "Nonrepresentative" metal was terminated and "normal" metal, processing commenced on September 16. The transition was accomplished by recycling two batches, (~10 tons) of "cold" uranium to reduce the plutonium inventory in the system. In addition, during the final dissolver heel cuts of "Nonrepresentative" metal, continuity of operation was maintained by recycling two "short" batches (~8 tons) of "cold" uranium to the HAF Tank.

The Final Uranium Cycle was shutdown at equilibrium for a brief period on September 17, due to mal-operation of the 20F Flow System.

On September 20, failure of the HA Column Interface Float Jumper permitted the entire contents of the HA Column to drain to the floor, necessitating a crash shutdown of the HA and HS Columns. The remainder of the First Cycle and the Final Plutonium Cycle were shutdown at equilibrium. The Final Uranium Cycle was operated until the heel flush of rework uranium from P3 Tank was processed. It was then shutdown at equilibrium.

Failure of the HAX Control Valve during shutdown operations necessitated its replacement.

2. Flowsheet

- (a) On September 14, the 2DF, 2DX, and 2EX rates were increased about eight percent to increase the rate of rework of off-standard uranium product solution.
- (b) On September 15, the HAX and HAF rates were reduced about five percent.
- (c) On September 17, the HA-NaNO₂ was increased from 0.63 to 0.78 flows.

- (d) On September 18, control of the HA Column was accomplished by varying the HAX flow. Normal control by varying the HAF flow was reverted to on September 19.
- (e) On September 18, rework of 1WW was commenced at a rate approximately 300 gallons in every feed batch.
- (f) On September 19, the 3WB specific gravity was increased from 1.25 to 1.28.

3. Feed Preparation

Dissolver operation continued to use 57% acid for dissolution and 1000 gallon rinse following coating removal. Non-representative metal heels were cut and all dissolvers started normal metal dissolution by 9/16.

When dissolver A3 was charged with its first charge of normal metal to establish heel, the uranium loss was .026 pounds per gallon in the coating waste. The dissolver was recharged and the coating removal procedure repeated. The coating waste this time contained .015 #U/gal. The first loss could have been caused by insufficient rinsing of dissolver and metal but the second loss probably resulted from either dissolution of uranium by caustic or a poor sample from TK-D2. However, a batch of nitrate solution, over-butted with caustic on 8/30, was sent through TK-D2 to underground storage. Analysis of the sample showed only .0009 #U/gal. which indicated the dissolver flushing might have been effective, and the cesium in TK-D2 coating removal solution results from caustic attack on the uranium slugs.

4. Solvent Extraction

Solvent extraction performance was generally good during the report period, in spite of the "upsets" encountered. Periodic partial plugging of the HAF flow system resulted in severe cycling of the organic saturation in the HA Column. This cycling produced periods of excessive losses followed by periods of negligible loss and excessive activity in the HSP Stream. Consequently, the plutonium product activity exceeded limits by 50 per cent during two periods. Also, high losses via the HAW Stream required 10,000 gallons of 1WW be segregated for rework.

Decontamination performance of the Final Plutonium Cycle continued to be marginal. Samples of 1BP and 2AF taken concurrently were in good agreement with respect to fission product activity and plutonium concentration, contradicting the hypothesis of inadequate agitation proposed in the last summary.

Performance of the Final Uranium Cycle continued to be excellent. No adverse effect was noted from the rework of the off-standard uranium from TK-P2. All uranium product continued to meet shipment specifications.

No adverse effect on performance of the First Cycle from 1WW rework was noted.

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5. Final Plutonium Processing

Operation of N-cell continued excellent during the report period. On September 16, the transition from unclassified to normal plutonium was made "on the fly" with segregation being based on the estimated normal plutonium holdup. Analysis of product batches indicated the segregation was very effective.

6. Waste Concentration and Acid Recovery

Acid recovered by single cycle waste concentration continued to be satisfactory during the report period except for a sixteen hour period on September 20 when the gamma activity level unexplainably jumped approximately ten-fold in Zr-Nb and four-fold in Ru. For the majority of the time, the Ru level remained at about 3×10^4 $\mu\text{c/gal}$ and the Zr-Nb at about 7×10^5 $\mu\text{c/gal}$.

Equilibrium concentrator operation was interrupted once on September 14, when the F5 to F3 jumper was changed and once on September 18, when the F6 bottoms were jettied to F7 for reclaiming plutonium that had been lost in HAW. On September 18, rework of 1WW to D5 also began which could have an affect on future F-Cell activity. On September 19, the E-F6 tray water was made 0.085 M in nitrite and the 1WF nitrite was cut in half to .0025 M in the feed. The net effect was to increase the total nitrite to E-F6 by 50 percent.

7. Fission Product Recovery

Head End

Strontium recovery run series 47, consisting of three sulfate runs, one oxalate run and one concentration run, was completed with a recovery of 72 kilocuries of strontium 90. The 1WW feed for this run series was two to three-fold above normal in cerium content while the zirconium-niobium and ruthenium content was below normal. The unusual fission product distribution in the 1WW apparently resulted from F11 flushes that were routed to F6 when F11 was bypassed.

The high cerium content of the 1WW resulted in excessive heat loads in the centrifuge during the first two sulfate runs and cake removal was difficult. The batch size was reduced for the third sulfate run and centrifuge performance was normal.

The large accumulation of cerium in the carbonate product from the three sulfate runs required making the oxalate run centrifugation in three parts to limit the centrifuge heat load to an acceptable level.

Strontium run series 48 was a single sulfate test run aimed at achieving strontium crude, acceptable for Strontium Semiworks feed, in a single step. The flowsheet differed from a standard sulfate run as follows:

- (a) The pH was adjusted to the range 2.5-3.5 instead of 0.7-1.5. Actual pH was 2.8;

- (b) The precipitation feed was adjusted to 0.20 M H_2O_2 and digested for thirty minutes after the pH adjustment and before² the lead addition;
- (c) After the lead was added the solution was digested one hour at 50°C instead of one hour at 90°C;
- (d) The centrifugation temperature was 50°C instead of 65°C; and
- (e) The cake was metatherized with a mixture of 5.0 M NaOH - 1.0 M Na_2CO_3 instead of straight 1M Na_2CO_3 .

The single cycle test failed to obtain a DF from cerium but strontium recovery was 89 percent which is considered good. The efficiency of the caustic carbonate metathesis solution for removing lead was not measured because of a bad sample.

Because 1WW feed was not available, an oxalate run was made on the single batch of carbonate product and 25 kilocuries of strontium 90 was sent to storage.

Strontium Semiworks

The HAPO II-1 cask was moved from the Strontium Semiworks to the Purex loadout station, leak tested and decontaminated. A calorimeter test showed a cask load of 94 kilocuries of Sr90.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 9/21/62 TO 2400 ON 9/27/62

I. Production

A. HAF Processed: 71.4 Tons

B. Uranium Product

1. Solvent Extraction

	<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, pphp U</u>	<u>Tons</u>	<u>Shipped To</u>
Split	60	1.49	2.3	3.6	E6
Batch	60	1.49	2.3	3.4	P4
	61	1.78		7.0	P4
	62	1.81	3.0	7.1	P4
	63	1.34	2.0	7.5	P4
	64	1.0	1.7	7.7	P4
	65	1.06		7.4	P4
Split	66	1.38	1.8	5.1	E6
Batch	66	1.38	1.8	2.4	P4
	67	1.03	3.1	6.9	E6
	68	1.09		7.1	E6
	69	1.10		7.2	P4
	70	1.33	3.08	7.3	P4
	71	1.11		6.3	P4

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
30	5.89	<.001		6	4	Hold
31	3.81	.002		5	* 30	Hold
32	3.83	.003		5	* 25	Hold
33	4.48	<.001		7	* 30	Hold

* Unshielded Cans

D. Overall Waste Loss ÷ % of Total Production

U = 0.5% Primarily G8 → 104   Pu = .4%

E. Rework - % of Total Production

U = 26.4%

Pu = 54%

Out of Spec. Mat'l. L11 - E6- 44%
 Sumps and Flushes - 10%

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II. Waste TreatmentA. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	364	104-A
Cell Dr	83	106-A
FP	54	106-A
IWW	0	
CW	270	103BX

- B. IWW Flows: Accurate determination of IWW flow impossible because of IWW rework.

Notes1. Plant Operating Information

The plant remained shut down (due to the failure of the HA Interface Float Jumper) until Sept. 24. Startup occurred at a nominal 3.0 CF with HAF on at 1525. The first batch of HAF was approximately 50 percent "hot" feed and 50 percent "cold" recycled uranium. The second batch was entirely "hot" feed.

Mal-operation of the Plutonium Ion-Exchange Facility after startup necessitated routing the 2BP Stream to the L Cell Package on Sept. 26. The concentrated 2BP was reprocessed via the HAF makeup tank and required recycling three batches (~15 tons) of "cold" uranium. Processing of "hot" feed was resumed on Sept. 27. Rework of IWW continued with ~250 gallons added to every "hot" batch of HAF.

2. Flowsheet

- a) The plant startup was with the same flowsheet as was in effect at shutdown, with the following exceptions:
- (1) The 2A pulse frequency was 75 rather than 87 cpm.
  - (2) The 2AF acidity was 3.6 rather than 3.2 M. This acidity was maintained by addition of fresh HNO₃ to the 2AF Tank.
- b) On September 24, the HA Pulse frequency was reduced from 82 to 79 cycles per minute.
- c) On Sept. 24, the 1BX pulse frequency was reduced from 56 to 50 cycles per minute to "break" a flood. It was increased back to 56 cycles per minute later the same day.

- d) On September 24, the 2A Pulse frequency was reduced from 75 to 50 cycles per minute to break a flood at startup. It was increased back to 70 cycles per minute later the same day, and to 86 cycles per minute on Sept. 25.
- e) On Sept. 25, the HA-NaNO₂ was increased from 0.77 to 0.63 flows.
- f) On Sept. 25, the 1CX temperature was increased from 57 to 60 degrees C and the 1CX rate was increased from 430 to 460 flows.
- g) On Sept. 26, the 2DS was reduced from 69 to 66 flows. It was further reduced to 64 flows on Sept. 27.
- h) On Sept. 26, the 2BP was routed to the L-Cell Package. It was routed back to the Ion-Exchange System on Sept. 27.
- i) On Sept. 27, the 2AF Tank was flushed back to the 3WB Tank and the 2A Interface was purged to the 3WF Tank.

3. Feed Preparation

Dissolver operation continued to use 57% acid and 1000 gallon fresh water rinse after coating removal.

During the temporary plant shutdown (Sept. 21-24) TK-D2 was flushed by digesting ~1400 gallons of 12% HNO₃ - 2% tartaric acid for 24 hours, then neutralized and sent to U.G.S. The acid flush was sampled (on the basis of Cs, the U was .006 #U/gal). However, after the flush, the coating waste loss reverted back to same general level of 0.16 #U/gal.

HAF specifications for E6 make-up were changed from 3.4 #U/gal. to 3.57 #U/gal. to compensate for dilution during jet transfer of the feed from TK-E6 to TK-H1.

4. Solvent Extraction

Solvent-extraction performance after startup was generally excellent. The severe problem of plugging in the HAF system, which was experienced prior to the shutdown, was virtually nonexistent. Consequently, control of both waste losses and activity in the First Cycle was excellent. Final Uranium Cycle performance remained excellent, with all uranium product meeting shipment specifications. Decontamination performance of the Final Plutonium Cycle was rather poor immediately after startup, probably due to the flooding in the 2A Column at startup. However, the decontamination performance improved greatly by the end of the report period.

Rework of IWW produced no noticeable adverse effects on solvent extraction performance.

5. Final Plutonium Processing

Following the plant shutdown on September 20, N-cell continued to run on rework plutonium introduced into TK-N1 via TK-L9. The 2B-2BP stream was allowed to run into TK-N1 to produce sufficient volume for normal XAF flow. Twenty-seven cans of out-of-specification material were processed before the Backcycle Waste System critical mass limitations required rework to stop. The operation of the magnetic flowmeter on the L9 to N1 feed stream was excellent during the rework period. N-cell decontamination performance during rework was a factor of about four below that experienced during normal processing. This points to the possibility that ZrNb not removed in the first pass is not as susceptible to future decontamination efforts.

On September 24, after the unit was stripped, the expansion of the resin created a problem by filling the push tank and resin receiver. During the subsequent startup, the unit did not push properly in spite of removal of some resin and consequently N-cell operation was extremely erratic. On September 27, after diverting the 2BP to the L-cell package, the entire resin inventory was replaced with a mixture of 75% 20-40 mesh and 25% 10-20 mesh Permutit SK resin.

6. Waste Concentration and Acid Recovery

During the first four days of the report period F-cell equipment was used for accumulating and concentrating J-cell sumps into rework batches. Following normal startup on September 24 and through the end of the report period, the gamma levels of the F-cell recovered acid remained well in the satisfactory range with both ZrNb and Ru concentrations analyses generally below 1×10^{-4} uc/gal.

Recovery of the HA Column contents from the floor, after the interface jumper failure, was accomplished by combining these wastes with sump rework in TK-F13 and rework 1WW in the unused F11 Waste Concentrator. Refluxing of these combined wastes was started on 9/26 by routing the condensate to TK-12 for use as 1WF dilution water. Rework of high plutonium 1WW continued through most of the run period at a ratio of approximately one part 1WW to 20 parts feed.

7. Fission Product RecoveryHead End

Strontium run series 49 was in progress. Hydroxyacetic acid was scheduled to be tested in run 49-S-4 and 49-S-5 as a substitute for tartaric acid.

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Strontium Semiworks

The HAP0 II-2 cask (strontium shipment #9) containing 94 kilocuries of strontium 90 was prepared for release to the AEC. The composition of the cask contents was as follows:

Curie Ratio - Isotope to Sr90

Sr-89 (9/1/62)	5.0×10^{-3}
Ce-144	$< 3.0 \times 10^{-3}$
Ru-106	$< 6.0 \times 10^{-4}$
ZrNb-95	$< 8.0 \times 10^{-4}$

Gram Ratio - Cation to Strontium

Calcium	0.01
Barium	0.002
Iron	0.0032
Manganese	0.018

Isotopic Ratio

Sr 90 to Total Strontium 0.563

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 9/28/62 TO 2400 ON 10/4/62

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	5.0	3.6			.003	.02
Final	2.0	3.1	10.0	0.9	.002	
Ion Exchange		1.5		3.5		
Overall	7.0	8.2	10.0	4.4	.005	.02

II. Production

A. HAF Processed: 202 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>
72	1.04	3.6	7.4	P4
73	0.613		7.5	P4
74	1.63	0.58	6.6	E6 *
75	1.87		7.5	P4
76	1.46		7.5	P4
77	1.40		7.3	P4
78	1.41	6.4	7.1	P4
79	1.49		7.5	P4
80	<.90	5.4	7.6	P4
81	1.66		7.1	P4
82	1.37	6.1	7.5	P3
1	1.66		7.5	P3
2	1.46	6.0	7.3	P3
3	1.81		7.5	P4
4	2.03	3.5	7.5	P4
5	1.71		7.5	P3
6	1.37	1.8	7.3	P3
7	1.50		7.5	P4
8	1.11	2.7	7.3	P4

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<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
9	1.36		7.6	F4
10	1.28	3.5	7.5	P4
11	1.41		7.5	P4
12	1.36		7.5	P4
13	1.22		7.6	P4
14	1.24	6.8	7.6	P4
15	1.87		7.7	P4
16	1.48	4.8	7.5	P4

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
34	25.8	0.014		5		Hold
40	8.97			6	9	Hold
> 42	8.09	0.003		4	6	Hold
43	4.48	0.002		5	6	Hold
44	3.71	0.003		5	* 30	234-5
45	3.19	0.001		5	* 22	234-5
46	1.62	0.007		5	2	234-5
1	3.95	<0.001		5	4	Hold
2	2.08	0.008		6	* 20	234-5
3	1.65	0.003		5		234-5
4	2.15	0.002		6	* 22	5 Cans 234-5
5	3.28	0.01		6	1	5 Cans 234-5
6	1.88	0.002		6	* 20	4 Cans 234-5
7	2.3	0.002		5	* 17	2 Cans 234-5

D. Overall Waste Loss - % of Total Production

U = 0.2%   Pu = 0.3%

E. Rework - % of Total Production

U = 3.3%
 Pu = 24.2%
 Fl6 → D5 0.3%
 Sumps 1.8%
 Out of Spec. 22.1% L9 → L11 → E6

III. Waste Treatment

A. Volumes to WGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	244	104-A
Cell Dr	30	106-A
FP	70	106-A
IWW	0	
CW	270	103BX

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B. IWW Flows: 10.3C. TK Farm Boil-off Rates

<u>Waste TK</u>	<u>Boil-off, Gal/Min</u>
101	2.3
102	2.6
103	0.3
104	3.3
106	14.0

Notes:1. Plant Operating Information

The plant was maintained at a nominal 3.3 (actual 3.4) C.F. during the report period. Rework of IWW continued until October 3 at a rate of ~250 gallons per seven-ton batch of HAF. All of the pulsers were inadvertently shut off for about two minutes on October 2, but no ill effects were noted. A batch (~6 tons) of "cold" uranium was re-cycled to the HAF Tank on Sept. 28 to absorb plutonium-bearing sump material.

2. Flowsheet

- a) On Sept. 28, the HAS, HAX, HAF, 2DF, and 2DX flows were increased ~10% to a nominal 3.3 CF rate.
- b) On Sept. 28, the 3WB Sp. Gr. was reduced from 1.28 to 1.25.
- c) On Sept. 28, the HAF uranium concentration was increased from 3.4 to 3.57 lb/gal.
- d) On Sept. 30, the 2EX was increased from 424 to 447 flows. It was further increased to 462 flows on Oct. 1.
- e) On Oct. 1, the 2DF-ferrous sulfamate addition was increased from 0.39 to 0.46 flows. It was reduced back to 0.39 flows on Oct. 2.
- f) On Oct. 3, the HAX and 2DX rates were increased slightly as the overall production rate was increased to ~3.0 C.F.
- g) On Oct. 3, rework of IWW was terminated.
- h) On Oct. 3, the 1BXP was reduced from 19.2 to 18.4 flows. It was increased back to 19.7 flows on October 4.

3. Feed Preparation

The weekly average age of metal charged was 160 days old, with 185 max. and 122 minimum age.. I₁₃₁ emission was 0.34 curies for period.

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4. Solvent Extraction

Solvent-extraction performance was generally very good during the report period. First Cycle plutonium losses remained very low, averaging less than 0.03 percent, and decontamination performance remained at about the same level as last report period. A noticeable increase in the HSP stream activity occurred when the IWW rework was terminated. This was attributed to the decrease in salting strength in the HA Column and the consequent increase in acidity required to maintain the same solvent saturation. Performance of both the Final Plutonium and Final Uranium Cycles remained at essentially the same level during the report period. All uranium product met shipment specifications. The plutonium product gamma activity was still above limits for Ion Exchange treatment at the start of the report period, but decreased to tolerable levels within two days.

The effects of the increase in the HAF uranium concentration are still somewhat nebulous due to continuing variations from batch to batch. However, it appears to have made it considerably easier to control the HSP stream activity without over-saturating the solvent and causing high plutonium losses.

5. Final Plutonium Processing

N-Cell decontamination performance was good during the report period but difficulties were encountered in the operation of XAF pumping system and XAW jet system. The routing of N-Cell waste was frequently switched between TK-F10 and TK-J1.

6. Waste Concentration and Acid Recovery

After four days of stable operation during which the F3 acid activity remained constant at about 4×10^4 $\mu\text{c/gal}$ Ru and 9×10^3 $\mu\text{c/gal}$ ZrNb, the acid activity started to climb as a result of the IWW rework which began on Sept. 24.

By the end of the report period, Ru activity had peaked at about 1.2×10^5 $\mu\text{c/gal}$ and ZrNb at about 2.0×10^4 $\mu\text{c/gal}$. During this period the IWW flow rate did not exceed the 7.5 flows which was established as the target overflow rate. Because of IWW rework a higher than normal SpG boiling temperature occurred in E-F6. Analysis of IWW revealed the activity levels were nearly double that of normal IWW.

7. Fission Product Recovery

Head End

Run series 49, consisting of 6 sulfate runs, two oxalate runs, and one concentration run was completed. A total of 172 kilocuries of strontium 90 were recovered with a decontamination factor of 12 from cerium.

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Head End

Hydroxy acetic acid was tested as a substitute for tartaric acid in runs 49-S-4 and 49-S-5. Equipment operation during these two runs was normal. Waste loss performance for the test runs compared favorably with tartrate runs, as shown below:

<u>Fission Product</u>	Waste Loss - Percent of IWW					
	Run Series 49					
	<u>S-1</u>	<u>S-2</u>	<u>S-3</u>	<u>S-4</u>	<u>S-5</u>	<u>S-6</u>
Sr	44	11	39	<u>33</u>	<u>47</u>	26
Ce	10	12	54	<u>51</u>	<u>36</u>	25

Note: Underlined numbers are the two test runs.

No firm conclusions can be made about the affect of hydroxy acetic acid on waste loss behavior. The tests are scheduled to be repeated in run series 50.

Sulfate waste losses were unusually high for all runs and are believed due to abnormal IWW. A gallon sample of this material sent to HLO also exhibited high losses in laboratory runs.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 10/5/62 TO 2400 ON 10/11/62

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	3.8			.002	.04
Final	3.1	2.9	11.5	6.3	.002	
Ion Exchange		1.5		0.5		
Overall	6.9	8.3	11.5	6.8	.004	.04

II. Production

A. HAF Processed: 174 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>
17	1.54		7.5	P3
18	1.70	7.8	7.5	P4
19	1.61	5.4	7.4	P4
20	1.56	5.8	7.5	P4
21	1.78	4.5	7.6	P4
22	1.78	3.2	7.5	P4
23	2.00		6.6	E6
24	1.98		6.6	E6
25	1.99	2.7	6.7	P3
26			6.7	E6
27	1.87		6.3	P3
28	1.25	2.2	7.5	P3
29	1.37		7.7	P3
30	1.02	2.2	7.1	P4
31	1.63		7.4	P4
32	1.38	3.2	7.7	P4
33	1.12		7.5	P4
34	1.50	2.1	7.6	P4
35	1.72		7.5	P4
36	1.78	2.5	7.6	P4

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
37	1.51		7.7	P4
38	1.82	2.6	7.8	P4
39	1.36		7.6	P4
40	2.12	1.8	7.7	P4
41	1.63		7.5	P4
42	1.29		7.6	P4
43	1.67		7.8	P4

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
8	1.4	.002		6		Hold
9	1.55	.002		6	20	Hold
10	1.78	.001		5	1	234-5
11	2.13	.001		5	*18	2 Cans-234-5
12	2.15			4	*18	1 " "
13	7.2			4	*300	Hold
14				4	.100	Hold
--	Directly to E6					
16				4	*700	Hold
17				2	.50	Hold
18				4	*500	Hold
--	Directly to E6					
--	Directly to E6					
21	2.54	.002		5	25	1 Can-234-5
22	2.1	.002		5	20	Hold
23	1.41			5	*15	Hold
24	1.64			6	.2	Hold

D. Overall Waste Loss - % of Total Production

U = 0.3

Pu = 0.4

E. Rework - % of Total Production

U = 11.4

Pu = 23.7

Sumps, Flushes - 2.5%

SN Cans - 2.7%

Out-of-Spec. Cans - 18.5%

III. Waste Treatment

A. Volume to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>TK</u>
Wash	19	106 A
	237	104 A

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<u>Source</u>	<u>Gal/Ton</u>	<u>TK</u>
Cell Dr.	47	106 A
FP	131	106 A
1WW	67	106 A
CW	270	103 BX

B. 1WW Flows: 3.8Notes:1. Plant Operating Information

The plant was maintained at a nominal 3.3 (actual 3.6) CF during the report period. Mal-operation of the Plutonium Ion-Exchange Unit necessitated the routing of the Second Plutonium Cycle product (2BP) to the L-Cell concentrator package and the recycle of three batches (~20 tons) of "cold" uranium to the HAF in order to decrease the in-process plutonium inventory.

2. Flowsheet

- a) On October 5, the 2BP was increased from 1.88 to 2.1 flows.
- b) On October 5, the 2DS was increased from 59 to 62 flows. It was reduced back to 59 flows on October 6.
- c) On October 6, the 1C Pulse Frequency was increased from 35 to 36 cycles per minute.
- d) On October 6, the 2A Pulse Frequency was increased from 88 to 89 cycles per minute. It was further increased to 91 cycles per minute on October 7.
- e) On October 7, SO_4 = - free 2 BX was started and the 2BX and 2BP flows were adjusted to concentrator "package" conditions as the 2BP was routed to the concentrator "package".
- f) On October 8, the concentration of the recovered acid had dropped off to ~54%. It was increased back to 57% on October 10.
- g) On October 8, the 2A Pulser was off for about five minutes.
- h) On October 9, the 2BP was routed into the Plutonium Ion Exchange Unit and the 2BP composition and rate were adjusted accordingly.
- i) On October 10, the 2BP was increased from 1.85 to 2.1 flows.
- j) On October 10, the ICX was increased from 440 to 455 flows.

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3. Feed Preparation

The weekly average metal age as charged was 154 days with 192 maximum, and 143 minimum. The maximum amount of I_{131} charged in one 24 hour period was 15 curies. I_{131} output was at low detection level.

The final dissolver fresh water rinse was heated to boiling on 11 charges in an attempt to reduce the coating waste losses. Although the coating waste loss was reduced from average 0.17% to 0.13%, the test was terminated because the extra time required (45-60 minutes) produced a feed shortage.

4. Solvent Extraction

Solvent-extraction performance remained generally good during the report period. Losses from the First Cycle showed some minor fluctuations, but averaged less than 0.03 percent. The activity of the uranium product stream (1UC) remained essentially constant - about two-fold above normal, but the plutonium product stream (1BP) activity showed a gradual decrease of about 25 percent. Final Uranium Cycle performance remained excellent, with all product meeting shipping specifications. The 2DS flow rate was reduced about five percent (while the processing rate was increased ~20 percent over the past three weeks) without any noticeable effect on product purity.

Decontamination performance of the Final Plutonium Cycle remained marginal, in spite of increases in the 2A Column pulse frequency and slightly higher-than-normal losses to the Backcycle Waste System.

5. Final Plutonium Processing

N-Cell mechanical operation became progressively worse during the report period and by Oct. 7 resin pushing ceased and the 2BP stream was routed to the L-Cell package. Although it did not appear at first that the condition of the resin was a contributing factor, replacement of the resin restored N-Cell to normal operating condition.

During the seven shifts N-Cell was down, a crash program was conducted to repair solenoids, air cylinders, and valves. When the unit was started on Oct. 10, a plug in the XAF line and a defective XAW valve were discovered and repaired.

Mechanical operation of N-Cell following startup was excellent. Lower push pressures (about 28 lbs.) and less push water were required per push than at any time in the past year. However, losses via the XAW and XSW were higher than normal, due to a high plutonium level in the 2BP stream and improper manipulation of the units operating cycle.

6. Solvent Treatment

Reuse of the No. 2 aqueous solvent wash wastes in the No. 1 solvent wash system was initiated during the report period. Excessive solvent losses from R Cell to G Cell apparently from R1 to G1 jetting operations necessitated procedure changes, such as, less frequent aqueous changes in TK-R1, less volume per change, different operating temperatures for the wash

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TK-R1 between 40 to 50 degrees C, and varying the length of time the recirculation pump is off before transferring the carbonate wash from TK-R1 to TK-G1. None of the procedure changes significantly reduced the solvent loss from the No. 2 to the No. 1 solvent system; however, preliminary data indicate plant overall solvent losses have been reduced about 300 to 200 gallons per day and overall wash volumes have been reduced by about 30% at current rates.

7. Waste Concentration and Acid Recovery

Rework of IWW was stopped on Oct. 4 and midway through the report period E-F6 specific gravity was reduced to near normal and the activity of the recovered acid decreased. By Oct. 11 the Ru activity had declined from a peak of $1.2 \times 10^5 \mu\text{c/gal}$ to about $5 \times 10^4 \mu\text{c/gal}$ and the ZrNb from $2.0 \times 10^4 \mu\text{c/gal}$ to about $1.3 \times 10^4 \mu\text{c/gal}$. The IWW overflow rate was maintained near 7.5 flows for the period.

While removing the TK-F13 agitator, it was noted that the vessel contents had evaporated to dryness. The vessel contained approximately 8,000# of U which had been collected from draining the HA column when the interface jumper failed combined with sump wastes and rework IWW. Approximately 1200 gallons of 60% HNO_3 were added to TK-F13 to dissolve the solids on the bottom of the tank.

8. Fission Product Recovery

Head End

Run series 50 was started. Substitution of hydroxy acetic acid for tartaric acid was again being tested. In addition, a double batching procedure which involved precipitation from twice the normal batch. The centrifugation was then made in two equal parts. The possible advantages of this procedure include:

- 1) A two-fold reduction in the number of IWW samples, and pH adjustment steps;
- 2) Elimination of half of the digestion periods; and
- 3) A reduction in chemical make-ups and attendant loss of chemicals to header flushes.

The disadvantage of the double batch procedure is it does not permit increasing the batch size to the full capability of the centrifuge when the IWW is dilute.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 10/12/62 TO 2400 ON 10/18/62

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			.002	.025
Final	3.1	3.5	9.0	3.4	.002	
Ion Exchange		1.5		11.3		
Overall	6.9	8.9	9.0	14.7	.004	.025

II. Production

A. HAF Processed: 202.9 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	1.97	2.4	7.6	P4
45	1.55		7.2	P3
46	1.98	3.0	7.4	P4
47	1.73		7.4	P4
48	1.58	2.6	7.6	P4
49	1.71		7.4	P4
50	1.72	2.03	7.5	P3
51	1.6		7.4	P3
52	1.66	2.73	7.8	P3
53	1.44		7.0	P3
54	1.09	5.0	7.6	P3
55	1.28		7.5	P3
56	0.99	7.2	7.5	P3
57	1.48	5.5	7.5	P3
58	1.51	4.6	7.7	P3
59	1.20		7.7	P4
60	1.37	3.15	8.2	P4
61	1.23		7.2	P1
62	1.33	2.9	7.5	P4

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
63	1.01	3.15	7.2	P4
64	1.47	4.5	7.6	P4
65	1.38		7.6	P4
66	1.25	2.8	7.7	P4
67	1.26		7.5	P3
68	1.23	2.6	7.7	P3
69	1.70		7.5	P3
70	1.29	2.3	7.4	P4

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
25	1.89	.002		5	20	Hold
26	2.0	.002		6	4	234-5
27	1.51	<.001		4	15	3 Cans 234-5
28	1.53			6	15	3 Cans 234-5
29	1.76	<.001		4	15	Hold
30	3.05	.001		5	18	2 Cans 234-5
31	1.39	<.001		5	18	234-5
32	0.64	<.001		6	15	Hold
33	0.36	.001		5	2	2 Cans 234-5
34	0.49	.001		5		2 Cans 234-5
35	0.39			5	2	Hold
36	0.40	.001		5	12	Hold
37	0.49	.002		5	1	Hold
38	0.88	.003		5	1	2 Cans 234-5
39	0.48	.001		5	10	234-5
40				5		2 Cans 234-5
41	0.41			6	13	Hold

D. Overall Waste Loss - % of Total Production

U = 0.2%   Pu = 0.2%

E. Rework - % of Total Production

U = Nil

Pu = 24.7%

Sumps = 0.4%

SN = 1.1%

High Cans via L11 23.2%

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	235	104A
Cell Dr	12	106A

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A. Volumes to UGS (continued)

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
FP	117	106A
IWW	40.1	106A
CW	300	103BX

B. IWW Flows: 7.1

Notes:

1. Plant Operating Information

The plant was maintained at a nominal 3.3 (actual 3.6) CF during the report period. A severe burst of activity from the Second Plutonium Cycle necessitated diverting the 2BP Stream from the Anion-Exchange System to the L-Cell package for about seven hours.

2. Flowsheet

- a) On October 12, the HNO₃ addition to the 2DF Tank was changed from Purex Recovered Acid to UO₃ Plant Recovered Acid.
- b) On October 16, the 3WB nitric acid concentration was reduced ten percent.
- c) On October 16, the 2EX temperature was reduced from 48 to 42 deg. C. At the same time, the 2EX rate was increased from 420 to 434 flows.

3. Feed Preparation

The weekly average metal cooling time was 158 days, with a 200 day maximum and 121 day minimum. The maximum I₁₃₁ input to the dissolvers was 128 curies for a 24 hour period. The total I₁₃₁ output was 0.36 curies for the period, with 0.27 curies released in the last 2 days.

4. Solvent Extraction

Solvent-extraction performance was excellent during the report period. Waste losses from the First Cycle were generally below analytical detection limits and averaged less than 0.03 percent. Activity of the plutonium stream (1BP) remained essentially constant while the uranium stream (1UC) showed a twenty-percent reduction in activity, despite a general increase in activity of the feed stream.

Performance of the Final Uranium Cycle continued excellent, with all solvent extraction product meeting shipping specifications. While no severe changes were made in the 2DS flow rate, the scrubbing efficiency of the 2D Column did not appear to be particularly sensitive to a decrease in the 2DS rate.

Decontamination of the Final Plutonium Cycle continued to be marginal until October 13. At that time a severe burst of activity was emitted from the 2B Column. This burst presumably originated in either the 2AF Tank or the 2A Column - the column is considered the more likely source. The column was immediately shut down and the 2AF Tank was drained and flushed. After startup, the operating characteristics of the 2A Column had changed significantly. The extraction section appeared to be holding up less aqueous and the scrub section considerably more - an indication of a more efficient scrubbing operation. The activity of the 2BP stream decreased rapidly to a level three-to-four-fold lower than typical of recent weeks and remained there. Decontamination factors since startup after the burst have ranged from three to four thousand.

5. Final Plutonium Processing

Mechanical operation of N-Cell equipment remained excellent during the report period. Decontamination performance was also outstanding with DF's ranging between 40 and 50.

On Oct. 18 erratic NaF pump operation necessitated turning off the NaF flow and in view of the low product activity (product averaging about 0.6×10^{-11} $\frac{\text{ZrNb}}{\text{AT}}$) the fluoride flow was left off until activity levels would warrant its resumption.

6. Waste Concentration and Acid Recovery

Early in the report period the IWW rate increased to about 8.2 flows (vice 7.5) and the effect was readily visible in the activity levels of the recovered acid when the Ru dropped almost a factor of two to 2.5×10^4 $\mu\text{c/gal}$ and the ZrNb a factor of three to about 4×10^3 $\mu\text{c/gal}$.

On Oct. 17 the nitrite addition to the LWF was turned off (the E-F6 tower tray nitrite remained on) to test the effect on Ru concentration in the recovered acid. Deletion of this flow reduced the LWF nitrite addition one third, from 0.01 M to 0.0065 M. By the end of the report period the Ru activity had increased two fold to 9×10^4 $\mu\text{c/gal}$ while the ZrNb stayed essentially unchanged. During this test IWW flows were held to about 7.5 flows.

7. Fission Product Recovery

Run series 50 consisting of six sulfate runs was completed through the oxalate step. The double batch procedure described last week was used for the entire run series, and no equipment difficulties or process problems were encountered. Waste losses from the second centrifugation were lower than from the first centrifugation, thus

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indicating that digestion periods greater than one hour may be beneficial.

Hydroxy acetic acid was again tested as a substitute for tartaric acid to complex iron. The strontium and cerium loss behavior is shown in the following table.

Fission Product	Waste Loss - Percent of IWW					
	Run Series 50					
	<u>S-1</u>	<u>S-2</u>	<u>S-3</u>	<u>S-4</u>	<u>S-5</u>	<u>S-5</u>
Sr	22.2	9.0	<u>12.2</u>	<u>9.2</u>	~(1)	8.7
Ce	5.4	<2.6	<u>6.2</u>	<u>2.8</u>	~(1)	13.1

- Notes: (1) Not sampled
(2) Underlined numbers are hydroxy acetic acid testruns.

As shown in the above table, waste loss performance between tartaric acid and hydroxy acetic acid is identical. Substitution of hydroxy-acetic acid for tartaric acid will reduce the chemical cost for the purex strontium recovery by 45 percent at the current purchase price. Bulk purchase prices should be even more favorable.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 10/19/62 TO 2400 ON 10/25/62

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	3.5			.002	.07
Final	3.0	3.6	8.0	2.8	.002	
Ion Exchange		1.3		2.1		
Overall	6.7	8.4	8.0	4.9	.004	.07

II. Production

A. HAF Processed: 201.9 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>
71	1.30		7.5	P4
72	1.07	2.03	7.6	P3
73	1.38		7.5	P3
74	1.32	1.7	7.6	P4
75	0.99		7.6	P4
76	1.34	1.2	7.5	P4
77	<1.2		7.5	P4
78	1.69	1.8	7.7	P1
79	2.35		7.5	P3
80	2.13	1.7	7.9	P3
81	1.71		7.7	P1
82	1.02	2.7	7.6	P1
83	1.65		7.8	P1
84	1.69	2.1	7.5	P1
85	1.35		7.5	P4
86	1.82	3.1	7.7	P4
87	1.46		7.2	P4
88	1.91	3.0	7.5	P1
89	1.18		7.6	P4
90	0.91	2.8	7.7	P4

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
91	1.18	4.2	7.5	P4
92	2.18		7.4	P4
93	1.76		7.7	P4
94	2.06		7.6	P4
95	2.59	2.9	8.1	P4
96	2.7		7.1	P1
97	2.24		7.2	P1

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
41	0.40	.002		5	11	234-5
42	0.59			6	1	234-5
43	0.45	.002		6	10	234-5
44	1.04	.003		5	18	2 Cans 234-5
45	1.03	.003		5	15	1 Can 234-5
46	1.37	.002		5	15	3 Cans 234-5
47	1.27	.004		5	1	2 Cans 234-5
48	1.01			6	10	6 Cans 234-5
49	0.74	.008		5	12	3 Cans 234-5
50	1.13			6	12	6 Cans 234-5
51	1.16	.002		5	15	1 Can 234-5
52	1.43	.001		6	15	1 Can 234-5
53	1.30	.02		6	5	3 Cans 234-5
54	1.68	<.001		5	25	5 Cans 234-5
55	1.77	.001		6	1	6 Cans 234-5

D. Overall Waste Loss - % of Total Production

U = 0.17   Pu = 0.30

E. Rework - % of Total Production

U = 11.6% (K5 → E6)

Pu = 10%*

SN - 3.8%

Sumps, Flushes, Out of Spec. Cans - 6.2%*

* Recycle transfer sheets unavailable
for period 10/19 - 10/21.

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	288/29	104-A/106-A
Cell Dr	60	106-A
FP	155	106-A
IWW	42	106-A
CW	300	103BX

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B. IWW Flows: 6.6C. Tank Farm Boil-off Rate

<u>Tank No.</u>	<u>Est. Boil-off, Gal./Min.</u>
101	2.3
102	2.6
103	0.3
104	3.3
106	14.0

Notes:1. Plant Operating Information

The plant was maintained at a nominal 3.3 (actual 3.6) CF during the report period. The reprocessing of ~2800 gallons of digested waste solution was accomplished during the report period. Malfunctions in the power converter systems for the pulse generators resulted in three separate temporary losses of pulsing on October 25. The only significant effect on solvent-extraction operation was a flood in the 2A Column, which was alleviated by reducing the frequency and 2AF flow.

2. Flowsheet

- a) On October 19, the 2EX temperature was increased from 48 to 55 degrees C and the 2EX rate was reduced from 385 to 353 flows.
- b) On October 20, rework of recovered, digested waste from TK-F8 was commenced at a rate of ~150 gallons per batch of HAF.
- c) On October 22, the 2DF-ferrous sulfamate was reduced from 0.37 to 0.29 flows. It was further reduced to 0.24 flows on Oct. 23.
- d) On Oct. 25, the 2EX temperature was reduced from 55 to 45 degrees C and the 2EX flow rate was increased from 353 to 404 flows.
- e) On Oct. 25, rework of the digested waste solution was terminated.
- f) On Oct. 25, the 2A Pulse Frequency was reduced from 91 to 81 cycles per minute to "break" a flood. caused by a temporary stoppage of the pulser. It was further reduced to 75 cycles per minute when another loss of pulse caused a second flood.

3. Feed Preparation

Dissolver charging for this run period was completed on 10/24. The I₁₃₁ emission was 1.51 curies for the report period despite the addition of mercuric nitrate to the metal solution tanks starting 10/18. The daily charge of I₁₃₁ was 160 curies on 10/19 and 10/20, but then dropped to 109-135 curie per day until charging ended. Cooling times varied from 123 to 204 days.

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4. Solvent Extraction

Solvent extraction performance was generally excellent during the report period. Rework of the digested waste material in TK-F8 caused an approximately five-fold increase in the HAW loss (from 0.02% to 0.10%, ave.) in addition to a two-fold increase in the activity of the product (1BP and 1CU) streams. The momentary losses of pulsing on October 25 upset the 2A Column considerably but no problems were noted with the other columns. However, a small volume of organic may have been transferred to the 1CU and 2EU Concentrators during the last outage (which was about twice as long in duration as the other outages).

Performance of the Final Plutonium Cycle remained excellent until the upset mentioned earlier. The D.F. decreased about two-fold (from ~7000 to ~4000) during the report period, but the product remained within shipment specifications without fluoride addition in the Plutonium Anion-Exchange unit.

Performance of the Final Uranium Cycle also remained excellent with the DF's averaging about 1500 for the week. The activity of both the feed and product streams increased 50 percent, but the product remained within specifications without silica gel treatment.

5. Final Plutonium Processing

The operation of N-Cell equipment continued to be excellent during the report period. Based on product loadout, the average N-Cell plutonium throughput was equivalent to a uranium processing of 4.5 CF for the entire report period. Because of these high processing rates XAW losses necessarily showed some signs of erratic behavior as the "A" column color line operated nearer the XAW outlet.

The fluoride flow remained off almost the entire report period. On Oct. 25 rising 2BP activity levels prompted restoration of the addition. Removal of the fluoride reduced the DF from about 35 to about 8. However, because of the low 2BP activity, all N-Cell product remained within shipping specification.

6. Solvent Treatment

The 2EX temperature was raised from 45 degrees C to 55 degrees C to test the effect on emulsification in TK-R1. Solvent losses to G1 continued to represent about 30 to 40% of the volume transferred from R1 to G1 regardless of procedure changes.

Pulser outage on 10/25 resulted in minor column upsets and a solvent loss of about 1100 gallons presumably to the concentrators.

7. Waste Concentration and Acid Recovery

On Oct. 19 nitrite addition to the 1WF via TK-F12 was restored

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7. Waste Concentration and Acid Recovery (continued)

(page 196 weekly Oct. 12-18) but the Ru activity in the recovered acid did not decrease but remained at about 9×10^4 $\mu\text{c/gal}$. Although the initial increase in Ru activity seemed clearly associated with reducing the nitrite, the fact that it did not decline when the flow was restored makes such a conclusion questionable.

On Oct. 21 the ZrNb activity increased by a factor of two and one half to about 1.8×10^4 $\mu\text{c/gal}$, but the Ru activity level remained unchanged. The sharp increase was attributed to F8 rework material which was started on Oct. 20. IWW rates were gradually reduced to about 6.5 flows by the end of the report period in an effort to make IWW of higher Sr90 content for fission product recovery. This also had a tendency to increase the activity of the recovered acid.

8. Fission Product Recovery

During run series 50, a total of 166 kilocuries of strontium 90 were recovered with a decontamination factor of 16 from cerium. Total time cycle for the double batch operation was about equal to two single batches.

During run 51-S-1, some material from TK E3 was lost to the sump during the 90 degree C digestion period. After half of the contents of TK E3 was centrifuged and the temperature of the remainder was adjusted to 90 degree C, material was again lost to the sump. It was postulated that the material was foaming out of TK E3 because the tank was boiling but efforts to establish that the thermohm was reading incorrectly were inconclusive. The rest of the run series was completed without incident using a digestion temperature of 80 degrees C. Subsequently, a short was found in the cold side of the temperature transmitter.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 10/26/62 TO 2400 ON 11/1/62

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	2.9	3.2			.002	02
Final	3.5	3.5	6.8	2.9	.002	
Ion Exchange		1.3		2.1		
Overall	6.4	8.0		5.0	.004	02

II. Production

A. HAF Processed: 32.6 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>
98	2.21		7.5	P3
99	2.56		7.3	P1
100	2.17	3.1	2.9	P1
100A	--	--	5.8	E6
101	3.35		6.8	E6
102	3.17	5.6	3.3	P1
103	5.63		7.7	P1
104	4.15		7.5	P1
105	6.01		7.6	E6
106	12.3		7.5	E6
107	7.49		6.1	P1
108	7.19	7.0	6.0	E6
109	5.76		4.9	P1
110	4.46	4.9	7.7	P1
111	3.13		6.7	P1
112	5.24	3.7	4.5	P1
113	10.9		5.7	P1

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C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./Hr.</u>	<u>Shipped To</u>
56	1.78	.001		4	2	3 Cans 234-5
57	1.04	<.001		5	10	3 Cans 234-5
58	4.57	.002		5	3	Hold
59	7.96			7	5	Hold
60	4.5	.002		6	25	1 Can 234-5
61				5	45	L11

D. Overall Waste Loss - % of Total Production

U = 0.20   Pu = 0.30

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	264	104/106A
Cell Dr	116	106A
FP Waste	196	106A
IWW	22	106A
C.W.	300	103BX

B. IWW Flows: 6.6

C. TK Farm Boil-off Rate

<u>Waste TK</u>	<u>Est. Boil-off, Gal/Min.</u>
101A	2.6
102A	3.0
103A	0.3
104A	3.8
106A	14.2

1. Plant Operating Information

The plant was maintained at a nominal 3.3 (actual 3.6) CF until the planned shutdown on October 28. Continued malfunction in the electrical converter systems for the pulse generators (mentioned in the previous report) caused a thirty-minute pulser outage early on October 26 and a five minute outage on October 28. Five batches of "cold" uranium were recycled from the Final Uranium Cycle (TK-K6) to the HAF makeup tank prior to shutdown. Two of the batches contained organic resulting from the pulser outage, one was recycled to maintain continuity of operation during dissolution of the metal "heels" in the dissolvers, and two were recycled to purge the activity from the system. Processing of "hot" feed was terminated at about 0200 on October 28. The HAF was shut off at 1430 on October 28.

The solvent extraction system was completely shut down by about 1200 on October 29.

2. Flowsheet

- a) On October 26, the 2A pulse frequency was reduced from 75 to 65 cycles per minute to eliminate instability induced by the successive pulser outages. It was increased to 84 cycles per minute later in the day and to 88 cycles per minute on October 27.
- b) On October 26, the 2DF-ferrous sulfamate addition was reduced from 0.24 to 0.21 flows.
- c) On October 26, the 2EX temperature was increased from 44 to 55 degrees C. It was further increased to 57 degrees on October 28. (These changes were to test the effect of temperature on phase disengagement in the second solvent system.)
- d) On October 26, the 2EX was reduced from 404 to 359 flows.
- e) On October 26, two batches of "cold" uranium containing organic were recycled to the HAF. On additional batch was recycled on October 27 and two batches on October 28.

3. Feed Preparation

A Cell silver reactor was regenerated on 10/28 after processing off gases from 956 tons of irradiated uranium metal. No dissolution occurred during this period. The I_{131} emission remained at the lower level of detection for the entire period.

4. Solvent Extraction

Solvent extraction performance was considerably below normal the last three days of the operating period because of the pulser outages and the resultant column upsets and degraded organic in the "cold" feed which was recycled. In spite of the upsets, however, no severe activity bursts occurred and waste losses remained below or at the analytical detection limit. Decontamination performance of the First Cycle suffered a two-to-three-fold reduction, due partly to the upsets directly and partly to the more dilute "cold" feed.

An abrupt change in the generally decreasing decontamination performance of the Second Plutonium Cycle (mentioned in the previous report) was noted the last days of the operating period. The DF across the 2A and 2B columns jumped from about 2000 to about 8000 in a two-day period.

Decontamination performance of the Final Uranium Cycle remained good until shutdown, in spite of organic carry-over from the 1C Column and 1CU Concentrator during the long pulser outage. The DF's obtained remained above 1000 and increased to about 1500 by shutdown. Performance (both efficiency and stability) of the 2E Column remained excellent in spite of the numerous rapid changes in 2EX temperature and flow rate during tests in the Second Solvent Treatment System.

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5. Final Plutonium Processing

Operation of N-Cell became erratic just prior to the plant shutdown. Pushing became difficult and the XAW flow to TK-J1 could not be maintained, except for short intervals. Following shutdown of the unit on Oct. 28, the resin was stripped, jetted to TK-R8, and new resin of the normal blend (75% 20-40 mesh; 25% 10-20 mesh) added.

6. Fission Product Recovery

Run series 51 was completed recovering 173 kilocuries of strontium 90. The double batch procedure described previously, was used during this series of runs. In addition, hydroxy acetic acid was substituted for tartaric acid in runs 51-S-2 and 3 and 51-S-7. No adverse effect was observed on either decontamination or waste loss as a result of double batching or the use of hydroxy acetic acid.

The volume of 2.5 M nitric acid used for removing the carbonate cake in the sulfate step was reduced from 240 to 160 gallons. This reduction in volume permits increasing a run series to seven consecutive sulfate runs before the E1 vessel is filled to capacity with carbonate product.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 11/2/62 TO 2400 ON 11/8/62

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	4.1	3.5			.002	.04
Final	2.9	3.8	6.5	4.0	.002	
Ion Exchange		1.3		0.17		
Overall	7.0	8.6	6.5	4.2	.004	.04

II. Production

A. HAF Processed: 99.4 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	6.09		6.1	P1
2	1.65	2.1	4.3	P4
3	0.971		7.0	P3
4	1.16		6.8	P3
5	0.98		7.6	P4
6	.94	2.8	7.0	P3
7	<1.0		7.2	P3
8	1.49	2.3	7.2	P3
9	1.80		7.2	P4
10	1.46	7.7	7.1	P4
11	1.84	21.0	7.5	P4
12	1.1	34.5	7.4	P3
13	1.21	29.8	6.3	P3
14	1.51	18.4	7.3	P1
15	1.43	7.5	7.0	P3
16	1.1	4.3	7.5	P4

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C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
1						L11
2	1.95			5	15	234-5
3	1.25	.002		5	12	234-5
4	0.80	.001		6	1	5 Cans 234-5
5	1.20	.006		4	1	234-5

D. Overall Waste Loss - % of Total Production

U = 0.24%

Pu = 2.0% * Includes a reported but unconfirmed 1348 units - F16 → E3

E. Rework - % of Total Production

U = 20.0% (Primarily P1 → K1 - High Pu and Organic bearing UNH caused by pulser failure)

Pu = 27.9%

Cans 21.8%

SN 4.7%

Sumps 1.4%

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/T</u>	<u>Waste TK</u>
OWW	265	102A
Cell Drainage	120	106A
FP Waste	190	106A
IWW	-	-
CW	300	102A → 103BX

B. IWW Flow - Undetermined (Startup)

Notes:

1. Plant Operating Information

The plant was shut down until November 5 for routine maintenance work and physical integration of the Neptunium Recovery Package with the regular system.

The plant was started up with "cold" HAF on Nov. 5. "Hot" feed was started at 0600 on Nov. 5. The plant was started up at a 3.0 C.F. and after conditions had stabilized the rate was increased to a nominal 3.6 C.F. on November 6.

1. Plant Operating Information (continued)

Prior to the general startup, the Final Uranium Cycle was started up and the uranium which was segregated prior to shutdown (because of high organic activity, and plutonium content) was processed.

A three-hour equilibrium shutdown on Nov. 7 was necessitated by failure of the HAF Pump. Startup was accomplished without undue difficulties.

2. Flowsheet

- a. The plant was started up under the same flowsheet conditions that existed at shutdown, except for the following:
  - (1) The 2AX flow was not started until analysis indicated plutonium in 2AF. This was done to prevent the usual startup activity burst from entering the Final Plutonium Ion-Exchange System.
  - (2) The 2DF-ferrous sulfamate was 0.16, rather than 0.21 flows.
  - (3) The 3WB-HNO₃ concentration was ~ 9.5 rather than 6.5 lb./gal.
  - (4) The 2DW losses were maintained at 6 to 8%, rather than 10-12%.
  - (5) The 2EX temperature was 55 degrees C at startup.
- b. On November 5, the IBX pulse frequency was reduced from 54 to 49 cycles per minute to stabilize the operation. It was increased back to 55 cycles per minute on Nov. 6.
- c. On November 5, the 2D pulse frequency was reduced from 69 to 60 cycles per minute to maintain stable operation during rework of the last of the organic-bearing uranium.
- d. On November 6, the HAF, HSS-H₂O, HAX, HAO, and ICX streams in the First Cycle and the 2DF, 2DX, and 2EX Streams in the Final Uranium Cycle were increased as the production rate was increased from 3.0 to 3.6 C.F.
- e. On November 6, the 2D pulse frequency was increased from 60 to 67 cycles per minute. It was further increased to 69 cycles per minute on Nov. 7.
- f. On Nov. 7, the 2DF-ferrous sulfamate was increased from 0.16 to 0.36 flows. It was reduced back to 0.29 flows on Nov. 8.
- g. On Nov. 8, rework of the solution from the recovered HA Column leak on Sept. 20, was commenced with 150 gallons added to a batch of HAF. After a two-batch interruption for observation of possible deleterious effects, rework was continued at a rate of 300 gallons per batch (~ seven tons) of HAF.

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3. Feed Preparation

The line from the coating waste collection tank TK-D2 to UGS line became plugged. The procedure of coating waste handling calls for combining the coating waste and the dissolver rinse in TK-D2, sampling and receipt of the sample results prior to disposal of the waste from TK-D2. Such a procedure tends to permit formation of solids which could plug the jet-out line. The line flushed by using coil water but the flow was very slow. Steaming the line was also ineffective. After installation of the F12 pump, (suction 12" from bottom of tank), the waste pumped slowly through the line at first but after two hot caustic flushes were transferred, the line opened up. Jetting of the coating waste was again started. Mercuric nitrate was no longer used because the metal was nearly all greater than 140 days old. I_{131} emission for the period was 0.16 curies.

4. Solvent Extraction

Solvent extraction performance was generally good during the report period. Only a mild and very short-lived activity burst occurred as a result of "stripping" the solvent in the HA and HS columns at shutdown. Another short and relatively mild burst occurred as a result of the temporary shutdown for replacement of the HAF pump on Nov. 7. Waste losses were maintained low during both startups, averaging less than 0.05 percent for the report period. Performance of the Second Plutonium Cycle was excellent. The product stream (2BP) remained well within limits for final cleanup in the Ion-Exchange System, in spite of the burst which occurred on Nov. 7 from the temporary shutdown of the First Cycle.

Performance of the Final Uranium Cycle was also good, in spite of a slight instability in the 2D Column resulting from rework of the organic-bearing uranium at startup. It was necessary to reduce the pulse frequency about 15 percent to correct the instability. However, by the end of the report period, the "do bads" which had accumulated in the solvent from the rework operation were purged from the system and the frequency was returned to the normal 69 cycles per minute. During the approximate two-shift lag between the time the 2D Column stabilized and the time the frequency was increased, the column scrubbing performance was subnormal and six batches of uranium exceeded specifications with respect to plutonium contamination. The problem was quickly corrected by the frequency increase.

5. Continuous Neptunium Recovery

In preparation for continuous neptunium recovery in the J-Cell Package, the 3WB acidity was increased to ≥ 8 molar and the 2DW uranium loss was reduced from ~ 10 to 12% to 6 to 8 percent, nominally. No process problems have resulted from these changes, but some difficulty with control was evidenced, especially with the 3WB acidity. A potential problem of limiting concentrator capacity at the higher nitric concentration was also evidenced.

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6. Final Plutonium Processing

Prior to plant startup the scheduled N-Cell maintenance was completed. Operation following startup was good except that the XAW stream would not jet to TK-J1, and thus the flow was routed to TK-F10 for essentially the entire report period. Investigation of the problem points to a defective jet and replacement of the jet assembly as soon as possible has been recommended.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 11/9/62 TO 2400 ON 11/15/62

I. Typical Solvent-Extraction Performance by Cycles

	Gamma dF		% Recycle		% Loss	
	U	Pu	U	Pu	U	Pu
First	3.9	3.8			.002	.02
Final	2.8	3.4	8.0	3.5	.002	
Ion Exchange		1.6		0.17		
Overall	6.7	8.8	8.0	3.7	.004	.02

II. Production

A. HAF Processed: 170.3 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>
17	<1.0	3.4	7.3	P4
18	<1.0	4.0	7.6	P3
19			7.5	P3
20	1.07	4.2	7.1	P4
21	0.815		7.3	P4
22	1.28	9	7.6	P4
23	1.36	16.4	7.5	P4
24	1.11	18.1	7.5	P1
25	1.49	12.7	7.5	P4
26	1.21	9.5	7.5	P4
27	1.08	6	8.0	P4
28	1.16		7.3	P4
29	2.21		6.3	P1
30A	4.13	29	2.3	P1
30B			6.7	K1 ←
31	8.08	22.4	6.8	P1
32A	5.22	17.0	6.9	P1
32B	5.22		2.3	K1 ←
33	4.27	12.1	7.8	P1

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
34	3.72		7.8	P1
35	3.92		7.8	P1
36	4.51	<u>11</u>	7.6	P1
37	3.31	8.1	7.7	P1
38	3.61	6	7.4	P1
39	3.23	5.1	7.3	P1
40	3.20	4.3	7.4	P1

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
6	0.82	.008		5	1	234-5
7	1.13	.006		5	1	234-5
8	0.80	.003		5	1	234-5
9	0.43	.003		5	10	3 Cans 234-5
10	0.53	.004		6	2	234-5
11	0.76	.07		5	11	4 Cans 234-5
12	0.86	0.17		5	1	234-5
13	0.47			5	2	234-5
14	0.53	0.008		5	2	234-5
15	0.47	0.009	850	6	1	234-5
16	1.15	0.005		5	1	234-5
17	1.08	<.003		6	1	234-5
18	1.44	.004		5	1	234-5

D. Overall Waste Loss - % of Total Production

U = 0.3   Pu = 0.4

E. Rework - % of Total Production

U = 4.8% (K6 → K1)
 Pu: Total 15.1% (Cans-8.5%)
     (Sumps, Flushes 1.5%)
     (SN - 5.0%)
     (F16 → D5 0.1%)

III. Waste TreatmentA. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	241	102A
Cell Dr	44	106A
FP	107	106A
IWW	0	
CW	300	103BX

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B. IWW Flows: 6.2C. TK Farm Boil-off Rates

<u>Waste TK</u>	<u>Est. Boil-off, Gal/Min.</u>
101	2.6
102	3.0
103	0.3
104	3.8
106	14.2

Notes:1. Plant Operating Information

The plant was maintained at a nominal 3.6 (actual 3.6) CF. during the report period, except for three temporary outages. A temporary power outage within the building necessitated an emergency shutdown for approximately ten minutes on November 10. The solvent-extraction system was shut down at equilibrium on Nov. 11 because of a failed 3WF Pump. Continuing troubles after the pump replacement - first with the 3WF jumper (containing the control valve), then with numerous valves on essential jet systems - prolonged the shutdown period for twenty hours. A third temporary (20-minute) shutdown of the First Cycle to permit changing a failed diaphragm on the HSR Jet steam supply occurred on Nov. 14.

Rework of the recovered product-bearing solution previously refluxed in E-F11, commenced on Nov. 8, continued until the shutdown on Nov. 11. The remaining ~2000 gallons was transferred to the Waste Rework Tank (F8) for additional hydrolysis because of adverse effects on the process (high plutonium loss and increased gamma activity in intercycle streams).

2. Flowsheet

- a) On Nov. 9, the HAO was increased from 77 to 88 flows.
- b) On Nov. 9, the 2DF-ferrous sulfamate was reduced from 0.30 to 0.17 flows. It was increased to 0.24, then to 0.40 flows on Nov. 10.
- c) On Nov. 9, the 3WB HNO₃ was reduced from 10 M to ~8.7 M.
- d) The plant was shut down on Nov. 11 because of failure of the 1WF Pump. Startup attempts early on Nov. 12 were unsuccessful due first to inability to obtain HSR flow, then to insufficient 3WF flow. The plant was started up successfully at 3.6 CF. late on Nov. 12 at the same flowsheet conditions that existed at shutdown, with the following exceptions:

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2. Flowsheet (Continued)

- (1) The 2AF flow was not started until ~ seven hours after plant started up. The 1BP was routed to the 3WB Tank to prevent excessive activity from entering the Plutonium Ion-Exchange Unit.
  - (2) The wash solution in the No. 1 solvent wash tank (G1) was put on a six hour change cycle.
  - (3) The 1BXP was 21, rather than 18 flows.
  - (4) Rework of the reclaimed product-bearing solution was terminated just prior to shutdown.
- e) On Nov. 13, the 1BXP was reduced from 21 to 18 flows.
- f) On Nov. 13, the 2DF, 2DX, and 2EX-H₂O rates were increased ~ 20% to a CF of about 4.4 and rework of "cold" uranium through the Final Uranium Cycle was commenced.
- g) On Nov. 14, the Final Uranium Cycle rate was reduced back to 3.6 and rework activity was terminated.
- h) On Nov. 14, the 2D pulse frequency was increased from 69 to 71 cycles per minute. It was further increased to 76 cycles per minute on Nov. 15.
- i) On Nov. 14, the 2A pulse frequency was increased from 84 to 86 cycles per minute. It was further increased to 88 cycles per minute on Nov. 15.

3. Feed Preparation

The D2 waste routing plugged again. The F12 pump was again installed on 11/15, but pumpout rate is slow. Metal age varied from 124 - 229 days. Total I₁₃₁ emission for the period was 0.61 curies.

4. Solvent Extraction

Solvent-extraction performance was generally very good, in spite of the numerous outages and equipment difficulties. The plutonium product was two-to-four-fold below shipment specifications throughout the report period, a result of recycling the 1BP to the 3WB Tank during the severe gamma burst at startup on Nov. 12. The uranium product activity increased to a maximum four-fold above shipment limits as a result of the burst, but had decreased to near normal by the end of the report period. The losses averaged slightly above normal during the report period, as a result of the rework operation (see Note 1). When the rework was terminated, however, the losses rapidly returned to normal (<.04).

Performance of the First Cycle reflected the shutdown and false startups.

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4. Solvent Extraction (continued)

The activity of the 2DF stream peaked at 9000 gamma ratio after startup on Nov. 12 and it was necessary to recycle ~seven batches of LBP to the 3WB tank to prevent a burst in the Final Plutonium Cycle. Even so, the LBP activity peaked about eight-fold above normal after the 2AF flow was started. By the end of the report period, however, the activity of both product streams had returned to near normal.

Performance of the Final Plutonium Cycle was excellent during the report period, with DF's ranging from 2000 to 7000, depending on the 2AF activity (high 2AF activity - high DF).

Performance of the Final Uranium Cycle was generally excellent. The CF remained essentially constant at ~1200, so that the product exceeded shipment specifications after startup. An instantaneous capacity in excess of 4.4 was demonstrated during rework operations. Losses at the high rate remained below detection, but the decontamination performance appeared to be slightly poorer than at 3.6 CF rates.

5. Final Plutonium Processing

Several weeks ago the XAW jet cooling water was hooked up to the XAW to TK-J1 line to intermittently pressure flush the line. On Nov. 9 a directive to cease such flushing was issued based on the possibility of plutonium polymer formation in the line.

On Nov. 12, while the plant was down, the XAW jet and steam DOV were replaced. Following the replacement and for the remainder of the report period the XAW stream was successfully routed to TK-J1.

6. Fission Product Recovery

Head End

Completed run series 52 recovered 154 kilocuries of strontium 90 with a decontamination factor of 32 from cerium. The carbonate cake from the fifth sulfate precipitation could not be removed from the centrifuge by standard dissolution techniques. The cake was finally removed with a combination of 6 M nitric acid and 1 M oxalic acid. Origin of the sticking cake was traced to processing the F6 concentrator bottoms. A review of previous cake removal difficulties indicates the cake stuck whenever F11 concentrator bottoms were processed.

Strontium Semiworks

The HAPO II-1 cask, returned from Quehanna, was flushed with 0.1 M HNO₃ air dried and the void volume measured as 7.98 liters. During an attempt to load the cask with the same strontium product as was loaded in the HAPO II-2 cask in September, the filter plugged prematurely. Such behavior indicated an off-standard condition existed

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Strontium Semiworks (continued)

with this material, consequently, it was set aside for rework. The cask and TK's 67 and 64 were thoroughly flushed before proceeding to new product in TK 1. Cask loading with strontium product from TK-1 was underway at the end of the report period.

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PROCESS SUMMARY OF PUREX PLANT OPERATION FROM 0000 ON 11/16/62 TO 2400 ON 11/22/62

I. Typical Solvent-Extraction Performance by Cycles

	Gamma dF		% Recycle		% Loss	
	U	Pu	U	Pu	U	Pu
First	3.9	3.5			.002	.02
Final	2.9	3.8	8.0	2.1	.002	
Ion Exchange		1.2		0.6		
Overall	6.8	8.5	8.0	2.7	.004	.02

II. Production

A. HAF Processed: 221.2 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>
41	2.41	3.6	7.3	P1
42	2.52	3.0	7.4	P1
43	2.45	2.2	7.2	P1
44	1.80	1.7	7.6	P4
45	1.73	1.8	5.6	P1
46	2.3	3.0	7.1	P1
47	2.15	2.6	7.3	P1
48	2.07	2.7	7.4	P4
49	1.7	2.6	7.3	P4
50	2.01	2.2	7.3	P4
51	1.50	1.8	7.6	P4
52	1.20	1.7	7.8	P4
53	*Acid Flush	--	1.3	P1
54	1.21	1.33	7.1	P4
55	1.15	1.8	7.4	P4
56	1.20	1.5	7.5	P4
57	1.02	1.8	7.0	P4
58	1.05	1.9	7.5	P4
59	1.31	4.0	7.6	P4
60	1.17	2.4	7.4	P4

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
61	1.02	2.8	7.4	P3
62	<1.10	2.8	7.3	P4
63	1.16	2.0	7.5	P4
64	1.09	2.4	7.7	P4
65	1.11	2.2	7.8	P4
66	1.23	2.4	7.5	P4
67	0.98		7.6	P3
68	0.89	1.8	7.5	P3
69	1.23		7.2	P3
70	1.20	1.68	7.6	P4
71	1.04		7.5	P4

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
19	0.627	.007		6	1	234-5
20	1.13	.006		5	13	234-5
21	.73	.007		5	2	234-5
22	0.65	.01		5	1	2 Cans 234-5
23	.66	.01		5	1	2 Cans 234-5
24	.62	.013		6	1	234-5
25	.55	.008		5	1	1 Can 234-5
26	.55	.006		5	1	4 Cans 234-5
27	.57	.006		5	15	All 234-5
28	.44	.008		6	12	All 234-5
29	.38	.005		6	14	Hold

D. Overall Waste Loss - % of Total Production

U = 0.2%

Pu = 0.3%

E. Rework - % of Total Production

U = 0.04%

Pu = 3.8%

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	204	102A
Cell Dr	14	106A
FP	134	106
IWW	35	106A
CW	300	103BX

B. IWW Flows: 6.4

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Notes:

1. Plant Operating Information

The plant was maintained at a nominal 3.6 (actual 3.6) CF during the report period. An emergency shutdown of approximately 30 minutes occurred on Nov. 16 when power to the pulse generators was inadvertently turned off. A batch of relatively dilute IWW was successfully reworked at a rate of ~300 gallons per batch of HAF. A temporary loss of electric power in the No. 2 Solvent System caused a brief upset in the Final Uranium Cycle on Nov. 21.

The continuous Neptunium Recovery System was completely integrated into the Solvent Extraction System on Nov. 20 when a portion of the 3WB stream was diverted to the 3AF Tank. The system will be referred to as the Neptunium Recovery Cycle.

2. Flowsheet

- a) A number of adjustments was made in the frequency of the 2D Column during the report period. The adjustments were in the range of 69 to 75 cycles per minute.
- b) On Nov. 20, the TBP concentration in the No. 2 solvent system was increased from 29 to 33 percent. It was reduced to ~30 percent by dilution over the succeeding 24 hours.
- c) On Nov. 20, the 3WB flow to the HA Column was temporarily reduced from about 36 flows to 30 flows, as the 3WB was diverted to the 3AF Tank. The rate of the HA Column was then increased to about 36 flows over the next 24 hours.

3. Feed Preparation

The coating waste routing from TK-D2 continued to give transfer problems. Caustic flushes have not improved solution flow. The pump is still being used in place of the jet for transfer. A temporary Hex jumper in 152 CR Div. Box was replaced with used rigid jumper in an attempt to improve the flow. During the report period, metal age varied from 126 - 235 days old. An average of 52 curies/day of I₁₃₁ was charged and .085 curies/day discharged from stuck for an average I₁₃₁ DF of ~600. No mercuric nitrate was used.

4. Solvent Extraction

Solvent-extraction performance was excellent during the report period. Both inter-cycle and product activities showed a two-fold decrease. Waste losses were excellent with the HAW plutonium loss averaging less than 0.04 percent. All solvent-extraction cycles recovered rapidly and completely from the series of upsets reported in the

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4. Solvent Extraction (continued)

previous summary. Decontamination performance in the First Cycle showed an apparent three-fold improvement which was attributed to purging from the system, activity which resulted from the previous upsets. Performance of the Final Plutonium Cycle remained essentially stable at a DF of about 3000. Decontamination performance in the Final Uranium Cycle decreased by about ten percent during the report period as the 2DF activity came down. The uranium produced averaged well under the shipping limits.

5. Neptunium Recovery

The Neptunium Recovery Cycle was completely integrated into the process on Nov. 20 when 3WB flow was routed to the 3AF Tank at a rate of 12 flows (~60% of designed capacity). No severe problems were encountered when the transition to "hot" feed was made. The differential pressure indicators for the 3B Column fluctuated considerably after the transition, but the apparent instability was not reflected in the static pressure indication. Some problems were encountered in "cold" solution make-up operations due to inoperable equipment and different requirements, but the troubles were eliminated as they occurred.

6. Final Plutonium Processing

Operation of N-Cell during the report period was excellent with DF's ranging between 25 and 35 and all product below 1^{-11} ZrNb/AT.

The new XAW jet installed Nov. 12 apparently solved the problem of maintaining the XAW flow to TK-J1.

7. Silica Gel

After being shut down for 20 weeks, silica gel operation was started on Nov. 17, to process out-of-specification uranium produced during plant upsets following unscheduled shutdowns.

8. Demineralized Water

Between Nov. 9 and Nov. 20 a carbon filter was installed downstream of the main demineralized water filter with the system set to continually filter 250 ml/min. of demineralized water. At the completion of the run the filter was removed and sent to Hanford Laboratories (John Skarpelos) to be analyzed for the amount and types of organics present on the filter.

Studies of our anion resin (Nalco WBR-weak base) conducted by John Skarpelos, indicate that the resin presently in use is of extremely low capacity (two hour breakthrough vs fifty hours for new resin), probably because of organic fouling. Attempts to recondition it so as to restore normal capacity have been unsuccessful. He has recommended that Duclite A-30B (medium base resin) be installed in the demineralizer anion units.

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9. Fission Product Recovery

Loading of the HAPO II-1 cask with 170 kilocuries of strontium 90 from TK-1 was completed at the strontium semiworks. This loadout was the first from CPD prepared product (vice Hanford Laboratories prepared product) and demonstrated that satisfactory product can be prepared by hydrogen peroxide treatment of the citrate HCP solution as it comes from the column rather than allowing decomposition of the citrate by radiolysis before peroxide treatment. The loadout solution was also observed to be free of black material noted in all previous loadouts.

At the end of the report period HAPO II-2 was being prepared for loading.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 11/23/62 TO 2400 ON 11/29/62

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	4.2	3.9			.002	
Final	2.9	3.5	8.0	2.9	.002	.03
Ion Exchange		1.2		.07		
Overall	7.1	8.6	8.0	3.0	.004	.03

II. Production

A. HAF Processed: 208.9 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>
72	1.17	1.4	7.3	P3
73	<1.13		7.4	P4
74	1.44	1.4	7.6	P4
75	1.04		7.7	P3
76	1.04	1.5	7.5	P3
77	1.34	1.4	7.5	P4
78	<1.11	1.8	7.8	P3
79	1.25		7.5	P3
80	1.28	1.1	7.4	P3
81	1.28		7.7	P3
82	1.12	1.9	7.6	P3
83	1.21		7.4	P3
84	1.27		7.4	P4
85	1.63		7.5	P3
86	2.28	3.6	7.7	P1
87	1.98		7.5	P1
88			7.5	P1
89	2.09		7.8	P1
90	2.06	3.4	7.6	P1

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
91	2.02		7.4	P1
92	1.86	3.6	7.5	P1
93	1.91		7.5	P3
94	1.96	2.1	7.6	P3
95	2.02	7.0	7.2	P3
96	2.02	2.9	7.3	P3
97	1.92		7.4	P1
98	1.92	3.6	7.9	P1
99	2.20	4.4	7.6	P1

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
30	.59	.005		6	13	4 Cans 234-5
31	.77	.004		5	15	1 Can 234-5
32	.93	.02		6	1	Hold
33	1.17	<.001		6	20	Hold
34	1.29			5		234-5
35	1.38	.004		6	1	2 Cans 234-5
36	2.78			5	2	2 Cans 234-5
37	2.77			5	2	4 Cans 234-5
38	3.05	.002		4	2	2 Cans 234-5
39	1.58	.004		5	2	5 Cans 234-5
40	3.61	.003		5	2	4 Cans 234-5
41	1.26	.002		5	3	2 Cans 234-5
42	1.20	.003		5	2	Hold

D. Overall Waste Loss - % of Total Production

U = 0.04%   Pu = 0.1%

E. Rework - % of Total Production

U = <0.1%

Pu: Total 2.0%

F16 → D5 0.4%

Sumps, Flushes 0.1%

SN 1.5%

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	175	102-A
Cell Dr	58	106-A

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A. Volumes to UGS (continued)

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
FP	157	106-A
IWW	78	106-A
C.W.	300	103-BX

B. IWW Flows: 6.4

C. Tank Farm Boil-off Rates

<u>Waste TK</u>	<u>Est. Boil-off, Gal./Min.</u>
101	1.3
102	2.4
103	0.3
104	3.4
105	
106	16.1

Notes:

1. Plant Operating Information

The plant was maintained at a nominal 3.6 (actual 3.65) CF during the report period.

2. Flowsheet

- On Nov. 23, the 2D pulse frequency was increased from 69 to 71 cycles per minute. It was increased to 73 cycles per minute on Nov. 24, then reduced to 71 cycles per minute on Nov. 25.
- On Nov. 26, the 2DF-ferrous sulfamate was reduced from 0.40 to 0.37 flows. It was further reduced to 0.33 flows on Nov. 28.
- On Nov. 26, the 1BXP was increased from 17.8 to 18.6 flows.
- On Nov. 26, the KMnO₄ concentration was reduced from 0.25 to 0.12 molar and the addition rate to the 10F tank was increased two-fold.

3. Feed Preparation

On 11/23/62 the jet on TK D2 to UGS line was reinstalled. TK-D2 jet out was satisfactory until 11/28/62 when the jetting ceased and the long shafted pump (pump suction 2" from bottom of tank) was installed. Pumping was slow but better than jetting. The metal age varied from 114-219 days cooled. An average of 70 curies/day I₁₃₁ was charged and .084 curies/day discharged from stack for an average I₁₃₁ DF of 830. No mercuric nitrate was used.

4. Solvent Extraction

Solvent-extraction performance continued at a high level during the report period. A general upward trend in activity throughout the plant was noted, but both the uranium and the plutonium product remained within shipment limits without further treatment. Waste losses remained very low, averaging less than 0.05 percent and the "losses" to the Backcycle Waste System remained well within the control range.

First cycle performance was generally excellent. No serious problems have occurred from the integration of the Neptunium Recovery Cycle into the process. A general upward trend occurred in the plutonium contamination in the uranium (LCU) stream for no obvious reason; however, by the end of the report period the trend was back downward and no problems resulted.

Performance of the Final Plutonium Cycle continued at the same high level as in the previous week. An anticipated problem of high activity being introduced to the LBX column from the organic waste (3WB) stream from the Neptunium Recovery Cycle did not materialize.

Performance of the Final Uranium Cycle was unchanged from the previous report period. The DF across the columns were essentially constant at 800 to 1000, in spite of a steady increase in the 2DF activity. "Losses" to the Backcycle Waste System (3WB) were in the range of five to nine percent - more variation than desirable for optimum performance of the Neptunium Recovery Cycle, but not severe enough to cause any problem.

5. Neptunium Recovery

The Neptunium Recovery Cycle was operated continuously during the report period. Due to shipment commitments, the "hot" operability tests were postponed and the primary emphasis was on maintaining control of the process and operational stability. However, valuable qualitative information on column stability and performance and general operational control was gained. Following are items of noteworthy interest:

- a) The maximum operating capacity of the columns exceeds that of the flow control equipment.
- b) The columns appear to be quite stable - at least with high nitric acid and low uranium concentrations.
- c) The 3A Column extraction efficiency is more than adequate, but the scrubbing efficiency is rather low - at least in the range of pulse frequencies which has been used to date.
- d) There appears to be a problem with excessive 3BW losses. This is probably dependent on the uranium and nitric concentration in the 3B Column as well as the flow ratio.

5. Neptunium Recovery (continued)

No serious problems have been encountered to date and the minor problems will undoubtedly be resolved with more operating experience and quantitative operability data.

The neptunium accumulation in the "Package" was on a steady upward trend until the last day of the report period, when the product stream analysis dropped sharply.

6. Final Plutonium Processing

N-Cell operated satisfactorily until mid-report period when pushing became difficult. Conditions continued to get worse until Nov. 28 when a resin change was successfully made "on-the-fly". Operation improved following the change and the unit was running smoothly by the end of the report period.

An analysis was made of operating experience since changing to the mixed resin (25% 10-20 mesh and 75% 20-40 mesh) and evidence points to an overall shortening of resin life from about sixty days to about three weeks operating time. Based on this analysis it was decided that future resin changes would be made with 20-40 mesh resin only.

7. Waste Concentration and Acid Recovery

During the month of November, F-Cell has operated at steady equilibrium conditions with nitrite being added to make the LWF .01 M - NaNO_2 and the E-F6 overflow rate held to about six flows LWW. During this period the gamma activity of the F3 recovered acid remained relatively constant with ZrNb running about $1.3^4 \mu\text{c/gal}$ and Ru about $1.0^5 \mu\text{c/gal}$. No testing of variables was attempted.

8. Fission Product Recovery

Head End

Run series 53 was completed recovering 175 kilocuries of strontium 90 with a decontamination factor of 17 from cerium. At the completion of the sixth sulfate run, 3500 gallons of LWW was jetted from TK F7 to TK E3 to TK E5 to TK F18 to underground storage. This transfer was necessary since rework was being held in TK F16. The LWW transfer thoroughly contaminated the centrifuge even though the centrifuge was not rotated while the flow was routed through it. The centrifuge was flushed several times with both 6 M nitric acid and 1 M oxalic acid in order to return the Beckman readings to normal. Much of the activity appeared to be held up in the diverter jumper.

In preparation for the fifth cesium production campaign, the E-Cell equipment was decontaminated with nitric acid, caustic tartrate, and mixed oxalic-nitric acid solutions. Beckman readings on G-E2 was reduced to less than 100 Mr./hr. Laboratory analysis on the final water flushes indicated all fission product concentrations to be less than 0.5 curies per gallon.

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Strontium Semiworks

Loading of the HAP0 II-2 cask with 165 kilocuries of strontium 90 was completed. Except for difficulties with leaks in the Hanford miniature connector, the loadout was routine. Filtrate losses were less than 0.10 percent.

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Series A.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 11/30/62 TO 2400 ON 12/6/62

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	4.1	5.5			.002	.06
Final	2.8	3.1	6.3	5.0	.002	
Ion Exchange		0.9		0.2		
Overall	6.9	9.8	6.3	5.2	.004	.06

II. Production

A. HAF Processed: 178.3 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>
100	1.67	5.1	7.3	P4
101	1.61	3.8	7.3	P4
102	1.47	3.5	7.5	P4
103	1.70		7.6	P4
1	1.95	<1	7.4	P1
2	1.7	4.06	7.4	P4
3	1.71		7.5	P4
4	1.76	2.70	7.3	P1
5	1.58		7.3	P1
6	2.2	6.6	7.5	P1
7	1.80		7.4	P1
8	1.61	4.5	7.4	P1
9	1.57		7.5	P1
10	1.28	4.1	7.6	P1
11	1.91		7.4	P1
12	2.03		7.5	P4
13	2.32		7.3	P1
14	3.12	3.5	7.9	P1
15	2.64		7.3	P1

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
16	2.74		7.3	P1
17	2.82		7.6	P1
18	2.52	2.1	7.6	P4
19	2.45		7.0	E6 *
20	2.14	2.2	7.1	E6 *
21	2.05		6.9	E6 *
22	1.88		7.3	E6 *
23	1.65		7.1	E6 *
24	<1	0.70	7.4	P4

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
43		.003		5	1	Hold
44	0.40	.005		6	8	2 Cans 234-5
1	0.32	.003		5	10	4 Cans 234-5
2	0.53	.001		5	12	4 Cans 234-5
3	0.29	.001		6	12	Hold
4	0.66	.001		6	4	Hold
5	1.60			5	22	234-5
6	1.60			6	22	4 Cans 234-5
7	2.00			5	25	1 Can 234-5
8	2.70			6	2	Hold
9	1.20			5	20	Hold
10	0.73	.004		5	15	2 Cans 234-5
11				5	12	2 Cans 234-5
12	1.76	.003		5	10	4 Cans 234-5

D. Overall Waste Loss - % of Total Production

U = 0.26% Pu = 0.33%

E. Rework - % of Total Production

U = Nil

Pu = 3.8% Total

SN = 3.1%

Cans, Sumps = 0.7%

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	187	102A
Cell Dr	60	106A
F.P.	63	106A
IWW	74	106A
C.W.	300	103BX

B. IWW Flows: 6.5 to 7.5

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C. TK Farm Boil-off Rates

<u>Waste TK</u>	<u>Est. Boil-off, Gal./Min.</u>
101	1.3
102	2.4
103	0.3
104	3.4
106	16.1

Notes:1. Plant Operating Information

The plant rate was maintained at a nominal 3.6 (actual 3.70) CF during the report period. Pluggage of the coating waste line to UGS on December 15 temporarily interrupted the metal dissolution operation. An alternate route was established and metal dissolution started on December 6. Five batches of "cold" uranium were re-cycled to the HAF Tank to maintain continuity of operation.

Transition of the Neptunium Recovery process from accumulation phase (Phase I) to the decontamination phase (Phase II) was accomplished on November 5.

2. Flowsheet

- (a) On November 30, the 2DF-ferrous sulfamate rate was reduced from 0.32 to 0.27 flows. It was increased to 0.34 flows on Dec. 5.
- (b) On Dec. 1, the 1BXP rate was increased from 18.5 to 19.0 flows. It was further increased to 19.6 flows on Dec. 2.
- (c) On Dec. 2, the 1BXP-HNO₃ rate was increased from 7.7 to 9.23 flows. It was further increased to 10.2 flows on Dec. 3.
- (d) On Dec. 2, the addition of 50 percent HNO₃ to the 3WB Tank was commenced at a rate of 3.65 flows. It was reduced to 2.0 flows on Dec. 3.
- (e) On Dec. 4, the 2DS rate was increased from 48 to 53 flows.
- (f) On Dec. 5, the recycle of "cold" uranium was commenced. It was terminated on Dec. 6.
- (g) On Dec. 5, the 2D pulse frequency was increased from 71 to 72 cycles per minute.
- (h) On Dec. 5, the 3WB flow to the HA Column was temporarily increased by about 30% (37 to 47 flows) as the Phase I to Phase II transition was completed in the Neptunium Recovery Cycle. It was reduced to about 43 flows on Dec. 6.

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3. Feed Preparation

The TK-D2 pumpout rate continued to deteriorate until transfer of coating waste stopped completely on 12/4/62. Attempts to unplug the line with coil water pressure failed to open the line. In order to maintain plant operating continuity, the strontium transfer line to the CR Diversion Box was connected to the D2 jet and transfer via this line started on 12/6/62. In order to prevent reoccurrence of the plugging, a change to the original coating waste procedure, jet out the coating waste after sampling followed by a line flush with the rinse water, was made. Dissolution was suspended for about 36 hours while the coating waste transfer line was out of service.

Mercuric nitrate was added to some batches of metal solution during this period. The metal age varied from 115 - 261 days cooled. An average of 88 curies/day I₁₃₁ was charged and .061 curies/day discharged from the stack for an average I₁₃₁ DF of 1440.

4. Solvent Extraction

Solvent-extraction performance was generally very good during the report period. The plutonium losses via the HAW stream were somewhat above normal at the start, but were rapidly reduced to generally below detection limits for the remainder of the report period. The plutonium carry-over in the LBU stream was two-fold above normal but slowly decreased during the run period. Intercycle gamma activities were considerably higher than characteristic of recent weeks - probably due partly to the higher activity of the recovered acid and partly to the lower cooling time of the metal feed.

Decontamination performance of the Final Plutonium Cycle continued to be excellent, with all product well within activity limits for processing in the Plutonium Anion Exchange Unit. Fission product decontamination performance of the Final Uranium Cycle was somewhat lower than typical, in spite of moderate adjustments in both the 2DS rate and the pulse frequency. Plutonium decontamination efficiency however, was excellent, with contamination in the uranium product decreasing about four-fold (from ~4 to ~1 ppb) during the report period. This decrease was in spite of a 2DF plutonium concentration which increased to about two-fold above normal. This, together with generally excellent neptunium decontamination, indicates the current problem of fission product decontamination involves more complicated phenomena than the 2D Column scrubbing efficiency.

5. Neptunium Recovery

Operation of the Neptunium Recovery Cycle operated continuously. Neptunium accumulation, which had tapered off at the end of the last report period, started to increase again at a very satisfactory rate after adjustments in the flow ratios of the aqueous and organic streams. Transition to Phase II (the decontamination phase) was completed on Nov. 5, and the product was sufficiently decontaminated after 12 hours of operation. Transition to Phase III (concentration and load-out) was

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5. Neptunium Recovery (continued)

delayed, however, until installation of jumpers to route the 3BN to the L-Cell Package (L3 and L4) was completed.

Operability of the equipment was generally very good. However, the stability and efficiency of the columns appear to be quite sensitive to processing variations. Following are additional interesting items which were noted during the report period:

- (a) The stable operating frequency of the 3B Column is sensitive to uranium saturation. Increasing the uranium saturation from 0.04 to 0.30 molar in the 3BN reduced the stable operating frequency from > 100 cycles per minute to 80 cycles per minute. It should be kept in mind, however, that increasing uranium saturation results in a corresponding reduction in the acid concentration.
- (b) Stability of the 3A Column is excellent under Phase I conditions, but decreases considerably under Phase II conditions. However, the capacity was more than adequate.
- (c) The plutonium decontamination efficiency in the 3A Column is closely related to pulse frequency. The efficiency improved greatly as the frequency was increased from 60 to 80 cycles per minute.
- (d) During transition to Phase II, the 3A Column flooded, apparently due to a small amount of air which was entrained with the 3AF as the 3AF Tank liquid level was being lowered. Reduction of the sparger air flow to minimum eliminated the problem.
- (e) It is necessary to maintain a uranium saturation in the 3BN of > 0.05 M ($\sim$ 0.1 Molar) to strip the neptunium product from the organic phase and prevent high losses via the 3BW stream.

6. Final Plutonium Processing

N-Cell operation during the report period was good. Beginning early in the week, however, it became apparent that the CRC instrument was not working properly and XCP time was set manually on the timer.

On Dec. 5 rework of 8 cans of Z-Plant MR material (dissolved skulls) began. After moving four cans successfully through TK-L9 to TK-L11 to TK-E6, the TK-L9 sample lines and transfer lines became plugged. Investigation revealed that the cans contained considerable amounts of solids as represented by a definite sludge layer on the bottom of the can.

7. Waste Treatment and Acid Recovery

Formaldehyde treatment of IWW was started on 12/3/62, using 100 ppm Dow Corning Antifoam B (a water dispersible silicone) as a defoamer added to the IWW prior to the reaction pot. Performance was erratic

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7. Waste Treatment and Acid Recovery (continued)

due to air locks in the antifoam addition system and also due to filter plugging in the formaldehyde system; however, rates approaching flowsheet were obtained. Periodic losses of anti-foam resulted in pressure surges which subsided as soon as the anti-foam flow was re-established.

8. Fission Product Recovery

Purex Plant

Two STT casks, ORNL numbers 44 and 47, were loaded with 44 and 49 kilocuries of cesium 137, respectively, from centrifuged TK-103A supernatant solution from tank 105A. The E1 tank temperature was held at a minimum and varied between 20 and 30 degrees C. Waste analyses indicated the casks had not reached break-through.

Strontium Semiworks

The solvent extraction columns were decontaminated and maintenance work started in B-Cell.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 12/7/62 TO 2400 ON 12/13/62

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	3.6			0.002	0.03
Final	2.9	3.3	7.0	4.2	0.002	
Ion Exchange		1.5		0.3		
Overall	6.8	8.4	7.0	4.5	0.004	0.03

II. Production

A. HAF Processed: 210.1 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>
25	< 1		7.3	P4
26	< 1		7.3	P4
27	1.08		7.3	P4
28	< 1	0.7	7.2	P4
29	1.27		7.2	P4
30	1.25	1.3	7.2	P4
31	1.60		7.0	P4
32	1.89	1.6	7.5	P4
33	2.21		7.9	P1
34	2.94	2.2	7.5	P1
35	2.91		7.8	P1
36	3.05	1.4	7.2	P1
37	2.95		7.3	P1
38	3.16	1.3	7.3	P1
39	3.37		7.0	P1
40	2.85	0.77	7.1	P1
41	2.59		7.1	P1
42	3.16		6.9	P1
43	2.34		6.9	P1
44	2.20	1.3	6.9	P1

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<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
45	1.89		7.2	P4
46	1.84	1.8	7.1	P4
47	1.80		7.2	P4
48	1.82	0.91	7.2	P4
49	1.76		7.3	P4
50	1.97	1.4	7.1	F1
51	2.40		7.4	F1
52	1.91	1.12	7.2	F4
53	1.58	.5	7.6	P4

2. Silica Gel

Gamma Ratios: Feed = 2.32
Product = 1.46

Tonnage: 83 Tons

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppm Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
13	1.80	.004		4	15	Hold
14	0.85			6	12	Hold
15	1.41	.002		5	15	Hold
16	1.45			6	12	234-5
17	0.91	.004		5	1	234-5
18	1.39	.003		5	20	234-5
19	0.94	.002		5	1	234-5
20	1.69	.004		5	2	234-5
21	1.77	.005		5	2	234-5
22	1.92			6	2	234-5

D. Overall Waste Loss - % of Total Production

U = 0.2%   Pu = 0.5%

E. Rework - % of Total Production

U = 17.2% (K6→E6)

Pu = 10.3%

SN = 0.3%

Internal = 10% (~4500 units of total recycle was a calculated value based on E6)

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	190	102A
Cell Dr	231 *	106A

DECLASSIFIEDA. Volumes to UGS (continued)

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
FP	99	106A
IWW	101	106A
CW	300	103BX

* Increased volume due to plug in vac. pump drain line.. Water going to M1.

B. IWW Flows: 6.7Notes:1. Plant Operating Information

The plant maintained a nominal 3.6 (actual 3.66) CF during the report period. The first neptunium recovery run was successfully completed in the new continuous neptunium recovery cycle, with more than 1900 units being isolated and decontaminated.

2. Flowsheet

- (a) On Dec. 7, the 2DF-ferrous sulfamate flow was reduced from 0.34 to 0.29 flows.
- (b) On Dec. 8, the HSS-HNO₃ concentration was reduced from 2.9 to 1.9 molar (it had drifted up over the preceding four shifts).
- (c) On Dec. 9, the 2D Column pulse frequency was increased from 72 to 74 cycles per minute.
- (d) On Dec. 9, the 3WB-HA rate was temporarily reduced from 48 to 37 flows, as the transition from Phase III to Phase I was made in the Neptunium Recovery Cycle. The 3WB-HA was increased to 43 flows later in the day.
- (e) On Dec. 10, the acid addition to the 2DF was changed from Purex Recovered nitric to UO₃ nitric.
- (f) Commencing on Dec. 11, the 2DW-Uranium loss was increased from ~6% to a peak of ~14% as part of the operability tests of the Neptunium Recovery Cycle.

3. Feed Preparation

The coating waste solutions were transferred to underground storage without difficulty via the strontium line to C.R. Div. Box.

Mercuric nitrate was added to all metal solution starting Dec. 8. On Dec. 11, mercurous nitrate was substituted for mercuric nitrate. The metal age varied from 110 to 196 days cooling. An average of 222 curies/day I₁₃₁ was charged and 0.27 curies/day discharged from the stack for an average I₁₃₁ DF of 820.

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4. Solvent Extraction

Solvent-extraction performance continued at a generally high level during the report period. All plutonium product met shipment specifications after processing through the Final Ion-Exchange System. Fourteen batches of uranium were slightly above shipping limits and required silica gel treatment.

Performance of the First Cycle was excellent. Gamma activity of the product streams was about two-fold above normal at the start, but had returned to normal by the end of the report period, in spite of the continuing downward trend in cooling time of the feed. Plutonium losses were excellent, averaging less than 0.03 percent for the report period.

Performance of the Final Plutonium Cycle continued excellent, with product activity within limits for the Plutonium Ion-Exchange Unit, in spite of above normal activity in the feed stream. "Losses" to the Backcycle Waste System were very low for most of the report period, but were on an upward trend during the last half of the period. The increasing losses were due to poor control on the nitric acid addition to the LBXP stream and on the 2AX stream and required much closer attention than normal.

Performance of the Final Uranium Cycle remained at about the same level as characteristic of recent weeks. Typical decontamination factors ranged from ~1000 (early in the report period when the 2DF activity was three-fold above normal) down to ~500 at the end of the report period. As in recent weeks, the plutonium and neptunium decontamination continued to be excellent, indicating the problem of fission product decontamination to be more complex than simple scrubbing efficiency.

5. Neptunium Recovery

Operation of the Neptunium Recovery Cycle continued, with completion of Phase III and transition to Phase I on Dec. 9. The first recovery run was very successful, despite numerous upsets and flowsheet imbalances resulting from the dual nature of the run (hot shakedown and normal recovery). Decontamination of the product from both plutonium and fission-product activity was very good. The uranium contamination was above normal, but well within limits for subsequent purification by ion exchange.

Accumulation since the transition has been at a normal rate, in spite of the lower inventory in the Backcycle Waste System. Flooding studies were continued in an effort to define more completely the capacity and flooding characteristics of the columns. The 3B Column again exhibited instability at a lower frequency with higher uranium saturations.

6. Final Plutonium Processing

Except for erratic operation of the CRC instrument, operation of N-Cell remained good during the report period.

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7. Waste Treatment and Acid Recovery

Installation of a 40 mesh screen in place of the fiberglass fullflow filter resulted in a more controllable formaldehyde flow to the denitration prototype unit. Operation of the unit was generally smooth while maintaining antifoam concentrations of about 30 to 70 ppm in the IWW feed; however, samples of the treated waste show acidities of 3-4 M HNO_3 vs. desired 1.0 M HNO_3 . The poor fission product DF experienced, especially during pressure surges, produced recovered acid which analyzed 10^5 to 5×10^6 Zr-Nb uc/gal and 10^4 - 10^6 uc/gal Ru.

This acid required reprocessing in F-Cell. Because direct additions to the F6 concentrator resulted in concentrator control problems, a flexible jumper was installed to jet recovered acid from the prototype unit into the IWF feed Tank F12. Thus, the acid was blended with HAW and dilution water and the feed flow into the concentrator was maintained constant.

8. Waste Management

On Dec. 12, 1962 a sludge monitor was installed in the 12 inch riser just west of pump pit on TK-241-A-105. The monitor has facilities for measuring the sludge temperature at eleven locations on 5 inch radius over a depth of 33 inches and also for determining the thermal heat transfer coefficient of the sludge. Three sludge sample containers each connected to a separate wire can be used to withdraw 5", 10", and 15" samples of sludge one inch in diameter.

9. Fission Product RecoveryPurex Plant

Two STT casks, ORNL numbers 48 and 45A were each loaded with 54 kilocuries of cesium 137. The feed to cask 48 consisted of a blend of 90 percent supernatant solution from tank 105A and 10 percent from tank 101A. Blending TK 101A with TK 105A in this ratio (9:1) increased the cesium 134 to 137 curie ratio from 0.015 to 0.017. The feed for cask 45A was obtained entirely from Tank 101A which has a cesium 134 to 137 curie ratio of 0.030. No difference in loading behavior or cask dose rate was observed between the two feeds.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 12/14/62 TO 2400 ON 12/20/62

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	3.9			.002	.05
Final	2.7	3.4	8.5	7.3	.002	
Ion Exchange		0.2		0.3		
Overall	6.7	7.5	8.5	7.6	.004	.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>
54	1.71	0.35	7.3	P4
55	1.14		7.6	P4
56	1.16	1.2	7.5	P4
57	1.52		7.5	P4
58	1.47	1.6	7.1	P4
59	1.70		7.2	P4
60	1.86	1.4	7.0	P3
61	1.59		7.4	F1
62	2.08	1.3	7.3	P1
63	2.14		7.4	P1
64	2.78	1.1	7.2	P1
65	3.35		7.2	P1
66	3.28	1.0	7.3	P1
67	4.32		6.9	P1
68	4.21	0.8	6.7	P1
69-A	5.17		4.1	E6 *
69-B	5.17		2.6	P1
70-A	5.4		5.1	E6 *
70-B	5.4		2.0	P1

1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
71-A	5.51		3.3	E6 *
71-B	5.51		3.7	P1
72	5.24	1.61	7.0	P1
73	4.01	1.25	7.1	P1
74	2.12		7.2	P1
75	2.70		7.1	P1
76	2.73	1.5	7.3	P1
77	4.32		7.4	P1
78	3.30		7.4	P1
79	3.85		7.2	P1
80	3.71		7.1	P1
81	3.30		6.	E6 *
82	3.47	1.3	4.5	E6 *

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
23	0.70	.01		5	2	234-5
24	0.66	.006		4	1	234-5
25	13.42	.16		Not Canned Out		L11
26	13.3	.001		2	8	Hold
27	1.04	.006		5	14	234-5
28	14.6	.008		Not Canned Out		L11
29	0.33	.01		6	1	
30	12.2	4.56		4	11	Hold
31	3.54	.03		6	20	234-5
32	12.2			3	90	Hold
33	59.0	.09		4	30	Hold
34	2.06	< .001		4	15	234-5
35	60.0			4	40	Hold
36	.73	< .001		5	3	234-5

D. Overall Waste Loss - % of Total Production

U = 0.2%   Pu = 0.58%

E. Rework - % of Total Production

U = 11.3% (K6 → E6)
Pu = 11.0% (Primarily out of Spec. Cans)

III. Waste Treatment

A. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	194	102A
Cell Dr	154	106A

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A. Volumes to UGS (continued)

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
FP	83	106A
IWW	38	106A
CW	300	103BX

B. IWW Flows: 5.31. Plant Operating Information

The plant maintained a nominal 3.6 (actual 3.7) CF. during the report period. Due to maloperation in the Plutonium Ion-Exchange Unit, the plutonium inventory in the Backcycle Waste System became abnormally high and it was necessary to recycle "cold" uranium on two occasions to reduce the plutonium level. Four batches in all were recycled during the report period. Also, due mostly to the difficulties mentioned above, the 2BP was diverted to the L-Cell Package on three occasions..

2. Flowsheet

- (a) On Dec. 14, L-Cell Package 2BX was started to the 2B Column and the 2BP was routed to the Package. It was temporarily diverted back to the Ion-Exchange System on Dec. 15 and permanently on Dec. 16 after the resin had been changed in the ion-exchange unit. The 2BP was diverted to the L-Cell Package again on Dec. 17, partly because of high losses in the Ion-Exchange System and partly because of a mild activity burst in the Final Plutonium Cycle.
- (b) On Dec. 15, the 2DX was increased to reduce the 2DW uranium recycle from ~12% to ~6%.
- (c) Commencing on Dec. 16, routing of the carbonate wash solution from the No. 2 solvent cycle to the No. 1 solvent cycle wash tank (G1) was terminated temporarily because of high solvent inventory in the No. 1 system.
- (d) On Dec. 17, the nitric acid addition to the 2DF was stopped for ~three hours. After flow was restored it was held at twice normal rate for ~an hour, then reduced to normal rate.
- (e) On Dec. 17, "cold" uranium was recycled from the Final Uranium Cycle (K6) to the HAF to purge excessive plutonium from the solvent extraction system. "Hot" feed was started again on Dec. 18.
- (f) On Dec. 18, the 2BP was reduced from 2.7 to 1.5 flows when it was routed into ion exchange from the L-Cell Package. It was increased back to 1.9 flows later in the day to reduce the plutonium reflux in the column.
- (g) On Dec. 18, the Neptunium Recovery Cycle was shut down at equilibrium to permit installation of a 3BW sampler jumper. It was started up early on Dec. 19.

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2. Flowsheet (continued)

- (h) On Dec. 20, the change frequency of the carbonate wash solution in the No. 1 solvent cycle was extended from 8 to 10 hours.
- (i) On Dec. 20, the change frequency of the carbonate wash solution in the No. 2 solvent cycle was extended from 24 to 30 hours.
- (j) On Dec. 20, 1-1/2 batches of "cold" uranium were recycled from the Final Uranium Cycle to the HAF tank to purge excessive plutonium from the system.
- (k) On Dec. 20, the 2D pulse frequency was increased from 73 to 75 cycles per minute.
- (l) On Dec. 20, the Neptunium Recovery Cycle was shut down because of inability to maintain 3WB flow to the feed tank. Startup occurred later in the day on Synthetic 3AF at half rate and at 40 cycles per minute frequency on both columns. The 3WB flow was restored at ~1800, at which time the flow rates and pulse frequencies were increased to 90% CF and 80 cycles per minute, respectively, simultaneously over a 40 minute period.

3. Feed Preparation

No difficulty was experienced in jetting coating waste through the strontium line to C.R. Div. Box.

Metal solution treatment with mercurous nitrate continued during the report period. The metal age varied from 105 to 196 days cooling. An average of 620 curies/day of I₁₃₁ was charged to the dissolvers, with an average discharge of 0.37 curies/day from the stack for an average I₁₃₁ DF of 1675.

4. Solvent Extraction

Solvent-extraction performance continued to be generally good during the report period. Plutonium losses remained at a very low level (<0.03%) until the last day of the period, when a sharp upward trend was noted. Plutonium contamination in the First Cycle uranium product stream was still slightly above normal, but on a downward trend by the end of the week with no problems resulting in the Final Uranium Cycle. Considerable difficulty was experienced with high plutonium levels in the First Cycle and three and a half batches of "cold" uranium were recycled to prevent exceeding the critical mass control limits in the Backcycle Waste System. The high plutonium levels presented no problems with respect to partition performance, however.

Performance of the Final Uranium Cycle was marginal with respect to fission product decontamination. Plutonium and neptunium decontamination performance, however, continued to be excellent. There appears to be a correlation between the uranium saturation in the 2D column and fission product decontamination. However, this correlation may be only one contributing factor to the problem. No beneficial effects were noted from the 2 cycle-per-minute increase in the 2D frequency (to 75 cycles per minute).

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4. Solvent Extraction (continued)

Performance of the Final Plutonium Cycle continued to be excellent, in spite of poor process control resulting from malfunction of the 1BXP-HNO₃ flow meter. DF's obtained across the 2A and 2B Columns varied from ~1000 (when the 2AF activity was low) to ~4000 (when the 2AF activity and 2AW loss were normal).

5. Neptunium Recovery

The second recovery run in the new facility continued with additional study of the performance characteristics of the 3A and 3B columns. At the start of the report period, flooding studies were in progress at conditions of high uranium-to-nitric ratios in the 3AF stream. As noted previously, the high uranium saturation adversely affected the neptunium recovery performance of both columns. Also, indications are not as apparent when the 3B column floods at excessive uranium saturation as at normal (flowsheet) conditions. The flooding study resulted in the 3AF tank becoming nearly filled with organic from the 3B column without any significant indication of a flood.

Neptunium accumulation in the "Package" was reduced by about 50 percent by the upset, but was increasing again at the normal rate by the end of the report period. Considerable trouble was experienced during the report period with plugging in the 3WB addition line to the 3AF tank. One shutdown of the system and numerous other minor upsets resulted.

6. Final Plutonium Processing

On Dec. 14 and Dec. 15, N-Cell operation quickly deteriorated when pushing became extremely difficult. Symptoms pointed to probable plugging in the "A" column. On the 15th the 2BP was diverted to L-Cell Package and N-Cell was stripped of Pu. On the 16th the resin was completely replaced while the unit was down. The last of the mixed resin on hand (75 percent 20-40 mesh, 25 percent 10-20 mesh) was used in the change.

Following startups of N-Cell, pushes were steady but high plutonium losses were encountered via the XSW stream because of an improper XSW time setting when the CRC instrument failed. Efforts to adjust the cycle were also only partially effective. On Dec. 20, the unit was shut down and a new CRC probe installed.

7. Waste Treatment and Acid Recovery

Reversing the agitator blade in the antifoam addition tank so solution would be pushed toward the top of the tank rather than drawing it down toward the bottom of the tank apparently reduced the amount of air drawn into the system and reduced air lock problems in the antifoam addition line. An antifoam concentration of approximately 50-75 ppm was sufficient to control foaming in the reactor and permitted attainment of flowsheet rates for formaldehyde. However, the large antifoam flow rotometer made operation at higher-than-desired antifoam rates necessary and diluted the IWW by about 10 percent. Thus, the actual IWW flow rate to the prototype unit should be reduced by 10 percent when figuring the IWW HCHO rates. The following data summarizes the information

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7. Waste Treatment and Acid Recovery (continued)

accumulated during approximately three weeks of continuous prototype unit operation:

<u>Stream</u>	<u>Desired</u>		<u>Actual</u>		<u>γ Activity μc/gal (av.)</u>	
	<u>Flow</u>	<u>H⁺ M</u>	<u>Flow</u>	<u>H⁺ M</u>	<u>Zr</u>	<u>Ru</u>
IWW	5	5	7.3	5.7-7.2	2.0 ⁹	8 ⁸
Formaldehyde	0.6		0.72			
Treated Waste	3.2	0.5	7.5	3-4		
Reflux Water	1.7		2.0			
Recovered Acid	4.1	3.5	2.4	2.5-3.5	2 ⁵ -2 ⁶	1 ⁴ -2 ⁵

High activity in the recovered acid was mainly due to activity bursts during periods when the antifoam rate was low or off. Assuming normal smooth operation, the activity will probably produce acid of about 2×10^5 μc/gal Zr and 1×10^4 μc/gal Ru.

During a three hour shutdown to revise the pump safety interlock system, the feed spray ring to the tower apparently plugged, necessitating extensive flushing operations to free the line.

8. Fission Product RecoveryPurex Plant

A 200 gallon HLO "Bowling Ball" cask was filled with 175 gallons of un-centrifuged IWW and shipped to the 325A Building for Waste Calcination Studies.

Strontium runs were resumed with run series 54 underway at the end of the report period.

Strontium Semiworks

Batch extraction operations in TK 21 recovered 65 kilocuries of Sr-90 from waste rework solutions. The rework solution consisted of strontium product that exhibited poor precipitation and filtration behavior plus cask filtrate and sump solutions containing recoverable quantities of strontium. Batch extraction behavior was good with overall strontium recovery of 97 percent and decontamination factors from iron, sodium, cerium, zirconium-niobium, and ruthenium exceeding 50.

When the rework operation was completed, the organic was washed with a 0.5 M nitric acid - 0.75 M oxalic acid solution and cribbed. The recovered strontium solution was retained and will be used for washing strontium from the HCW stream in strontium run number 3.

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PROCESS SUMMARY OF PUREX PLANT OPERATION
FROM 0000 ON 12/21/62 TO 2400 ON 12/31/62

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	4.0			.002	0.10
Final	2.8	3.4	8.0	3.3	.002	
Ion Exchange		1.2		5.6		
Overall	6.7	8.6	8.0	8.9	.004	0.10

II. Production

A. HAF Processed: 245 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>
83	3.00		6.1	P1
84	3.10	0.91	7.1	P1
85	3.00		7.7	P1
86	2.71	2.0	7.4	P1
87	3.00		7.1	P1
88	2.59	2.45	7.2	P1
89	3.12		7.6	P1
90	3.09	1.96	7.4	P1
91	3.31		7.3	P1
92	3.10	2.00	7.2	P1
93	2.95		7.3	P1
94	3.75	1.4	7.3	P1
95	4.01	1.4	6.5	P1
96	4.73		6.9	P1
97	5.30		7.5	P1
98	5.00	1.47	7.4	P1
99	4.12		7.4	P1
100	4.27		7.1	P1
101	3.52		7.3	P1
102	3.30	1.5	6.5	P1

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1. Solvent Extraction (continued)

<u>Batch</u>	<u>Gamma Ratio</u>	<u>Pu, ppbp U</u>	<u>Tons</u>	<u>Shipped To</u>
103	2.48		7.4	P1
104	2.48	0.77	6.9	P1
105	2.33		7.7	P1
106	2.63	1.0	7.2	P1
107	2.23		7.1	P1
108	2.90		7.2	P1
109	2.68		7.8	P1
110	3.06	0.50	7.6	P1
111	2.79		7.6	P1
112	2.69	0.42	7.4	P1
113	3.60		7.3	P1
114	3.64	0.4	7.3	P1
115	4.30		7.5	P1
116	4.64		6.0	E6 *
117	4.91		5.9	E6 *
118A	4.74		2.9	E6 *
118B	4.74		1.3	E6 *
119	4.02		7.5	P1
120	3.2		6.8	P1
121	2.02		7.0	P1
122	1.68		7.4	P1
123	1.91		3.1	P1

C. Plutonium Product

<u>Batch</u>	<u>Zr-Nb/ATx10⁻¹¹</u>	<u>% U in Pu</u>	<u>Fe, ppmp Pu</u>	<u>No. Cans</u>	<u>Average Mr./hr.</u>	<u>Shipped to</u>
37	1.07			3	50	2 Cans 234-5
38	54.4	0.16		3	10	Hold
39	1.0	< .001		6	2	234-5
40	2.37	.001		4	1	234-5
41	.80	.001		6	10	234-5
Flush				4	25	Hold
42	.52	.002		5	2	234-5
43	.32	.003		6	1	234-5
44	.43	.002		5	0	234-5
45	.72	.004		6	1	234-5
46	.90			6	4	234-5
47	.75			5	13	234-5
48	1.1			6	15	234-5
49	1.17	.001		6	12	234-5
50	.68	.001		5	15	234-5
51	.87			6	11	234-5
52	2.54			6	18	234-5
53	2.28	.003		4	20	234-5
54	2.64	.004		5	20	2 Cans 234-5
55	1.3	.01		5	3	3 Cans 234-5
56	.82	.005		5	1	Hold
57	1.15	< .001		5	15	Hold
58	1.78	.015		4	11	Hold

D. Overall Waste Loss - % of Total Production

U = 0.4% Primarily D Cell Flush - 1200#
 Pu = 0.4%

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E. Rework - % of Total Production

U = 5.7% (K6 → E6, F8 → D5)
 Pu = 3.0%

III. Waste TreatmentA. Volumes to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste TK</u>
Wash	282	102A
Cell Dr	156	106A
FP	140	106A
IWW	36	106A
CW	300	103BX

B. IWW Flows: 6.3Notes:1. Plant Operating Information

The plant was maintained at a nominal 3.6 (actual varied from 3.4 to 3.7) CF until shutdown on Dec. 30. "Hot" feed was terminated at 0800 on Dec. 29. After recycling two batches of "cold" uranium to purge residual plutonium from the system the HAF was shut off at 2300 on Dec. 29, and the HA and HS Columns stripped and emptied in preparation for scheduled maintenance activities. The Partition Columns, Final Uranium Cycle, and Final Plutonium Cycle were shut down on Dec. 30.

2. Flowsheet

- (a) On Dec. 21, the 2D pulse frequency was reduced from 75 to 72 cycles per minute.
- (b) On Dec. 23, the acidity in the HA Column was reduced by ~12%, to the flowsheet valve (HAW-HNO₃ ~3 M) after having gradually increased over a several-day period.
- (c) On Dec. 25, addition of F8 rework solution to the HAF was commenced at a rate of 100 gallons in the first batch of HAF. It was increased to 150 gallons in the next batch and to 250 gallons per batch in subsequent batches. The rework was concluded on Dec. 28.
- (d) On Dec. 26, the 3WB flow to the HA column was temporarily increased by about 30 percent (from 40 to 53 flows) as the transition from Phase I to Phase II was completed in the Neptunium Recovery Cycle. It was reduced to 42 flows on Dec. 27.

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2. Flowsheet (continued)

- (e) On Dec. 27, the Neptunium Recovery Cycle was reduced from Phase II to Phase III flow rate. Phase III operation was completed and the system was shut down on Dec. 29.

3. Feed Preparation

Routing of coating waste to C Farm via the unplugged original line was restored Dec. 21; however, the line still gave indication of being restricted. The pump was reinstalled to finish out the run period. The last dissolver was charged on 12/26/62. Metal age varied from 106 to 116 day old. Average of 480 curies/day of I₁₃₁ was charged; with an average of 0.47 curies/day emission from the stack the average DF was 1020.

4. Solvent Extraction

Solvent-extraction performance continued at about the same level as during the previous report period. The source of the increasing problem of flow control and instrument maloperation was traced to an accumulation of alumina powder in the instrument air system. First Cycle product activity and losses were both above normal during the report period. A general improvement (~two-fold) in decontamination performance coincided with addition of F8 rework material to the HAF. No noticeable effect on losses resulted from the rework.

Performance of the Final Plutonium Cycle continued to be excellent, in spite of the flow control problem mentioned above. Rapid fluctuations in the 2AW "losses" resulted in corresponding fluctuation in the 2BP activity, indicating a correlation between solvent saturation and DF in the 2A Column. However, even with "losses" less than one percent, and at maximum product activity, the DF was approximately 1200. During periods of high losses (~seven percent) the DF was approximately 10,000.

Performance level of the Final Uranium Cycle continued without change. All product required silica gel treatment to reduce the gamma activity to shippable limits.

5. Neptunium Recovery

The second recovery run in the new facility was completed successfully. An estimated 1350 units were loaded out and are awaiting transfer to 222-S for final purification. (Final analyses indicate 2270 units were recovered in the first run.) Decontamination from both plutonium and fission products continued to be excellent during Phase II operation. Operational studies indicated higher capacities and higher stable operating frequencies than were demonstrated in the "cold" development studies. Another significant aspect of the system is the apparent minimum level in the Backcycle System to which the neptunium concentration can be reduced. Accumulation in the

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5. Neptunium Recovery (continued)

J-Cell Package appears to level out when the inventory in the Backcycle (3WB) System reaches ~500 units. This appears to be independent of the inventory in the J-Cell Package.

Neptunium accumulation in the Backcycle System has shown a significant improvement since integration of the J-Cell Package into the process. Loss via the HAW and 2EU streams are at least two-fold less than they were prior to operation of the "Package".

6. Final Plutonium Processing

The CRC installed "on-the-fly" on Dec. 20 did not operate well enough to be used for automatic control and by Dec. 22 the unit was again operating on the manual timer. Adjustments in cycle time, however, seemed to correct excessive XSW losses, and the unit operated exceptionally well through shutdown on Dec. 30.

N-Cell DF. fluctuated widely during the report period as fluoride flow was suspended for a short period during the midportion of the period.

7. Solvent Treatment

The following tabulation compares solvent treatment flowsheet information for Dec. 1962, June 1962 and that given in the control specification HW-56933. The information shown for June 1962 reflects improvements made between the issuance of HW-56933 in Nov. 1958 and June 1962. The information shown for Dec. 1962 reflects the reuse of R-Cell organic wash wastes in G-Cell as initiated in September 1962. The tabulation at the bottom depicts the volumes of organic wash waste generated at each of the selected dates and the amount of carbonate and permanganate solution required.

<u>Stream</u>		<u>Dec. 62</u>	<u>June 62</u>	<u>HW-56933</u>
10B (Na ₂ CO ₃)	Batch to G8 598-8 hrs.			
	598-8 hrs. 2.5% CO ₃	6.6	7.2	19.2
	272-8 hrs. 0.62% KMnO ₄			
10B (KMnO ₄)	Continuous to G1 2% KMnO ₄	0.78	1.6	1.7
10S (water	Batch ~600 gallons		34.1	
HNO ₃)	every 12 hrs. from R2	5.33*	0.5	38.1
10S (HNO ₃)	Direct for acid butt	0.2		0.45
10C (1% NaOH)	Continuous	7.6	7.6	11.9
10D	Continuous	7.6	7.6	11.9

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7. Solvent Treatment(continued)

<u>Stream</u>		<u>Dec. 62</u>	<u>June 62</u>	<u>HW-56933</u>
20B (2.5% Na ₂ CO ₃)	360 gal/24hrs. to G1	1.61		-
(0.62% KMnO ₄)	for reuse			
20S (water HNO ₃)	Semi-batch 600 gal/12 hrs.	5.33	2.7	(Na ₂ CO ₃) 6.1
20S (acid)	Direct for acid butt	0.2		

* Not a fresh stream - comes from R-Cell

Total waste HW-56933 flowsheet = 77.35 flows

June '62 flowsheet prior to R1→G1 R2→G2 = 61.3 flows

Total waste current flowsheet = 22.32 flows

Flowsheet carbonate + KMnO₄ = 27 flows

June '62 flowsheet prior to R1→G1 R2→G2 = 11.5 flows

Current flowsheet carbonate + KMnO₄ = 8.99 flows

8. Fission Product Recovery

Purex Plant

Strontium run series 54 consisting of seven sulfate precipitations - one oxalate precipitation and one concentration precipitation - was completed recovering 206 kilocuries of strontium 90. A decontamination factor of 26 was obtained from cerium. Strontium run series 55 was underway at the end of the report period.

Sodium bisulfate was tested as a substitute for sodium sulfate in runs 54-S-6 and 54-S-7. No difference in process performance was observed with the sodium bisulfate. Since sodium bisulfate is about three-fold more soluble than sodium sulfate, the volume of chemicals added during the sulfate strike can be reduced thus permitting the double batch procedure described previously to be used to advantage even with dilute IWW.

Strontium Semiworks

The HAP0 IB-1 cask was loaded in late December 1962 with 470 kilocuries of Sr-90 as the carbonate. The strontium carbonate cake contained about 240 kilocuries from Run SSW #1 and 230 kilocuries from the combined Run SSW #2 and TK-21 rework. The cask loading operation was not as difficult as with HAP0 II type casks. The ease of loading was attributed to the larger filter surface area and thinner cake thickness (7/16" versus 5/8"). The loading operation was normal and uneventful except the precipitate from the second bicarbonate strike (SSW #2 and TK-21 rework) settled very slowly. However, no difference was noted in the filtration characteristics of the two precipitates and the filtrate losses in either case were less than 0.3 percent.

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Strontium Semiworks (continued)

Prior to filtering the second precipitation, the batch was butted again with potassium bicarbonate. As a result, this batch received a total potassium bicarbonate addition that was 200% in excess of the theoretical requirement. Despite the additional bicarbonate in the solution, the settling rate of the precipitate was improved very little. Although laboratory tests do not confirm the results, operating experience indicates a precipitate more difficult to filter is formed when strontium is precipitated from a basic rather than a neutral or acidic solution.

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11 / 4 / 93

END