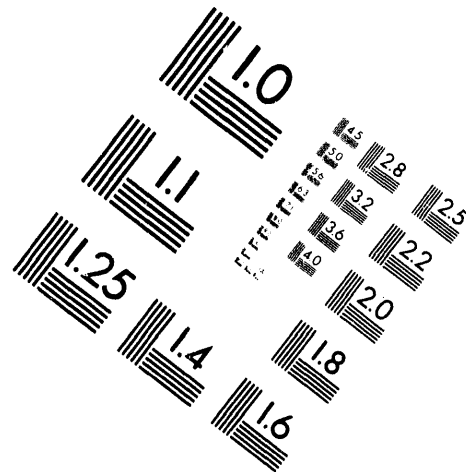
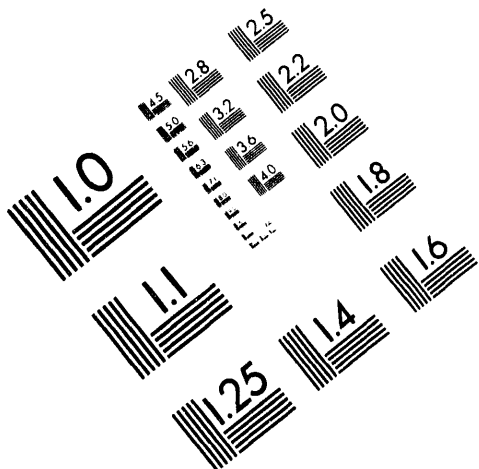




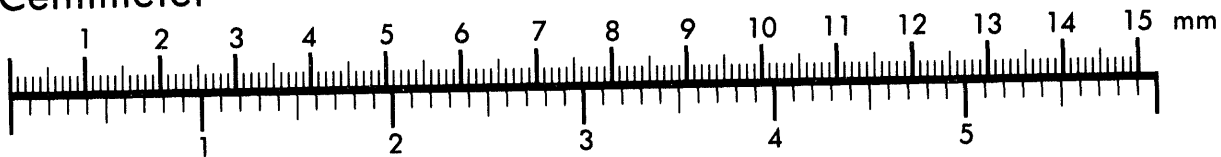
**AIM**

**Association for Information and Image Management**

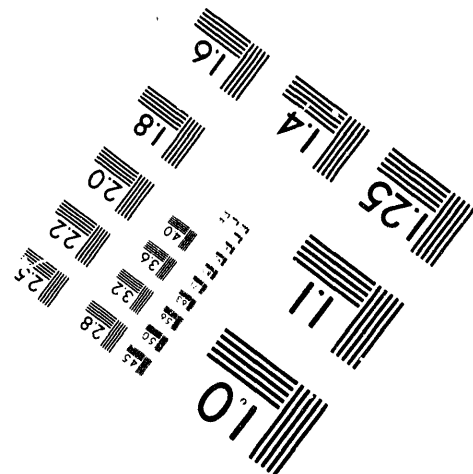
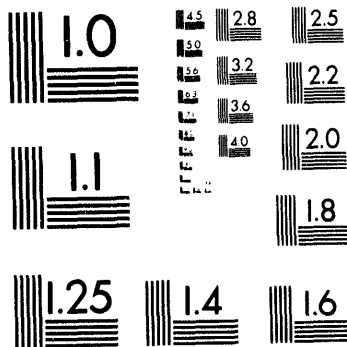
1100 Wayne Avenue, Suite 1100  
Silver Spring, Maryland 20910  
301/587-8202



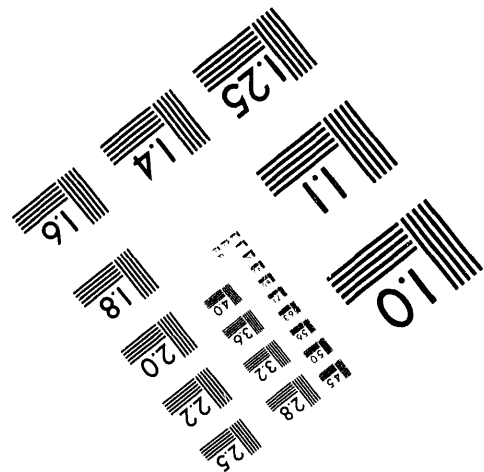
Centimeter



Inches



MANUFACTURED TO AIM STANDARDS  
BY APPLIED IMAGE, INC.



**1 of 3**

# DECLASSIFIED

(CLASSIFICATION)

## GENERAL ELECTRIC

HANFORD ATOMIC PRODUCTS OPERATION - RICHLAND, WASHINGTON

DOCUMENT NO.

HW-76912

SERIES AND COPY NO.

A 4

DATE

1-15-63

**RESTRICTED**  
THIS DOCUMENT CONTAINS RESTRICTED DATA AS DEFINED IN EXECUTIVE ORDER 12812 OF 1954. IT IS TRANSMITTED AND STORED AS AN UNAUTHORIZED PERSON'S PROPERTY.

TITLE

PUREX PROCESS PERFORMANCE SUMMARY  
JANUARY 1963 THRU DECEMBER 1963

**OTHER OFFICIAL CLASSIFIED INFORMATION**  
THIS MATERIAL CONTAINS INFORMATION AFFECTING THE NATIONAL DEFENSE. IT IS TRANSMITTED WITHIN THE MEANING OF THE LAWS, TITLE 18, U. S. C., SECS. 793 AND 794, THE TRANSMISSION OR REVELATION OF WHICH IN ANY MANNER TO AN UNAUTHORIZED PERSON IS PROHIBITED BY LAW.

AUTHOR

B. F. Judson

ISSUING FILE

*Handwritten stamp:*  
CLASSIFIED  
RETURN COPY  
TECHNICAL INFORMATION FILES

THIS DOCUMENT MUST NOT BE LEFT UNATTENDED OR WHERE AN UNAUTHORIZED PERSON MAY HAVE ACCESS TO IT. WHEN NOT IN USE, IT MUST BE STORED IN AN APPROVED LOCATION. IF YOU ARE NOT AUTHORIZED TO BE WITHIN AN APPROVED GUARDED AREA, YOU MUST OBTAIN A SIGNED RECEIPT FROM THE PERSON RESPONSIBLE FOR THE CARE OF THE CLASSIFIED FILES. IT IS YOUR RESPONSIBILITY TO RETURN THE DOCUMENT WITHIN THE LIMITS OF THIS PROJECT AND FROM AN UNAUTHORIZED PERSON. ITS TRANSMITTAL TO, AND STORAGE AT YOUR PLACE OF RESIDENCE IS PROHIBITED. IT IS NOT TO BE DUPLICATED. IF ADDITIONAL COPIES ARE REQUIRED, OBTAIN THEM FROM THE RELATED ISSUING FILE. ALL PERSONS READING THIS DOCUMENT ARE REQUESTED TO SIGN IN THE SPACE PROVIDED BELOW.

ROUTE TO:	PAYROLL NO.	LOCATION	FILES ROUTE DATE	SIGNATURE AND DATE
B. F. Judson	60952	3034		
	60946	2024	DEC 17 1963	
	60958		MAY 5 1970	
G. A. Nicholson	60991		JUN 8 1971	<i>G. A. Nicholson</i>
<b>RECORD COPY</b>				

**MASTER**

CONTINUOUS PAGINATION FOR HW

AS OF	<u>3-22-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>22</u>	PAGES
AS OF	<u>4-1-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>38</u>	PAGES
AS OF	<u>4-1-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>47</u>	PAGES
AS OF	<u>4-27-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>62</u>	PAGES
AS OF	<u>5-28-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>68</u>	PAGES
AS OF	<u>6-12-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>81</u>	PAGES
AS OF	<u>6-14-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>87</u>	PAGES
AS OF	<u>7-20-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>93</u>	PAGES
AS OF	<u>7-18-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>107</u>	PAGES
AS OF	<u>8-1-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>113</u>	PAGES
AS OF	<u>10-1-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>123</u>	PAGES
AS OF	<u>12-30-63</u>	(DATE)	THIS DOCUMENT CONSISTS OF	<u>198</u>	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES
AS OF	_____	(DATE)	THIS DOCUMENT CONSISTS OF	_____	PAGES

THIS CONTINUOUS PAGINATION FORM IS TO BE USED FOR REVISING CLASSIFIED MANUALS OR ACCUMULATING CLASSIFIED WORKING PAPERS IN A BINDER ISSUED AND CONTROLLED BY CLASSIFIED FILES.

DECLASSIFIED

[REDACTED]  
- 1 -

HW-76912

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - OF Hill
  - 4 - RF Judson
  - 5 - RW McCullugh
  - 6 - PR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling

Classification Cancelled and Changed To

**DECLASSIFIED**

By Authority of CG-NMP-1

By AS Lewis 5/16/94  
JD Prisher 5/17/94  
Verified By JL Phillips 5/17/94

This document consists of  
198 Pages. ~~Number of~~  
~~Pages in Series~~

PUREX PROCESS PERFORMANCE SUMMARY  
JANUARY 1963 THRU DECEMBER 1963

By

B. F. Judson

Purex Process Engineering  
Research and Engineering  
Chemical Processing Department

January 15, 1963

DECLASSIFIED

This document contains Restricted Data as defined in the Atomic Energy Act of 1954. Its transmittal and disclosure of its contents in any manner to an unauthorized person is prohibited.

# DECLASSIFIED

HW-76912

This document consists  
of No. 1 of 10 copies.  
Series A.

- 2 -

Distribution:  
1 - OF Beaulieu  
2 - WS Frank  
3 - OF Hill  
4 - BF Judson  
5 - RW McCullugh  
6 - PR McMurray  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling

## PUREX PROCESS PERFORMANCE SUMMARY FROM 0000 ON 1/1/63 TO 2400 ON 2/3/63

### I. General - G. A. Nicholson

The Purex Plant was not operated for normal production during this report period because of scheduled maintenance work. However, various rework and fission product recovery operations were carried out on a special procedure basis. Descriptions of these operations are included below.

### II. Performance Data - D. C. Leyson

#### A. Volume to UGS

<u>Orig. Tank</u>	<u>U</u>	<u>Pu</u>	<u>Gallons</u>	<u>Waste Tank</u>
F18	1765	747	25 360 (FP) 170 367 (CD)	106-A 106-A
G8	13	30	39 021	102,4-A
R8	31		55 086	102,4-A
F16	<u>23</u>	<u>56</u>	(Vol. Acct. for in F18)	106-A
Total	1832	833	289 834	

#### B. Tank Farm Boil-off

Tank 101 = 2.1 Gal/Min.  
102 = 4.1  
103 = 0.3  
104 = 1.8  
105 = -0-  
106 = 15.8

### III. Flowsheet - G. A. Nicholson

No permanent changes.

# DECLASSIFIED

This document contains restricted data as defined in the Atomic Energy Act of 1954. Its transmission or the disclosure of its contents in any manner to an unauthorized person is prohibited.

IV. Feed Preparation - G. J. Raab

A new bath tub design, 15,000 gallon tank was put into the TK-D3, D4 position. Tank D4 was placed in a concrete and steel storage cylinder and saved as a spare tank. Tank D3 was put into the TK-D2 position. The existing TK-D2 was removed and buried. Considerable solids were removed during decontamination of TK-D2.

Back-up filters were installed in each of the dissolver off-gas lines. Considerable condensate built up in the filters when air was initially pulled through them. It appeared that the condensate failed to drain from the filters because the SX-3 sump filled to overflow. The SX-3 instrumentation and automatic jets were malfunctioning. The condition appears to have been fixed.

Silver Reactor B2 was regenerated after processing 910 tons.

Dissolving was started on nonrepresentative metal on Jan. 28, 1963. The first coating waste through the line to UGS either plugged the line or an old plug still existed. The UGS line was flushed both ways from the 152-A diversion box with water at a rate as high as 180 gpm; a plug appeared to be removed. At the end of the report period, coating wastes were jetting well. No mercuric nitrate was used as metal age was 200 days or older.

V. Solvent Extraction - G. L. Nicholson

Rework of several tons of recovered uranium through the Final Uranium Cycle was completed successfully during the shutdown period. The uranium came from the following sources:

- (a) About four and a half tons recovered as a dilute (0.05 molar) waste solution (20W Stream) from the Second Solvent System several months ago.
- (b) About twelve tons of concentrated (~ 2.0 molar) solution recovered from sumps, etc. in 224-U Building.
- (c) About five tons of uranium recovered from the transfer incident in May 1962 which contained large quantities of decontaminating solutions.

Items of interest regarding the rework operation are included below:

- (a) Losses were negligible until just prior to completion of the operation when the 2EW losses increased about 100-fold - undoubtedly due to addition of degraded organic to the system when the storage tank was emptied.
- (b) Both plutonium and fission product contamination were excessive in the first batch of product so it was recycled to the 2DF Tank. However, successive batches were only slightly above gamma activity specifications and were blendable.

- (c) Solvent quality remained excellent until the very end of the rework operation, when the plutonium retention increased about 5000 fold.

Following the rework operation, the ICU Concentrator and 2DF Tank were water-flushed and the 2D and 2E Columns were given a decontamination flush with caustic-tartrate solution followed by oxalic-nitric solution, then an organic-removal flush with hot carbonate followed by nitric acid.

Considerable maintenance and related work was performed on the Solvent-Extraction System during the shutdown. Activities of interest include the following:

- (a) A new HAF jumper was installed and the HAF Pump Tank (H1) was water-flushed.
- (b) The HA Pulse Generator was replaced because of a leak.
- (c) The 3WF Concentrator (EH4) was emptied and given a mild thermal shock treatment according to recommendation by Facilities Engineering.
- (d) A new 2AX jumper was installed to permit operation closer to mid-range on the flow recorder.
- (e) All instrument air lines were purged and all pneumatic transmitters were dismantled and cleaned to remove deposits of powdered alumina which accumulated during the previous operating period after a new air drier was installed.

#### VI. Product Treatment - R. W. Lambert

##### Final Plutonium Processing

Immediately following the regular plant shutdown, N-Cell was stripped and a special series of flushes made in N1, N6, N7 and L10 to reduce the representative plutonium content of the system. An estimated 500 grams of normal plutonium were left in the N-Cell-L10 complex. Following the plutonium cleanout, the resin was discarded to UGS via Tank R8.

Maintenance work during the shutdown period included the following items:

- (a) Removed, checked and reinstalled CRC probe.
- (b) Removed and checked Durco valves for possible defects.
- (c) Installed new air supply lines to Durco valve air cylinders.
- (d) Installed pressure relief line on the top of the push tank, N5.
- (e) Inspected XAW screen and push tank top-hat.
- (f) Installed new large XAF spare pump.
- (g) Made normal checks of solenoids, air operated valves, and air cylinders.

**DECLASSIFIED**



Following the maintenance work, new 20-40 mesh resin was added and the unit was run in preparation for start-up.

VII. Solvent Treatment - G. J. Raab

During the shutdown, the interface probe and static pressure jumper was replaced in the G2 Column. Also, the jumper bypassing G3 Centrifuge was reinstalled. The No. 2 Solvent System was operated during the rework of organic wash waste from Tank P2 in the Second Uranium Cycle. Before the run, R-Cell solvent had a plutonium retention value of  $\sim 1 \times 10^7$ . After the run, the plutonium retention was  $1 \times 10^{11}$  (unstrippable). The organic heel in P2 which went through the concentrator was blamed for degrading the solvent. After the rework, Tank R1, R2 Column and Tank R2 were flushed with 5% oxalic - 3% nitric. Tank R1 showed 300 gallons off calibration so it was reflushed with 5% oxalic, 3% nitric. This flush went to Tank G1 to Tank G8. The degraded solvent in R-Cell failed to respond to treatment. The degraded R-Cell solvent inventory was stored in R5 and new solvent was made up in the system. This used up all except 600 gallons of the mixed Soltrol, spray-base diluent.

VIII. Waste Treatment and Storage - R. C. Forsman

Batch Denitration of IWW

The batch denitration of 2,400 gallons of IWW wash was accomplished by continuously adding a 1.4 molar sugar solution during a 48 hour period to the hot (100° C) constantly agitated IWW batch. The reaction was very smooth with tank vacuum readings remaining at greater than 15 inches of vacuum during the entire test. Sizing of sugar addition equipment which limited flow rates to about one-half the assumed flowsheet rate and cooling the reaction vessel for sampling extended the treatment over a 48 hour period vs. the desired period of 12 hours reaction time followed by a 12 hour digestion period.

The free nitric concentration was reduced from 4.5 M  $\text{HNO}_3$  to 0.68 M  $\text{HNO}_3$  for an efficiency of about 18 moles of nitrate destroyed per mole of sugar. Samples taken 24 hours after the final sugar addition indicated no change in nitrate concentration while digesting at 70° C.

IX. Fission Product Recovery - W. C. Schmidt

A. Strontium Recovery

Run Series 55 comprised of three sulfate precipitations, one oxalate precipitation, and one concentration precipitation was completed. Approximately 75 kilocuries of strontium were recovered with a df of 6 for cerium.

B. Cerium Recovery

Run Series 56, Cerium Recovery #1, was processed to recover cerium. Minor chemical adjustments to the strontium recovery flowsheet (no lead as co-carrier and reprecipitation with sulfate after the oxalate precipitation) were performed to recover 1.6 megacuries of cerium as

a double sulfate cerium precipitate. The waste losses were 50 per cent.

Loading of the precipitate into the HAPD-IB-2 cask proceeded with difficulty.

In the initial attempt to load the cask, approximately 70 kilocuries of cerium-144 was transferred into the cask when the differential pressure across the filtering screen prevented any additional loading. Later examination indicated that sufficient agitation in the loadout tank (TK-E1) was not available. The cake was removed from the cask with hot 4 M nitric acid and the agitator in TK-E1 replaced.

X. Fission Product Purification - J. B. Kendall

A megacurie strontium-90 purification run was completed at the Strontium Semiworks at instantaneous processing rates of 200 to 270 kilocuries of Sr-90 per day. Performance of the solvent extraction system is shown below:

AVERAGE WASTE LOSSES - PER CENT

Stream

HA Column Effluent	6.7
HC Column Organic Effluent	0.4

DECONTAMINATION FACTORS

Impurity

ZrNb	3 000
Ru	400
Ce	19 000
Ba	1.0
Ca	30
Fe	3 000

Instantaneous HA Column waste losses were generally around 2 - 4 per cent. Difficulties in adjusting the HAF pH at start-up resulted in initial HA Column losses as high as 25 - 50 per cent averaging 6.7 per cent for the entire run. Most of the waste containing the high loss was segregated and reworked by batch contact reducing the net HA Column loss to 4.4 per cent.

The reasons for the apparent lack of barium decontamination are under investigation, however, barium concentration in the purified product were well within specifications.

The HAPD-II-1 cask was loaded with 170 kilocuries of Sr-90. Precipitate filtration behavior was excellent and the loadout was routine. This represents the fifth load for this cask with no apparent decrease in capacity.

**DECLASSIFIED**

# DECLASSIFIED

This document consists  
of No.      of 10 copies.  
Series A.

- 7 -

HW-76912

Distribution:  
1 - OF Beaulieu  
2 - WS Frank  
3 - OF Hill  
4 - BF Judson  
5 - RW McCullugh  
6 - PR McMurray  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling

## PUREX PROCESS PERFORMANCE SUMMARY FROM 0000 ON 2/4/63 TO 2400 ON 2/10/63

### I. General - G. A. Nicholson

Normal processing was resumed on February 8 at a nominal 3.0 (actual 2.5) CF after two "false" start-ups and subsequent equilibrium shutdowns, necessitated by equipment malfunctions. "Cold" HAF was turned on at 0120 and "hot" feed started at 1030. However, processing of "cold" HAF was resumed with the subsequent batch and continued until 1030 on February 10 to provide receiver space for various plutonium-bearing solutions. The "hot" feed material which was processed contained above-normal Pu-240 content.

### II. Performance Data - D. C. Leyson

#### A. Solvent-Extraction Performance by Cycles (Typical)

Insufficient data available to determine typical performance.

#### B. Production

(1) HAF Tons = 42.7

(2) U Production = 6.27 (U Product, inc. flushes)

#### C. Overall Waste Loss - % of Total Production

U = 438#

Pu = 131

#### D. Rework - % of Total Production

U = (K6→E6) 46.1 T (Inc. 11.3 T P3→K6)

Pu = (L11→E6) 2,074

#### E. Volume to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	6 079	104-A
Cell Dr.	699	105-A
FP	1 722	105-A
IWW	1 124	105-A
CW	2 043	103-BX

F. IWW Flow

Not to equilibrium.

G. Tank Farm Boil-off

(See Monthly Summary - Page 2)

III. Flowsheet - G. A. Nicholson

- (a) The plant was started up under the same flowsheet conditions that existed at shutdown, except for the following:
- (1) The 2BP Stream was routed to the "L-Cell Package" after an initial burst of activity during the first start-up attempt.
  - (2) The 2AX flow was left off until a significant amount of plutonium appeared in the 2AW Stream.
  - (3) The IBX Column was started at a frequency of 44 rather than 54 cycles per minute.
  - (4) The 2A Column was started at a frequency of 64 rather than 86 cycles per minute.
- (b) On February 7, the 2A pulse frequency was increased from 64 to 78 cycles per minute. It was further increased to 89 cycles per minute on February 8.
- (c) On February 8, the IBX pulse frequency was increased from 44 to 54 cycles per minute.
- (d) On February 9, the 2BP stream was routed to N-Cell; the 2BX composition and 2BX and 2BP rates were adjusted accordingly.

IV. Feed Preparation - G. J. Raab

At start-up, the vacuum fractionator jets produced only about 100 inches of vacuum. The inter-condenser cooling water line had either been frozen or corroded so a leak neutralized the effect of the final jet. A temporary repair was made by use of a rubber hose to replace the cooling water line.

Dissolver operation was normal except the B2 electric heater controller went out of control and the silver reactor was heated to 460° F over a period of about an hour, then shut off. The system has high temperature alarms but no high-temperature cut-off. The B2 controller has been replaced three times since start-up. I<sub>131</sub> emission has been very low (only detectable limit) as metal age has been 180 - 200 days. No mercuric nitrate has been used.

V. Solvent Extraction - G. A. Nicholson

The Solvent-Extraction System was somewhat erratic during the initial start-up on February 3 as was expected. The chief problem was in the

DECLASSIFIED

HW-76912

- 9 -

Instrumentation System. Contamination of the air system with alumina during the last operating period and the subsequent rehabilitation program resulted in a number of instruments out of calibration, and various other malfunctions. Primarily as a result of the instrumentation problems, the IC Column flooded at start-up and operated about three hours before the flood was detected. An estimated 4,000 gallons of organic was transferred to the ICU Concentrator during the IC flood, necessitating a shutdown to permit its disposition.

After stripping the uranium from the organic and segregating both for future recovery, the plant was started up a second time on February 7, but failure of the control valve in the newly installed 2AF Jumper necessitated a second equilibrium shutdown nine hours later. Except for the failed jumper, operation of all equipment during the second start-up was excellent. The third start-up, following replacement of the failed jumper, also proceeded smoothly and all cycles were up to rates and "leveled out" eight hours after start-up preparations commenced.

First Cycle performance was slightly erratic until processing of "hot" feed commenced on February 10. While no significant activity burst occurred, waste losses were above normal, requiring segregation of about one and a half batches of IWW for rework. The typical mild instability of the IBX Column during start-up was experienced, but a ten-cycle per minute reduction in the pulse frequency quickly stabilized the column.

Performance of the Final Plutonium Cycle was generally good after start-up. Losses were erratic because of erratic control of the IBXP-HNO<sub>3</sub> Stream. Activity in the product (2BP) stream rapidly dropped to normal, after the initial burst on February 3, and the 2BP was routed back to the Anion-Exchange System as soon as that system was flushed.

Performance of the Final Uranium Cycle was excellent. Some high-activity solution was apparently left in the 2EU Concentrator as the initial batches of concentrated uranium product were several-fold over limits with respect to activity. Activity of the uranium product decreased rapidly, however, and was within limits for treatment through the Silica Gel Facility by the end of the report period.

#### VI. Product Treatment - R. W. Lambert

##### Plutonium Purification - N-Cell

During start-up on February 3, but prior to starting the HAF flow, N-Cell experienced a severe gamma burst via the 2BP Stream. The source of the gamma appeared to be crud which were dislodged from the IBX, IBS, and 2A Columns during the start-up activities. For the remainder of the start-up and until February 9, the 2BP Stream was diverted to the L-Cell Package.

Following the gamma burst, N-Cell was stripped and flushed in an effort to reduce the radiation levels. On February 4, efforts to return N-Cell to normal operation failed when severe pushing problems developed. A laboratory examination of a resin sample disclosed a large fraction of cracked beads as well as a sizeable portion of 10-20 mesh resin which had been eliminated from the make-up. Based upon this evidence, the

resin was changed out and on February 6, a new charge of 20-40 mesh resin added. The discarded resin had been used for only 72 hours of actual N-Cell operation.

Immediately after the changeout, N-Cell again encountered problems in the form of a tight resin plug in the XAF line between the XA Column and the remote head of the XAF pump. The plug is believed to have been created by resin which had been forced through the XAF distributor screen when the unit was inadvertently started up with the scrub streams on and the XAW and XSW valves closed. By February 9, the unit was operating satisfactorily with the exception that it was difficult to route the XAW stream to TK-J1.

VII. Solvent Treatment - G. J. Raab

The organic recovered from the IC Column flood was washed eight times in KI with water, dilute nitric acid and sodium carbonate. The 3,900 gallons of organic was 48 per cent TBP; however, Process Chemistry tests indicate this solvent compared favorably with plant solvent when butted with diluent to specification and permanganate-carbonate washed.

Solvent from R Cell was transferred to G-Cell to make up for the solvent lost during the flood. R-Cell has  $2 \times 10^6$  plutonium retention value.

The G-Cell solvent was still very low in activity (241 Zr, 146 Ru).

VIII. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

Following the plant start-up, it was quickly apparent that the maximum obtainable boil-off rate in the IWW Concentrator (E-F6) was only about 70 per cent of that during the previous run period. In addition the factor limiting the capacity was the tube bundle steam pressure rather than pressurization of the concentrator as experienced previously.

This reduction in boil-off capacity apparently resulted from the installation of two new 600 ft<sup>2</sup> titanium tube bundles in E-F6 during the January shutdown. The previous bundles were standard 1650 ft<sup>2</sup> stainless steel bundles.

IX. Fission Product Recovery - W. C. Schmidt

Considerable difficulty was encountered in moving the slurry from TK-E1 to the cask in the second loading attempt. The loadout pot jet dip-leg and jet continually plugged. A preliminary calorimeter test with a wet cake indicated 107 kilocuries of cerium-144 was loaded into the cask.

This document consists of No. of 10 copies. Series A.

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - OF Hill
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - PR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 2/11/63 TO 2400 ON 2/17/63

I. General - G. A. Nicholson

The plant was operated at a nominal 3.0 (actual 2.5) CF at the beginning of the report period. On February 12, the rate was increased to a nominal 3.3 (actual 3.27) CF. Processing of "nonrepresentative" feed material continued until February 15. After recycling two batches of "cold" uranium to the HAF through the system, the plant was shut down at equilibrium on February 16 to change the tube bundles in the IWW Concentrator (E-F6). Start-up preparations were in progress late on February 17.

II. Performance Data - D. C. Leyson

A. Solvent-Extraction Performance by Cycles (Typical)

Data not typical due to upsets and recycle.

B. Production

- (1) HAF Tons = 97.8
- (2) K6 to P Tank: 103.5 T
- (3) L10 Loadouts: 91,340

C. Overall Waste Loss - % of Total Production

U = 447# - 0.22%  
 Pu = 227 - 0.25%

D. Rework - % of Total Production

U = 22.0 T - 22.1%  
 Pu = 11,278 - 12.3%

E. Volume to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	337	104-A
Cell Dr.	188	105-A
FP (Ge Flushes)	22	105-A
IWW	-0-	
CW	300	103-BX

F. IWW Flow

Not to equilibrium.

G. Tank Farm Boil-off

(See monthly Summary - Page 2)

III. Flowsheet - G. A. Nicholson

- (a) On February 12, the HAX was increased from 385 to 427 flows. The HAF rate was increased accordingly to adjust the production rate to 3.0 CF.
- (b) On February 12, all process streams, except the IBXP, IBP, IBS, and Final Plutonium Cycle streams were increased by ten per cent to a nominal 3.3 CF.
- (c) On February 12, rework of IWW was commenced with about 200 gallons in the first batch of HAF. After skipping a batch, rework was resumed at a rate of 300 gallons in each six-ton batch of HAF.
- (d) On February 15, recycle of "cold" uranium from the Final Uranium Cycle (Tank K6) to the HAF Make-up Tank (Tank E6) was commenced to reduce the in-process plutonium inventory in preparation for transition to normal feed material.
- (e) On February 16, the plant was shut down at equilibrium.
- (f) On February 17, start-up of the plant from the equilibrium shutdown was commenced with the Final Plutonium Cycle "on stream" at about 2230. The 2AX flow was left off until the plutonium saturation in the 2A Column reached normal level.

IV. Feed Preparation - G. J. Raab

Charging of nonrepresentative metal was completed and the heels removed. Only dissolvers A3 and B3 were used. The metal solution tank heels were flushed forward into the last of the hot feed.

The coating waste line appeared to be building up a plug again. Jetting became slower and on February 14, flow became intermittent. The first 25% caustic flush jetted slowly but a second flush (~ 3000 gallons) jetted well. It was recommended that it become routine that a flush of 5% caustic be made weekly.

An investigation of the vacuum fractionator tail water sampling point showed that it had been disconnected and the line through the wall used for an instrument function. There was no I<sub>131</sub> emission of significance.

V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance was excellent during the report period. Following the difficulties experienced during and immediately after start-up (discussed in the preceding summary), the operation "steadied



out" and process control improved greatly. The only major problem was that of excessive plutonium levels in the process, engendered by abnormal "losses" in the Plutonium Anion Exchange Cycle. No processing difficulties resulted from the high levels, however.

Decontamination performance of the First Cycle was generally good. The activity level of the product streams was about two-fold below normal, due primarily to the increased cooling time of the "nonrepresentative" feed material. Waste losses, initially low, showed an increasing trend until shutdown, due primarily to below-normal acidity in the HA Column.

Decontamination performance of the Final Plutonium Cycle was excellent in spite of considerable fluctuation in the "losses" to the Backcycle System. Typical decontamination factors ranged from  $2 \times 10^3$  to  $6 \times 10^3$ . The abnormal fluctuation in "losses" was due primarily to a malfunction in the flow control system on IBXP- $\text{HNO}_3$  addition stream. All plutonium product met specifications after processing through the Plutonium Anion Exchange Cycle, with the Zr/Al ratio generally less than  $1 \times 10^{-11}$ .

Performance of the Final Uranium Cycle has been excellent, with plutonium contamination in the final product less than two parts per billion parts of uranium and activities within shipment limits without further treatment. The great improvement in performance was attributed to the extensive flushes which were given the columns during the January Shutdown Period. Two standard decontamination flushes followed by a hot five per cent carbonate flush to remove the organic film from the sieve plates and a ten per cent nitric pretreatment flush were given the 2D and 2E Columns. The hot carbonate and nitric flushes were primarily intended for the 2D Column Scrub Section (to restore the aqueous-wetting characteristics of the stainless steel sieve plates), but were routed through the 2E Column for convenience.

#### Neptunium Recovery

Neptunium accumulation in the Solvent-Extraction System was normal during the report period. Typical losses averaged ten and three per cent for the HAW and 2EU Streams, respectively. The Continuous Neptunium Recovery Cycle (J-Cell Package) was not operated during the report period.

### VI. Product Treatment

#### A. Plutonium Purification (N Cell) - R. W. Lambert

Except for difficulties in routing the XAW Stream to TK-31, N-Cell performance was good during the report period. As a means of monitoring resin quality, a program has been initiated to sample the N-Cell resin three times a week with the resin being sent to Separations Chemistry Laboratory for evaluation. Samples checked during this report period showed no increase in the per cent of broken beads from that of the start-up sample. The color of the resin did change from nearly colorless to golden.

DECLASSIFIED

HW-76912

-14-

Flushing of the XAW line to TK-J1 failed to improve the waste routing problem. A flow of about 2.5 gpm to TK-J1 was obtained using the L11 to TK-E6 jet pump as the motive power for the flush.

N-Cell did experience slightly higher than desired XAW losses during the report period. This was a result of the plant's high plutonium throughput rate created by processing high exposure metal while simultaneously reworking out-of-specification nonrepresentative plutonium from the L-Cell Package. The N-Cell plutonium Pu rate reached an equivalent 4.5 CF based on the processing of normal uranium.

On February 17, N-Cell was stripped and all the product tanks were emptied to effect segregation of the nonrepresentative plutonium.

For several weeks, the operation of the L11 to TK-E6 transfer system has been marginal with transfers being made sporadically and at greatly reduced rates. To date, efforts to correct the problem have consisted of overhauling the jet pump, acid flushing the trench line, and checking valve operation. A new jet is on order, but not currently available. No noticeable improvements have resulted.

B. Neptunium Purification - S. M. Nielson

Functional testing of the Q-Cell equipment was completed and "cold run" system testing was started this report period. The "cold runs" simulate regular operation in that standard flow rates, operating times and chemicals are used.

Cold run tests completed to date include (1) operation of the E-Q2 3BN Concentrator, (2) resin add to the T-Q4 3X Column, (3) resin pre-treatment step, and (4) the feed make-up step. The resin loading step was started the last day of the report period.

The steps were all run at rated flows except the E-Q2 3BN Concentrator which was run at a limiting .44 gallons per minute (88% of rated flow). This step will be rerun at rated flow later in the "cold runs".

VII. Solvent Treatment - G. J. Raab

A layer of ice on the bottom of TK-40 had given false diluent inventory readings so that 5400 gallons (rather than 600 gallons) of mixed diluent (Soltrol 170, Shell E-242) in TK-40 had to be added to the plant system before a shipment of Soltrol could be unloaded. About 6800 gallons of solvent (30% TBP) was added to R-Cell, about 2000 gallons of solvent was transferred to G-Cell, and about 1200 gallons was left in TK-R1A, all made up from the contents of TK-40.

TK-40 was flushed with high pressure water to the chemical sewer and a 20,000 gallon tank car of Soltrol was unloaded.

The one per cent sodium hydroxide heel in TK-G7 was changed out on February 15 as samples of IOD were very hot.

VIII. Acid Recovery and Waste Concentration - R. W. LambertF-Cell Acid Recovery

During the report period, it became apparent that the reduced boil-off capacity of the IWW Concentrator E-F6 was limiting the plant rate to approximately a 3.0 CF in terms of acid recovery efficiency and increased salt disposal to the waste tank farm. Therefore, on February 16 and 17, following an equilibrium plant shutdown, the stainless steel tube bundles in E-F11 and the titanium E-F6 bundles were interchanged. Subsequent plant operation demonstrated the return of E-F6 capacity to its previous rates (~ 54 gpm boil-off) with pressurization of the concentrator again limiting.

Also during the report period, the E-F6 Concentrator was twice successfully cooled and jetted while the plant was operating. On February 12, it was emptied to segregate IWW for rework and on February 14, it was jetted to provide IWW needed for meeting tank farm heating requirements.

IX. Waste Treatment and Storage - R. C. ForsmanA-Farm Operation

Recent temperature excursions in Tanks 241-A-104 and 106 have apparently been controlled by increased circulation and dilution of the tank contents. The temperature in Tank 104-A increased from about 130° C to 224° C, then slowly decreased to 213° C. The temperature in Tank 106-A increased from about 190 - 195° C to 220° C before slowly decreasing to about 215° C.

To conform with the 2° F per day (or 40° F incremental) temperature increase limit which is established to minimize thermal stresses during the initial underground storage tank filling operations, Tank 105-A was partially filled with hot water and two batches of waste from the December operating period. During the report period, IWW representing approximately 70 tons of production was transferred to Tank 105-A to increase the temperature from 140 - 145° F to 175 - 185° F. Building surge space will allow storage of IWW for 10 - 14 days which will allow a sufficient digestion period at this temperature before the continuous addition of IWW will raise the temperature the final 40° F to boiling.

X. Fission Product Recovery - W. C. SchmidtCerium Recovery

The cerium cask was heated to 314° F (internal), 271° F (side) with steam coils. The Woods Metal cavity pressure gauge dropped from a pre-set 8 psig to 4.1 psig.

A calorimeter check of the dried cake indicated that 90 kilocuries of cerium-144 was loaded into the cask.

DECLASSIFIED

HW-76912

-16-

XI. Fission Product Purification - J. B. Kendall

Strontium Semiworks

An 11 gallon cask was loaded and transferred with  $47.5 \pm 2.5$  kilocuries of Sr-90 in a volume of 23.2 liters. The cask loaded very slowly. Whether this was due to an extremely high pressure drop in the vent Hoke valve as leaks in the loading system is not known. No other problems were encountered.

# DECLASSIFIED

HW-76912

This document consists of No.      of 10 copies. Series A.

-17-

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - OF Hill
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - PR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 2/18/63 TO 2400 ON 2/24/63

I. General - G. A. Nicholson

The plant was started up with "cold" feed at a nominal 3.3 CF on February 18, with "hot" feed of normal irradiation history to the HA Column at 0930. On February 19, the processing rate was increased to a nominal 3.6 (actual 3.50) CF. The actual rate was increased to 3.75 CF over the next three days.

II. Performance Data - D. C. Leyson

A. Solvent-Extraction Performance by Cycles

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>U</u>	<u>% Loss</u>	<u>Np</u>	<u>Np Acc.%</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>		<u>Pu</u>		
First	2.5	3.7			0.002	0.03	15.0	
Final	3.0	3.4	7.5	3.7	0.002		5.0	
IE		1.1		0.3				
Overall	5.5	8.2	7.5	4.0	0.004	0.03	20.0	24.0

B. Production

- (1) HAF Processed = 206.1 T
- (2) Capacity Factor = 3.5-3.6
- (3) U Loadouts = K6 to P Tank: 193.3 T
- (4) Pu Loadouts = I9, 10 Loadout: 97,246
- (5) Np Loadouts = Q7 Loadout: -0-

C. Overall Waste Loss - % of Total Production

U = 0.2%  
 Pu = 0.3%  
 Np = F16, 18 - 125  
       K6 - 11  
       -----  
       136

D. Rework - % of Total Production

U = 2.4%\*  
Pu = 1.3%\*\*

\* 98% K6 → E6  
\* 2% F16 → E6  
\*\* 65% L11 → E6  
\*\* 37% F16 → D5

E. Volume to UGS

<u>Source</u>	<u>Gal/Ton (Proc.)</u>	<u>Waste Tank</u>
OWW	176	101-A
Cell Dr.	103	105-A
FP	29	105-A
CW	300	103-EX
IWW	-0-	

F. IWW Flow

5.0

G. Tank Farm Boil-Off

(See Monthly Summary - Page 2)

III. Flowsheet - G. A. Nicholson

- On February 18, rework of IWW was temporarily suspended with completion of the first batch. Rework of the second batch was commenced on February 20 and completed on February 24.
- On February 24, rework of hydrolyzed waste (F\*) material was commenced with a 100-gallon addition to a batch of HAF.
- On February 24, the 2LF-ferrous sulfamate was reduced from 0.29 to 0.24 flows.

IV. Feed Preparation - G. J. Raab

Dissolver operation was subnormal during the week due to poor vacuum control. I131 emission was 0.51 curies for the period, with metal cooling times of 130 to 160 days. The HAF nitric acid analysis results were erratic, possibly associated with waste rework operations, a faulty E6 acid meter, or sampling difficulties.

V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance following the start-up was somewhat erratic, but by the end of the report period, had improved to the general high

level of the previous report period. Following a generally smooth start-up, the "heel" of the first batch of IWW was added to the HAF, producing inextractable plutonium and high waste losses. In an attempt to minimize the losses, the organic saturation in the HA Column was reduced until the fission product activity of the product (HSP Stream) was about seven-fold above normal. While the system was recovering from the above increase in activity, the HSS-H<sub>2</sub>O flow was temporarily lost, resulting in another mild burst of activity. As a result, the activity of both plutonium and uranium product streams increased to a maximum of about two-fold above specifications.

Performance of the Final Plutonium Cycle has continued at an excellent level, with decontamination factors ranging between 2500 and 4000 despite continuing fluctuations in the acidity in the 2A Column. This also resulted in wide fluctuations in the 2AW "losses". The primary source of the problem, a faulty converter in the signal transmission system of the IBXP-HNO<sub>3</sub> Stream, was located and corrected.

Performance of the Final Uranium Cycle has also continued to be excellent, with plutonium contamination in the final product below the detection limits ( $\frac{1}{1}$  ppb) since start-up and neptunium losses via the 2EU Stream are about a factor of 3 below the average losses for the past 6 - 9 months. Final uranium product activity "peaked" about two-fold above shipping limits requiring operation of the Silica Gel Facility, but decreased back to near limits by the end of the report period.

#### Neptunium Recovery

Neptunium accumulation in the Backcycle Waste System (3WB) has continued at the normal rate. The below-normal losses to the Final Uranium product have been offset by the high loss via the HAW Stream during the IWW rework.

### VI. Product Treatment - R. W. Lambert

#### A. Plutonium Purification

The Plutonium Ion Exchange Unit, N-Cell, operated satisfactorily during the report period. Efforts failed, however, to route the XAW waste to TK-J1 and the waste stream was routed to TK-F10 throughout the week. The decontamination factors obtained during the period (12-15) were somewhat lower than optimum, but this was probably a reflection of the erratic and low fluoride flow caused by malfunctioning of the NaF pumps.

Small amounts of resin continued to be lost from the unit. A portion of this resin escaped through the feed screen which resulted in periodic plugging of the XAF feed line. A "dutchman" was installed in the XAF line to facilitate draining and flushing the resin from the line.

Continued sampling of the N-Cell resin disclosed fewer cracked beads and fewer larger beads. This indicates that the beads originally

B. Final Uranium Treatment - R. W. Lambert

On February 22, the Silica Gel unit was started up to process out-of-specification uranium product. Effluent from Silica Gel was well within shipping specifications.

C. Neptunium Purification - S. M. Nielson

Cold run systems testing of Q-Cell equipment continued during the report period. The resin loading step, Pu wash step, and fission product wash step were completed by the end of the report period with no major problems involved. The E-Q2 Concentrator was operated, but still unable to reach flowsheet rates when using top overflow from concentrator. Flowsheet rates are possible when the concentrator is allowed to overflow from the bottom jet-out leg.

VII. Solvent Treatment - G. J. Raab

Performance of the solvent systems was excellent with G5 activity levels of 300 uc/gal Zr-Nb and 800 uc/gal Ru. Solvent losses from the complete system totalled about 500 gallons for the week. About 600 gallons of solvent was lost from the No. 2 Organic System to the No. 1 Organic System, resulting in a net gain of about 100 gallons in the No. 1 System.

VIII. Acid Recovery and Waste Concentration - R. W. Lambert

The F-Cell acid recovery operation continued to operate at satisfactory rates following the tube bundle switch on Feb. 17. The activity level in the AAA acid also remained satisfactory with the equilibrium Ru reaching about  $3.0 \times 10^4$  uc/gal and the Zr-Nb reaching about  $1.2 \times 10^4$  uc/gal.

On Feb. 18, a special sampling program was initiated in an effort to identify the activity being added to the swamps and cribs from the recovered acid system. The program calls for composite samples of the E-F5 overheads, A10 crib, B swamp, Gable Mt. swamp, fractionator tail water, and U1-U2 recovered acid to be run once per week for four weeks for HNO<sub>3</sub>, gross beta, Ru, Zr-Nb, Sr, and Cs.

IX. Waste Treatment and Storage - R. C. ForsmanA-Farm Operation

Transfers of IWW to TK-105-A increased the temperature from the 60 to 65° C range to 80 to 85° C range. Further transfers are being delayed until Feb. 28 which should allow the tank to remain within the desired 2° F per day temperature increase during initial filling operations to minimize thermal stress in the structure.

The temperature in TK-241-A-104 decreased from a maximum of 224° C to 183° C. New thermocouple and temperature bulb systems installed in TK-241-A-106 indicate temperatures of 132° C and 131° C, respectively, while previous temperatures had been reported in the 210 to 225° C range.



Attempts to rotate the shaft on the 103-A sluice pump have indicated the impellers are jammed or bearings are frozen. Efforts to replace the pump are being placed on a crash program basis.

X. Fission Product Recovery - W. G. Schmidt

A. Cerium Cask - HAPO TB-2

After drying the cake, a calorimeter check of the cerium cask was completed. With a flow rate of 1/2 gallon per minute, a differential temperature between the inlet and outlet was measured as 11 degrees F. With a cerium-141 to cerium-144 ratio of 0.57, the data indicate the cask to be loaded with 90 kilocuries of cerium-144.

During the heating cycle, the Wood's Metal cavity pressure instrument decreased. After cooling the instrument was checked, the pilot valve replaced, and the cask reheated to 170° F. With steam on the coils, no pressure increase was observed in the cavity. The cask was cooled, purged with helium, and sealed. The pressure buildup due to radiolytic hydrogen is less than 1/2 pound per square inch per day.

B. Cesium Loadout (Bldg. 801-C)

The new cesium loadout facility was activated on 2/21. The feed pump delivered only 2.5 gpm. After resetting the impeller clearances, the loading jet limited the maximum flow to the cask at 3.5 - 4.0 gallons per minute. The feed sampler does not operate satisfactorily. The volume of the sample bottle is not sufficient to draw a sample into the bottle when the vacuum is released. The magnetic flowmeter on the feed to the cask has been erratic. To date the total volume of feed to a cask has been determined by liquid level measurements in the 103-C Tank.

Two casks were loaded, one with approximately 60 kilocuries of cesium and one with approximately 50 kilocuries of cesium. The temperature of the feed was easily adjusted and at 75° F, the waste losses were less than .05 per cent.

DECLASSIFIED

HW-76912

This document consists of No.      of 10 copies. Series A.

-22-

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - OF Hill
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - FR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 2/25/63 TO 2400 ON 3/3/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.6 (actual 3.7) CF during the report period. The HA and HS Columns were shut down at equilibrium on March 1 to replace a leaking HAF Jumper. The HAF was shut off at 1020 and back on at 1430.

II. Performance Data - D. C. Leyson

A. Solvent-Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Acc. %</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.8	3.4					5.4	
Final	2.9	3.5	7.5	5.35	.004	0.04	5.0	
Ion Ex.		1.2		0.08	.002			
Overall	6.7	8.1	7.5	5.43	.006	0.04	10.4	73.0

B. Production

- (1) HAF Processed = 215.7 T
- (2) Capacity Factor = 3.6
- (3) U Loadouts = K6 - 207.7 T
- (4) Pu Loadouts = L10 - 123,962 Gm .

C. Overall Waste Loss - % of Total Production

U = 0.27% (1,117#)  
 Pu = 0.44% (545 Gm)  
 Np = F16, 18 - 31% (147 Gm)  
       K6 - 4% (19 Gm)

D. Rework - % of Total Production

U = 1.7% (7,029#) F8 → D5  
 Pu = 5.1% 2,459 Gm  
       3,868 Gm L11 → E6

**E. Volume to UGS**

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	170	101-A
CD	50	105-A
FP	97	105-A
IWW	36	105-A
CW	300	103-BX

**F. IWW Flow**

6.0

**G. Tank Farm Boil-Off**

<u>Tank</u>	<u>Gal/Min</u>
101	2.5
102	2.2
103	.4
104	2.5
105	-
106	14.4

**III. Flowsheet - G. A. Nicholson**

- (a) On February 25, the 3WB-HA was temporarily increased from about 34 to 50 flows as the Neptunium Recovery Cycle was started up on "cold" feed. The 3WB-HA rate was reduced back to about 35 flows over the following twelve hours as 3WB was diverted to the 3AF Tank.
- (b) On February 25, the rate of rework of F8 solution was increased from 100 gallons to 200 gallons per seven-ton batch of HAF.
- (c) On February 25, the 3A Column pulse frequency was reduced from 80 to 75 cycles per minute.
- (d) On February 26, the 3B Column pulse frequency was reduced from 80 to 75 cycles per minute.
- (e) On February 27, the 2DF and 2DX rates were reduced by about ten per cent to build up an inventory in the 2DF Tank in preparation for the temporary shutdown of the HA and HS Columns. The rates were returned to normal on February 28.
- (f) On March 1, the 3AF rate was reduced from 26 to 23.5 flows. It was further reduced to 20.8 flows on March 2.
- (g) On March 1, the 2AX rate was reduced by 50 per cent and the 2AF flow shut off during the temporary shutdown of the HA and HS Columns. After startup, the first two batches of 2AF were jetted to the 3WB Tank (TK-J1).
- (h) On March 2, the 2DF-ferrous sulfamate rate was reduced from 0.25 to 0.21 flows.

IV. Feed Preparation - G. J. Raab

Dissolver operation was improved somewhat when the vacuum jet steam supply was raised from 55 psig to 75 psig. However, the marginal vacuum on A3 Dissolver was not improved by regulating the charging lid. The cell filter B1 showed signs of plugging but the DP started to decrease at weeks end; a steam leak in the B2 Heater could have caused these symptoms. I<sup>131</sup> emission was 0.52 curies for the week with metal cooling times of 140 to 160 days.

V. Solvent Extraction - G. A. Nicholson

Generally superior performance with respect to both waste losses and decontamination performance was characteristic for the report period. Plutonium losses via the HAW Stream were much more constant and lower than during the previous report period although an upward trend was evidenced at the end of the report period. The losses averaged less than 0.04 per cent. The upward trend was coincident with termination of the rework of F8 hydrolyzed waste material. A general increase (~ 60 per cent) in activity of the First Cycle product streams was also noted upon termination of rework.

Performance of the Final Plutonium Cycle continued at an excellent level. The increase in First Cycle product (IBP) activity was not reflected by a corresponding increase in the 2BP activity. DF's ranged from a low of 2800 to 4000 at the end of the report period despite a general increase in "losses" to the Backcycle Waste System.

Performance of the Final Uranium Cycle continued at about the same general level as during the previous report period. A sharp, 35 per cent decrease in Zr-Nb activity of the final product stream occurred at the beginning of the report period, but rate changes on February 27 coupled with increasing activity in the 2DF Stream resulted in a two-fold increase toward the end of the report period and it was necessary to commence Silica Gel treatment of the uranium product.

Neptunium accumulation in the Backcycle Waste System showed considerable improvement after termination of the IWW rework. Losses via the HAW Stream were three-fold less than the previous report period, averaging about four per cent. Losses via the 2EU Stream, however, were generally slightly higher, averaging about five per cent.

The Continuous Neptunium Recovery System (J-Cell Package) was started up on February 25. Accumulation in the system was somewhat slower than normal due to a high uranium-to-nitric acid ratio in the 3WB Stream. Flow adjustments were in progress to accommodate this condition.

VI. Product TreatmentA. Plutonium Purification (N-Cell) - R. W. Lambert

Operation of the N-Cell ion exchange unit continued to be satisfactory during the report period with no increases in push time or push pressure noted.

# DECLASSIFIED

HW-76912

-25-

Separations Chemistry Laboratory reported the following information based on a visual inspection of resin samples. The figures are only approximations.

<u>Sample Date</u>	<u>% Beads Smaller Than 50 Mesh</u>	<u>% Beads 35-50 Mesh</u>	<u>% Beads 25-35 Mesh</u>	<u>% Beads Larger 25 Mesh</u>
Feb. 11	7%	26%	54%	13%
Feb. 18	15%	50%	25%	10%
Feb. 25	18%	60%	21%	1%

These figures confirm previous observations that the large beads in the resin are more prone to physical destruction than the small beads. Microscopic examination also reveals that the per cent of cracked beads present in the resin had declined since startup. This indicates the rate of current bead breakup is small.

No progress has been made in establishing a satisfactory flow of XAW to TK-J1 and the waste remained routed to TK-F10 during the report period.

On March 2, the TK-L11 to TK-E6 jet was removed, rodded out and reinstalled. Subsequent operation of the transfer system was exceptionally good. Previous difficulty with the system can probably be attributed to a plugged jet.

## B. Uranium Silica Gel - R. W. Lambert

On March 1, the Silica Gel unit was shut down and the regeneration procedure began. On March 3, regeneration was completed and the unit put back on the line.

## C. Neptunium Purification - S. M. Nielson

Cold run systems testing of Q-Cell equipment was continued this report period. The Fluoride Scrub Step and the Forecut and Elution Step were completed. Testing of the T-Q4 Ion Exchange Column will be complete with the running of the Resin Removal Step. This test will be run during the period that Tank Q5 maintenance work is being done.

The E-Q2 Concentrator was operated using top overflow and lower vacuum (8 in.). At flowsheet rates, the concentrator will not maintain a constant weight factor, but continued to drop off to the low weight factor cutoff point.

## VII. Solvent Treatment - G. J. Raab

Performance of the solvent systems was good. The activity levels of G5 were trending upward to 650 uc/gal Zr-Nb and 1300 uc/gal Ru. Solvent loss from the system totalled 4070 gallons for the week. The No. 2 System lost about 1980 gallons to the No. 1 Organic System resulting in a net loss of about 2,090 gallons (0.03% of gallons processed) in the No. 1 System.

0.376

VIII. Acid Recovery and Waste Concentration - R. W. LambertF-Cell System

Activity levels in the AAA remained fairly stable during the report period with the Zr-Nb activity steady at  $1.2 \times 10^4$  uc/gal and the Ru rising slightly to  $5 \times 10^4$  uc/gal.

On March 3, the nitrite addition to the trays of the IWW Concentrator E-F6 was stopped and the nitrite addition to the IWF Tank TK-F12 doubled. The total nitrite added remained unchanged, .01 M  $\text{NaNO}_2$  in IWF, but the change in addition point will test the effectiveness of tower addition versus pot addition.

IX. Waste Treatment and Storage - R. C. Forsman

Operation of the formaldehyde unit has been delayed due to failure to place the instrumentation in operating condition.

The sludge temperature in Tank 241-A-104 continued to decrease reaching  $154^\circ \text{C}$  at the end of the report period. Although some erratic readings as high as  $258^\circ \text{C}$  have been recorded in Tank 241-A-106, readings taken with new calibrated thermocouples and temperature bulbs indicate the general temperature to be about 120 to  $130^\circ \text{C}$ . Efforts to obtain reliable readings in the tanks continue.

X. Fission Product Recovery - W. C. SchmidtA. Cesium Recovery

The remaining two STI casks were loaded with approximately 50 kilocuries of cesium per cask. Difficulties experienced with the previous two casks (sampling and volume of feed through the cask) were corrected. The samplers will draw sufficient liquid in the sample bottle by a sequenced operation of opening and closing appropriate valves. The magnetic flowmeter was repaired and appears to be working properly. The loading of these two casks proceeded very smoothly and indicates the Cesium Loadout Station is an excellent facility.

B. Strontium Recovery

Recovery of strontium from IWW was resumed in E-Cell of the Purex Building. Hydroxyacetic acid has been substituted for tartaric acid as the iron-complexant. Four separate sulfate precipitations (run series 57) were made. The fourth precipitation was discarded prior to the sulfate centrifugation when it was noted that the caustic for neutralization had been added prior to the addition of hydroxyacetic acid for complexing iron.

Waste losses for this series were extremely high ranging from 43 to 81 per cent for strontium and 58 to 83 per cent for cerium. Two abnormal conditions were noted. Caustic ratios of the sulfate waste as obtained

DECLASSIFIED

HW-76912

[REDACTED]  
-27-

from the Analytical Laboratory ranged from 1.7 to 2.2 lbs per gallon, whereas all previous runs had ranged from 0.5 to 0.9 lbs. per gallon. This indicates the pH meter may not have been operating properly. The thermohm or associated equipment in the precipitator tank has not been responding properly. It was shown by test that both water and LWV were boiling when the thermohm indicated the temperature to be 85° C. The CVT has been repaired and plans to replace the thermohm are being made.

This document consists  
of No. 1 of 10 copies.  
Series A.

[REDACTED]  
-28-

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - OF Hill
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - PR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 3/4/63 TO 2400 ON 3/10/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.6 (actual 3.8) CF until March 10, when the rate was reduced to 3.3 CF. Two batches of cold uranium were recycled to the HAF Tank - one on March 8 and the other on March 10 - to maintain continuity of operation.

II. Performance Data - D. C. Leyson

A. Solvent-Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Acc. %</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.5	3.4			.002	.25	6.8	(J1) - 11%
Final	3.2	3.3	7.0	2.8	.002		4.0	(3BN) - 64%
Ion Ex.		1.1		9.0				
Overall	6.7	7.8	7.0	11.8	.004	.25	10.8	75%

B. Production

- (1) HAF Processed = 206.6
- (2) Capacity Factor = 3.6
- (3) U Loadouts = K6 to P Tank: 195.8
- (4) Pu Loadouts = L9, 10: 137,790

C. Overall Waste Loss - % of Total Production

- U = 898# - 0.23%
- Pu = 515 gms - 0.37%
- Np = 128 gms - 28%
- K6: 14 gm (3%)
- F16: 104 gm (25%)

[REDACTED]



D. Rework - % of Total Production

U = 13.4 T - 6.8%  
 Pu = 21,847 gms - 15.9%

E. Volume to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	147	101-A
Cell Dr.	98	105-A
FP	88	105-A
IWW	73	105-A
CW	300	103-BX

F. IWW Flow

6.1 - Ave. HNO<sub>3</sub> Conc. 6.0 M

G. Tank Farm Boil-Off

(See Monthly Summary - Page 23)

III. Flowsheet - G. A. Nicholson

- (a) On March 5, the Neptunium Recovery Cycle rates were reduced by 55 per cent.
- (b) On March 6, the continuous addition of KMnO<sub>4</sub> to the IOF Tank was terminated.
- (c) On March 6, the ZDS was increased from 48 to 52 flows.
- (d) On March 7, the Neptunium Recovery Cycle was shut down at equilibrium.
- (e) On March 7, the ZDF temperature was found to be at 20° C due to a faulty instrument. The fault was corrected and the temperature increased back to 50° C.
- (f) On March 10, all flows, except the IBX, IBS, 2AS, 2AF, 2AX, and 2BX, were reduced by about eight per cent, as the CF was reduced from 3.6 to 3.3.

IV. Feed Preparation - G. J. Raab

Dissolver A3 operation was subnormal during the week due to low pot vacuum. The differential pressure across the A-Cell heater-reactor has increased to 17 - 18 inches and will require flushing and regenerating during the next outage. I<sub>131</sub> emission was 0.90 curies for week. Because of a sudden increase in apparent I<sub>131</sub> output, mercuric nitrate was added to the metal solution starting on March 8. . Coating wastes were jetted to UGS without difficulty during the week.

V. Solvent Extraction - G. A. Nicholson

Process performance during the report period was characterized by generally higher activity in the First Cycle product streams, resulting from decreased organic saturation in the HA Column. The HA Column saturation was decreased in an attempt to reduce the apparently high plutonium losses via the HAW Stream (0.4 to 0.8 per cent). The problem had not been completely resolved by the end of the report period, but the following factors appeared to be contributing to the problem:

1. Plutonium rework operations.
2. Flow pot vent plugging.
3. HNO<sub>3</sub> acid flow control problems.
4. Sampling difficulties.
5. Laboratory reagent contamination.

To compound the problem discussed above, upsets in HA and HS and 3A and 3B Columns occurred on March 6 and 7. The upset on March 7 was severe enough that the Neptunium Recovery Cycle was shut down to avoid losing the neptunium which had been accumulated. The cause of the problem was subsequently traced to an inter-connecting overflow line between the seal pots in the HSR, HSS, and 3AS Addition Systems. Gassing in the 3AS System was apparently causing gas - and possibly 3AS entrainment in the HSS and HSR Streams - thus, upsetting the HA and HS Columns. The problem has not reappeared since the 3A and 3B Columns were shut down.

Performance of the Final Plutonium Cycle has continued to be very good, with the DF holding steady at about 2700. The 2AW loss decreased sharply about the middle of the run period from about five per cent to 1.5 per cent, due to an increase in the acidity in the column.

Performance of the Final Uranium Cycle improved considerably during the report period. Typical DF's increased from about 700 to about 1550 at the end of the report period. This was not sufficient to overcome the excessive activity of the 2DF Stream, however, and it was necessary to continue silica gel treatment of the final product. Plutonium and neptunium contamination in the uranium product showed a slight increase in the middle of the week but had returned to near non-detectable levels by the end of the report period.

Neptunium accumulation in the Backcycle Waste System continued at the same general rate as during the previous report period. Somewhat higher losses via the HAW Stream (about seven per cent) were offset by improving 2EW losses, which had decreased to less than two per cent by the end of the report period. Analysis of the 3WB and 2DW Streams showed no unusual loss of neptunium from the "Package" during the upsets, but verification by analysis of the 3AF or 3BN is not reliable because of the unsteady conditions at the time of the shutdown of the "Package".

VI. Product TreatmentA. Plutonium Purification (N-Cell) - R. W. Lambert

Operation of the plutonium ion exchange unit, N-Cell, continued to be

satisfactory during the report period. Decontamination performance, however, remained marginal as the fluoride addition to the feed was frequently interrupted by pump problems.

Examination of M-Cell resin samples by Separations Chemistry Laboratory indicated that the resin characteristics did not change during the report period. The shift in the size distribution of the resin has apparently stabilized.

High plant rates coupled with a high plutonium rework rate resulted in an extremely high plutonium feed rate to M-Cell. The 2BP plutonium content indicated that this rate averaged in excess of an equivalent 5.5 GF based on the processing of normal uranium.

The problem of routing the XAW flow to EK-71 remained unsolved. The pump which supplies the EK-111 to EK-E6 jet was again used to flush the routing, but at 160 lbs. pump pressure, a flow of only about 2.3 gpm was obtained. It appears that the line is partially plugged.

B. Uranium Silica Gel - R. W. Lambert

On March 11, the product from the Silica Gel unit reached the specification limits and the unit was shut down for regeneration. Approximately 200 tons of uranium was treated by Silica Gel during the eight days the unit operated.

C. Neptunium Purification - S. M. Nielson

Testing of Q-Cell equipment was discontinued during the report period while the waste collection Tank EK-Q5 was being repaired.

VII. Solvent Treatment - G. J. Raab

Solvent activity in the No. 1 System rose from values of  $6.77 \times 10^2$  and  $1.25 \times 10^3$  uc/gal to  $1.75 \times 10^3$  and  $2.58 \times 10^3$  uc/gal, Zr-Nb and Ru respectively during the week. Activity levels had risen by a factor of 1.5 before addition of  $KMnO_4$  to TK-G1 was stopped on day shift March 6. The activity has appeared to have leveled off the higher level. The net organic loss from the system was 900 gallons. Fresh solvent (3300 gallons) was added to the No. 2 System from RIA Make-up Tank.

VIII. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell System

On March 4, one day after stopping the nitrite addition to the trays of the IWW Concentrator E-F6, and doubling the nitrite flow to the E-F6 Pot, the ruthenium activity in the AAA acid dropped by a factor of two to about  $2 \times 10^4$  uc/gal. The ruthenium activity remained at this level for the rest of the report period. As a result of the test, the ruthenium in the acid absorber overheads E-F5 also dropped by a factor of about two. The zirconium-niobium activity level in both the acid and E-F5 overheads was not affected by the change in the nitrite addition method.

IX. Waste Treatment and Storage - R. C. Forsman

A. Denitration of IWW

Startup of the formaldehyde unit was delayed by a closed valve in the formaldehyde prototype instrumentation and an apparent plug in the IWW feed system to the reactor. By backflushing the feed jumper to the feed Tank TK-F7, the line was cleared and the formaldehyde unit started on March 8. After four hours of operation, the unit was shut down due to higher than normal pressure readings in the formaldehyde reactor. The differential pressure across the reactor tower, which normally records pressure surges prior to the reactor instrumentation, did not show pressure increases; therefore, the unit will not be started until instrumentation is corrected.

B. Tank Farms

Temperatures in the 241-A-101, 104 and 106 underground storage tanks have apparently returned to their normal 100 to 140° C range. New temperature bulbs were installed in all but TK-104; however, the 102 and 105 readings are still low (about 85° C).

X. Fission Product Recovery - W. C. Schmidt

Strontium Recovery

Run series 57 was completed recovering ~ 190 kilocuries of strontium-90. Laboratory analyses for both strontium and the other fission products were extremely erratic but later analysis of the carbonate and oxalate products indicated the high waste losses as reported last week were in error. Investigation by laboratory personnel found one of their extraction solutions to be in error which caused the erratic strontium results. No satisfactory explanation has been found for the erratic results on the other fission products.

The thermohm in the precipitator tank was changed. The new thermohm appears to be working satisfactorily. The installation of the submerged jet, which permits centrifuging the sulfate precipitated fission products at elevated temperature was completed. Initial tests indicate the jet will transfer solution at a rate of 10 - 15 gpm at a temperature of 75° C.

XI. Fission Product Purification - J. B. Kendall

Between March 6 and 13, the HAPD-III-2 filter cask was loaded with 130 kilocuries of Sr-90.

# DECLASSIFIED

HW-76912

This document consists  
of No. 4 of 10 copies.  
Series A.

-33-

Distribution:  
1 - OF Beaulieu  
2 - WS Frank  
3 - OF Hill  
4 - BF Judson  
5 - RW McCullugh  
6 - PR McMurray  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 3/11/63 TO 2400 ON 3/17/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.3 CF until March 15 when the rate was increased to a nominal 3.6 (actual 3.65) CF.

II. Performance Data - D. C. Leyson

A. Solvent-Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>		<u>Np</u>	<u>Np Acc. %</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>		
First	3.7	3.3			.002	.30	25.0	57%
Final	3.2	3.4	6.5	3.59	.002		3.0	-0-
Ion Ex.		1.1		0.5				
Overall	6.9	7.8	6.5	4.09	.004	.30	28.0	57%

B. Production

- (1) HAF Processed = 206.7 T
- (2) U Loadouts = (K6 → P Tk) - 198.5 T
- (3) Pu Loadouts = (L10) - 108,832 Gm.
- (4) Np Loadouts = -0-

C. Overall Waste Loss - % of Total Production

	<u>Units</u>	<u>% of Total Prod.</u>
U =	1 183 #	0.28
Pu =	598 Gm.	0.55
Np = F16, 18:	106 Gm.	23.0
	K6 8 Gm.	2.0

# DECLASSIFIED

HW-76912

-34-

## D. Rework - % of Total Production

	<u>Units</u>	<u>% of Total Prod.</u>
U =	2 152 #	.54
Pu =	1 032 Gm.	.95

## E. Volume to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	197	101
Cell Dr.	124	105
FP	111	105
IWW	26	105
CW	300	103-BX

## F. IWW Flows

6.4 - Ave.  $\text{HNO}_3$  Conc. 6 M

## G. Tank Farm Boil-Off

<u>Waste Tank</u>	<u>Boil-Off Rate, Gal/Min</u>	<u>Temperature</u>
101-A	2.5	165°
102-A	2.2	137°
103-A	.4	95°
104-A	2.5	140°
105-A	-	96°
106-A	14.4	125°

## III. Flowsheet - G. A. Nicholson

- On March 11, the HA pulse frequency was increased from 74 to 79 cycles per minute. It was reduced to 76 cycles per minute on March 12 to stabilize the column. It was further reduced to 64 cycles per minute on March 14 to dissipate another flood and increased back to 70 cycles per minute from March 15 to March 17.
- On March 12, the  $\text{HNO}_3$  addition to the 2DF Tank was changed from  $\text{UO}_3$  Plant recovered acid to fresh acid.
- On March 12, the HSR- $\text{HNO}_3$  was increased by about twenty per cent. It was further increased to 17.4 flows between March 13 and March 17.
- On March 12, the  $\text{KMnO}_4$  addition to the IOF Wash Tank was started at 1.6 flows.
- Commencing on March 13, hydrolyzed high-uranium waste solution was added to the HAF from the spare concentrator (E-F11). The first batch was about 80 gallons. Rework was deleted from the following batch, then added to each succeeding batch at a rate of about 150 gallons per batch.

- (f) On March 15, the 2DF-HNO<sub>3</sub> was increased from 0.45 to 0.55 flows.
- (g) On March 17, the HSS-H<sub>2</sub>O Control System was malfunctioning. After replacement of some components and repair of others, the HSS-H<sub>2</sub>O flow was returned to normal.

#### IV. Feed Preparation - G. J. Raab

Dissolver operations were normal except for A3 Dissolver which still has limited vacuum due to high d/p across the silver reactor. I<sub>131</sub> emission was 0.39 curies for the week with metal cooling times of 122 to 167 days. Since the high I<sub>131</sub> emission numbers reported late last week were not confirmed, mercuric nitrate addition to metal solution was stopped on March 12. The coating waste jet system performed normally.

#### V. Solvent Extraction - G. A. Nicholson

Process performance was considerably improved over the previous report period. The plutonium losses via the HAW Stream increased to a maximum of about 0.5 per cent early in the report period, but had decreased by three-fold by the end of the period. The activity of both uranium and plutonium product streams showed a general decrease during the report period. Investigation of the plutonium loss problem is continuing. In addition to the sampling and/or analysis difficulties mentioned in the previous report, factors which are suspected to be contributing to the problem of high HAW losses are listed below:

1. Abnormal nitric acid concentration in the HA Column. A check of influent stream flow rates and analyses indicates excessive acid, but HAW analyses have been generally about 20 per cent low and variable.
2. HA Column instability.
3. Introduction of "do-bad" material into the Backcycle Waste System via the 3WF Tank.
4. Rework of hydrolyzed material from the spare concentrator. While this has not caused any apparent problems, the general unsettled condition of the HA Column could be masking more subtle effects.
5. The problem which permitted gassing in the 3AS System to upset the HA and HS Columns might be affecting HSR-HNO<sub>3</sub> and HSS Stream flows without any indication on the flow recorders.

Concurrent with the appearance of the HAW loss problem, the ruthenium activity in the IBP Stream increased for no apparent reason to a peak about three-fold above normal, but has been on a downward trend since the middle of the report period.

Performance of the Final Plutonium Cycle continued at essentially the same level as the previous report period. The 2AW recycle has increased from 1.5 to about 4 per cent early in the period and remained at that level. An upward trend in the activity of both the IBF and 2BP Streams

appeared at the end of the report period, with a concurrent slight decrease in the DF across the cycle. Plutonium product activity was somewhat above normal, but remained within blending limits.

Performance of the Final Uranium Cycle was excellent, with typical DF's between 1500 and 2000. Ruthenium contamination in the uranium product (two-fold higher than normal) necessitated silica gel treatment during the first few days of the report period, but both ruthenium and zirconium levels had decreased to shipment limits by the middle of the week. The plutonium and neptunium contamination in the uranium product remained near the analytic detection limits until near the end of the report period, when an upward trend was noted. The temporary excursion of plutonium contamination appeared to be caused partly by the increased acidity in the 2D Column and partly by an increase in plutonium contamination in the IBU Stream.

Neptunium accumulation the Solvent Extraction System continued at about the same rate as during the previous report period. Accumulation in the Back-cycle Waste System appeared to be at a higher-than-normal rate, but losses via the HAW Stream averaged three-fold higher than during the previous report period. It is suspected that a slight leak into the J-Cell Package equipment has filled the 3AF Tank and caused it to overflow to the 3WB Tank, thus "bleeding" a considerable quantity of neptunium back into the Backcycle Waste System.

## VI. Product Treatment

### A. Plutonium Purification (N-Cell) - R. W. Lambert

Operation of the plutonium ion exchange unit, N-Cell, continued to be satisfactory up until March 13, when indications of sluggish resin movement began to appear. By March 15, push pressures had been raised to 50 psig and the decision was made to change out the resin. Twenty gallons of resin were changed out without interrupting the normal N-Cell operation. The resin changed out had been in use 37 days. Operating conditions following the changeout were not vastly different as the unit was still operated at 43 psig push pressure.

### B. Final Uranium Treatment - R. W. Lambert

On March 12, following regeneration, the Silica Gel Unit was started up. In spite of abnormally high ruthenium activity in the feed, the unit operated the remainder of the run period producing in-specification uranium product.

### C. Neptunium Purification - S. M. Nielson

The resin was removed from the T-Q4 Ion Exchange Column to the Q8 Sump Tank on March 13. A swirling water mixer was added to Tank Q8 and removal of the resin was successfully demonstrated.

Maintenance of Q5 Waste Tank and other items continued during the report period.



VII. Solvent Treatment - G. J. Raab

Solvent activity was  $1.74 \times 10^3$  and  $2.89 \times 10^3$  uc/gal ZrNb and Ru, respectively at the beginning of the week. Potassium permanganate flow was started to IOF Wash Tank on March 12. The solvent activity did not respond to the treatment rapidly as solvent activity was still in the range of  $1.0 \times 10^3$  and  $2.60 \times 10^3$  uc/gal ZrNb and Ru, respectively at the end of the week. The net solvent loss from the system was 1640 gallons (6%). The loss from the No. 2 System to the No. 1 System was in balance with the loss from the No. 1 System to UGS. <sup>0.12%</sup>

VIII. Waste Treatment and Storage - R. C. ForsmanA. Denitration

The formaldehyde denitration prototype unit was operated with only minor interruptions from March 11 until failure of the reactor thermohm necessitated shutdown on March 17. Erratic IWW flow was overcome by increasing the flow rate from 0.8 gal/min to ~ 1 gal/min and reducing the preheater temperature from 100° C to 96° C. Due to inability of sampling the formaldehyde treated waste in the receiving tank (TK-F15), samples taken in Tank F16 include mixtures of untreated IWW and variable treated IWW batches; hence, efficiency calculations could not be made. Best estimates indicate the IWW was reduced from ~ 6 M HNO<sub>3</sub> to 3 M HNO<sub>3</sub> by formaldehyde treatment for an efficiency of 2.2 moles nitric destroyed per mole formaldehyde added. Antifoam rate of about 70 ppm was sufficient to control foaming.

B. A-Tank Farm

Calculations of the sodium molarity (based upon plant records) indicate the recent temperature excursion in Tank 104 started when the liquid level was allowed to decrease until the sodium concentration reached 8.5 M. Maximum temperatures were attained at about 9.0 M Na<sup>+</sup>, then returned to normal after dilution to about 7.0 M Na<sup>+</sup>.

It appears that faulty thermohms in Tank 106 falsely indicated a temperature excursion in the vessel sludge. Subsequent temperature measurements taken with newly calibrated bulbs and thermocouples indicated temperatures in the 120-140° C range, rather than >220° C. In addition sodium calculations indicate the maximum concentration attained was about 7.2 M Na<sup>+</sup> which is well below the concentration that normally produces high sludge temperatures.

IX. Fission Product RecoveryA. Strontium Recovery - W. C. Schmidt

Run series 58 is in progress. With the use of the prototype high temperature jet, the solution in the sulfate precipitation step is being transferred to the centrifuge at 75° C and the resulting solution temperature in the centrifuge is ~ 95° C. The waste losses have

DECLASSIFIED

HW-76912

-38-

decreased a factor of two for both strontium and cerium (from ~ 20% to ~ 10%). Promethium analyses have been requested and will be forthcoming for further evaluation of centrifugation at the elevated temperatures.

B. Strontium Semiworks - J. B. Kendall

The fourth CFD strontium purification run was started on March 13. Startup was very smooth and initial process performance was excellent, both with respect to waste losses and fission product decontamination. Blending of strontium rework with the HAF was started on March 15 with no significant effect on the process. The strontium being reworked (approximately 100 kilocuries) had been recovered by batch contact from miscellaneous wastes generated during SSW run number 3.

This document consists of No. 4 of 10 copies. Series A.

- Distribution:
- 1 - CF Beaulieu
  - 2 - WS Frank
  - 3 - CF Hill
  - 4 - EF Judson
  - 5 - RW McCullugh
  - 6 - PR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 3/18/63 TO 2400 ON 3/24/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.6 (actual 3.45) CF during the report period.

II. Performance Data - D. G. Leyson

A. Solvent-Extraction Performance by Cycles (Typical)

	Gamma dF		% Recycle		% Loss			Np Acc. %
	U	Pu	U	Pu	U	Pu	Np	
First	3.7	3.3			.002	.09	3.0	173%
Final	3.4	3.1	7.0	4.4	.002		8.0	-138%
Ion Ex.		1.5		0.06				
Overall	7.1	7.9	7.0	4.46	.004	.09	11.0	35%

B. Production

- (1) EAF Processed = 194 Tons
- (2) U Prod. (K6 to P Tanks) = 186.8 T
- (3) Pu Prod. (L10 Loadouts) = 126,310
- (4) Np Prod. = -0-

C. Overall Loss

	Units	% of Total Prod.
U:	612 Lbs	0.21
Pu:	481 Grams	0.38
Np: F16, 18	153 Grams	36.0
K6	18 "	4.2
Solvent:	550 Gals.	0.04

(Based on virgin input)

# DECLASSIFIED

EW-76912

-40-

D. Rework

	<u>Units</u>	<u>% of Total Prod.</u>
U:	2.1 Tons (F11 to D5)	1.1
Fu:	12,803 Grams	10.1

E. Volume to UGS

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
JWW	212	101-A
Cell Dr.	21	105-A
FP	95	105-A
IWW	71	105-A
CW	300	103-BX

F. IWW Flows

6.5                  Ave. HNO<sub>3</sub> Conc. - 5.0 M

G. Waste Tank Boil-Off, Gal/Min

<u>Waste Tank</u>	<u>Boil-Off Rate, Gal/Min</u>	<u>Temperature °C</u>
101-A	2.5	160
102-A	2.2	120
103-A	.4	94
104-A	2.5	139
105-A	-	96
106-A	14.4	124

III. Flowsheet - G. A. Nicholson

- (a) On March 18, the EA Column pulse frequency was reduced from 72 to 67 cpm to dissipate a flood. It was further reduced to 64 cycles per minute on March 21 to dissipate a second mild flood and to 60 cycles per minute on March 23. The frequency was increased to 62 cycles per minute on March 24.
- (b) On March 19, the ESR-HNO<sub>3</sub> was reduced from 17.4 to 10.9 flows.
- (c) On March 19, rework of the hydrolyzed solution in the spare concentrator was terminated. Rework of acid flush solution from the concentrator commenced on March 22 and continued through March 24.
- (d) On March 19, the 2DF-HNO<sub>3</sub> was reduced from 8.5 to 7.9 flows. It was further reduced to 7.2 flows on March 20 and to 6.0 flows on March 24.
- (e) On March 23, the ICK-H<sub>2</sub>O was reduced from 436 to 423 flows. It was further reduced to 410 flows on March 24.
- (f) On March 23, the Neptunium Recovery Cycle was started up at a 2.0 CF rate. The 3WB-EA flow rate was adjusted accordingly.

IV. Feed Preparation - G. J. Raab

Dissolver operation was normal during the week except for the A3 Dissolver.  $^{231}\text{Pu}$  emission totalled 0.91 curies, with metal cooling times of 118 to 167 days. The maximum amount of  $^{231}\text{Pu}$  charged was 115 curies/day.

V. Solvent Extraction - G. A. Nicholson

Process performance during the report period was marked by a continuing improvement throughout the plant. Performance of the First Cycle had returned to near normal (EAW losses averaging less than 0.05 per cent and HSF activity at normal levels) operation by the end of the report period. The unusual ruthenium burst through the Final Uranium Cycle dissipated early in the week and the level has remained near normal since. Evaluation of the process and analytical data concerning EA Column performance indicates that the following factors were of primary effect:

1. Excess acidity in the EA Scrub Section which decreased the scrubbing efficiency and, in an effort to control the activity of the ESP Stream, the saturation was increased to the point that HAW losses were excessive. The HAW- $\text{HNO}_3$  analyses, apparently invalid, were generally about 25 per cent low. In an effort to increase the HAW- $\text{HNO}_3$  to the flowsheet value of 2.75 molar, the HSR- $\text{HNO}_3$  flow was increased by about 60 per cent. After the HSR- $\text{HNO}_3$  flow was returned to normal and the saturation in the column adjusted, the losses commenced to decrease.
2. Instability in the HA Scrub Section. When the scrub section acidity was returned to normal, the scrub section stability decreased markedly, to wit - it was necessary to reduce the frequency by 10 cycles per minute to maintain stable operation. The maximum stable frequency is presently 20 cycles per minute lower than during previous run periods. The reason is probably fouling of the nozzle plates by crud accumulations. Activities which are planned for the shutdown should eliminate the problem.

Performance of the Final Plutonium Cycle has been below normal for the past week. About the time the performance of the First Cycle began to improve, the performance of the Final Plutonium Cycle began to decline. The EF is still above levels typical of a few months ago, but nearly two-fold below the previous report period. The decrease in EF does not appear to correlate with either organic saturation or acidity.

Performance of the Final Uranium Cycle continued at the same high level as during the previous report period. A slight upward trend in Zr-EB activity of the final product occurred, but was compensated for by the decrease in ruthenium activity and all product met shipment limits without silica gel treatment. The upward trend in plutonium and neptunium contamination in the uranium product, noted in the previous report, appeared to coincide with the increased acidity of the 2LE Stream.

Neptunium accumulation in the Solvent Extraction System was normal during the report period. An increase in the accumulation rate in the Backcycle Waste System was due to a continual "bleeding" from the 3-Bell Package after it was shut down. Apparently, the 3AX valve leaked through, causing the

3A Column to overflow to the 3B Column. The 3BNR valve also leaks through to the 3AF Tank which overflows to the 3WB Tank. The Neptunium Recovery Cycle was started up on March 23 but, because of the problem discussed above, the system was not yet at equilibrium by the end of the report period and significant accumulation of neptunium had not commenced.

## VI. Product Treatment

### A. Plutonium Purification (N-Cell) - R. W. Lambert

Operation of the Ion Exchange Unit, N-Cell, was satisfactory during the report period with decontamination performance improving noticeably over the previous several weeks. The zirconium-niobium decontamination factor for the report period ranged between 25 and 35.

The resin movement characteristics of the unit continued to be marginal with push pressures averaging near 45 psig. A study of resin samples by Separations Chemistry Laboratory indicated that 50 per cent of the used resin was left in the unit following the resin change made on March 16. A review of the last change-out procedure also confirmed that the change was not done in the most efficient manner.

### B. Final Uranium Treatment - R. W. Lambert

The Silica Gel Unit continued to operate until March 22 when the unit was shut down for lack of feed and for regeneration. Approximately 250 tons of uranium was processed during the ten days the unit operated.

### C. Neptunium Purification - S. M. Nielson

Maintenance work and repair of Tanks Q5 and Q8 were completed during the report period. Minor maintenance work such as painting, cleaning, installing lucite panels in hot cell, and installing viewing windows and glove ports in the maintenance hood continued throughout the report period.

On March 20, 21 and 22, preparations were made and work started on tying in the hot process lines to Q-Cell.

## VII. Solvent Treatment - G. J. Raab

Solvent activity has not responded significantly to restoring potassium permanganate flow to the ICF Wash Tank. The Turbomix Tank aqueous phase (one per cent caustic) was changed out on March 21. The activity of the solution had reached  $2.9 \times 10^4$  and  $1.5 \times 10^4$  cc/gal Zr-Nb and Ra, respectively and may have been recontaminating the solvent. The solvent loss for the week was 0.04 per cent of the solvent used in the plant (550 gallons net loss). The systems were out of balance by 360 gallons (the No. 2 System lost 360 more gallons to the No. 1 System than the No. 1 System lost to TGS).

VIII. Acid Recovery and Waste Concentration - R. W. LambertF-Cell Acid Recovery

On March 19, the  $\text{NaNO}_2$  flow to Tank F12 was increased 30 per cent which increased the nitrite in the IWF to 0.013 M. The ruthenium activity in the AAA acid immediately decreased by a factor of 2 and remained at about  $5 \times 10^3$  uc/gal until the end of the report period. The zirconium-niobium activity remained essentially unchanged. The IWF overflow rate was held constant during the test.

IX. Waste Treatment and Storage - R. C. ForsmanDenitration

After replacing the thermohm in the formaldehyde reactor (TK-F22), the unit was placed in service on March 19. Operation of the equipment was excellent; however, sampling difficulties and delays precluded accurate efficiency calculations. The acid in the treated IWW remained high at  $\sim 1.8$  M vs. desired 0.5 M. Approximately 100 parts antifoam per million parts IWW were added to control foaming in the reactor. Gamma activity in the recovered acid was generally a factor 100 greater than the normal plant recovered acid.

X. Fission Product RecoveryA. Strontium Recovery - W. C. Schmidt

Run series 58 continued during the week. Only three sulfate precipitations were made due to delays encountered in removing the wastes to underground storage via either F-16 or F-18. The sulfate waste analyses still indicate the strontium and cerium waste losses to be  $\sim 10$  per cent with the feed tank temperature held at  $75^\circ \text{C}$  during centrifugation.

B. Strontium Semiworks - J. B. Kendall

Between March 13 and 20, about one megacurie of strontium-90 was purified at the Strontium Semiworks. About ninety per cent of the feed was vault crude concentrate and the remainder was Tank 76 rework. No processing or equipment problems were encountered except with Tank 6, the HCP Concentrator. The product was well within the cerium specification, but other product analyses have not been completed.

The HAW and HCW losses were only 1.2 and 1.3 per cent, respectively, but a product loss of about 15 per cent resulted from foaming during treatment of the product with nitric acid. The latter material was set aside for rework.

Tank 6, the HCP Concentrator, became inoperative after about six days either because of plugging in the concentrator tower and/or foaming in the concentrator pot. The product was later treated in Tank 1 and Tank 6 with nitric acid and hydrogen peroxide. No difficulties were encountered during the treatment with hydrogen peroxide, but the preliminary treatment

with nitric acid in all cases was characterized by a sudden and unpredictable release of gases accompanied by a severe foam. In virtually every respect, this behavior was identical to the behavior of HLO product in 1961.

The cause or causes of the unusual characteristics of the strontium product apparently are related to insufficient nitric acid in the concentrator solution which in turn was caused by a change in the citrate destruction technique with hydrogen peroxide. In this run, as in previous runs, the HCP Concentrator solution was 3.0 M  $\text{HNO}_3$  at start-up. In the past, the hydrogen peroxide flow was started and maintained at five flow mols per flow mol of citrate when the HCP Concentrator solution reached 1.0 M citrate. In this run, the hydrogen peroxide flow, however, was not started until the HCP Concentrator solution reached 2.0 M citrate and was maintained at only 2.5 flow moles per flow mol of citrate. It is believed this processing technique allowed a much greater fraction of the citrate to be destroyed by radiolysis and thus in turn destroyed the nitric acid by reaction with radiolysis products, presumably carbonates. This hypothesis is supported by the apparently low acid and citrate concentrations of the product at shutdown. The prognosis of low acid and citrate concentrations are based on (a) the violent gassing and reaction of the product solution with nitric acid, and (b) the quantity of hydrogen peroxide required after shut down before the solution  $\text{SpG}$  stopped falling was only 1.0 mole per mole of the total citrate added.

In addition to the changes in the hydrogen peroxide treatment, three other process variables were evaluated. They were (a) the effect of rework, (b) the effect of reduced "free" DEPA concentration, and (c) the effect of modifying the organic treatment technique. The results of these tests were as follows:

- (a) No changes in process performance was noted from the addition of rework to the HAF Stream.
- (b) Reducing the "free" DEPA in the extraction section from 0.065 to 0.050 increased the quantities of cerium in the HAF, HCP, and HCW Streams. However, since the HAW pH decreased from 4.75 to 4.65 and the HA Column temperature varied from 32 to 42° C, the results of the test are not considered conclusive.
- (c) Efforts to reduce the quantity of aqueous periodically added via the HAX Stream was only partially successful. Apparently some batches of organic were not adequately settled and decanted before use.



# DECLASSIFIED

HW-76912

This document consists of No. 4 of 10 copies. Series A.

-45-

Distribution:

- 1 - OF Beaulieu
- 2 - WS Frank
- 3 - OF Hill
- 4 - EF Judson
- 5 - RW McCullugh
- 6 - PR McMurray
- 7 - SG Smolen
- 8 - RE Tomlinson
- 9 - AJ Waligura
- 10 - MT Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 3/25/63 TO 2400 ON 4/1/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.6 (actual 3.6) CF during the report period. Commencing on March 29, "cold" uranium was recycled from Tank K6 to the HAF in preparation for the scheduled shutdown. A total of about 3500 gallons of F8 Rework solution was processed through the Solvent-Extraction System between March 29 and April 1. Shutdown of the Solvent Extraction System commenced on April 1 with the HAF off at 1145 and was completed with the Final Uranium Cycle and the Second Solvent Treatment System down about 1200 on April 2.

II. Performance Data - D. C. Leyson

A. Solvent-Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np. Acc. %</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.5	3.3			.002	.04	3.5	-269%
Final	3.2	3.1	6.5	4.05	.002		4.0	+336%
Ion Ex.		1.5		0.2				
Overall	6.7	7.9	6.5	4.25	.004	.04	7.5	67%

B. Production

- (1) HAF (Virgin) = 133 T
- (2) U Production = 136 T
- (3) Pu Production = 138 Kg (Inc. High Can Rework)
- (4) Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Prod.</u>
U:	580 Lbs	0.2
Pu:	327 Grams	0.2
Np (F16, F18):	94 Grams	32.0 (Based on virgin
(K6)	6 Grams	2.0 input)
Solvent:	2742 Gals.	0.17 (Gain)*

\*Column empty-out for shutdown.

**D. Rework**

	<u>Units</u>	<u>% of Total Prod.</u>
U: (K6 to E6)	60 T	44
(F8 to D5)	6 T	4.4
Pu: (High Cans L11 to E6)	41,673 Grams	30.2
(F8 to D5)	2,322 Grams	1.7

**E. Volume to UGS**

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	201	102-A
	117	101-A
Cell Dr.	93	105-A
FP	121	105-A
IWW	-0-	
CW	300	103-EX

**F. IWW Flows**

6.5                      Ave. HNO<sub>3</sub> Conc. - 6.2 M

**G. Waste Tank Boil-Off, Gal/Min**

<u>Waste Tank</u>	<u>Boil-Off Rate</u>	<u>Temperature °C</u>
101	1.7	147° (Fulb)
102	2.2	92° "
103	0.3	94° "
104	3.6	139° "
105	1.5	96° "
106	<u>14.7</u>	124° "
	24.0	

**III. Flowsheet**

- (a) On March 26 and 27, the ICX was increased from 410 to 430 flows.
- (b) On March 26, the nitric acid addition to the 3WB was started at 6.4 flows. It was stopped on March 27.
- (c) On March 26, the processing rate in the "C-Cell Package" was increased from 2.0 to 3.6.
- (d) On March 27, the HA pulse frequency was reduced from 62 to 60 cycles per minute. It was further reduced to 58 cycles per minute on March 28.
- (e) On March 29, rework of the hydrolyzed waste solution in EK-F8 was commenced at a rate of 500 gallons in each batch of "cold" uranium recycled from TK-K6. The rework was terminated on March 31.

-47-

- (f) On March 30, the 2DW uranium loss was decreased from about 6 per cent to less than 0.1 per cent.
- (g) Commencing on March 30, the HNO<sub>3</sub> concentration in the 3WB Stream was reduced from ~ 8 to ~ 4.5 molar. It was increased back to 8 molar on April 1.
- (h) On March 30, the transition from Phase I (accumulation) to Phase II (decontamination) was made in the J-Cell Package.
- (i) On March 30, the J-Cell Package rates were reduced by 50 per cent.
- (j) On March 31, the 2DF-ferrous sulfamate was increased from 0.21 to 0.33 flows.

#### IV. Feed Preparation - G. J. Raab

Dissolver charging for the run period was completed on March 27. The metal heels were removed by boiling 400 gallons of 50% nitric acid in the dissolvers for 4 - 6 hours after the last heel cut indicated the metal was removed. The acid indicated insignificant removal of metal (by lack of specific gravity increase). The flush of A2 Silver Reactor with sodium thiosulfate was started on March 31. The I<sub>131</sub> emission was 0.46 curies for the period with metal cooling times of 116 to 175 days. The coating waste jet appeared to be plugging at the end of the run period. A hot caustic flush was jetted through the line.

#### V. Solvent Extraction - G. A. Nicholson

Process performance was generally good during the report period. The plutonium losses via the First Cycle Waste (HAW Stream) averaged less than 0.04 per cent until March 29, when a temporary over-saturation during the transition to cold feed resulted in high losses (>0.5 per cent) for about sixteen hours. Activity of the HSP Stream was generally excellent, except during the periods of instability in the HA Column. As a result of the short, mild bursts, the activity of the First Cycle product streams (especially the ICI) showed a decided upward trend. The instability in the HA Column appeared to be caused by an emulsifier which was slowly progressing down the column. The previous floods have occurred in the scrub section, while the two instabilities the past week were in the extraction section. Also, perhaps coincidentally, the stability of the HA Column appeared to decrease when the rates in the J-Cell Package were increased, indicating a probable continuing cross-contamination problem.

Performance of the Final Plutonium Cycle has remained at essentially the same level as during the previous report period. Losses to the Backcycle Waste System were fairly steady at about four per cent during the report period.

Performance of the Final Uranium Cycle was slightly lower than reported for the previous week. An increasing trend in plutonium contamination coincided with a decreasing fission product decontamination performance. The plutonium contamination in the final product "peaked" at about six ppb and the activity increased to about 70 per cent above shipping limits at shutdown - well within silica gel treatment limits. Analysis of the general performance of the

Solvent-Extraction System indicates the entire system has become "crudded" up and is in need of extensive flushing.

Neptunium in the Solvent-Extraction System was normal during the report period, with losses averaging two and four per cent, respectively, via the HAW and ZEC Streams. Accumulation in the Continuous Recovery System was excellent, once the uranium to nitric acid ratio was reduced to flowsheet value (by adding HNO<sub>3</sub> to the 3WB Tank) and the rates were increased to 3.6 CF. The "Package" was shut down (with approximately 1500 units isolated) at equilibrium pending hot startup of the Neptunium Purification Cycle.

## VI. Product Treatment

### A. Plutonium Purification - N-Cell - R. W. Lambert

The mechanical operation of N-Cell was satisfactory during the report period although push pressures of about 50 psig were required to obtain resin movement. During the first seven days of the period, the inventory of Purex out-of-specification plutonium was substantially reduced by reworking large amounts of plutonium from EK-L11 to the feed make-up tank, TK-E6.

On March 27, a short gamma burst reached N-Cell via the ZBF Stream. The activity in the three loadout batches following the burst exceeded product specifications. On April 1, normal processing of plutonium in N-Cell was completed and stripping of the unit was begun in preparation for shutdown and making a resin change.

### B. Neptunium Purification - S. M. Nielson

The hot process line tie-ins to Q-Cell were completed on March 25. Minor maintenance continued throughout the report period. Most work was associated with sealing of the lucite panels in the Hot Cell.

## VII. Solvent Treatment - G. J. Raab

The No. 1 Solvent System activity was  $1.6 \times 10^3$  and  $3.1 \times 10^3$   $\mu\text{c/gal}$  Zr-Nb and Ru, respectively at the beginning of the week and continued at about this level until cold feed was started. The activity in the No. 1 System had dropped to  $1.1 \times 10^2$  and  $6.17 \times 10^2$   $\mu\text{c/gal}$  Zr-Nb and Ru just before shutdown. The No. 2 Solvent System had a plutonium retention value of  $8.94 \times 10^6$  on March 28. The solvent loss was well balanced in the systems. The loss represented 0.13% (1690 gal.) of the volume circulated in the two systems for the period.

## VIII. Acid Recovery and Waste Concentration - R. W. Lambert

### F-Cell Acid Recovery

Failure of the DOW in the IWF jumper on March 24 caused an upset in the acid recovery system which increased the AAA acid ruthenium level by a factor of two to about  $1.2 \times 10^4$   $\mu\text{c/gal}$ . By March 28, the system had recovered somewhat and the ruthenium activity fell to about  $7 \times 10^3$   $\mu\text{c/gal}$ . During the upset, the zirconium-niobium activity remained relatively stable at

$1 \times 10^4$  uc/gal. The nitrite in the IWW remained at 0.013 M during the report period.

Results of the special sampling program conducted during the February-March run period indicate that the Sr-90 concentration in the Purex process condensate is about  $1.5 \times 10^{-7}$  uc/ml. This is approximately one-fifth the 40 Hr. Occupational MPC limit. The Ce-137 content appears to be slightly less than the MPC limit of  $1 \times 10^{-4}$  uc/ml.

IX. Waste Treatment and Storage - R. C. Forsman

A. Denitration

Operation of the formaldehyde unit essentially duplicated pilot plant experience producing an efficiency of 2.5 moles of acid destroyed per mole of formaldehyde added to the IWW. Approximately 100 ppm of anti-foam were added to the IWW which was preheated to 98° C before entering the reaction vessel at 6.5 flows where the temperature was maintained at 94 - 96° C. Continuous formaldehyde addition reduced the free acid concentration of the IWW from 6.1 M to 0.66 M. The treated waste was maintained at 70° C to insure complete destruction of the formaldehyde. Although the activity in the recovered acid had decreased by a factor of 10, the Zr and Ru were still approximately a factor of 10 above normal plant recovered acid.

A severe leak on the IWW pump necessitated shutting down the unit on March 29.

B. A-Farm

To determine the reliability of the temperature bulbs used for measuring underground storage tank temperatures, one bulb was subjected to  $3 \times 10^6$  r/hr from a Cobalt-60 source for 5 days with no deleterious effects. Normal tank radiation readings have shown activity as high as  $5 \times 10^4$  r/hr.

X. Fission Product Recovery - W. C. Schmidt

Strontium Recovery

Run series 58 was completed recovering approximately 200 kilocuries of strontium-90. Centrifugation of the sulfate precipitation at 75° C has reduced the losses to ~ 10 and ~ 25 per cent for strontium and promethium, respectively.

Run series 59 was initiated. Considerable difficulty was encountered in removing the pre-sulfate precipitation solids from the centrifuge bowl. The IWW used for these runs was formaldehyde treated and the acidity was less than 0.5 lbs/gallon. The cell Beckmans indicate there is a large increase in the amount of fission products precipitated in this low acid IWW.

XI. Fission Product Purification - J. B. Kendall

Between March 22 and 24, the strontium lost by foaming in the strontium product was reprocessed through the solvent extraction batteries. The run was conducted without incident until the product volume after citrate destruction was reduced. At this time, the boil-up rate of the concentrator was allowed to greatly exceed the capacity of the concentrator tower, and this in turn caused a 50 per cent product loss to contaminated vessels. Although analytical results of the accumulated rework from two different tank samples showed between 165 and 200 kc of Sr-90 in the feed, only 110 kc of Sr-90 could be accounted for at the completion of the run. Since the HCW-TK-21, HAW-TK-69, and HCP-TK-6 material balance agreed with each other, it appears that only 110 kc of Sr-90 were actually processed. On this basis, the HAW and HCW losses were 1.0 and 2.5 per cent, respectively. This HCW loss was combined with the HCP concentrator loss for reprocessing.

This document consists of No. 4 of 10 copies. Series A.

Distribution:

- 1 - DE Beallien
- 2 - WS Frank
- 3 - DP Hill
- 4 - RF Johnson
- 5 - RW McCullough
- 6 - PR McMurray
- 7 - SG Smolen
- 8 - RE Tomlinson
- 9 - AD Waligura
- 10 - ME Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 4/2/63 TO 2400 ON 4/24/63

I. General - G. A. Nicholson

The plant was shut down during the report period except for a two-day uranium rework operation in the Final Uranium Cycle and intermittent operation of the Neptunium Recovery System.

II. Performance Data - B. G. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

(None - Shutdown)

B. Production

- (1) UAF Processed = ---
- (2) U Production = 31.6 E
- (3) Pu Production = 8.4 Kg
- (4) Np Production = ---

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
(1) U	702	
(2) Pu	647	
(3) Np, F16, F18 K6	80 15	
(4) Solvent	5761 gal.	

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
(1) U	1391 #	
(2) Pu (In E6 for Startup)	7.3 Kg.	

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gallons</u>	<u>Waste Tank</u>
OWW	101,318	102-A
Cell Dr.	45,288	105-A
FP	32,082	105-A
IWW	-0-	
CW	-0-	

F. IWW Flows  
(None)

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	2.2	148
102-A	1.2	94
103-A	.3	92
104-A	1.3	138
105-A	4.8	92
106-A	9.3	123

III. Feed Preparation - G. J. Raab

The first sodium thiosulfate flush of the A2 reactor appeared to be incomplete by crane observation. The flush was repeated and considerable silver was removed on the second flush as the solution was very black. The A3 dissolver system failed to hold vacuum when reassembled. A number of remote flanges were gasketed and the dissolver air bleed jumper was replaced. The A3 dissolver system still has only marginal vacuum and the in-cell filter indicated higher than normal D/P, an indication of air leakage into the system. The press of other crane work and startup have curtailed other work on the system.

The dissolver and TK-26 nitric acid addition meters were calibrated with water.

When dissolver operation was started the coating waste did not jet well to UGS. A caustic flush has apparently freed the line.

IV. Solvent Extraction - G. A. Nicholson

Rework of about 15 tons of uranium, recovered from the 10 Column flood (Page of this document) and UO<sub>3</sub> Plant sump wastes, was completed successfully during the shutdown period. Instantaneous losses via the 2EW and 2EW streams appeared to be negligible. However, analysis of the concentrated waste accumulated from the operation indicated an average loss of about two per cent. Consequently, the waste was held for hydrolysis and eventual rework. The solvent used during the rework operation was the same as that used during the January rework operation. The uranium produced during the rework operation was within silica gel treatment limits and below plutonium contamination limits.



Following the rework operation, all solvent-extraction columns, with the exception of the IBX, IBS, 3A, and 3B Columns, were given caustic-tartaric and nitric-oxalic decontamination flushes. The 2D and 2E Columns were flushed with hot carbonate and hot nitric solutions following the decontamination flushes. Also, following the decontamination flushes, the ICU Concentrator was given a hydrazine flush in preparation for the special hydrazine test planned for shortly after startup.

On April 24, preparations commenced for startup of the Solvent-Extraction System.

The Neptunium Recovery Package was started up under Phase II conditions on April 15. After the system had "steadied out", transition to Phase III was made and the 3BN was diverted to the Neptunium Purification Cycle. However, only about 35 per cent of the neptunium which was in the system (C Cell Package) at shutdown appeared in Q Cell. The remainder ended up distributed among the Backcycle Waste System (3WB), the 3BW (in the IBXF Tank), and J-Cell Package. The Neptunium Recovery Package was started up again under Phase I conditions immediately before startup of the plant and all of the Backcycle Waste was processed to remove the neptunium and, thus, eliminate the possibility of excessive losses via the HAW Stream at startup. This time, due to the excessive acidity in the system, the bulk of the neptunium remained with the organic phase (3BW Stream).

V. Product Treatment - R. W. Lambert

A. Plutonium Purification - N-Cell

Following the plant shutdown on April 1, the N-Cell resin was given a final strip and removed from the columns. Extensive flushing of the columns was done to insure a complete removal of the old resin.

Maintenance items completed during the shutdown include:

1. Installation of new one-inch line from discharge of XAW jet to pipe chase. This completed new routing from XAW jet to the trench-side of L-Cell.
2. Removed old absorptometer box from hood.
3. Rerouted XSW line to XAW sample pot.
4. Repaired water adds to XAF pump heads.
5. Checked feed screens and top-hat screens.
6. Flushed TK-N1 of resin residuals and extensively flushed TK-N6, TK-N7 and TK-L10.
7. Completed normal checks and replacements of air cylinders.

The screen check revealed the XAF feed screen to be ruptured with resin tightly packed on the wrong side of the screen. This screen failure

probably occurred at the very beginning of the February run period and was responsible for the intermittent XAF pumping problems encountered during the run.

During the April shutdown, 42 cans (~ 19 Kg) of Z-Plant ion exchange product were concentrated in N-Cell. The rework line from EK-L9 to EK-N1 was modified such that EK-L9 was pumped directly to the N6 Concentrator. In spite of previous flushing of EK-L9, enough residual activity was present to make the concentrated product slightly out-of-specifications.

Following maintenance work, a new batch of Permutit SR 20-40 mesh resin was added to the unit and it was run-in pending startup of normal processing.

#### VI. Solvent Treatment - G. J. Raab

The degraded solvent in EK-R5 was used while processing the uranium rework from EK-P2. After the rework operation, the R5 solvent was spun through the No. 2 Solvent System for three days with no apparent improvement in its quality, so this complete rework solvent inventory was pumped into EK-R5 for future use during rework or disposal. Separations Chemistry samples indicate little hope of this solvent being recoverable as plant solvent.

The solvent recovered from EK-K1, which had been stored in EK-R8 since plant startup in February, was blended back into the regular No. 2 Solvent inventory and butted with diluent. A strong reducing agent associated with this solvent apparently killed all the permanganate in the solvent wash tank and resultant  $MnO_2$  tended to autocatalyze further reduction and generally plugged up the system, including the tanks in the G-Cell which receive R-Cell aqueous waste. An oxalic acid flush of EK-R1, EK-R2, ER-2, EK-G1, E-G2 and EK-G2 removed the  $MnO_2$ . In order to expedite startup, a portion of the No. 2 Solvent inventory was jettied to EK-R8 and EK-R7 was butted with fresh solvent. The resulting inventory after spinning in R-Cell proved acceptable for startup. The solvent in EK-R8 should be acceptable when blended into the inventory in small batches.

#### VII. Acid Recovery and Waste Concentration - R. W. Lambert

##### A. Titanium Tube Bundle

On April 11, the titanium tube bundle was pulled from EK-4, the Backcycle Concentrator, and inspected from the canyon greenhouse. The bundle was heavily crudded, with the area between the tubes at the bottom end of the bundle completely packed with solids. The shroud was also coated with a heavy layer of crud. The tubes at the upper end of the bundle were clear. The shroud prevented seeing how high the crud extended up into the bundle.

On April 15, a flake sample of the solid crud was obtained and sent to the Separations Chemistry Laboratory. It was found to be essentially insoluble in everything but 30 per cent hydrofluoric acid. While a complete chemical analysis is not yet available, there are indications that it contains a good portion of silica.

B. F-Cell Acid Recovery

The reported results on four samples of A-10 (process condensate) crib solution taken during the week of March 18, 1963 and run by Radiological Analysis are as follows:

(All results are in  $\mu\text{c/ml}$ )

<u>Total Sr</u>	<u>Sr-90</u>	<u>Zr-95-Nb-95</u>	<u>Cs-137</u>	<u>Ru-103</u>	<u>Ru-106</u>
1.6	.214	41.1	8.6	$1.09 \times 10^5$	$7.0 \times 10^4$
1.7	.161	38.0	8.1	$1.12 \times 10^5$	$6.8 \times 10^4$
1.2	.149	18.4	7.8	$1.87 \times 10^5$	$1.3 \times 10^5$
1.7	.175	32.7	13.2	$1.86 \times 10^5$	$1.3 \times 10^5$

The occupational 40-hour MPC limit for Sr-90 is  $1 \times 10^{-6}$   $\mu\text{c/ml}$  and for Cs-137 is  $1 \times 10^{-4}$   $\mu\text{c/ml}$ . Activity levels in F-Cell during the time when these samples were taken were the lowest of any equilibrium operating period, since conversion to single-cycle.

VIII. Waste Treatment and Storage - R. C. Forsman

Efforts to obtain greater vacuum in TK's F15 and F16 by connecting the vessels to the condenser vent system with a flexible jumper were unsuccessful due to a severe leak in the jumper. After installation of the normal vessel vent jumper and replacement of an unreliable thermohm in TK-F16, sugar treatment of IWW commenced on April 17, 1963. Due to the limiting vessel vent vacuum and the low acid concentration in the feed, no efficiency or reaction time data was obtained. Approximately 4,500 lbs. of  $\text{HNO}_3$  were destroyed in two batches of waste by sugar addition.

IX. Fission Product Recovery - W. C. SchmidtA. Strontium Recovery

During the week of March 31 to April 6, run series 59 was completed recovering approximately 100 kilocuries of strontium-90. The temperatures of the centrifugation of the sulfate precipitate was increased from 75 to 80° C to further improve waste losses. Laboratory analyses indicate the strontium losses to have increased from ~ 10 per cent to the old ~ 20 per cent. Since the IWW feed was low in nitric acid (~ 0.5 lb/gal) and gave considerable difficulty in removing the precipitated cakes from the centrifuge, additional tests will be performed at 80° C to evaluate this process change.

During the week of April 7 to April 14, the first two sulfate precipitations of run series 60 was completed. The sulfate waste losses continue to be ~ 20 per cent although centrifugation was done at 80° C. Considerable idle time was encountered due to lack of feed and availability of F18 for waste disposal.

During the week of April 15 to April 21, the third sulfate precipitation of run series 60 was completed. This batch was centrifuged in two portions due to excessively high radiation readings on the centrifuge. Laboratory analyses indicate the waste loss for strontium was reduced approximately a factor of two for the second batch. This phenomena is possible due to longer digestion time at high temperatures, but analyses of the other fission products in the same sample were low which indicated a non-representative sample rather than low waste losses. Considerable idle time was encountered due to lack of feed and availability of F18 for waste disposal.

During the week of April 22 to April 28, run series 60, which had only 3 sulfate precipitations due to lack of feed, was completed recovering approximately 76 kilocuries of strontium-90.

The oxalate cake (lead, cerium, and trivalent rare earths) was successfully slurried from the centrifuge with 160 gallons of water rather than the regular 4 M nitric acid. This test will be repeated for future use at B-Plant.

B. IWW Sample

During the week of April 7 to April 14, approximately two hundred gallons of uncentrifuged formaldehyde treated IWW was loaded into a cask for shipment to HLO. No process or equipment difficulties were encountered.

C. Promethium

Laboratory analyses of the centrifuged IWW, sulfate waste, and carbonate product samples of strontium series 58, 59, and 60 indicate the promethium recovery to vary between 40 and 80 per cent. These same analyses give poor material balances for promethium whereas the associated fission products material balances are within acceptable limits. This indicates the methods and/or techniques for promethium analyses need investigation.

X. Fission Product Purification - J. R. Kendall

About 65 kilocuries of rework Sr-90 was processed through the solvent extraction columns. Column operation was erratic because of periodic plugging in the EAW system. Although the EAW losses were negligible, the overall EAW loss was about 8 per cent. The product from this run was combined with the products from the preceding rework and vault crude runs in TK-6.

All waste in the building was discarded (no rework remaining) and limited flushing operations were started.

The HAPD-III-2 cask was loaded with 170 kilocuries of Sr-90 and shipped to ORNL on April 15.

XI. Miscellaneous - R. W. Lambert

A. Water Treatment

During the April shutdown, the recently installed No. 3 Anion Unit

containing amberlite IR-45 weak base resin was preconditioned with sulfuric acid and placed in service.

Also during the shutdown, the Nalco WBR resin in the No. 2 Anion Unit was replaced with Duolite A-308 intermediate base resin. Measurements indicated a 15 per cent loss of WBR resin since its installation in March of 1962. The No. 2 Unit was also pretreated and put into service.

The No. 1 Unit was removed from service pending arrival of WGR resin and plans are to make the resin change during the upcoming operating period. Following this change, all three beds will contain a different type of resin and in-service evaluation tests of the resin will be made to determine their resistance to fouling.

Installation of equipment necessary to add  $KMnO_4$  to the filter plant flash mixer has been completed and treatment may begin when warranted by laboratory or plant data.

B. E-Cell Sump

During the April shutdown, 7 ft<sup>3</sup> of 1 per cent boron, stainless steel Raschig rings were added to the E-Cell sump.

This document consists of No. 4 of 10 copies. Series A.

-58-

Distribution:

- 1 - CF Beaulieu
- 2 - WS Frank
- 3 - CF Hill
- 4 - RF Judson
- 5 - RW McCullough
- 6 - PR McMurray
- 7 - SS Smolen
- 8 - RE Tomlinson
- 9 - AJ Waligura
- 10 - ME Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 4/25/63 TO 2400 ON 5/5/63

I. General - G. A. Nicholson

The plant was started up at 3.0 nominal CF on April 25 with EAF on at 0710 and "hot" feed commencing at 1615. However, a suspected leak in the HS Column was verified on April 26 and the plant was shut down on April 27 after recycling about ten tons of "cold" feed to purge plutonium and activity from the system.

II. Performance Data - D. G. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

(None - Shutdown)

B. Production

- 1. EAF Processed - 32.1 T
- 2. U Production - 9.6 T
- 3. Pu Production - 49.9 Kg
- 4. Pu Production - -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	598 #	
2. Pu	172 Gr.	
3. Pu, F16, F18 K6	80 /1	
4. Solvent	-0-	

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	16.5 T (in E6 for startup)	
2. Pu	3459 (39,509 - Ehru N-Sell)	

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gallons</u>	<u>Waste Tank</u>
OWW	52,087	101-A
Cell Dr.	38,027	105-A
FP	12,555	105-A
GW	~ 9,630	106-BX

F. IWW Flows

(None)

G. Wank Farm

<u>Waste Tank</u>	<u>Boil-Off, Gal/Min</u>	<u>Temp. - °C (Bulb)</u>
101-A	2.2	147
102-A	1.2	92
103-A	0.3	88
104-A	1.3	137
105-A	4.8	92
106-A	9.3	124

III. Flowsheet - G. A. Nicholson

The Solvent-Extraction System was started up under the same flowsheet in effect at shutdown on April 1, with the following exceptions:

- (a) The 2AX flow was left off until a significant concentration appeared in the 2AW Stream.
- (b) The 2A Column was started up at a frequency of 54 rather than 69 cycles per minute.
- (c) Nitric acid of about seven molar concentration was substituted for the regular 3WB Stream at startup.
- (d) On April 25, the synthetic 3WB was replaced by process 3WB.
- (e) On April 25, the 2A pulse frequency was increased from 64 to 69 cycles per minute.
- (f) On April 25, transition from Phase I to Phase II was completed in the Neptunium Recovery Cycle.

IV. Solvent Extraction - G. A. Nicholson

Startup of the plant was accomplished with minimal difficulty. The activity of the First Cycle product streams was about three-fold above normal, however, in spite of high solvent saturation in the HA Column which caused excessive HAW losses. Performance of the Final Plutonium and Final Uranium Cycles appeared to be very good during the short period they were operating.

A severe gamma burst occurred during the shutdown when the HA and HS interfaces were displaced to the IBXS Tank. The organic was displaced from the HA and HS Columns and the columns were emptied at shutdown. The Partition Columns and the Final Plutonium and Final Uranium Cycles were shutdown at equilibrium.

The original plan at shutdown was to recover the cartridge from the failed HS Column and install it in the new column. However, inspection revealed the top support "spider" to be loose from the cartridge proper. The cartridge commenced to fall apart when an attempt was made to pull it by means of one of the tie rods, so it was left in the column.

After the spare HS Column was installed, the HA Cartridge was inspected. Inspection revealed it also to be no longer intact. The top support spider was broken and an attempt to remove the cartridge resulted in about one-third of the plates falling back into the column. After an attempt to "fish" the plates out proved unfruitful, the HA Column was removed and replaced with the spare.

At the end of the report period, both columns were installed and preliminary steps were again underway for startup of the plant.

#### V. Product Treatment

##### A. Plutonium Purification - N-Cell - R. W. Lambert

On April 30, following the plant shutdown on April 27, rework operations were started in N-Cell using a ferrous sulfamate valence adjustment step. Twenty-one kgs of plutonium were reworked with approximately 10 per cent being lost to the backcycle via XAW losses and EK-19 heels. Analyses of the rework material showed a 30 per cent to 40 per cent Pu-VI content reflecting a long concentrator holdup time and only four days log storage time before being reworked.

##### B. Neptunium Purification - S. M. Nielson

At 1158 on April 14, the first "hot" 3BN from E-Cell Package was received in Q-Cell. The 3BN flow ran continuously until 2145 at essentially flowsheet value of 0.5 gpm. The concentrated material, collected in Tank Q3, was 1.72 g/l in neptunium. The total amount was approximately half that needed to constitute one batch. During the subsequent ion exchange steps, the flow rates averaged at the specified flowsheet values. The scintillation monitor, SM-Q4, and the Q4 line sampler were inoperable until the elution step, preventing the waste streams leaving the Ion Exchange Column T-Q4.

On April 17, the Resin Pretreatment step was run with the flow going to Tank Q5 instead of Q3. This was done to keep from diluting the feed any further. The feed in Tank Q3 was butted with ferrous sulfamate and hydrazine. The ferrous sulfamate concentration was four-fold higher than specifications due to a bad valve.

The Resin Loading step was started on April 18. A sample of the loading waste in Tank Q5, taken when the loading step was completed on April 19, showed a loss of 1.4 per cent during the loading step.



The Plutonium Scrub was made on April 22 using 7 column volumes of scrub instead of the regular 20 because of the low plutonium content in the feed. The total loss measured on completion of the Plutonium Scrub step was 3.7 per cent. The high loss is believed to be due to erratic flows during the Loading and Plutonium Scrub steps resulting from a faulty flowmeter.

The Fission Product Scrub was started on April 23 and completed on April 24. The Fluoride Scrub was completed on April 25.

Elution began on April 25. On April 26, after a total of 65.6 liters had been passed through the Ion Exchange Column T-Q4, the scintillation monitor, SM-Q4, on the effluent stream showed an increase in gamma count at the 0.11 mev level. The total counts per minute reached 25,200. The product stream was switched from Tank Q3 to Q6. After 0.60 column volume had been collected, the gamma monitor decreased rapidly. Further elution, routed to Q3, showed that the resin was unloaded. A sample of the Q5 Fission Product wash was analyzed and it was found that 90 per cent of the neptunium was stripped from the column during the step. A check of the Fission Product Scrub make-up showed that the  $\text{HNO}_3$  concentration was 5.0 M rather than 7.0 M. This is believed to be the reason for the loss.

On April 29, the E-Q2 Concentrator was started and the material concentrated. During the concentration step, the weight factor in E-Q2 was inadvertently allowed to get too low and the concentrator underflow lines became plugged. The plug appeared to be on both sides of the underflow jet. By using steam connected to a drain valve on the overflow line inside the maintenance hood, the plug was broken. The plug is believed to have been caused by the ANN that was in the Fission Product Scrub waste.

On May 3, the feed was butted to 0.1 M hydrazine and ferrous sulfamate. The Resin Pretreatment step was run and the Resin Loading step completed. A sample of the Q5 Waste Tank showed that only 0.17 per cent of the neptunium had been lost during loading.

#### VI. Solvent Treatment - G. J. Raab

The No. 1 Solvent System activity level was  $1.33 \times 10^3$  and  $1.32 \times 10^3$   $\mu\text{c/gallon}$  ZrNb and Ru respectively after "hot" plant startup and continued at about this level during the short plant operating period. However, when the plant was shutdown, the organic inventory was spun through the wash system and the level reached  $4.82 \times 10^2$  and  $4.97 \times 10^2$   $\mu\text{c/gallon}$ .

The No. 2 Solvent System demonstrated notable cleanup while processing cold uranium at plant startup. The plutonium retention ~~increased~~ <sup>decreased</sup> from  $2.34 \times 10^6$  to  $9.69 \times 10^6$ . This is possibly due to acid and uranium contact in the 2D Column or more complete turnover through the solvent wash system.

#### VII. Miscellaneous - R. W. Lambert

##### A. Crib Flows

On May 2, the fractionator tail water was diverted from the Chemical Sewer to the A-9 Crib.

DECLASSIFIED

HW-75912

-62-

B. Water Treatment - R. W. Lambert

Based on permanganate demand tests run by the Purex Laboratory, the chemical oxygen demand of the sanitary water increased from an average of about 0.6 ppm to about 1.1 ppm about April 12. The demand remained constant at the 1.1 ppm level up through the end of the report period.

Analyses for manganese has shown the sanitary water to contain routinely less than 0.004 ppm.

## Distribution:

- 1 - JF Beaulieu  
 2 - WS Frank  
 3 - CF Hill  
 4 - EF Judson  
 5 - RW McCullugh  
 6 - PR McMurray  
 7 - SG Smolen  
 8 - RE Tomlinson  
 9 - AJ Waligura  
 10 - MF Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 5/6/63 TO 2400 ON 5/12/63

I. General - G. A. Nicholson

Normal processing was resumed on May 6 with the HAF on at about 1100 at a nominal 3.0 G.F. Processing of "hot" feed was commenced at 2000. The rate was increased to a nominal 3.6 (actual 3.8 G.F.) on May 7 after the process stabilized and equipment operability was established. Commencing on May 8, a batch of fresh LW, representing the high EAW losses incurred during both the April 25 and the May 6 startups, was successfully reworked through the Solvent-Extraction System.

II. Performance Data - D. G. LeysonA. Solvent-Extraction Performance by Cycles (Typical)

	Gamma dF		% Recycle		% Loss			Np Accum.
	U	Pu	U	Pu	U	Pu	Np	
First	3.7	3.3			.013	.03	5.0	13.0%
Final	3.1	3.6	5.8	6.4	.002		2.5	-35.0*
Non Exch.		1.0		.2				
Overall	6.8	7.9	5.8	6.6	.015	.03		

B. Production

1. EAF = 194.2 T
2. U Production = 173.5 T
3. Pu Production = 88.7 Kg
4. Np Production = -0-

C. Overall Loss

	Units	% of Total Production
1. U	1,088 Lb	0.37
2. Pu	402 gr	.45
3. Np, F16, F18 K6	45 21	
4. Solvent	3,228	2.2

\*Includes ~ 500 units in Q-Sell but does not include ~ 500 units in Rework System.

I. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. W	3.9 T	2.6
2. Pa	4.0 Kg	9.0

E. Volumes to UGS, Gal/Con

<u>Source</u>	<u>Gal/Con</u>	<u>Waste Tank</u>
QWW	222	101-A
GD	74	105-A
IWW	10	105-A
FP	-0-	
CW	300	106-BX

F. IWW Flows

7.1 Avg. HNO<sub>3</sub> Conc. - 5.7 M

G. Bank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	2.2	147
102-A	1.2	166
103-A	0.3	102
104-A	1.3	137
105-A	4.8	94
106-A	9.3	288

III. Flowsheet - G. A. Nicholson

- (a) The plant was started up under the same flowsheet conditions in effect at shutdown, except for the following:
- (1) The 2A Column was started at a frequency of 69 rather than 89 cycles per minutes.
  - (2) The 2BP was routed to the "1-Bell Package" following a flood in the 2AX Column at startup, which was initiated by a temporary loss of pulse.
  - (3) The 2AX was maintained at a reduced rate until 2AW loss was up to the normal control level.
  - (4) The EA Column was started at a frequency of 74 rather than 58 cycles per minute.
- (b) In May 6, the EA pulse frequency was increased to 76 cycles per minute. It was further increased to 78 cycles per minute on May 7.

- (c) On May 7, the 2EP Stream was routed to the Plutonium Anion Exchange Cycle. The 2BX composition was adjusted accordingly.
- (d) On May 8, rework of IWW commenced with a 150-gallon addition to a batch of HAF. After skipping a batch, rework continued at a rate of about 300 gallons per batch of HAF.
- (e) On May 8, the Neptunium Recovery Cycle was started under Phase II conditions at about one-half the normal rates. On May 9, transition from Phase II to Phase I conditions was made.
- (f) On May 9, the EA pulse frequency was reduced from 78 to 71 cycles per minute and the HAF rate was reduced by about 25 per cent temporarily to dissipate a flood in the column. The frequency was increased back to 75 cycles per minute on May 10.
- (g) On May 8, the ICX-H<sub>2</sub>O and 2EX-H<sub>2</sub>O control instruments were determined to be malfunctioning and the flow rates were ten to twenty per cent high.
- (h) On May 11, the IEX pulse frequency was reduced from 54 to 51 cycles per minute. It was increased back to 54 cycles per minute on May 12.

#### IV. Feed Preparation - G. J. Raab

Dissolvers were started on May 7. The first two batches of coating waste jetted to UES with difficulty. A 25 per cent caustic flush, plus continued use appears to have removed the solids from the low spot in the line. Dissolver A3 still has subnormal vacuum; during vigorous reaction, low vacuum cuts off the cell air bleed. I<sub>131</sub> emission was 0.44 curies for the week. Cooling time of the metal was 125 to 172 days. The HAF has been dilute (3.25 to 3.4 pounds uranium/gallon), undoubtedly due to insufficient compensation in make-up calculation for the IWW rework.

#### V. Solvent Extraction - G. A. Nicholson

After initial startup at 3.0 C.F., performance of the Solvent-Extraction System was excellent. Decontamination performance of all cycles was back up to the high levels experienced approximately six months ago. As the processing rate was increased, however, the First Cycle E.F. dropped off about two-fold. Also, the ruthenium activity in the First Cycle product streams increased by about five-fold. Except for temporary losses at startup, the losses via the HAW Stream have been very good, averaging less than 0.05 per cent. The IWW which was segregated after startup was reworked without any problem. Indeed, a general improvement in both activity and losses was experienced during the rework operation, in spite of an approximate two-fold increase in activity of the HAF Stream. The instability in the HA Column, experienced on May 9, was due partly to erratic HAF flow and partly to erratic (cycling) ESR flow caused by faulty controls systems. Operation of the IZ Column at the current rates (~ 34 E/D in the First Cycle Columns) has been at absolute maximum capacity. The losses via the ICW have generally been near or below the analytical detection limit, but the ICX flow has been high and the column has been slightly unstable.

Performance of the Final Plutonium Cycle has been excellent, with typical D.F.'s in excess of 2000. Losses to the Backcycle Waste System have been maintained slightly higher than normal, but well under control.

Performance of the Final Uranium Cycle has also been very good, with typical D.F.'s about 1500. The plutonium activity in the final product has been well under limits - generally about 3 to 5 ppb - in spite of any increase in carryover in the IBU Stream toward the end of the report period. The increase in ruthenium contamination in the First Cycle product (IBU) stream was reflected in high activity in the final product also. The Zr-Nb activity in the final product also showed an increasing trend toward the last of the report period, and silica gel treatment was required for ten batches of product.

The Neptunium Recovery Cycle was started up on May 8 under Phase II flow-sheet conditions, but at one-half the normal Phase II rates. On May 10, transition to Phase I conditions was made. Neptunium accumulation in the plant since startup has appeared to be below normal, but addition of rework material and the general unsteady conditions in the plant since startup make an accurate estimate of the inventory extremely difficult.

## VI. Product Treatment

### A. Plutonium Purification - N-Cell - R. W. Lambert

Operation of N-Cell during the report period was good with no evidence of pushing problems. Three batches of out-of-specification product were produced during a period when no fluoride was being added and a slight gamma bump reached N-Cell via the 2BP Stream.

### B. Silica Gel - R. W. Lambert

The Silica Gel Unit was shut down for regeneration on May 12.

### C. Neptunium Purification - S. M. Nielson

The second purification of the first batch of "hot" material continued during the report period. Due to the high efficiency of the J-Cell Package, the plutonium content of the feed was low and no Plutonium Scrub was made. The Fission Product Scrub was started on May 6 and was completed on May 7. A sample of the waste from the Fission Product Scrub showed a 4.8 per cent neptunium loss during this step. The high loss to the backcycle system is believed to be due to fast and erratic flow rates caused by the faulty flowmeter.

On May 8, the Fluoride Scrub was completed and the Elution Step started. On May 9, after a total of 2.6 column volumes of elutant, the SM-Q4 Scintillation Monitor indicated that the neptunium was eluting. The gamma count reached a peak of ~ 42,000 c/m at ~ .09 mev. The product stream was switched to Tank Q6 and a total of 0.70 column volumes was collected when the gamma count began to decrease rapidly. Further elution to Tank Q3 of 0.95 column volumes showed that the resin was unloaded. A total of 83 per cent of the original feed was collected in the Q6 product tank.

Analysis of the collected product was within shipping specifications except for concentration which was a factor of 2.3 low. Since the feed consisted of only half a batch, this low concentration was expected.

The following shows the D.F.'s achieved:

Zr-Nb*	767
Ru*	13.0
Pu**	1.6
U**	1 590

\*Based on uc/gr Np

\*\*Based on gr/gr Np

During this run, the scintillation monitor exceeded expectations and proved to be excellent as a primary process control instrument. It was especially helpful in determining the point at which to switch from the Forecut to the Product Elution Step. The Resin Probe that indicates resin level in the column operates well and also gives an added service in that it indicates when the column is gassing.

VII. Solvent Treatment - G. J. Raab

The No. 1 Solvent System activity level was in the order of  $3 \times 10^2$  and  $3 \times 10^2$  uc/gallon Zr-Nb and Ru, respectively, from startup to May 8. On May 8, day shift IWW rework was started. The solvent activity rose to  $1.23 \times 10^3$  and  $2.2 \times 10^3$  uc/gallon Zr-Nb and Ru by graveyard May 9. The solvent activity has remained at about this level for the remainder of the period. The frequency of wash solution change was increased 30 per cent, but failed to improve the solvent quality. Excessive solvent loss (3,228 gallons) were also noted during the period. The losses may have been caused by increased solvent washing and instability of the IC Column.

The No. 2 Solvent System has been losing the normal 150 to 200 gallons per day to the No. 1 System in wash solutions. The plutonium retention for the No. 2 Solvent was 5.177 as of May 9.

An attempt was made to bleed the organic now stored in Tank R8 back into the system via a line between the Tank R8 and Tank R1 samplers as no route now exists between these tanks. No appreciable volume has been transferred via this route due to unknown difficulties. Fresh solvent (~ 1000 gallons) has been added to Tank R1 to hold the inventory at operating level.

VIII. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

During the report period, activity levels climbed continuously and by the end of the period, the Zr-Nb level was near the February-March run period equilibrium value while the ruthenium was a factor of three above the lowest equilibrium value of the past run. Operation of the system was somewhat nonstandard because of heavy IWW rework which essentially doubled the salt and activity level of the HAW.

IX. Fission Product Recovery - W. C. Schmidt

Special process test runs were started May 11 for recovery of Cs-137 and Tc-99 from 103-C supernatants and May 12 for recovery of a cerium-rare earth fraction from current IWW solution.

X. Miscellaneous - R. W. LambertA. Cribs

After diverting the fractionator tail water to the A9 Crib on May 2, the crib liquid level rose steadily and by the end of the report period, reached 26 inches. The water flow to the barometric condenser was cut from about 550 gpm to 450 gpm, with a corresponding temperature rise to 45° C in an effort to relieve the crib.

B. Water Treatment

The demineralizer units were started up using only the No. 2 and No. 3 Anion Units in conjunction with the two normal cation units. The No. 1 Anion Unit was held out of service pending replacement of the resin.

The new resins in the No. 2 and No. 3 Anion Units demonstrated excellent capacity with no sulfate breakthrough after processing approximately 300,000 gallons of water.



This document consists of No.      of 10 copies. Series A.

-89-

Distribution:

- 1 - CF Beaulieu
- 2 - WS Frank
- 3 - OF Hill
- 4 - RF Judson
- 5 - RW McCullugh
- 6 - PR McMurray
- 7 - SE Smolen
- 8 - RE Tomlinson
- 9 - AJ Waligura
- 10 - ME Walling

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 5/13/63 TO 2400 ON 5/19/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.6 (actual 3.7) CF during the report period. Rework of IWV was concluded on May 14 and rework of hydrolyzed waste (F8) solution commenced on May 16. The plant was shut down at equilibrium for about five hours on May 15 and the 2D Column was emptied for replacement of the 2DF Pump.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.6	3.5			.003	.025	4.0	13%
Final	3.2	3.2	6.5	4.7	.002		4.0	104% *
Ion Exch.		1.3		0.07				
Overall	6.8	8.0	6.5	4.8	.005	.025	8.0	117%

B. Production

- 1. HAF = 205.9 T
- 2. U Production = 209.3 T
- 3. Pu Production = 126.6 Kg.
- 4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	1 038 #	.25
2. Pu	327	.26
3. Np, F16, F18	26	5.6
K6	12	2.6
4. Solvent	1 655	.12

)Based on virgin (in-put

\*Includes ~ 400 units back into Process thru Rework System.

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	1 250 #	0.3
2. Pu	2 814	2.2
3. Np	400	87 (Based on virgin in-put)

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	77	101-A
"	171	102-A
CD	37	105-A
FP	86	105-A
IWW	7	105-A
CW	300	106-BX

F. IWW Flows

7.5 Ave. HNO<sub>3</sub> Conc. - 5.3 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	2.2	146
102-A	1.2	122
103-A	0.3	93
104-A	1.3	137
105-A	4.8	93
106-A	9.3	299

III. Flowsheet - G. A. Nicholson

- On May 13, the 2DF-ferrous sulfamate rate was reduced from 0.22 to 0.19 flows. It was further reduced to 0.16 flows on May 19.
- On May 13, a check indicated the 2EX-H<sub>2</sub>O flow controller to be malfunctioning and the flow rate to be about 20 per cent high. The rate was reduced back to normal.
- On May 14, a check indicated the HSS-H<sub>2</sub>O flow controller to be incorrect and the rate was 26.4 rather than the normal 50 flows. It was increased back to 45 flows on May 16.
- On May 14, the ICX-H<sub>2</sub>O was reduced from 485 to 465 flows. It was further reduced to 424 flows on May 15 and to 400 flows on May 16.
- On May 15, the HA pulse frequency was reduced from 78 to 73 cycles per minute. It was increased back to 77 cycles per minute on May 16.

On May 19, it was reduced to 68 cycles per minute, then increased back to 73 cycles per minute.

- (f) On May 16, the ICX temperature was increased from 66 to 68° C. It was further increased to 70° C on May 18.
- (g) On May 18,  $N_2H_4$  flow was started to the ICU Concentrator at 0.29 flows of 2.2 M solution.
- (h) On May 18, transition from Phase I to Phase II was completed in the Neptunium Recovery Cycle.
- (i) On May 18, recovered acid from the  $UO_3$  Plant was substituted for fresh  $HNO_3$  in the 2DF- $HNO_3$  Stream.

#### IV. Feed Preparation - G. J. Raab

Dissolver production has started to fall behind plant rates. The main delay has been charging dissolvers. The cooling time of the metal was 121 to 179 days. The  $I_{131}$  emission was 0.68 curies for the week. Rework of IWW was completed on May 14. Rework of hydrolyzed sump wastes via EK-F8 to EK-D5 (Metal Solution Sample Tank) was started May 16.

#### V. Solvent Extraction - G. A. Nicholson

Performance of the Solvent-Extraction System remained at the same high level as during the previous report period with respect to both decontamination and waste losses. Waste losses during the report period were typically less than 0.03 per cent. Activity of the First Cycle uranium product stream showed an upward trend - about a two-fold increase - during the report period, while the activity of the plutonium product stream increased only about 50 per cent. The activity of both streams took a sharp drop when rework of hydrolyzed waste solution was started, but commenced increasing almost immediately. The general decrease in decontamination performance of the First Cycle was attributed to the continuing difficulty with the HS Column Interface Controller and the resulting erratic HSR flow. Ruthenium contamination in the 2DF Stream decreased markedly about the middle of the report period. When the hydrazine flow was started to the ICU Concentrator, a mild ruthenium burst occurred, but rapidly dissipated.

Performance of the remainder of the Solvent-Extraction System has been excellent. Decontamination factors in the Final Plutonium Cycle have increased from about 2000 at the beginning to about 5000 at the end of the report period, coincident with a general decrease in losses to the Backcycle Waste System. Performance of the Final Uranium Cycle has remained fairly constant with decontamination factors typically 2000. Plutonium contamination in the final product, except for two brief excursions, has shown a steady downward trend. A process test of the effects on process performance of the substitution of hydrazine for ferrous sulfamate to reduce the residual plutonium in the 2DF Stream was initiated on May 18. The hydrazine is added to the ICU Concentrator to provide sufficient digestion time for complete reduction of the plutonium. Results so far show no adverse effects on Final Uranium Cycle performance. To the contrary, preliminary indications are that both plutonium and ruthenium decontamination may be improved.

Performance of the Neptunium Recovery Cycle has been excellent. After minor adjustments in rates at the beginning of the report period, accumulation in the system was rapid and the transition was made to Phase II on May 18 with an estimated 1500 units "captured". Transition to Phase II was smooth, except for persistent mild flooding in the 3A Scrub Section. This was alleviated by a 15 per cent reduction in the pulse frequency.

Decontamination performance during Phase II was normal and by the end of the report period, the product was within specifications for transfer to the Purification Cycle. Plutonium contamination in the product (3BN) stream was reduced to a non-detectable level. Losses to the Backcycle Waste System during the Phase II operation were negligible.

## VI. Product Treatment

### A. Plutonium Purification - N-Cell - R. W. Lambert

Operation of the ion exchange unit, N-Cell, continued satisfactorily during the report period. By the end of the period, however, there were some indications of the resin deteriorating as the push pressure necessary for good resin movement increased to 40 lbs.

### B. Uranium Silica Gel - R. W. Lambert

On May 15, regeneration of the Silica Gel units was completed and normal treatment of uranium started.

### C. Neptunium Purification - S. M. Nielson

During the report period, the product collected from the first Neptunium Purification run was concentrated from 17.7 g/l to 44.8 g/l. This was accomplished by heating the solution to 94° C and air sparging at 0.5 cfm with Tank Q6 vent vacuum at 10 inches of water. A material balance showed no product loss during the operation.

The faulty flowmeter in the T-Q4 Column feed line was removed and a new one prepared for replacement. Plans were made to place a temporary sampler in the T-Q4 outlet line inside the maintenance hood for use during the first few hot runs.

## VII. Solvent Treatment - G. J. Raab

The No. 1 Solvent System activity level was  $1.40 \times 10^3$  and  $3.47 \times 10^3$   $\mu\text{g/gal}$  Zr-Nb and Ru, respectively at the beginning of the period and  $6.75 \times 10^2$  and  $1.86 \times 10^3$  Zr-Nb and Ru by the end of the period. The activity level dropped suddenly to the lower level upon completion of EW rework. The flowsheet wash solution change schedule was restored on May 17. In order to have space to butt the fresh solvent in the Make-up Tank, 315 gallons of 46% TBP solvent was added to the No. 1 System. Solvent losses have been normal.

The No. 2 Solvent System Pu retention value was  $1.67 \times 10^7$  as of May 16. The Ru activity was 40  $\mu\text{g/gal}$ , about two-folds higher than normal. Additions totaling 4,432 gallons of fresh solvent were made to the system during the week. The transfer of the solvent from TK-R8 to TK-R1 via the samplers was unsuccessful.

[REDACTED]

VIII. Acid Recovery and Waste Concentration - R. W. LambertF-Cell Acid Recovery

During the report period, F-Cell ruthenium activity levels reached two equilibrium plateaus, one representing conditions during EWV rework and one representing conditions during F8 rework. The Ru activity in both the AAA Acid and the AAD Overheads was 50% higher during the F8 rework period. The activity level during the EWV rework was an estimated factor of two higher than during periods of plant operation with no rework.

Zirconium-niobium activity in the AAA Acid fell slightly after the transition from EWV rework to F8 rework but both levels remained near the expected normal concentration of  $1 \times 10^4$  ug/gal.

IX. Waste Treatment and Storage - R. G. ForsmanA. Denitration

Denitration of the first EWV batch after extensive rework operations during the first few days of startup difficulties was unsuccessful. Although no excessive pressures were encountered, severe foaming in the Reaction Vessel, EK-F15, prevented reasonable sugar addition rates. A second EWV batch treated in EK-F16 did not indicate foaming; however, the available time limited the nitric destruction to a final free acid concentration of 3.4 M vs. initial 6.8 M concentration.

Continuous denitration with formaldehyde was limited to approximately 30% normal rates due to apparent foaming in the reactor. Increasing the antifoam rates a factor of 4 did not reduce foaming nor did temperature reductions in the preheater and reactor reduce foaming.

A formaldehyde pump leak necessitated shutdown of the unit on May 19; however, EWV flow was continued to flush the reactor of possible solvent products resulting from the extensive rework operations which may have caused the foaming problems.

B. A-Tank Farm

Since the organic wash waste (EWV) rate is greater than individual tank boil-off rates, frequent routing changes are necessary to balance liquid levels and NaF concentration in Tanks 101, 102 and 104. Therefore, EWV was routed to Tank 102 on May 15, then routed back to Tank 101 on May 20.

Apparent temperature excursions in Tanks 102 and 106 necessitated immediate water dilution and increased air to circulators. The bulb temperature in Tank 102 increased from  $\sim 95^\circ \text{C}$  to  $\sim 172^\circ \text{C}$ , while the thermocouple which is 20 feet away did not change. A new temperature bulb in Tank 102 indicated the temperature to be about  $120^\circ \text{C}$ . Although the temperature bulb and also a new bulb exchanged to confirm readings in Tank 106 showed no temperature changes, the thermocouple temperatures increased from  $160^\circ \text{C}$  at 4 inches from tank bottom to  $310^\circ \text{C}$  while the thermomh 12 inches from tank bottom increased from  $145^\circ \text{C}$  to  $220^\circ \text{C}$ . Both thermocouple temperatures are decreasing since water dilution and increased circulation. The sodium molarity at the time of temperature increases were  $\sim 5.5 \text{ M}$  in Tank 102 and  $\sim 7.3 \text{ M}$  in Tank 106.

X. Fission Product RecoveryA. Cerium-Rare Earth Recovery - W. C. Schmidt

The cerium and trivalent rare earths in approximately 5,800 gallons of centrifuged LWW in three separate batches was precipitated with sulfate ion. No lead was used a co-precipitant. Waste losses were approximately 29, 23, and 63 per cent for promethium, cerium and strontium, respectively. The separation of the cerium and rare earths from strontium by an oxalate precipitation is in progress.

B. Cesium Recovery - W. C. Schmidt

Three of the four SMT casks have been loaded with cesium. Feed temperatures are between 70 - 75° F and the casks loaded to the beginning of breakthrough. Preliminary data indicates that each cask will hold approximately 45 kilocuries of cesium. A spare SMT cask loaded with IRA 401-A Amberlite resin was connected in series with the cesium casks for recovery of technetium and is described below.

C. Technetium and Antimony Recovery - J. B. Kendall

An SMT containing about 55 cubic feet of 20 - 50 mesh Amberlite 401 resin in the nitrate form was connected downstream of the cesium casks in order to adsorb technetium. The technetium cask plugged after processing about 6,000 gallons of Tank 103 supernate. The cask was unplugged but plugged again after processing about 1,000 gallons. The cask was again unplugged but plugged again after processing about 500 gallons. By this time, the four cesium casks were loaded and the technetium loading was terminated. After washing the technetium cask with water, radiation readings were quite low. Plans are underway to remove and inspect the middle leg of the technetium cask to see if the screened portion of this leg is plugged.

Although analytical data are incomplete, it appears that the technetium was poorly absorbed. The technetium loss was generally 30 - 50%, with occasional losses as high as 90% reported. In addition to technetium, the path of Sb-125 was followed. Again the analytical data are incomplete, but the Sb-125 concentration is about 50-fold below the theoretical value, indicating a large fraction must be associated with the tank farm sludge. About 30 per cent of the Sb-125 was apparently absorbed, but it is not known at present whether it was absorbed on the Decalso or the Amberlite.

XI. Miscellaneous - R. W. LambertCribs

The liquid level in the A9 Crib continued to increase during the report period. On May 19, when the level in one riser reached 42 inches, the blank to the chemical sewer line was removed and the fractionator tail water allowed to flow to both the A9 Crib and the Purex Swamp. The division of flow between the two has not been firmly established.

This document consists of No. 4 of 11 copies. Series A.

[REDACTED]  
-75-

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - OF Hill
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - PR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling
  - 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 5/20/63 TO 2400 ON 5/26/63

I. General - G. A. Nicholson

Following a severe upset early on May 20, the Solvent Extraction System was shut down at equilibrium to empty the uranium concentrators and recycle their contents back through the system. The plant was started up at a nominal 3.3 CF on May 21 after a 12-hour shutdown. The rate was increased to a nominal 3.6 (actual 3.6) CF on May 22 and maintained at that level for the remainder of the report period. Rework of hydrolyzed waste (TK-F8) solution was terminated on May 20.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	Gamma dF		% Recycle		% Loss			Np Accum.
	U	Pu	U	Pu	U	Pu	Np	
First	3.5	2.2			.002	.04	10%	- 106%
Final	3.1	3.8	6.8	5.4	.002		3	+ 47
Ion Exch.		1.2		0.2				
Overall	6.6	7.2	6.8	5.6	.004	.04	13%	- 59%

B. Production

- 1. HAF = 161.7 T
- 2. U Production = 160.1 T
- 3. Pu Production = 76.6 Kg.
- 4. Np Production = -0-

C. Overall Loss

	Units	% of Total Production
1. U	1 647#	0.51
2. Pu	363	0.47
3. Np, F16, F18	139	31.0
K6	10	2.2
4. Solvent	383 Gal.	.03

[REDACTED]

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	27.5 T	17.1%
2. Pu	16 Kg.	21.0

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	271	101-A
CD	58	105-A
FP	128	105-A
IWW	81	105-A
CW	300	106-BX

F. IWW Flows

6.5 Ave. HNO<sub>3</sub> Conc. - 5.9 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal</u>	<u>Temp. (°C)</u>
101-A	2.2	145
102-A	1.2	126
103-A	0.3	91
104-A	1.3	137
105-A	4.8	92
106-A	9.3	280

III. Flowsheet

- (a) On May 20, the HA pulse frequency was reduced from 73 to 51 cycles per minute in several adjustments.
- (b) On May 20, "cold" uranium from the Final Uranium Cycle was recycled to the HAF prior to shutdown and after start-up.
- (c) On May 20, the 2BP Stream was routed to the Plutonium Concentrator and the 2BX composition and flow adjusted accordingly. It was routed back to the Plutonium Anion Exchange Cycle on May 22.
- (d) On May 20, transition from Phase II to Phase III was completed in the Neptunium Recovery Cycle.
- (e) On May 21, the HA Column was started up from equilibrium shutdown at a pulse frequency of 62 cycles per minute. The frequency was increased to 65 cycles per minute on May 22 and to 67 cycles per minute on May 24.
- (f) On May 22, the 20 Column was started up at a pulse frequency of 69, rather than 72 cycles per minute.



- (g) From May 23 through May 25, the HAO rate was reduced from 86 to 81 flows.
- (h) On May 23, the 2AF HNO<sub>3</sub> concentration was increased by about ten per cent to 3.5 molar.
- (i) On May 23, the 2DF-ferrous sulfamate was reduced from 0.16 to 0.12 flows. It was turned off early on May 24 and turned on again at 0.22 flows later on May 24. On May 26, it was reduced to 0.16 flows.

#### IV. Feed Preparation - G. J. Raab

Dissolver operation has been normal, but just able to maintain plant rate. There has been time lost in charging. The coating waste to UGS has been modified to incorporate a long air blow to clear the line. The coating waste has jetted to UGS without difficulty during the week. I<sub>131</sub> emission was 1.01 curies for the period. Cooling time of the metal was 127 to 172 days. No IWW or hydrolyzed waste was reworked via HAF.

#### V. Solvent Extraction - G. A. Nicholson

Performance of the Solvent Extraction System took a sudden, temporary decline on May 20 when a severe upset of the HA Column resulted in an extended burst of activity throughout the system. The severe flooding condition, due to inclusion of organic crud with the hydrolyzed waste solution in two batches of HAF lasted for about sixteen hours. After the contaminated HAF was processed, a batch of "cold" uranium was recycled to purge the system of the excess activity and "do-bads". The Solvent Extraction System was then shutdown at equilibrium and the uranium concentrators emptied. The uranium solution in the ICU Concentrator and 2DF Tank was recycled to the First Cycle while the material in the 2EU Concentrator was transferred back to the 2DF Tank. During the shutdown, a new HA Column DP jumper and new HA and HS Column organic Sp.G. jumpers were installed.

Startup of the HA Column was somewhat "rough" because of improper calibration of the instruments (resulting from installation of the new jumpers), but the process quickly stabilized and general performance rapidly returned to normal. Waste losses via the HAW Stream during the startup were excessive but, for the remainder of the report period, averaged less than 0.02 per cent.

Performance of the Final Plutonium Cycle was generally excellent during the report period. Decontamination performance has remained at the high level reached near the end of the previous report period and losses to the Back-cycle Waste have remained at normal level (2 to 4%). The severe burst of activity from the First Cycle necessitated routing the 2BP Stream to the Concentrator "Package" temporarily. However, as soon as the burst dissipated, the 2BP Stream was routed back to the Plutonium Anion Exchange Cycle.

Performance of the Final Uranium Cycle declined somewhat during the report period to an average DF of about 1000. The process test of the substitution of hydrazine for ferrous sulfamate as a reducing agent in the 2DF was continued. A definite improvement in plutonium decontamination resulted when hydrazine was added to the ICU Concentrator concurrent with flowsheet

addition of ferrous sulfamate to the 2DF Tank. However, when the ferrous sulfamate flow was turned off, the plutonium activity in the 2EU Stream increased rapidly to above tolerable levels. When the ferrous sulfamate flow was re-established at flowsheet rate, the plutonium activity rapidly decreased to the prior level. Indications to date are that hydrazine addition has definite beneficial effects. However, whether it can be used as a substitute for ferrous sulfamate is questionable. The process test is continuing in an effort to define more precisely optimum conditions.

Transfer of a large quantity of decontaminated neptunium to the Neptunium Purification Cycle proceeded without incident. The product (3BN) quality was excellent, with no detectable plutonium contamination and fission product activity about ten-fold less than limits. Accumulation in the Solvent Extraction System has been normal since the process was stabilized after the startup. However, a relatively large quantity was lost via the HAW for about eight hours after startup. Accumulation in the Neptunium Recovery Cycle since transition to Phase I has been excellent with approximately 90 per cent of the virgin neptunium input transferring to the "package" in spite of a low concentration in the Backcycle Waste System.

#### VI. Product Treatment

##### A. Plutonium Purification - N Cell - R. W. Lambert

On May 22, the N-Cell resin was changed out to take advantage of the plant shutdown. Prior to the changeout, the N-Cell operation had been satisfactory with only minor indications of pushing problems. Following the resin change, operation was resumed with no difficulties.

##### B. Uranium Silica Gel - R. W. Lambert

Processing of uranium through Silica Gel was stopped on May 25 and the units prepared for regeneration. During the ten day run period, approximately 250 tons of uranium were treated.

##### C. Neptunium Purification - S. M. Nielson

On May 20, the J-Cell Package was switched to Phase III and the 3BN Stream reached Q-Cell at 1620. The E-Q2 Concentrator was operated continuously until 0900 on May 21 when the 3BN jet was turned off. Operation was excellent with no problems encountered. A total of 1759 units were transferred with nondetectable plutonium content, again showing good J-Cell decontamination.

The TMI sample results were received on the product from the first purification run and were 37 per cent above shipping limits. This product will be saved for blending later.

#### VII. Solvent Treatment - G. J. Raab

The No. 1 Solvent System activity level was in the range of  $6.0 \times 10^2$  and  $1.3 \times 10^3$  ZrNb and Ru, respectively while the solvent feed tank sampler was working. The G5 (solvent feed tank) sampler has been out of order since May 24. The net solvent loss from the two solvent systems was only 380 gallons (No. 1 System gained 480 gallons, No. 2 System lost 860 gallons).

VIII. Acid Recovery and Waste Concentration - R. W. LambertF-Cell Acid Recovery

Following the processing of cold uranium on May 21, the ruthenium activity in the AAA (TK-F3) recovered acid dropped a factor of five to  $5 \times 10^3$  uQ/gal and remained there for two days even though hot feed was restarted early on May 22. By May 24, the ruthenium activity had again increased to approximately  $2 \times 10^4$  uQ/gal. The ZrNb activity remained near  $1.5 \times 10^4$  uQ/gal during the report period.

No process changes were made in the F-Cell acid recovery system during the report period and IWW flows remained at about 6.5 flows.

IX. Waste Treatment and Storage - R. C. ForsmanA. Denitration

Due to the formaldehyde pump leak only sugar treatment of IWW wastes was conducted during the report period. Space and time limitations restricted nitric destruction such that the IWW was reduced from  $\sim 5$  M nitric to only  $\sim 4$  M  $\text{HNO}_3$ .

B. A-Tank Farm

Excessive temperatures in TK's 102 and 106 were returning toward normal with last reported temperatures of  $120^\circ\text{C}$  (high of  $172^\circ\text{C}$  - normal  $100^\circ\text{C}$ ) and  $289^\circ\text{C}$  (high of  $311^\circ\text{C}$  - normal  $160^\circ\text{C}$ ), respectively.

Hot water sluicing tests in TK-103 indicated relative soft sludge in the vessel. During the first test using a  $\sim 55$  foot long 2 inch pipe with a  $1/2$  inch sluicing nozzle, the tank bottom was reached after 5 to 10 minutes in three trials by sluicing with hot water ( $\sim 150^\circ\text{F}$ ) at only 5 to 10 psig water pressure. The 240 lb. sluicing assembly was suspended from a pulley containing a weight gauge. The sluicing unit was slowly lowered until the sludge at  $\sim 20$  inches from tank bottom supported the 240 lb. assembly. The assembly was raised and lowered to maintain a total weight of 220 to 240 lb. on the gauge while pumping hot water at 5 to 10 psig pressure until the tank bottom was reached. Sludge depth was easily detected by decreased total weight of the assembly and increased water pressure when the nozzle was submerged in sludge. The hardest sludge layer appears to be at the 12 to 16 inch level. An offset in the pipe assembly allowed sluicing in about a 30 inch diameter circle under the 12 inch riser used for the tests. Sludge depth measurements using a standard disc (1 inch steel plate by 3 inch diameter with a 1 inch hole in center) indicated sludge heights of  $\sim 41$  inches before sluicing and 7 inches after sluicing.

To preclude previous sampling and testing in the 12 inch riser having affected the sluicibility of the sludge, a 1 inch pipe weighing 115 lb. was lowered into a 6 inch riser which had never been used. Pumping hot water ( $75^\circ$  to  $95^\circ\text{C}$ ) at less than 2 psig water pressure in three positions resulted in sludge penetration in 9 to 14 minutes. Duplicating the previous test in the 12 inch riser, only 10 to 20 lb. of

weight was allowed on the assembly while slowly lowering the nozzle to the tank bottom. All tests indicated relative soft sludge from 41 inches from the bottom to about 16 inches from the bottom and again from 10 inches above bottom to the tank bottom. The 4 to 6 inch hard sludge layer required about 30 to 50 per cent of the sluicing time for penetration; however, no increased weight or water sluicing pressure was necessary to penetrate this layer.

X. Fission Product Recovery - W. C. Schmidt

A. Cerium - Rare Earth Recovery

Approximately 3 megacuries of cerium was recovered in the cerium-rare earth recovery run. Only 880 kilocuries was received at the Strontium Semiworks in the bowling ball cask. Tank El Beckman indicates a large percentage of the cerium was left in Tank El. The volume and acidity in El was kept low to facilitate transfer to Strontium Semiworks via the 200 gallon bowling ball cask. With no agitation and low acidity, the cerium apparently existed as a precipitate rather than a solution.

B. Strontium Recovery

The recovery of strontium was resumed. Four sulfate precipitations of run series 62 have been completed. Sulfate waste losses averaged 20 - 30 per cent. Considerable difficulty is being encountered in neutralizing the centrifuged IWW to the desired pH of 0.7 to 1.5.

XI. Fission Product Purification - J. B. Kendall

The HAPO-II-2 cask was loaded with 155 kilocuries of Sr-90 and shipped to the Martin-Marietta Company at Quehanna, Pennsylvania. Loading of the HAPO-I-1 cask from the new El precipitator was started.

A batch of lead-free oxalate product from the Purex Plant bearing about one megacurie of cerium and one hundred kilocuries of promethium was transferred to the Strontium Semiworks. Three oxidations with potassium permanganate and three batch extractions with SSW solvent of the quadrivalent cerium were made in order to separate cerium from the other rare earths. No difficulties were encountered with phase separation or ruthenium discharge to the environs, however, no extraction of cerium was achieved. The reasons for the failure are not known, but presumably are related to destruction of the potassium permanganate by reductants in the aqueous and/or organic solutions.

The technique used to separate the cerium from the rare earths is briefly as follows:

- (a) TK-5 tank was used as a caustic scrubber to remove ruthenium from the vessel vent off-gas during the operation. About 0.1 M NaOH at 40 - 50° C was the scrubbing solution.
- (b) About 400 liters of Purex oxalate product was contacted with about 750 liters of SSW solvent at 30 - 40° C for 20 minutes. Every five minutes potassium permanganate was added to butt the aqueous phase

DECLASSIFIED

-81-

HW-76912

to 0.005 M  $\text{KMnO}_4$  until the total addition would have made the aqueous phase 0.02 M  $\text{KMnO}_4$ . The acidity of the aqueous phase was about 1.3 M  $\text{HNO}_3$ .

- (c) The aqueous phase was decanted to another vessel and contacted with 150 liters of SSW solvent for 15 minutes. The aqueous phase was butted to 0.01 M  $\text{KMnO}_4$  at the start of the contact.
- (d) Item (c) above was repeated.
- (e) The aqueous phase was decanted to another vessel and treated with hydrogen peroxide.
- (f) The organic phases (three) from the contacts were separately washed with a 1.5 M  $\text{HNO}_3$  - 0.1 M  $\text{H}_2\text{O}_2$  solution at an aqueous to organic ratio of about one.

About 50 per cent of the cerium and promethium was found in the washes of the organic phases. This was caused by the very crude decanting operations. The aqueous phases from the organic washes were combined and a batch extraction operation was initiated in order to separate the manganese from the cerium and rare earths. In addition, laboratory studies were started in order to determine the reason for the process failure and the maximum manganese concentration that can be tolerated before repeating the operation for separating the rare earths and cerium.

## XII. Miscellaneous - R. W. Lambert

### Water Treatment

On May 26, 1963, conductivity recorders were placed in service on the effluent of the No. 2 and No. 3 anion units. The conductivity probes and recorder are on loan from Chemical Effluents Technology.

On May 22, 1963, the demineralized water filters were changed. This was the third time in sixteen days that the filters have been plugged. Prior to changing the anion resin during the April shutdown, the filters have gone between one and two months between changes.

This document consists of No. 2 of 11 copies. Series A.

Distribution:

- 1 - OF Beaulieu
- 2 - WS Frank
- 3 - OF Hill
- 4 - BF Judson
- 5 - RW McCullugh
- 6 - PR McMurray
- 7 - SG Smolen
- 8 - RE Tomlinson
- 9 - AJ Waligura
- 10 - MT Walling
- 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 5/27/63 TO 2400 ON 6/2/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.6 (actual 3.65) CF during the report period.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	2.4	3.5			.002	.03	7.5	- 30%
Final	4.0	3.5	8.0	4.0	.002		3.0	- 24%
Ion Exch.		1.3		0.1				
Overall	6.4	8.3	8.0	4.1	.004	.03	10.5	- 54%*

B. Production

- 1. HAF = 185.9 T
- 2. U Production = 210.5 T
- 3. Pu Production = 124.4 Kg
- 4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	1 187 Lb	0.28
2. Pu	295 g	0.24
3. Np, F16, F18	42	8.8 )Based on
K6	9	1.9 )virg.input
4. Solvent	1 320	0.09

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	Nil	--
2. Pu	9 394	7.5

\*Week-week Inv./Virgin Input.

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	63	101-A
OWW	166	104-A
CD	67	105-A
FP	83	105-A
IWW	14	105-A
CW	300	106-BX

F. IWW Flows

6.7 Ave.  $\text{HNO}_3$  Conc. -  $\sim 4.0 \text{ M}$

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (<math>^{\circ}\text{C}</math>)</u>
101-A	1.54	144
102-A	2.32	120
103-A	0.36	103
104-A	2.52	136
105-A	3.53	94
106-A	8.52	121

III. Flowsheet - G. A. Nicholson

- (a) On May 27, the  $\text{N}_2\text{H}_4$  addition to the ICU Concentrator was stopped. It was started again at the same rate on May 30. On June 1, the  $\text{N}_2\text{H}_4$  concentration was increased from 2.2 to 11.0 molar. The rate was maintained at 0.29 flows until June 2 when it was reduced to 0.06 flows.
- (b) On May 28, HA pulse frequency was increased from 67 to 69 cycles per minute. It was further increased to 74 cycles per minute on May 29 and to 76 cycles per minute on June 2.
- (c) On May 28, the 2D pulse frequency was increased from 69 to 72 cycles per minute.
- (d) On May 28, the 2DF-ferrous sulfamate was increased from 0.16 to 0.24 flows. It was reduced back to 0.16 flows on May 29.
- (e) On May 29, the Neptunium Recovery Cycle was reduced to a 1.6 CF nominal rate.
- (f) Between May 29 and June 1, the  $\text{HNO}_3$  concentration in the 3WB was gradually increased from 7.2 to 9.2 molar. It was reduced back to 8 molar on June 2. During this same time, the acidity in the HA Column was gradually increased by about 30 per cent because of anomalous sample analysis.

IV. Feed Preparation - G. J. Raab

Dissolver operation has been normal and have just been able to maintain plant rate.  $\text{I}_{131}$  emission was 1.46 curies for the period. A single bucket of 113-

day cooled metal was charged on May 27. The remainder of the metal cooling time was 128 to 188 days. No IWW or hydrolized waste was reworked via HAF. The rework of plutonium via HAF Make-up Tank E6 was heavy for most of the period.

V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance during the week was characterized by a general downward trend in the decontamination performance of both the First and the Final Uranium Cycles. Also, high acidity in the First Cycle caused the bulk of the activity to remain with the uranium stream. Action had been taken to alleviate the situation (reduction of the acidity and increase in pulse frequency of the HA Column), but the activity of the First Cycle product was still on the rise at the end of the report period. The problem this time, however, appeared to be caused by rework of some plutonium solution of incorrect valence, which resulted in temporarily high waste losses. In an effort to reduce the losses, the saturation in the HA Column was reduced with the consequent increase in activity of the product streams.

Performance of the Final Plutonium Cycle was excellent during the report period, with product activity well within the limits for processing through the Plutonium Anion-Exchange Cycle. The losses to the Backcycle Waste remained within control limits of two to four per cent. The acidity of the 2AF Stream was maintained about ten per cent above normal during the report period in an effort to determine the effect of acidity and extractant flow on the decontamination performance of the 2A Column. It is planned to reduce the acidity and increase the 2AX rate shortly to complete the investigation.

Performance of the Final Uranium Cycle showed some improvement over the previous report period, with D.F.'s typically about 1300. However, due to the high activity of the feed, final treatment by silica gel was necessary. The process test of the substitution of hydrazine for ferrous sulfamate in the Final Uranium Cycle continued. After the hydrazine flow was stopped, the plutonium contamination in the 2EU Stream immediately commenced increasing, while the activity remained essentially constant. When the hydrazine flow was reestablished, the activity commenced in the 2EU Stream and started increasing at a fairly fast rate; appearing to "peak" at about three-fold above the starting level. A five-fold increase in the hydrazine concentration in the ICU Concentrator resulted in an additional two-fold increase in the activity of the 2EU Stream. Results of the test to date are not overly encouraging. However, while it may not be feasible to substitute hydrazine for ferrous sulfamate completely, it may be possible to achieve superior plutonium decontamination with lower ferrous sulfamate concentrations in conjunction with a trace of hydrazine concentration in the 2DF.

Neptunium accumulation in the Solvent-Extraction System appeared normal during the report period. Waste losses were generally excellent, averaging five and three per cent via the HAW and 2EU Streams, respectively. Accumulation figures, based on the 3BN and 3WB analyses, however, were rather nebulous. The bulk of the neptunium had been transferred to the Neptunium Recovery System by May 29 and the rates were reduced. At about this time, the analyses became rather variable, so that neptunium recovery performance until the end of the report period was questionable.



VI. Product TreatmentA. Plutonium Purification - N-Cell - R. W. Lambert

Plutonium ion exchange (N-Cell) operated satisfactorily during the report period with all product well within specifications. Typical decontamination factors obtained during the period ranged between 15 and 25.

B. Uranium Silica Gel - R. W. Lambert

Regeneration of the units was completed on May 27 and normal silica gel processing started.

C. Neptunium Purification - S. M. Nielson

The first few days of the report period was spent replacing the faulty meter, cleaning the remote head of the metering pump and calibrating the new flowmeter.

The Resin Loading Step for Hot Run #2 was started at 1132 on May 31. This step continued until 0713 on June 1. The operation was excellent with the complete amount (1.8 batch) loading on the resin at a concentration of 64 gr per liter of resin. Waste losses were 0.91 per cent.

VII. Solvent Treatment - G. J. Raab

The No. 1 Organic System activity level based on a single sample on May 28 was  $7.99 \times 10^2$  and  $1.63 \times 10^3$  uc/gal ZrNb and Ru, respectively. The solvent feed tank sampler was unplugged, then became unusable again for the period. Solvent losses were normal for the period.

The transfer of solvent from TK-R8 to TK-R1 was still awaiting final completion of the route via temporary piping connections. The No. 2 Solvent System plutonium retention value was  $5.37 \times 10^7$ .

VIII. Acid Recovery and Waste Concentration - R. W. LambertF-Cell Acid Recovery

Operating conditions in the acid recovery system remained relatively unchanged during the report period with the AAA recovered acid ruthenium activity fluctuating between  $1.5 \times 10^4$  uq/gal and  $3.0 \times 10^4$  uq/gal and the ZrNb running about  $1.7 \times 10^4$  uq/gal.

IX. Waste Treatment and Storage - R. C. ForsmanA. Denitration

The formaldehyde prototype unit was down during most of the report period due to repair of steam leaks in the P & O Gallery preventing access to formaldehyde treatment equipment, loss of the reactor thermohm, and failure of the IWW feed valve. The failed thermohm jumper which was made of schedule 40 stainless steel was replaced by a schedule 80 stainless steel jumper as tests indicated corrosion could have caused the failure.

Sugar treatment was conducted in TK-F16 when cycle time and space permitted.

**B. 241-A Tank Farm**

Tank 102 temperatures have returned to the 120° C range while the apparent hot spot in TK-106 has decreased steadily to 265° C.

**X. Fission Product Recovery - W. C. Schmidt**

Run series 62 was completed. Approximately 170 kilocuries of strontium-90 was recovered. The path of both plutonium and promethium in the sulfate process still remains unknown. No promethium analyses were received and the plutonium material balance was very poor.

Run series 63 was initiated. With the reduction of iron in the IWW due to single cycle concentration and substitution of hydrazine for ferrous sulfamate, the amount of hydroxyacetic acid added to each batch was reduced by twenty per cent. The sulfate precipitation pH range was also raised to 0.7 to 2.0 with the anticipated reduction in IWW iron concentration. The addition of lead as a co-carrier has been changed to be added after the 90° C digestion period and while the solution is still hot rather than addition prior to heating for the digestion period. Sufficient runs to evaluate these changes have not been made.

**XI. Fission Product Purification - J. B. Kendall**

Batch contacts of the cerium-rare earth solution in Tanks 6 and 54 were made with SSW solvent in order to remove manganese. A total of 936 grams of manganese was added to the process during the attempt to oxidize and extract cerium. Laboratory analyses of TK-6 and TK-54 solution, however, showed a total of only 527 grams. The batch extraction operations are summarized below:

	<u>First Extraction</u>			<u>Second Extraction</u>		
	<u>Overall Summary</u>			<u>Overall Summary</u>		
	<u>Kilocuries</u>			<u>Kilocuries</u>		
	<u>Ce</u>	<u>Pm</u>	<u>Grams</u>	<u>Ce</u>	<u>Pm</u>	<u>Grams</u>
			<u>Mn</u>			
Feed	359	42	141	408	60	377
Losses	15	1.4	-	17	1.5	-
Product	259	24	9.3	369	58	42
Unaccounted	85	16	-	20	0.5	-

<u>Extraction</u>		<u>Extraction</u>	
Aqueous	400 Liters	Aqueous	400 Liters
SSW Solvent	750 Liters	Solvent from	First Extraction
pH	1.4	pH	1.4

<u>Waste (Kilocuries)</u>		<u>Waste (Kilocuries)</u>	
Ce	8	Ce	14
Pm	0.4	Pm	1.3

First Extraction  
Overall Summary

Solvent Scrub

Two 300 Liter Batches of  
Nitric Acid - pH = 1.6

Waste (Kilocuries)

Ce	1
Pm	0.1

Solvent Strip

Three 300 Liter Batches  
of 1.5 M HNO<sub>3</sub>

Strips 1 & 2 (Product)\*

Ce	259 Kilocuries
Pm	24 Kilocuries
Mn	9 Grams

Strip 3 (Waste)

Ce	6 Kilocuries
Pm	0.9 Kilocuries

Second Extraction  
Overall Summary

Solvent Scrub

Two 300 Liter Batches of  
Nitric Acid - pH = 1.6

Waste (Kilocuries)

Ce	2.7
Pm	0.2

Solvent Strip #1\*

One 300 Liter Batch  
of 1.5 M HNO<sub>3</sub>

Ce	289 Kilocuries
Pm	45 Kilocuries
Mn	7.5 Grams

Solvent Strip #2 (Product)

One 300 Liter Batch of 3.0 M HNO<sub>3</sub>

Ce	80 Kilocuries
Pm	13 Kilocuries
Mn	34 Grams

XII. Miscellaneous

Cribs

Even though the lines to both the A9 Crib and the Chemical Sewer were open, the crib liquid level continued to increase until by May 31, the liquid level in one of the crib resins reached 52 inches. The estimated flow to the crib at this time was about one-half the total fractionator tail water flow or about 250 gpm.

On June 2, the blank was replaced in the line to the A9 Crib resulting in the total tail water flow being routed to the Chemical Sewer.

\*These product solutions were combined in preparation for the oxidation and extraction of cerium.



This document consists of No.      of 11 copies. Series A.

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - OF Hill
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - PR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling
  - 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 6/3/63 TO 2400 ON 6/9/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.6 (actual 3.70) CF during the report period. However, it was necessary to recycle six batches of "cold" uranium to the HAF to maintain continuity of operation. An attempt to rework some hydrolyzed waste solution in the spare IWW Concentrator was not successful due to the resulting increase in activity of the HSP Stream.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.*</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.6	3.5			.002	0.1	8.7	+ 101%
Final	2.9	3.3	6.5	3.9	.002		2.5	- 77%
Ion Exch.		1.5		0.07				
Overall	6.5	8.3	6.5	4.0	.004	0.1	11.2	+ 24%

B. Production

- 1. HAF = 170.5 T
- 2. U Production = 173.4 T
- 3. Pu Production = 112.4 Kg.
- 4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	888 Lb	0.26
2. Pu	371	0.33
3. Np, F16, F18	62	16.5 (Based on
K6	11	2.9 (Input
4. Solvent	1 317	0.09

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	42.5 T	24.5
2. Pu	18.5 Kg.	16.4

\*Week End-Week End - Based on input.

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	73	104-A
OWW	169	101-A
CD	45	105-A
FP	113	105-A
IWW	13	105-A
CW	300	106-BX

F. IWW Flows

6.2 Ave.  $\text{HNO}_3$  Conc. - 5.0 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-Off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.5	142
102-A	2.3	121
103-A	0.4	89
104-A	2.5	135
105-A	3.5	93
106-A	8.5	130

III. Flowsheet - G. A. Nicholson

- (a) On June 3, the 2A pulse frequency was reduced from 86 to 56 cycles per minute to "break" a flood. It was increased back to 75 cycles per minute later the same day and to 86 cycles per minute on June 4.
- (b) On June 3, the 3AX was increased from 25 to 29 flows. It was reduced back to 25 flows on June 4.
- (c) On June 3, the HSR- $\text{HNO}_3$  was increased from 7.4 to 8.5 flows. It was reduced back to 8.0 flows on June 4.
- (d) On June 3, the HAO was increased from 83 to 94 flows. It was reduced back to 83 flows on June 4.
- (e) On June 6, the 2D pulse frequency was increased from 72 to 81 cycles per minute. It was reduced to 61 cycles per minute to break the resulting flood. The column was shut down temporarily on June 7, then started up at a pulse frequency of 61 cycles per minute. The pulse frequency was increased back to 71 cycles per minute later the same day.
- (f) On June 7, the hydrazine addition to the ICU Concentrator was turned off.

IV. Feed Preparation - G. J. Raab

Dissolver production lagged behind solvent extraction through the week. It was necessary to recycle six batches of cold feed to TK-E6. Approximately 16 hours was lost on dissolver A3 when 50 per cent NaOH was added to a charge

of jacketed slugs. This resulted in a major pressurization of the dissolver. The dissolver appeared to receive no serious damage, however, the lid bail was bent and a 17 lb plug was blown out of the silver reactor regeneration opening.

I131 emission was 2.36 curies for the period. The cooling time for metal was 120 to 156 days. The coating waste line plugging slowed jetting on June 3. A pump was installed in TK-D2 and the flow was slow until after a caustic flush was sent through. The pump was used to the end of the period.

#### V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance during the week stabilized at about the level at the end of the previous report period. Activity levels in the plant fluctuated considerably due to the intermittent recycling of cold uranium, with typical decontamination factors of 2500 and 800 for the Final Plutonium and Final Uranium Cycles, respectively. First Cycle waste losses were above normal for the week. As during the previous week, in an effort to minimize the loss, the HA Column organic saturation was reduced with the resulting decrease in the decontamination performance of the First Cycle. The high waste losses (averaging 0.20 per cent) were suspected of resulting from inextractable plutonium from rework solution.

Performance of the Final Plutonium Cycle continued at the same high level as during the preceding week, with all product well below shipping limits after processing in the Plutonium Anion-Exchange Cycle. The acidity in the 2A Column was maintained about ten per cent above the flowsheet value during the report period. During the report period, the 2AW losses were on the high side of the control range (generally, three to four per cent), but under good control and on a downward trend.

Performance of the Final Uranium Cycle was marginal, with typical decontamination factors about thirty per cent lower than the previous week. Analysis of the effect of the hydrazine addition to the ICU Concentrator during the first half of the report period was made extremely difficult by the intermittent recycling of cold uranium and the flood in the 2D Column. However, a general improvement in performance commenced when the hydrazine addition was terminated. The 2D pulse frequency was increased prior to termination of the hydrazine test to determine if the poor performance was a result of operation at too low a frequency. Flooding of the column after an increase of seven cycles per minute, however, indicated the normal pulse frequency to be near optimum for the current rates.

Neptunium accumulation in the Solvent-Extraction System during the week was apparently normal. Losses were normal (about 20 per cent overall) and both instantaneous losses (via HAW Stream) and accumulative (IWW) losses were within the normal variance. However, an accurate picture of the accumulation in the Backcycle Waste and Neptunium Recovery Systems was extremely difficult to obtain because of anomalous analytical data.

#### VI. Product Treatment

##### A. Plutonium Purification (N-Cell) - R. W. Lambert

Push pressures increased gradually during the report period and by June 7, resin movement was extremely sluggish. On June 9, the resin was changed

out without interrupting normal N-Cell operation. Due to some operating problems, however, optimum replacement methods could not be fully carried out and a significant per cent of old resin was left in the unit. Operation following the changeout was satisfactory although a push pressure of 35 lbs. was necessary to obtain adequate resin movement. The resin changeout had been in active use for only 20 days.

Decontamination performance during the week was excellent with typical DF's averaging near 30 and all product well within specifications.

B. Uranium Silica Gel - R. W. Lambert

On June 4, Silica Gel was shut down and regeneration begun. Approximately 200 tons of uranium were processed during the run.

C. Neptunium Purification - S. M. Nielson

The second purification run was successfully completed during the report period. Equipment operation was generally excellent. The final product was well within shipping specifications in all respects.

Decontamination was excellent even though no Plutonium Scrub was run and the Fission Product Scrub consisted of only 15 column volumes rather than the usual 20. A material balance and the SM-Q4 gamma monitor indicated that the fission products had been removed by the end of 15 column volumes of Fission Product Scrub. The following are the DF's\* attained for this run:

ZrNb	1300
Ru	630
U	48
Pu	Non-detectable in feed and $\leq 0.0004$ g/gr Np in Product
TMI	60

Waste losses were lower than experienced in the first run due to steady-stream flows controlled by the new flowmeter. The losses are listed as follows:

Loading Step	- 0.91%
FP & F1 Scrub Steps (Combined)	- 0.21%
Elution Forecut Step	- 2.2 %

The Elution step required 2.2 column volumes before the neptunium began to unload. The SM-Q4 gamma monitor indicated immediately when the neptunium began to unload. The maximum reading of the SM-Q4 reached 276,000 count/min (.09 mev), which corresponded to 61.5 g/l Np as measured by a line sample.

A total of 1.3 column volumes of product was collected in Tank Q6 when the effluent stream concentration fell to 0.05 g/l Np ( $\sim 50,000$  c/x) and the Elution step was stopped. Overall product concentration was 45.4 g/l Np.

\*On a per gram neptunium basis.

VII. Solvent Treatment - G. J. Raab

The No. 1 Solvent Feed Tank sampler operated intermittently. The latest sample was on June 7, with activity of  $8.11 \times 10^2$  and  $1.64 \times 10^3$  uc/gal ZrNb and Ru, respectively. Solvent losses were in balance between the two systems.

No solvent was added to the No. 2 System from TK-R8 so it was necessary to add 2,435 gallons of fresh solvent from TK-R1A to hold the inventory above agitator levels. The No. 2 Solvent System plutonium retention valve was  $2.32 \times 10^7$ .

VIII. Acid Recovery and Waste Concentration - R. W. LambertA. F-Cell Acid Recovery

During the report period, the ruthenium activity in the AAA recovered acid increased sharply on two occasions peaking once at  $5 \times 10^4$  uQ/gal and once at  $3.5 \times 10^4$  uQ/gal. No assignable cause for the increase was determined. By the end of the report period, the ruthenium had declined and was averaging about  $2 \times 10^4$  uQ/gal. The ZrNb activity remained at about  $1.7 \times 10^4$  uQ/gal during the period.

IX. Waste Treatment and Storage - R. C. ForsmanA. Denitration

Denitration by formaldehyde and/or sugar has reduced the IWW nitric from the 5 - 6 M range to 2 - 3 M. Efforts are continuing to reduce all IWW batches to 1 - 2 M nitric acid.

B. A-Tank Farm

The temperature "hot spot" in TK-106 continues to decrease with a current temperature of 255 °C (high was ~ 310 °C). All other temperature elements indicate normal readings.

Sluicing tests using the same 1 inch assembly tested in TK-103 were conducted in TK's-101 and 102. Standard sludge depth measurement in TK's-101 and 102 were 14 inches and 30 inches, respectively prior to the sluicing tests. Tests in TK-101 were inconclusive since the pipe assembly could not be lowered below 12 inches from tank bottom due to pieces of concrete and/or sludge depth measurement devices accumulated in this area. The riser used for the test was the only available riser in TK-101 and had been used for sludge depth measurements, supernate sampling, and sludge sampling.

Tests conducted in TK-102 indicated sludge to be slightly harder in the upper 4 - 5 inches of sludge than TK-103 sludge; however, once through this layer, the probe easily penetrated to the tank bottom. After 14 minutes of probing during the initial test, the probe was lowered from 20 inches to 17 inches when lack of sluicing water necessitated delay of testing until the following day. During the second day, the assembly was again supported by about 20 inches of sludge, but after seven minutes, the nozzle was about 16 inches above tank bottom. During an additional 6 minutes of sluicing, the nozzle reached tank bottom.

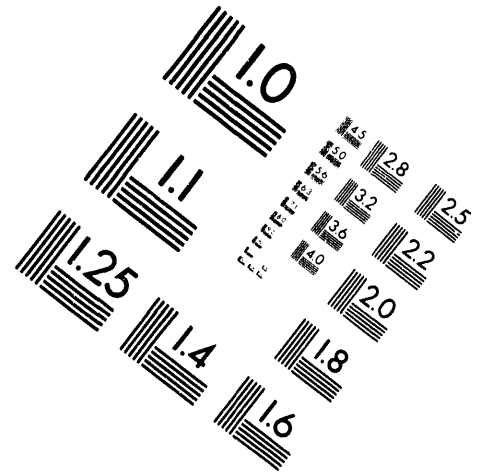
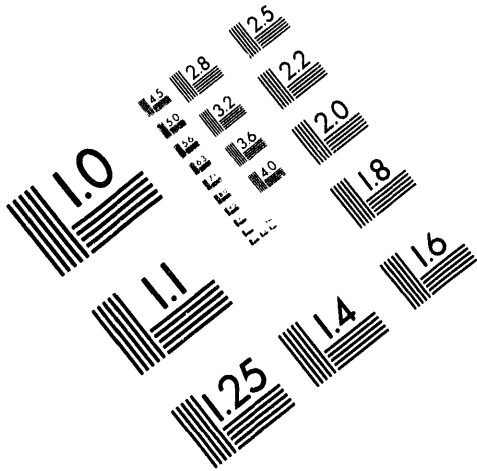




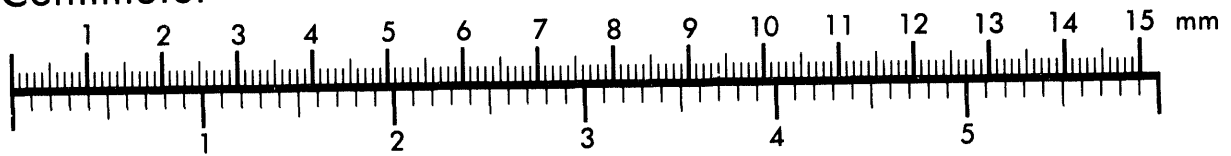
**AIM**

**Association for Information and Image Management**

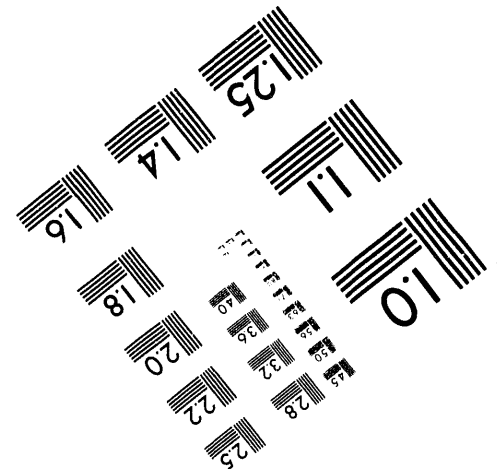
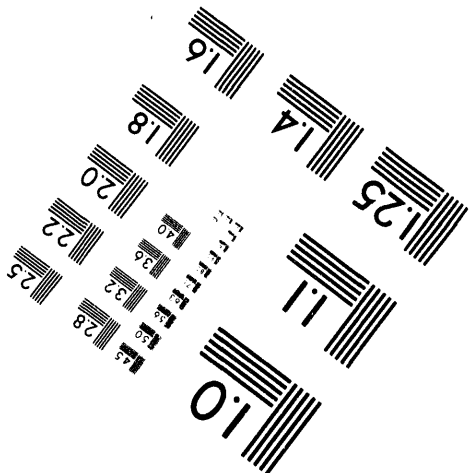
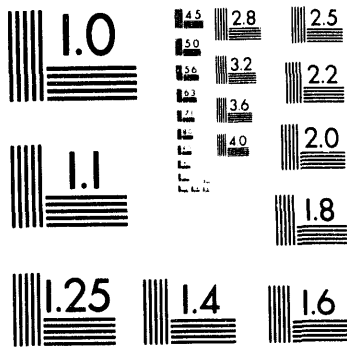
1100 Wayne Avenue, Suite 1100  
Silver Spring, Maryland 20910  
301/587-8202



Centimeter



Inches



MANUFACTURED TO AIIM STANDARDS  
BY APPLIED IMAGE, INC.

**2 of 3**

X. Fission Product Recovery - W. C. Schmidt

Run series 63 is in progress. The addition of lead after the sulfate solution has been heated to 90° C has not influenced the sulfate waste losses. Losses continue to average 20 - 30 per cent.

A new equation for adjusting the pH of the centrifuged IWW to 1.0 has been calculated from data obtained from Process Chemistry Operation. The new equation, gal of caustic = .126 (lbs nitric in centrifuged IWW) - .63 (gallons of centrifuged IWW), appears to be more nearly correct, especially for IWW which has been denitrated to an acidity of less than 1.5 lbs per gal.

XI. Fission Product Purification - J. B. Kendall

Two tests of oxidizing and extracting cerium were made. Both tests used sodium bismuthate as the oxidant, "fresh" SSW solvent as the extractant, and the product solutions from the manganese separation as feed. The feed composition was about 1.5 M HNO<sub>3</sub>, 0.013 TRE, and 0.007 M Ce. The solution used to strip the organic was always 1.5 M HNO<sub>3</sub>-0.2 M H<sub>2</sub>O<sub>2</sub> and the stripping operations were made at an aqueous to organic ratio of about 1.3.

The first oxidation-extraction operation was made at an L/V of 0.98 with a 0.046 M Na BiO<sub>3</sub> aqueous phase and a 30 minute contact. The aqueous phase was butted to a theoretical value of 0.052 M Na BiO<sub>3</sub> (L/V increased to 1.06) and recontacted for 15 minutes. About 45 per cent of the cerium was extracted in the first contact. The quantity of cerium extracted increased to 65 per cent in the second contact. Less than 5 per cent of the TRE (as measured by promethium) was extracted. Although data are not available, it would appear as the basis of the TRE extraction that the cerium fraction will not be appreciably contaminated with bismuth.

The second oxidation-extraction operation was made at an L/V of 1.12 with a 0.095 M Na BiO<sub>3</sub> aqueous phase and a 30 minute contact. About 97 per cent of the cerium was extracted. The path of the TRE is not clearly defined. (A material balance of 250 per cent was achieved) although not conclusive, it appears that a substantial fraction of the TRE (measured by promethium analysis) may have been retained by the sodium bismuthate sludge.

XII. Miscellaneous - R. W. Lambert

Water Treatment

On June 6, a batch of 1 per cent KMnO<sub>4</sub> was made up at the Power House Filter Plant and addition started into the filter plant feed at a rate of approximately 0.2 ppm KMnO<sub>4</sub>. The addition was made to check out the operability of the equipment and control procedures. The only difficulty encountered was obtaining complete dissolution of the KMnO<sub>4</sub> in the make-up tank because of insufficient agitation. Modification to the agitator or heating of the make-up may be necessary to correct the problem.

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - OF Hill
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - PR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling
  - 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 6/10/63 TO 2400 ON 6/16/63

I. General

The plant was maintained at a nominal 3.6 CF until June 12, when the rate was reduced to a nominal 3.4 (actual 3.45) CF. The rate was reduced to eliminate the necessity of recycling "cold" uranium to maintain continuity of operation. Rework of hydrolyzed sump waste solution was commenced on June 16 without any apparent adverse effects on the process.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	Gamma dF		% Recycle		% Loss			Np Accum.
	U	Pu	U	Pu	U	Pu	Np	
First	3.6	3.4			.002	0.1	6.1	-52%*
Final	2.8	3.4	8.0	3.4	.002		3.5	-36%*
Ion Exch.		1.5		.06				+165%*
Overall	6.4	8.3	8.0	3.46	.004	0.1	9.6	+77%*

B. Production

- 1. HAF = 187.3 T
- 2. U Production = 184.4 T
- 3. Pu Production = 105.2 Kg.
- 4. Np Production = 1365

C. Overall Loss

	Units	% of Total Production
1. U	944	0.25
2. Pu	429	0.41
3. Np, F16, F18	83	20.1*
K6	11	2.6*
4. Solvent	1288	.09**

\*Based on Virgin Input.  
 \*\*Based on Volume Pumped at 3.6 CF



D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	13.5 T	7.3
2. Pu	1.4 Kg.	1.4
3. Np	14 Gr.	3.4*

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	281	101-A
CD	50	105-A
FP	124	105-A
IWW	28	105-A
CW	300	106-BX

F. IWW Flows

6.8      Avg. HNO<sub>3</sub> Conc. - 5.3 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.54	140
102-A	2.32	120
103-A	0.36	87
104-A	2.52	137
105-A	3.53	92
106-A	8.52	130

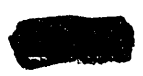
III. Flowsheet - G. A. Nicholson

- (a) On June 13, the 3AS-hydrazine concentration was reduced from 0.15 to 0.075 molar.
- (b) On June 13, the Neptunium Recovery Cycle was switched from Phase I to Phase II conditions.
- (c) On June 14, transition from Phase II to Phase III was completed in the Neptunium Recovery Cycle. The system was shutdown on June 15 after completion of Phase III operation.
- (d) On June 16, rework of hydrolyzed sump waste solution was commenced at a rate of about 100 gallons per batch of feed solution.

IV. Feed Preparation - G. J. Raab

Dissolver production has increased since several 15 bucket charges have been made to increase the heels. The dissolvers were current with extraction at end of report period. Accelerated train schedules were required to get extra

\*Based on Virgin Input.



metal in storage basin to make the larger charges. The I131 emission was 3.71 curies for the period. The cooling time was 121 - 191 days. The I131 content of metal charged was 1310 curies for the period for a DF of 350. The scintillation monitors showed little I131 coming from the dissolvers, however, the vessel vent system is unmonitored. Mercuric nitrate was started to the metal solution tanks on June 10.

#### V. Solvent Extraction

Solvent-extraction performance continued at essentially the same level as during the previous report period. The activity levels stabilized with the reduction in rates. First Cycle waste losses continued to be above normal, averaging about 0.2 per cent. While the analyses continued to fluctuate considerably, the acid control was improved, primarily because use of the analyses as primary control criteria was discontinued. The activity of the HSP Stream decreased rapidly when the first "shot" of hydrolyzed waste solution was processed, indicating an unusually high "tonic" effect.

Performance of the Final Plutonium Cycle was unchanged from the previous report period. However, the activity of the product (2BP) stream showed much less fluctuation and the losses via the 2AW Stream averaged slightly less than the previous report period.

Performance of the Final Uranium Cycle continued at about the same level as during the previous report period. A temporary decline in decontamination performance was noted early in the week as the product activity increased about two to three-fold while the activity of the 2DF remained essentially constant. However, after about two days, the Zr-Nb activity of the product stream commenced to decrease. Coincidentally, the Ru activity of the 2DF commenced to increase - to a maximum about three-fold higher - while the Ru activity in the product stream remained essentially constant. The net result was an improvement in decontamination performance to the level typical of the previous week.

Neptunium accumulation in the Solvent-Extraction System was normal during the report period. The neptunium which had accumulated in the "J-Cell Package" was transferred to the Neptunium Purification Cycle. About 650 units were transferred to "Q-Cell" for a recovery of about 90 per cent. The product (3BN) stream contained a total of five grams of plutonium and the activity, while about two-fold higher than the previous batch, was well below limits for final processing in Q-Cell.

#### VI. Product Treatment

##### A. Plutonium Purification - N-Cell - R. W. Lambert

During the report period, the mechanical operation of the ion exchange unit was satisfactory although push pressures were increased from 35 lbs. to 48 lbs. Decontamination performance remained excellent with DF's averaging between 30 and 35. On June 16, the N-6 weight factor line developed a corrosion leak and several liters of concentrated product escaped to the N-Cell floor sump before a new line could be fabricated.

B. Uranium Silica Gel - G. A. Nicholson

Regeneration of the Silica-Gel facility was commenced on June 11 and completed on June 14. About 125 tons of uranium were processed since the previous regeneration.

C. Neptunium Purification - S. M. Nielson

Product consisting of 1,360 units neptunium from the second purification run was loaded out and shipped on June 10. Shipping specifications were met in all respects.

On June 14, J-Cell Package was changed to Phase III and a small batch containing 710 units neptunium was transferred to Q-Cell. A Pu Scrub will be required since five units of Pu was received with this batch. Ion exchange treatment will begin during the next report period.

VII. Solvent Treatment - G. J. Raab

The No. 1 Solvent Feed Tank sampler is still inoperative. Solvent loss has been in balance between the two systems. The overall loss from the system was 1370 gallons. Approximately 1000 gallons of solution from TK-R8 was transferred to TK-R1 in 150 gallon batches. TK-R8 appeared to accumulate a small aqueous heel each time TK-G8 was jettted to UGS. This was also jettted to TK-R1. The No. 2 Solvent System activity level and Pu retention values did not appear to be affected adversely.

VIII. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

On June 12, the tray water to the E-F6 Concentrator was turned off. The Zr-Nb activity in the AAA recovered acid responded almost immediately increasing by a factor of two to about  $3.8 \times 10^4$  uq/gal. On June 16, the tray water was restarted at approximately 3 gpm and the Zr-Nb responded by beginning a sharp downward trend which leveled out at its pre-test plateau.

The Ru activity during the test was unaffected. The nitrite addition rate, boil-off rate and IWW flow rate were held constant during the test.

IX. Waste Treatment and Storage - R. C. Forsman

A. Denitration

Continuous denitration of IWW with formaldehyde has reduced the acid in IWW to approximately 1.5 M. Operation of the unit has been satisfactory; however, antifoam rates (~ 200 ppm) have been a factor of ~ 2 above the March operating period.

B. A-Tank Farm

The sludge temperature of TK-106 decreased 5° C to 250° C during the report period. The bulb reading remains normal at ~ 180° C.

Pressure tests using the Fire Department pumper truck have indicated the 1 inch sluice nozzle in TK-103 is operable.

X. Fission Product Recovery - W. C. Schmidt

Strontium run series 63 was completed with an approximate recovery of 242 kilocuries of strontium<sup>90</sup>. Waste losses continue to average 20 to 30 per cent on the sulfate precipitation. With a reduced volume of cake removal acid, eight sulfate precipitations were made in this series.

XI. Fission Product Purification - J. B. Kendall

Three batch extraction studies were made during the week for separating cerium and the rare earths.

The first study was made at an aqueous to organic ratio of about one. The aqueous phase was made 0.04 M lead dioxide and contacted for 10 minutes. The aqueous phase was made 0.02 M potassium permanganate and contacted for 10 minutes. The aqueous phase was increased to 0.04 M potassium permanganate and contacted for an additional 10 minutes. About 75 per cent of the cerium was extracted.

The second study was made at an aqueous to organic ratio of about one. The aqueous phase was made about 0.08 M sodium bismuthate and contacted for a total of 75 minutes. Aqueous phase samples were taken periodically in order to study the effect of agitation time on the oxidation and extraction of cerium. The analytical data were erratic and unexplainable. These analyses are being rerun. About 95 per cent of the cerium, however, was extracted.

The third study was made on the accumulated rare earth fraction from the above and preceding runs. This rare earth fraction was grossly contaminated with lead, manganese, and bismuth. The aqueous phase was contacted twice with two separate organic phases. The aqueous to organic ratios used were 2.3 and 3.5, respectively. The initial aqueous phase was made 0.075 M sodium bismuthate and contacted for 30 minutes. The aqueous phase was recontacted with a new batch of solvent for additional 30 minutes. About 96 per cent of the cerium was extracted. Data from the intercycle samples, however, were confusing. The aqueous phase after the first contact showed only 1.3 per cent of the cerium. After the second contact, 3.1 per cent of the cerium was in the aqueous phase. After transferring most of the aqueous phase to a storage tank, 3.9 per cent of the cerium was found.

XII. Miscellaneous - R. W. Lambert

Water Treatment

The main AMU demineralized water filters continued to plug at a greater than normal rate. The last three changes have been made after an average of 10 days of service.



This document consists  
of No.      of 11 copies.  
Series A.

-99-

**DECLASSIFIED** HW-76912

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - OF Hill
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - PR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling
  - 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 6/17/63 TO 2400 ON 6/23/63

I. General

The plant was maintained at a nominal 3.4 (actual 3.4) CF during the report period. Commencing on June 21, four and one-half batches of "cold" uranium were recycled to the HAF Tank to purge the plutonium from the system in preparation for processing feed material of a nonrepresentative irradiation history. Commencing on June 22, feed material of a nonrepresentative irradiation history was processed.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	Gamma dF		% Recycle		% Loss			Np Accum.*
	U	Pu	U	Pu	U	Pu	Np	
First	4.2	3.9			.002	.02	7.0	-58%
Final	2.8	3.1	8.5	4.7	.002		3.5	+109%
Ion Exch.		1.4		.35				
Overall	7.0	8.4	8.5	5.0	.004	.02	10.5	51%

B. Production

- 1. HAF = 157 T
- 2. U Production = 200 T
- 3. Pu Production = 91.8 Kg.
- 4. Np Production

C. Overall Loss

	Units	% of Total Production
1. U	795 Lbs.	0.25
2. Pu	495 Gr.	0.54
3. Np, F16, F18	167	37.0 (Based on Input
K6	39	8.7 (+ F8 Rework.
4. Solvent	2 095 Gal.	0.15 Based on Total Gal Pumped at Nom. 3.4 CF.

\*Based on Input + F8 Rework.

<u>D. Rework</u>	<u>Units</u>	<u>% of Total Production</u>
1. U - F8: 0.4 T P3: 39.0 T P2: 4.3 T	43.7 T	21.9
2. Pu	9.9 Kg.	10.8
3. Np - F8: 104 P3: 10	114 Gr.	25.4 (Based on Input + F8 Rework)

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	15	101-A
	22.3	104-A
Cell Dr.	29	105-A
FP	92	105-A
IWW	34	105-A
CW	300	106-BX

F. IWW Flows

~ 4.0 Avg. HNO<sub>3</sub> Conc. - 5.4 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-Off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.54	137
102-A	2.32	139
103-A	0.36	88
104-A	2.52	135
105-A	3.53	94
106-A	8.52	130

III. Flowsheet - G. A. Nicholson

- (a) On June 17, the Neptunium Recovery Cycle was started up under Phase I conditions at a 3.6 CF rate.
- (b) On June 17, the HA pulse frequency was reduced from 70 to 62 cycles per minute. It was increased back to 71 cycles per minute later the same day.
- (c) On June 19, rework of "out-of-spec" UNH solution through the Final Uranium Cycle was commenced. It terminated on June 20.
- (d) On June 19, the rates in the Final Uranium Cycle were increased to 4.4 CF. At this point the 2D Column became unstable and it was necessary to reduce the pulse frequency from 71 to 44 cycles per minute and the 2DF and 2DX rates by 20 per cent temporarily to re-stabilize the column.

The pulse frequency was increased back to 54 cycles per minute on June 20. Later the same day, the pulse frequency was reduced to 59 cycles per minute to restabilize the column again. It was again increased back to 64 cycles per minute after a short time.

- (e) On June 20, the 2DF ferrous sulfamate was increased from 0.17 to 0.26 flows. It was reduced back to 0.17 flows early on June 23, then increased back to 35 flows later the same day.
- (f) On June 21, the HA pulse frequency was reduced from 77 to 70 cycles per minute. It was increased back to 73 cycles per minute later on June 21 and to 76 cycles per minute on June 22.
- (g) On June 23, addition of recovered nitric to the 3WB was commenced at a rate of 4.2 flows.

IV. Feed Preparation - G. J. Raab

Dissolver operation was good. A large metal solution inventory allowed cutting of heels and transition to unclassified metal with no great difficulty. The heel of classified metal was removed from all dissolvers by June 19. The I<sub>131</sub> emission was 2.35 curies for the period. The cooling time was 124 to 248 days, with the unclassified metal being mostly 160 to 200 days old. The daily I<sub>131</sub> emission was down to .06 curies per day at end of the period.

V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance during the report period was characterized by a marked improvement in performance of the First Cycle, but essentially no change in the performance of the Final Plutonium and Final Uranium Cycles. The improvement in the First Cycle was attributed to the "tonic" effect of reworking the hydrolyzed sump waste solution. High HAW losses were experienced for the first day and a half of the report period (probably because of plutonium rework), but rapidly decreased to an average of less than 0.04 per cent for the remainder of the report period.

Little change was noted in the performance of the Final Plutonium Cycle. The activity of the product (2BP) stream showed much less fluctuation than normal. This was probably a result of maintaining a lower and more constant acidity in the 2A Column.

Performance of the Final Uranium Cycle continued at a marginal level, with the Zr-Nb decontamination factor typically about 500. The Zr-Nb activity of the product stream was well within limits for silica gel treatment, but ruthenium contamination continued to be a problem. Also, because of the flood in the 2D Column, the plutonium contamination in the uranium product was above specifications for a short time. While most of it was "blendable", it was necessary to segregate for batches for eventual rework. Another brief excursion in the plutonium contamination occurred at the end of the report period after a relatively large quantity of fresh solvent was added to the system at an above-normal rate. A brief capacity test in the Final Uranium Cycle resulted in the 2D Column flooding at a CF of 4.4. The flood occurred during a relatively fast increase in rates and while the 2DF

acidity was low. However, the 2D Column appears to be much more sensitive than it was eight months ago, indicating a possible problem with column internals. It is planned to continue the capacity test in the Final Uranium Cycle as scheduling permits.

The Neptunium Recovery Cycle was started up the first of the report period and the accumulation has been normal. Neptunium losses from the system averaged ten and five per cent via the HAW and 2EU, respectively.

## VI. Product Treatment

### A. Plutonium Purification (N-Cell) - R. W. Lambert

Between June 17 and June 19, the resin push pressure increased from 40 psig to 70 psig while the push time increased from 60 seconds to 90 seconds. Operation was continued under these severe conditions until June 22 when processing of nonrepresentative metal was begun affording an opportunity to strip out and change the resin. The 2BP Stream was diverted to the L-Cell Package during the resin change. Operation of N-Cell following the change was satisfactory with push pressures running about 25 psig.

### B. Uranium Silica Gel - G. A. Nicholson

The silica gel unit was regenerated on June 19 after about 125 tons of uranium had been processed. The Zr-Nb activity in the product prior to regeneration was 30 uc/gal. The regeneration was completed and normal operation commenced early on June 21.

### C. Neptunium Purification - S. M. Nielson

On June 17, the out-of-specification material produced during the first hot run was blended with the third batch bringing the total to 1179 units. The mixture was then butted to flowsheet specifications for resin loading.

On June 18, the feed was started to the ion exchange column at flow-sheet rate. A leak developed inside the maintenance hood at the remote head of the feed pump. A total of 266 units was collected in Sump Tank Q8. The leak was apparently caused by a loose flange. The material in Q8 was reconcentrated and butted and was loaded on the resin on June 21.

## VII. Solvent Treatment - G. J. Raab

The No. 1 Solvent System has operated normally with the exception of the IOF valve limiting solvent flow to about 3.5 CF. Water flushing the IOF valve restored flow temporarily. The activity level of ICW was  $5 \times 10^3$  and  $1 \times 10^3$  uc/gal Zr-Nb and Ru, respectively. The solvent feed tank sampler (G5) was inoperative.

The No. 2 Solvent System ruthenium level has remained high for the period (110 - 145 uc/gal). About 150 gallons of TK-R8 solvent was jettied to TK-R1 on June 20. The remainder of the TK-R8 inventory will be moved to the solvent system after shutdown.

VIII. Acid Recovery and Waste Concentration - R. W. LambertF-Cell Acid Recovery

On June 21, the E-F6 boil-off rate was decreased about 15 per cent (~ 57 gpm to ~ 50 gpm) to test the possible effect on overhead entrainment.

IX. Waste Treatment and Storage - R. C. ForsmanDenitration

A slight plug in the IWV flow pot resulted in a greater acid kill than desired. The acid concentration of the formaldehyde treated waste was 0.37 M (0.5 M desired) causing minor Sp.Gr. and Wt.Fac. dip-tube plugging due to solids precipitating at low acidity. Normal IWV rates were attained after flushing with water; however, failure of the reactor thermohm necessitated shutdown of the unit on June 20.

Denitration of IWV using sugar in TK-F16 will continue until plant shutdown or until the thermohm is replaced on the formaldehyde reactor.

X. Fission Product Purification - J. B. Kendall

A batch of normal Purex oxalate waste containing about 310 kilocuries of cerium and 25 kilocuries of promethium was transferred to the SSW for oxidation and extraction of the cerium.

The 1000 liter aqueous phase at 1.15 M  $\text{HNO}_3$  and 0.09 M  $\text{NaBiO}_3$  was contacted with 1500 liters of new solvent for 40 minutes and settled for 30 minutes. About 85 per cent of the cerium and about 10 per cent of the promethium were extracted. Since the quantity of the cerium extracted was expected to be  $\approx$  95 per cent, the cerium extraction efficiency may have been reduced by residual oxalate in the feed.

The quantity of lead in the feed was only 3300 grams (16000 grams expected), and the quantity of lead in the rare earth product fraction was 2100 grams.

About 19 curies of ruthenium was present in the feed. However, less than three curies of ruthenium were found in the off-gas scrub solution following the oxidation-extraction operation.

XI. Miscellaneous - M. R. SchwabWater Treatment

The potassium permanganate ( $\text{KMnO}_4$ ) addition system in the 283-E Filter Plant was operated during the report period and all equipment and control procedures proved satisfactory. During the test period, the concentration of  $\text{KMnO}_4$  in the Filter Plant feed was varied from 0.3 ppm to 1.0 ppm. During the addition of  $\text{KMnO}_4$ , normal prechlorination was discontinued.

Samples taken during the test period indicated that the manganese content of the Filter Plant effluent remained well below the recommended limit of 0.05 ppm. Sample results for chemical oxygen demand tests were inconclusive in determining the amount of organic material destroyed by the  $\text{KMnO}_4$ . During the tests, definite pink color was detected in the flash mixer and flocculators with the color line barely creeping into the settling basin during pink additions.

This document consists of No. 1 of 11 copies. Series A.

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - OF Hill
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - PR McMurray
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MF Walling
  - 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 6/24/63 TO 2400 ON 7/1/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.4 (actual 3.45) CF until June 28, when it was shut down under emergency conditions due to a loss of process water flow. Start-up was commenced about two hours later and normal processing continued until June 30, when the scheduled shutdown commenced. The HAF was shut off at 1330 on June 30 after two batches of "cold" uranium had been recycled to purge the plutonium and activity from the system. Shutdown of the entire system (except for the Neptunium Recovery Cycle) was completed by 1500 on July 1.

Rework of the remaining hydrolyzed waste solution in Tank F8 and a batch of hydrolyzed waste solution in the spare concentrator was concluded during the run period.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.*</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.8	3.9			.002	.03	2.5	23%
Final	3.1	3.0	6.0	5.0	.002		3.0	91%
Ion Exch.		1.4		0.15				
Overall	6.9	8.3	6.0	5.2	.004	.03	5.5	114%

B. Production

- 1. HAF = 161.5 T
- 2. U Production = 202.4 T
- 3. Pu Production = 143.3 Kg
- 4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	491 Lbs	.12
2. Pu	162 Gr	.11
3. Np, F16, F18	84 Gr	10.0*
K6	12 Gr	1.5*
4. Solvent	1411 Gal	0.1

\*Based on Input + Rework.

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	1270	0.3
2. Pu	3511	2.4
3. Np (From F11)	175	21.8*

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	250	104-A
CD	88	105-A
FP	151	105-A
IWW	10	105-A
CW	300	106-BX

F. IWW Flows

5.4                      Avg. HNO<sub>3</sub> Conc. - 5.7 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-Off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.39	132
102-A	1.76	123
103-A	0.21	86
104-A	1.40	136
105-A	9.28	92
106-A	9.80	128

III. Flowsheet - G. A. Nicholson

- (a) On June 25, the 2DF-ferrous sulfamate was reduced from 0.35 to 0.23 flows. It was increased back to 0.31 flows on June 26 and to 0.35 flows on June 27.
- (b) On June 29, the Neptunium Recovery Cycle was shut down and the 3A Column stripped and emptied to replace the interface float. The cycle was started up on June 30 under Phase II conditions.

IV. Feed Preparation - G. J. Raab

Dissolver operation was good. The dissolver heels were dissolved out at the end of the unclassified metal run, with all dissolvers down on June 28. The I<sub>131</sub> emission for the period was 0.48 curies. The metal age was 122 to 481 days. After the last coating waste was sent to UGS, the line and TK-D2 were flushed with 15 per cent caustic. Silver reactor, T-B2, was given a routine regeneration after dissolving was complete; 946 tons had been dissolved since the last regeneration.

\*Based on Input + Rework.

**V. Solvent Extraction - G. A. Nicholson**

Solvent-extraction performance during the report period showed considerable improvement over the previous period. The emergency shutdown on June 28 and the subsequent start-up resulted in a moderately severe activity burst - especially in the Final Plutonium Cycle. Except for the burst, First Cycle performance was generally very good. Both the HAW losses and activity were somewhat higher than the low values of the previous week, because of minor upsets in the HA Column, but were still about two-fold lower than typical of recent weeks.

Performance of the Final Plutonium Cycle continued essentially unchanged from the previous week. The activity burst quickly dissipated and the activity of the final product stream returned to normal.

Performance of the Final Uranium Cycle showed a substantial improvement during the report period. The activity of the uranium product was well within silica gel treatment limits and several batches were within shipment specifications without silica gel treatment. Plutonium contamination in the uranium product continued to be a problem of major concern in spite of a 2EF ferrous sulfamate addition rate two-fold above the flowsheet value.

Neptunium accumulation in the Solvent-Extraction System was improved during the report period. Instantaneous losses averaged about 8 per cent - two-fold lower than normal - and apparent accumulation was greater than 100 per cent of the input. This discrepancy was due, undoubtedly, to a combination of analytical and volume variances and rework of hydrolyzed waste solution. The temporary shutdown of the Neptunium Recovery Cycle (to replace the 3A Column Interface Float) was accomplished without losing a significant amount of the neptunium which had been accumulated in the "Package". At the end of the report period, a batch of about 1200 units had been accumulated and decontaminated and was ready for transfer to the Purification Cycle.

**VI. Product Treatment****A. Plutonium Purification - N-Cell - R. W. Lambert**

Operation of N-Cell was good during the report period. After being in use 11 days, the resin showed no signs of causing pushing problems with push times and push pressures remaining low. Product quality during the period was good with the exception of the final non-representative loadout. This batch reflected a short activity burst in the 2BP Stream near the end of the run.

**B. Uranium Silica Gel - G. A. Nicholson**

Normal operation of the Silica Gel Facility commenced on June 25 and continued through June 28. About 100 tons of uranium were processed before the facility was shutdown for regeneration. The Zr-Nb activity in the product stream prior to regeneration was 25 uc/gal. The regeneration was commenced at this relatively low activity level because of the high ruthenium contamination in the product (~ 100 uc/gal) and the necessity to maintain as low a total activity as possible in the product.



VII. Solvent Treatment - G. J. Raab

The No. 1 Solvent System was limited by IOF valve flow for the entire period. A portion of the solvent was jetted to the Turbomix Tank (G7) bypassing the IO Column in order to maintain rate. The activity level, however, was still about normal in the ICW Stream ( $4.0 - 6.0 \times 10^3$  and  $1.0 - 3.5 \times 10^3$  uc/gal Zr-Nb and Ru, respectively).

The No. 2 Solvent System operated normally. The ruthenium activity level dropped by a factor of two to 43 uc/gal. This may reflect the cold metal recycle and longer cooled unclassified metal feed.

VIII. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

On June 25, the IWW Concentrator, E-F6, boil-off rate was returned to its normal tower limited rate of about 57 gpm. Sample results indicated that the 15 per cent reduction in boil-off rate during the test did not noticeably reduce the activity entrained into the E-F6 overheads.

On June 26, antifoam was started into E-F6 via the IWF Stream. The antifoam addition rate was set so that the IWF contained 5 ppm of Dow-Corning Antifoam B. The conclusion reached from the test was that antifoam does not appreciably reduce the amount of overhead entrainment in E-F6. Although the test was marred by inconsistent antifoam flows and erratic F-Cell operation, it was felt that the test results were valid and further antifoam tests would be of little value.

IX. Waste Treatment and Storage - R. C. Forsman

Denitration

Installation of a vent jumper connecting the F16 Tank to the F24 condensate receiver tank, thus placing F16 under condenser-vent vacuum, resulted in greater than 5 inches (water) vacuum at F16. Tests are being conducted to increase sugar addition rates for shorter time cycles in the denitration process.

X. Miscellaneous - M. R. Schwab

Water Treatment

The conductivity meter installed last May has indicated that the Amberlite IRA-45 (weak base) resin in the No. 3 anion unit is not performing as desired. The IRA-45 appears to be sensitive to fluctuations in flow and the overall capacity of the resin appears to have decreased since installation. The IRA-45 resin produces greater than 100,000 ohm water for less than three hours after regeneration with the average for the run being between 40,000 and 25,000 ohms depending upon the flow through the bed.

The Duolite-A30R (intermediate base) resin in the No. 2 anion unit has produced 125,000 ohm water for periods exceeding the normal expected capacity.

**DECLASSIFIED**

HW-76912

**-108-**

The total water flow through the anion units has been greater than the 200 GPM prescribed with the No. 3 anion unit taking a major portion of the total flow. These conditions may have caused the observed decrease in the capacity of the IRA-45 resin. Further comparative capacity studies will be made when the new Rohm & Haas XE-225 resin is installed in the No. 1 anion unit.

The No. 1 anion unit has been out of service during the current run period awaiting the arrival of the XE-225 resin.

This document consists  
of No.      of 11 copies.  
Series A.

-109-

## Distribution:

1 - OF Beaulieu  
2 - WS Frank  
3 - OF Hill  
4 - BF Judson  
5 - RW McCullugh  
6 - PR McMurray  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 7/2/63 TO 2400 ON 8/18/63

I. General - G. A. Nicholson

The plant was shut down during the report period except for operation of the Neptunium Recovery and Final Uranium Cycles for a brief period immediately following the normal shutdown. The Neptunium Recovery Cycles were operated for seven days to remove the uranium and neptunium from the Backcycle Waste (3WB) Solution. The Final Uranium Cycle was operated for six days to rework the off-standard uranium product which had accumulated during the run period.

The plant was started up on August 6 with "cold" feed starting at 2150 and "hot" HAF starting at 0700 on August 7. However, failure of the HA Interface Float Jumper permitted the column contents to drain to the sump and necessitated an emergency shutdown of the First Cycle and normal shutdowns of the Final Plutonium and Final Uranium Cycles on August 8.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

-- None --

B. Production

1. HAF = 21.6 T  
2. U Production = 113 T  
3. Pu Production = -0-  
4. Np Production = 3333 (Shipped)

C. Overall Loss

	Units	% of <u>Total Production</u>
1. U	3250 Lb.	
2. Pu	670 Gr	
3. Np, F16, F18 K6	301 Gr 8 Gr	
4. Solvent	22,613	

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	109 T	
2. Pu	986 Gr	

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal</u>	<u>Waste Tank</u>
OWW	142,473	101-A
-	14,076	102-A
-	88,046	104-A
Cell Dr	125,425	105-A
FP	26,451	105-A
Flush	52,073	105-A

F. IWW Flows

-- None --

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	2.0	121
102-A	1.3	95
103-A	0.2	102
104-A	1.6	135
105-A	13.0	93
106-A	8.0	127

III. Flowsheet - G. A. Nicholson

-- None --

IV. Feed Preparation - G. J. Raab

During the plant outage, the A3 Dissolver chute was removed and the gasket was replaced. However, there are not any studs to hold the chute down; only the weight and dowel pins hold the chute in place. The vacuum on the A3 System is only about 5 inches and this drops to 2 to 2.5 inches at dissolving conditions.

The silver reactors as of 7/15/63 had the following use history:

<u>Dissolver</u>	<u>A3</u>	<u>B3</u>	<u>C3</u>
Tons Since Last Regeneration	493	0	1984
Regenerations Since Flush (Including Regeneration when Flushed)	1	3	1

The dissolvers were started up in anticipation of plant startup on 8/1/63. The tunnel was badly contaminated from a burial and delivery of metal was hindered. The coating waste line functioned normally.

V. Solvent Extraction - G. A. Nicholson

Rework of about 110 tons of uranium, which had been segregated during the previous run period because of excessive plutonium and ruthenium contamination, was completed successfully. While the instantaneous 2DW losses were generally less than 0.10 per cent, the cumulative losses were sufficient to warrant saving the concentrated 2DW for later recovery. The uranium product from the rework operation was within shipping limits after silica-gel treatment.

The Neptunium Recovery Cycle was operated for 24 hours under Phase III Flowsheet conditions, prior to the uranium rework operation to transfer about 1200 units of neptunium, accumulated and decontaminated during the past operating period to the Neptunium Purification Cycle. The "Package" was started up again on July 5 and operated for five days to recover the uranium and neptunium from the Backcycle Waste Solution. After decontamination, about 600 units were transferred to the Purification Cycle. Process performance was generally excellent. Failure of the interface float necessitated a temporary shut down and empty-out of the 2A Column, but negligible losses resulted.

After the regular shutdown on July 1, the HS Column was emptied and water-flushed in preparation for the scheduled cartridge replacement. Following the operations discussed above, the IC, 2D and 2E Columns were emptied and flushed in preparation for cartridge inspection and/or replacement. The HS, IC and 2E Cartridges were in good condition. The IC Cartridge was reinstalled and the HS and 2E Cartridges were removed and replaced before startup as previously scheduled. Many of the plastic plates in the 2D Scrub Cartridge were broken and a considerable amount of small broken pieces was found in the upper disengagement section of the 2D Column, in the 2DU overflow line and 2E Column organic distributor. The cartridge was replaced by one of similar design, but containing Fluorothene, rather than linear polyethylene plastic plates. Preliminary investigation indicates the problem to be one of embrittlement caused by the continuous flexing of the plates in the TBP solvent rather than radiation in nature.

During preliminary preparations for startup of the plant on August 2, the ICU Concentrator was found to be filled with a mixture of aqueous UNH and badly degraded solvent. Apparently the IC Column was operated without an interface for a considerable time during the uranium recovery operation in J Cell Package. The interface detector was "seeing" the liquid level rather than the organic-aqueous interface; the organic Sp.Gr. was reading zero, indicating the dip tubes were not submerged, and the static pressure was low. As a result, about 5000 gallons of solvent were routed to the ICU Concentrator along with the 2EU Stream. All of the diluent was evaporated and the TBP was simmered for two to three weeks in the UNH-HNO<sub>3</sub> environment.

The badly degraded "goop" appeared to be a stable emulsion, but contact with sodium carbonate solution separated the phases. Contact of this

[REDACTED]

two-phase mixture with nitric acid resulted in the formation of a gelatinous, or rubbery, insoluble material. Contact of the carbonate-treated mixture with caustic, however, precipitated the uranium as the di-uranate and this method was used to recover the uranium from the organic. Uranium losses by this method averaged about 20 per cent.

The mixture in the ICU Concentrator was transferred to the 20W (R8) Tank via the 2DF and 20F Tanks. The concentrator was then flushed with 2.5 per cent carbonate solution and water. The flushes were routed the same way until the tank was full; the remainder was routed from the 20F Tank to the 10W Tank (G8).

The above flush was not sufficient to remove the "do-bads" from the system as the Final Uranium Cycle, the No. 2 Solvent System, and eventually the No. 1 Solvent System were all grossly contaminated and it was ultimately necessary to discard several thousand gallons of solvent due to emulsification with the aqueous scrub and inability to clean it up. To complicate the picture further, the interface float on the HA Column became loose and allowed the column contents to drain to the cell floor. The uranium, plutonium and neptunium were recovered from this solvent by successive carbonate washes and the solvent was discarded. In all, about 20,000 gallons of solvent, 3200 lbs. of uranium, 420 grams of plutonium and 280 grams of neptunium were lost during the scheduled shutdown period and subsequent recovery operations. After the uranium recovery from the solvent was completed, the Backcycle Waste Solution (~ 8500 gallons) which was suspected to contain "do-bads" was routed through the IWW Concentrator for acid recovery. The bottoms were held with other solutions containing product for hydrolysis and eventual rework. Following completion of the above manipulations, startup preparations were commenced and the plant was started up on August 18 with cold HAF on at 1840 hours.

## VI. Product Treatment

### A. Plutonium Purification - N-Cell - R. W. Lambert

During the report period, all screens in the ion exchange unit were inspected and found to be free of defects. On July 25, new 20-40 mesh resin was added to the unit and it was spun in preparation for startup. Several minor leaks, defective valves and defective air cylinders were replaced.

### B. Uranium Silica Gel - G. A. Nicholson

Operation of the Silica Gel Facility continued during the entire month of July. The unit was regenerated three times during the month and a total of ~ 600 tons of uranium were processed. Much of the material processed was not within shipment limits after the first pass and was processed a second time.

### C. Neptunium Purification - S. M. Nielson

Run #3 was completed with the elution of 1341 units on June 28. The product was well within shipping specifications.

Phase III was again started on July 1 but a plugged 3BN jet necessitated reverting back to Phase II. On July 3, the 3BN jet was replaced and Phase III began at 0400. Approximately 1300 units were transferred. Due to the lack of plutonium in the feed, no plutonium scrub was made. The fission product scrub began on July 5. The product eluted on July 6 was high in ZrNb by a factor of 2. All other shipping specifications were met. The product, totaling 1173 units, was loaded out and shipped with Run #3 product on July 8.

On July 11, the plant inventory which had been decontaminated in the J-Cell Package was sent to Q Cell and concentrated. Due to poor decontamination in the J Cell Package, the ZrNb levels were a factor of 100 high. This material containing 641 units, including 53 units left from Run #4 Precut, was loaded on the resin. Approximately 750 units of Redox neptunium were then transferred from shipping cans to Q3 and loaded on the resin. The Redox neptunium was a factor of approximately 15 above specified ruthenium levels and a factor of 1.7 above ZrNb level.

During the wash steps, the high amounts of fission products caused excessive radiation in the maintenance room of Q Cell. Radiation levels reached 3 r through the glove-port at the feed pump, 700 mr at the CIA horn and 100 mr at the maintenance room door. These levels decreased as soon as the scrub waste was transferred to J1. By August 6, the radiation levels in the maintenance room were less than 6 mr.

On eluting the resin after standard scrubs, the ZrNb level in the product was a factor of about 10 high. The product was returned to Tank Q3 and butted to feed specifications and rerun using standard scrubs. The product eluted on July 31 was within shipping specifications. Only 819 units were produced from the run making a total recovery for the run of 59 per cent. The majority of the loss occurred during the first fission product scrub.

Plans are to flush the Q Cell equipment as soon as sufficient storage of flush material is available in the Purex Backcycle System.

#### VII. Acid Recovery and Waste Concentration - G. J. Raab

During the July outage, the recovered acid header was flushed with 5% oxalic - 3% nitric acid, water and 5% caustic - 2% tartaric acid flushes. The Vacuum Fractionator was not flushed; only TK-U1 was used for sending the flush through the system valve and acid meters to temporary hose drop legs into the cell. The pressure reducing valve and the Cuno filter were replaced. The Cuno filter which was installed had a 5 inch long by 2-1/2 inch diameter cartridge with leaf spacing of 0.008 inches giving an equivalent of 70 mesh size screen. The cartridge holder will accommodate a 10 inch cartridge but the only one available for immediate use was the 5 inch cartridge.

#### VIII. Waste Treatment and Storage - R. C. Fcrsman

Plutonium extraction from Purex IWW by trilaurylamine was successfully demonstrated in plant-scale tests. Although the concentration levels of neptunium and plutonium in the available feed batch were too low to

accurately determine plutonium recovery efficiency, data indicated approximately 60 per cent of the plutonium was recovered from 4900 gallons of Purex IWW. Laboratory contacts on the plant IWW and tri-lauryl amine used in the test indicated greater than 90 per cent of the plutonium should be recovered. Future tests will determine if poor phase mixing in plant equipment or analytical and sampling difficulties due to low product concentrations were the reason for low recovery efficiencies in the plant.

IX. Solvent Treatment - G. J. Raab

After the plant shutdown, the No. 1 Solvent System was spun from time to time to provide solvent for the neptunium recovery run. During the neptunium recovery run, 5100 gallons of solvent disappeared from the No. 1 Solvent inventory due to sending solvent to ICU Concentrator. The No. 1 Solvent System suffered no loss of quality until the first attempt to start up the plant on August 6 when the contaminated 3WB flow was started to the HA Column. Typically, the plutonium retention August 1 was  $3.84 \times 10^8$  and on August 8, it was  $1.30 \times 10^{10}$ . After the forced plant shutdown, the solvent system was spun until the August 18 startup, the plutonium retention was  $4.48 \times 10^8$  at this time. The G3 centrifuge failed to start on August 13 so it was bypassed; the flow now goes from T-G2 to TK-27. The centrifuge has not responded to attempts to start it and appears to have frozen bearings or a bind on the bowl at the lip. The IOF Tank, the IOF valve and rotameter were flushed out during the outage and full flow was restored to the system.

The No. 2 Solvent System was operated to process the UNH stored in TK-P2. During this operation, the solvent became highly contaminated with crud from the material in TK-P2; a typical plutonium retention value was  $1.95 \times 10^{11}$ . Continued spinning through the solvent recovery system reduced the plutonium retention value to  $6.59 \times 10^7$ . Tank R1, Column R2 and Tank R2 were flushed with oxalic and nitric acids to remove the excess  $MnO_2$  deposited during the above operation. The degraded solvent which had been stored in Tank R5 had aged to the point where it was usable. However, some solvent salvaged from the sump was mixed with the Tank R5 solvent to make Tank R8 available to store the degraded solvent from the J8 Concentrator. The very thick syrupy degraded solvent from E-J8 was routed to Tank R8 via Tank K1 and Tank R1. This solvent mass plus carbonate washes proved to be unusable as solvent and was eventually recovered for its uranium value in the head-end facilities ordinarily used for fission product recovery. When the Final Uranium Cycle was started on August 7, the uranium introduced via 2EW losses started showing in the No. 2 Solvent System. The centrifuges plugged up and the solvent supply ran out in the 2EX Tank forcing the Final Uranium Cycle to shut down. The solvent was washed with 1 volume 2-1/2 per cent sodium carbonate to 5 volumes of solvent until the solvent contained less than 0.01 lb. uranium per gallon. Then the solvent was spun through the system. The loss of solvent was so great during the washing that the solvent previously stored in Tank R5 was introduced into the system and spun into the inventory. This solvent appeared to clean-up the system as well as the regular inventory.

During the shutdown period July 1 to August 18, a total of 19,100 gallons of solvent was discarded or lost in washes and maloperation.



X. Miscellaneous

A. Water Treatment - M. R. Schwab

During the shutdown period, the following work was done on the Purex water demineralizers:

1. The WBR resin in No. 1 Anion unit was replaced with Amberlite XE-225 weak base resin and the new resin pretreated.
2. The No. 2 Anion unit containing Duolite A-30B resin was inspected and although the resin was about four inches below the desired level, no additional resin was added.
3. The No. 3 Anion unit containing IRA-45 was inspected and found to be short about 12 feet of resin (~ 30% low). The bed was restored to normal depth by adding some WBR resin already on hand.
4. Equipment for measuring and recording the effluent flow from each of the three anions was installed and put into service.

B. Cribs - R. W. Lambert

While the plant was shut down, a four inch orifice was installed in the line from the Vacuum Fractionator to the A9 Crib. Experience during the May-June run period indicated that the crib could only handle a portion of the tailwater flow and thus the orifice was calculated to allow about 200 gpm to go to the crib.

This document consists of No. 1 of 11 copies. Series A.

~~TOP SECRET~~  
-116-

DECLASSIFIED

HW-76912

Distribution:

- 1 - OF Beaulieu
- 2 - WS Frank
- 3 - OF Hill
- 4 - BF Judson
- 5 - RW McCullugh
- 6 - PR McMurray
- 7 - SG Smolen
- 8 - RE Tomlinson
- 9 - AJ Waligura
- 10 - MT Walling
- 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 8/19/63 TO 2400 ON 8/25/63

I. General - G. A. Nicholson

The plant was started up on August 18 with "cold" HAF on at 1840. Recycling of cold feed through the First Cycle continued until August 20 when the Final Uranium Cycle was started. "Hot" feed was started to the HA Column at 1340 on August 20 and continued at a nominal 3.2 CF rate until August 24, when the plant was shut down to change out the 2DF Flow System (pump, rotameter, and valve) which had plugged up. Normal processing was resumed on August 25, with HAF on at 1510, at a nominal 3.2 (actual 2.8) CF.

Rework of hydrolyzed sump and miscellaneous solutions commenced on August 22 at a nominal rate of 200 gallons per batch of HAF.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.8	4.1			.002	.02	2.5	90%*
Final	3.2	3.1	7.0	5.3	.002	.	1.5	
Ion Exch.		1.1		.04				
Overall	7.0	8.3	7.0	5.3	.004	.02	4.0	90%

B. Production

- 1. HAF = 109.4 T
- 2. U Production = 93.8 T
- 3. Pu Production = 40.4 Kg
- 4. Np Production

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	574 Lbs	0.3
2. Pu	230 Gr	0.5
3. Np, F16, F18	4 Gr	1.0 )% of Total
K6	3 Gr	>1.0 )Input
4. Solvent	2967**	

\*% of Total Input.

\*\* ~ 1600 Gals Retained in R8 & is Recoverable.

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	8.6 T	9.1
2. Pu	2788 Gr	6.9
3. Np	155 Gr	64 (% of Virgin Feed)

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	219	104
	466	106
Cell Dr	81	105
Flush	39	105
CW	300	106-BX

F. IWW Flows

Data Inconclusive

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	2.1	123
102-A	1.3	116
103-A	0.2	86
104-A	1.6	138
105-A	13.0	93
106-A	7.0	127

III. Flowsheet - G. A. Nicholson

(a) The plant was started up under the same flowsheet conditions existing at shut down, except for the following:

- (1) The HAO was 80 rather than 73 flows.
- (2) The HSS was 80 rather than 56 flows.
- (3) The IBX was 20 rather than 17 flows.
- (4) The IBXP-HNO<sub>3</sub> was 10 rather than 7 flows.
- (5) The IBX was 24 rather than 17 flows.
- (6) The 2AS was 4.5 rather than 3.0 flows.
- (7) The 2A pulse frequency was 64 rather than 89 cycles/minute.
- (8) Initially, the IUC was routed back to the HAF Tank.
- (9) When the Final Uranium Cycle was started up, the 2DS was 41 rather than 48 flows.
- (10) The 2BP flow was left off until "hot" feed was started to the HA Column.

(b) On August 20, the 2A pulse frequency was increased from 64 to 72 cycles per minute. It was increased to 84 cycles per minute on August 21.

(c) On August 20, the 2BP was started at 2.6 flows.

- (d) On August 24, the First and Final Plutonium Cycles were shut down at equilibrium. The Final Uranium Cycle was shut down and the 2D Column was stripped and emptied.
- (e) Normal processing was resumed later the same day under the same flow-sheet conditions existing before the shutdown.

#### IV. Feed Preparation - G. J. Raab

Dissolving started again on August 21. The dissolvers were not pressed to capacity as the plant rate was low and no 15 bucket charges were made. The coating waste jet to UGS has operated well. Metal age varied from 124 to 187 days. The I<sub>131</sub> emission was 0.33 curies for the period.

#### V. Solvent Extraction - G. A. Nicholson

The solvent-extraction performance picture has been considerably clouded since startup by the flow control problems with the HAF, 3WB, 2AX and 2DF Streams and the temporary shutdown. Continuing improvement has been apparent, however, with losses very low since startup. First Cycle performance has been generally very good with product (HSP) activity about 50 per cent lower than normal and waste losses averaging less than 0.03 per cent. Partition performance has also been excellent with plutonium carryover in the IBU Stream about 80 per cent lower than normal. This is probably due to the lower processing rate and increased aqueous-to-organic flow ratio in the IBX Column.

Performance of the Final Plutonium Cycle has been excellent. Since steady-state operation was reached about August 21, the D.F. has averaged about 2000 and a severe burst from the First Cycle late in the report period caused only a temporary two-fold increase in the product activity. Losses to backcycle were about two-fold higher than normal during the report period due to varying 2AF acidity and 2AX flow problems, but no adverse effects resulted.

Performance of the Final Uranium Cycle has shown considerable improvement with the new scrub cartridge with the D.F. averaging about 1000 in spite of the upsets due to the erratic 2DF flow. Contamination of the final product with plutonium and Zr-Nb was high immediately after startup, but decreased rapidly throughout the report period. The shutdown and subsequent startup on August 24 produced another burst of plutonium contamination in the product. However, this was declining rapidly and the gamma activity of the product was within shipping limits without further treatment by the end of the report period.

Neptunium accumulation in the plant was considerably above normal during the report period. Losses totalled less than five per cent, including the extra neptunium added with the rework. Estimated accumulation based on samples of the 3WB verified the low losses.

#### VI. Product Treatment - R. W. Lambert

##### A. Plutonium Purification (N-Cell)

During the first part of the run period, N-Cell was run-in and several leaks were detected and fixed. Plutonium was routed to N-Cell on

August 20 and the unit performed well the remainder of the report period with all product within specifications.

B. Uranium Silica Gel - G. A. Nicholson

The Silica Gel unit was operated for about two days, then shut down for lack of feed.

VII. Solvent Treatment - G. J. Raab

The No. 1 Solvent System activity level went down very fast after plant startup as shown in values below:

<u>Date</u>	<u>Time</u>	<u>Pu Ret.</u> <u>AT/Gal.</u>	<u>ZrNb</u> <u>Uc/Gal.</u>	<u>Ru</u> <u>Uc/Gal.</u>
8/19/63	0105	1.01 <sup>9</sup>	2370	853
8/19/63	1300	9.29 <sup>8</sup>	1170	289
8/20/63	0910	9.38 <sup>8</sup>	643	206
8/24/63	1300	-	445	714

The No. 1 Solvent System is operating without the use of centrifuge.

The No. 2 Solvent System had been spinning several days before startup and reached a Pu retention value of  $1.41 \times 10^8$  by 8/19/63; by 8/22/63, it had improved to  $5.66 \times 10^7$ . The loss of solvent from the system to the No. 1 System via solvent washes was about 390 gallons per day which is higher than normal.

VIII. Miscellaneous - R. W. Lambert

Cribs

On August 21 after only being in use for six days, the A9 Crib liquid level reached its maximum limit and it was necessary to blank off the line carrying the fractionator tail-water to the crib. The crib filled in spite of the orifice which was installed in the line to limit the flow to only about one-third of the total tail-water flow. With the A9 Crib being blanked, the entire tail-water flow was again routed to the Purex Swamp via the Chemical Sewer Line.

This document consists of No. 1 of 11 copies. Series A.

-120-

Distribution:

- 1 - OF Beaulieu
- 2 - WS Frank
- 3 - OF Hill
- 4 - BF Judson
- 5 - RW McCullugh
- 6 - PR McMurray
- 7 - SG Smolen
- 8 - RE Tomlinson
- 9 - AJ Waligura
- 10 - MT Walling
- 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 8/26/63 TO 2400 ON 9/1/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 2.6 (actual 2.7) CF during the report period. Rework of hydrolyzed sump solution continued until August 29. The Neptunium Recovery Cycle was started up on August 30 under Phase I flowsheet conditions.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	4.0	4.3			.002	.02	7.3	108%
Final	8.8	3.4	7.5	2.7	.002		3.9	
Ion Exch.		1.1		.03				
Overall	7.3	8.8	7.5	2.7	.004	.02	11.2	108%

B. Production

- 1. HAF = 144.7 T
- 2. U Production = 151.4 T
- 3. Pu Production = 72.6 Kg
- 4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	792 Lbs	0.26
2. Pu	406 Gr	0.56
3. Np, F16, F18	11.3 Gr	2.5 )% of
4. K6	9.4 Gr	2.1 )Input
4. Solvent	201 Gain	.02

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	4020 Lbs	1.3
2. Pu	470 Gr	0.6
3. Np	157 Gr	

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	391	106-A
C.D.	95	105-A
FP	61	105-A
Flush	9	105-A
IWW	19	105-A
C.W.	300	106-BX

F. IWW Flows

6.0                      Avg.  $\text{HNO}_3$  Conc. = 4.75 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	2.06	123
102-A	1.3	107
103-A	0.2	86
104-A	1.6	137
105-A	13.0	93
106-A	7.0	126

III. Flowsheet - G. A. Nicholson

- (a) On August 26, all First Cycle streams, except the IBS, were reduced from nominal 3.2 to 2.6 rates. The IBS was left at the same rate, or at 30 flows at a nominal 2.6 CF.
- (b) On August 28, the IBXP was reduced from 24 to 20 flows. It was increased back to 21 flows on August 29.
- (c) On August 28, the IBXP- $\text{HNO}_3$  was reduced from 13.4 to 10 flows. It was increased back to 12.3 flows on August 31.
- (d) On August 27, the 2DS was reduced from 53 to 47 flows.
- (e) On August 27, the 2DF-ferrous sulfamate was reduced from 0.31 to 0.22 flows. It was increased back to 0.28 flows on August 29, then reduced again to 0.22 flows on August 31.
- (f) On August 26, the 2AS was increased from 5.1 to 5.7 flows.

IV. Feed Preparation - G. J. Raab

One dissolver holding most of the time; only one 15-bucket charge made to improve time cycle. Metal age varied from 128 to 202 days. The  $\text{I}_{131}$  emission was 1.60 curies for the period. The rate of emission was higher than it should be for the age of the metal charged.

DECLASSIFIED

HW-76912

V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance during the report period has been excellent. Waste losses have continued to average lower than during the previous run period ( 0.03 per cent) and product purity has been excellent. Plutonium carryover in the IBU Stream continued at a level about five-fold lower than the previous run. The reduction in IBXP flow on August 28 was followed by a two-fold increase in plutonium carryover, but the new level represented only 0.2 per cent of the total in the HAF Stream. The plant processing rate was still limited by a flow restriction in the 2DF Stream.

Performance of the Final Plutonium Cycle continued at a high level, with losses to recycle averaging about normal (~ 2.5 per cent) and product (2BP) purity well below the limits for processing in the Anion Exchange Cycle.

Performance of the Final Uranium Cycle continued to improve during the first of the report period. The DF reached about 2500 and remained at this level for the last half of the run period in spite of numerous minor upsets when the 2DF control valve was exercised and the pump "bumped" in efforts to increase the 2DF flow. Plutonium and ruthenium contamination in the final product showed an increasing trend late in the run period, but was not severe and all uranium product met shipping specifications without further treatment.

Neptunium accumulation in the Solvent-extraction System remained above normal for the report period. However, losses via both the HAW and 2UC Streams increased by about two-fold over the previous report period. The increase in HAW losses occurred the last three days of the report period in plutonium losses, and were caused by flow control problems in both the 3WB and recovered acid addition (HSS-HNO<sub>3</sub> and HSR-HNO<sub>3</sub>) Streams. The 2UC losses were associated with difficulties in maintaining proper saturation in the 2D Column, due to the 2DF flow control problem. Accumulation in the Neptunium Recovery System since its start up on August 30 has been in doubt due to a faulty 3BN Sampler.

VI. Product Treatment

A. Plutonium Purification - N-Cell - R. W. Lambert

Plutonium quality improved during the run period reaching near record levels.

On August 27, the fluoride was turned off in N-Cell and remained off for the remainder of the report period. N-Cell DF, in spite of the low feed activity and absence of fluoride, remained unexpectedly high at between 10 and 15.

B. Uranium Silica Gel - G. A. Nicholson

The Silica Gel Unit was shut down during the report period due to lack of feed material.



DECLASSIFIED

HW-76912

-123-

VII. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

After reaching equilibrium conditions, the ZrNb activity in the AAA recovered acid (TK-F3) reached its normal single stage waste concentration level of about  $1.5 \times 10^4$  uc/gal. The ruthenium activity, however, reached only about one-half of its expected level and by the end of the report period was running about  $4.0 \times 10^3$  uc/gal. The IWW rate for the period was near normal running about 6.0 flows.

The lower ruthenium activity level may be the result of operating F-Cell with high IWF dilution made possible by the overall low plant operating rate. The high dilution rate decreases the E-F6 bottoms acid concentration and this in turn tends to reduce the amount of ruthenium volatilization.

**DECLASSIFIED**

Distribution:  
1 - OF Beaulieu  
2 - WS Frank  
3 - AM Platt  
4 - BF Judson  
5 - RW McCullugh  
6 - WM Harty  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 9/2/63 TO 2400 ON 9/8/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 2.6 (actual 2.6) CF rate during report period.

II. Performance Data - D. J. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.9	4.2			.002	.02	4.7	-19%
Final	3.5	3.4	5.5	1.3	.002		2.0	+66%
Ion Exch.		1.0		.04				
Overall	7.4	8.6	5.5	1.34	.004	.02	6.7	47%

B. Production

1. HAF = 150.7 T
2. U Production = 145.8 T
3. Pu Production = 93.8 Kg
4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	655 Lbs	0.2
2. Pu	386 Gr	0.4
3. Np, F16, F18	43 Gr	13.0
K6	9.3 Gr	2.8
4. Solvent	90 Gal	∟ 0.01

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	Nil	
2. Pu	1.5 Kg	1.7

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	102	104-A
OWW	192	106-A
CD	84	105-A
FP	120	105-A
IWW	6	105-A
CW-	300	109-B

F. IWW Flows

8.1 Avg. HNO<sub>3</sub> Conc. - 5.1 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	2.1	123
102-A	0.8	92
103-A	0.25	85
104-A	2.65	137
105-A	8.4	96
106-A	7.7	127

III. Flowsheet - G. A. Nicholson

- (a) On September 2, the 2D pulse frequency was increased from 71 to 72 cycles per minute. It was further increased to 74 cycles per minute on September 3.
- (b) On September 3 and again on September 5 and 6, the 2DW uranium loss dropped off to one to three per cent, vs. the normal control range of five to seven per cent.
- (c) On September 6, the 3WB HNO<sub>3</sub> concentration was decreased from 10 M to 8 M.
- (d) On September 6, the 2EX temperature was reduced from 55 to 52° C. It was further reduced to 50° C on September 7.

IV. Feed Preparation - G. J. Raab

On September 1, C3 dissolver was charged with 15 buckets of metal and when coats were removed, dissolving was started about 0800 September 2. The C3 I131 monitor, then the 293 Bldg. monitor went off-scale. Dissolving was stopped 35 minutes after steam was started by turning on full cooling water flow. The specific gravity leveled out at 1.55. The 100-C Area reported 3 buckets of 18-day old metal had been sent by mistake. The partial dissolved cut was jetted to TK-D4. TK-D4 had about 2600 gallon heel of good metal solution to which 10 pounds of mercuric nitrate had been added. The total volume in TK-D4 was 3990 gallons. Five more pounds of HgNO<sub>3</sub> was added to TK-D4 bringing concentration to .0013 M Hg<sup>++</sup>. The dissolver heel was covered with water and enough 50 per cent NaOH was added to make solution 5 per cent NaOH. The C3 dissolver and TK-D4 will remain out of service until the I131 has decayed sufficiently to process.

A total of 63.74 curies of I131 was emitted from the stack for the report period; 46.25 and 13.80 curies of the total were emitted on the day and day following the incident.

The remaining two dissolvers were routed to TK-D3 and were able to dissolve sufficient metal to meet the present plant capacity. One 15-bucket charge was made to improve time cycle.

V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance continued at an excellent level, with waste losses averaging less than 0.03 per cent and minimal contamination in both product streams. The 3WB (and, consequently HA Column) acidity was slightly above normal early in the report period because of an inoperable sampler. Also, on several occasions, the recovered acid flow to the HSS, HSR and IBXP was lost because of filter pluggage problems. However, neither of these conditions affected the process significantly. The plutonium carryover in the IBU stream remained about two to three-fold below normal.

Final Plutonium Cycle performance was excellent, with the DF averaging about 2500 despite lower-than-normal losses to the Backcycle System ( $\angle$  0.5 per cent) during the latter half of the report period. The final product (2BP) stream averaged only about three-fold above shipment limits, so was well below the limits for processing in the Ion Exchange Cycle.

Final Uranium Cycle performance continued at an excellent level during the report period. The flow impediment in the 2DF flow system continued to restrict the plant rate and periodic attempts to improve the flow by "bumping" the pump tended to increase the plutonium contamination in the final product. All product was within shipping limits without further treatment, however. The DF averaged greater than 3000 in spite of widely varying losses (1.5 to 10 per cent) to the Backcycle System. Plutonium contamination in the 2EU stream, reported as increasing last week, returned to the normal three to six ppb level. Ruthenium contamination, however, remained about two-fold above normal.

Neptunium accumulation in the Solvent-Extraction System remained at about the same level as during the previous report period. During the two periods of low saturation in the 2D Column, the 2EU losses were above normal and the acid flow problems in the First Cycle increased the HAW losses somewhat, but the total losses averaged less than eight per cent. Accumulation in the Neptunium Recovery Cycle continued at a normal rate with approximately a half of a batch accumulated since start-up of the system on August 27.

VI. Product Treatment

A. Plutonium Purification - N-Cell - R. W. Lambert

The performance of N-Cell during the report period continued good although by September 3, push pressures had been increased to 48 lbs and indications of resin problems were present. Fluoride was not used in N-Cell during the entire report period but DF remained near 10 and all product remained well within specifications.

During the latter half of the report period, about 9 gallons of resin were replaced "on the fly" in an effort to keep push pressures from increasing further.

**DECLASSIFIED**

B. Uranium Silica Gel - G. A. Nicholson

The Silica Gel Facility remained shutdown due to lack of feed solution.

**DECLASSIFIED**

Distribution:  
1 - CF Beaulieu  
2 - WS Frank  
3 - AM Platt  
4 - HF Judson  
5 - RW McCullugh  
6 - WM Harty  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 9/9/63 TO 2400 ON 9/15/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 2.6 (actual 2.65) CF during the report period. Addition of hydrolyzed waste rework solution from E-F11 to the HAF commenced on September 14, but jetting difficulties prevented regular additions to succeeding batches.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>NP Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	4.0	4.1			.002	.04	5.0	14
Final	3.5	3.4	5.6	1.9	.002		2.5	105
Ion Exch.		0.9		0.03				
Overall	7.5	8.4	5.6	1.9	.004	.04	7.5	119

B. Production

1. HAF = 143.8
2. U Production = 155.1
3. Pu Production = 71.6 Kg
4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	604 Lbs	0.19
2. Pu	239	0.33
3. Np, F16, F18	11	3.5 *
K6	9	2.8 *
4. Solvent	3000 Gal	0.3

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	899	0.29
2. Pu	132	0.18
3. Np	35	11.0 **

\*Based on total input, virgin feed and rework.

\*\*Based on virgin feed.

DECLASSIFIED

E. Volumes to UGS, Gal/Ton

	<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
	OWW	250	50/50 *
	Cell Drain	40	105-A
	FP	147	105-A
	CW	300	109-B

F. IWW Flows 7.0 Avg. HNO<sub>3</sub> Conc. - 4.75 M

G. <u>Tank Farm</u>	<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
	101-A	2.1	122
	102-A	0.8	90
	103-A	0.3	88
	104-A	2.7	135
	105-A	8.4	93
	106-A	7.7	126

III. Flowsheet - G. A. Nicholson

- (a) On September 9, the 2EX temperature was reduced from 50 to 47° C. It was further reduced to 45° C on September 10.
- (b) On September 9, the 2DF temperature was increased from 50 to 60° C. It was reduced back to 50° C on September 10.
- (c) On September 9, the 2DF uranium concentration was increased by about 10 per cent to 2.2 M.

IV. Feed Preparation - G. J. Raab

Dissolver C3 was holding to age the "green" metal charged September 1. Dissolvers A3 and B3 were able to maintain the plant rate, but three 15-bucket charges were made to keep cycle time to ~ 24 hours. The metal age varied from 130 to 201 days. The I131 emission was 1.96 curies for the period. The amount of I131 coming from the C3 dissolver is minimal as shown by the C3 iodine monitor.

V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance continued at the same excellent level as during the previous report period. Losses were up slightly, averaging about 0.05 per cent until the rework commenced when they increased about two-fold. Process control has shown considerable improvement since the first of the run period. Losses to the Backcycle Waste System have fluctuated less and plutonium levels are much lower than typical of recent months. Plutonium carryover in the IBU has remained exceedingly constant at about 0.3 per cent.

Final Plutonium Cycle performance has continued at the same level as during the previous period, with an average Zr-Nb DF of 2500 and losses averaging about 1.2 per cent. All product was well within limits for processing in the Anion Exchange Cycle without the addition of fluoride.

\*Split assumed between 104-A and 106-A.

Final Uranium Cycle performance has also continued at an excellent level, with all product within shipping limits with respect to gamma activity without silica gel treatment. Zr-Nb DF's improved by about 15 per cent during the report period, from about 3500 to about 4000. A temporary increase in Zr-Nb product activity resulted from the increased 2DF temperature, but quickly decreased when the temperature was returned to normal. The ruthenium contamination in the product showed a downward trend after the reduction in the 2EX temperature to 45° C. A minor problem of plutonium contamination in the product arose early in the report period, but returned to normal when the 2DF temperature was returned to normal. Segregation of five batches that were above the 10 ppb limit was necessary, but it should be possible to blend them off in the near future.

Neptunium accumulation in the Solvent-Extraction System showed a slight improvement, with instantaneous losses averaging about 4 and 2.5 per cent via the HAW and 2EU streams, respectively. The inventory in the Backcycle System has been reduced to an apparent base level of about 300 units and the accumulation in the "J-Cell Package" has been restricted to the input to the plant since the end of the previous report period.

## VI. Product Treatment

### A. Plutonium Purification - N-Cell - R. W. Lambert

During the report period, resin was replaced on an intermittent basis (15 gallons in all) and pushing conditions improved. In addition, the vent valve on the push tank was replaced and the operation improved markedly.

The NaF flow remained off during the report period and N-Cell DF continued to run about 10. All plutonium product was well within specifications.

### B. Neptunium Purification - S. M. Nielson

During the report period, the following items were completed in the Neptunium Purification Unit:

1. Transferred neptunium inventory to Tank J1 (~ 100 units).
2. All process vessels were thoroughly flushed with 30 per cent nitric acid.
3. The E-Q2 concentrator was given a deep-flush with nitric acid for plutonium control.
4. The T-Q4 resin column was stripped with 1 M HNO<sub>3</sub>.
5. The coils in Tank Q3 and Q5 and the jacket on Column T-Q4 were checked for leaks.

The flushes reduced the radiation readings in the Hot Cell to less than 15 mr per hour. Readings at the T-Q4 resin column were 4 mr at the top and 12 mr at the bottom. Essentially no plutonium (4.9 x 10<sup>7</sup> counts per minute per gallon) was found in the E-Q2 concentrator flush. The coil and jacket leak test over a period of eight hours revealed no leaks.



DECLASSIFIED

VII. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $5.17 \times 10^2$  uc/gal and  $2.77 \times 10^3$  uc/gal Zr-Nb and Ru, respectively. The over-all loss from the two systems was 3000 gallons. During this period, the TK-R1 changes were changed from 24 hr/change to 36 hr/change. This was done to reduce loss of organic from R-Cell to G-Cell. The No. 2 solvent activity was 3 uc/gal and 23 uc/gal Zr-Nb and Ru, respectively.

VIII. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

During the past two weeks, the activity in the AAA recovered acid slowly increased. By week's end, the Zr-Nb activity was about  $2 \times 10^4$  uc/gal and the Ru activity had climbed to about  $1 \times 10^4$  uc/gal.

IX. Fission Product Purification - J. B. Kendall

A successful R & D batch run was made at the SSW during the week of August 12 for separating the rare earths and cerium. A cerium df of 250 was achieved from the rare earths by oxidation of the cerium to the quadravalent ion in a  $0.2 \text{ M } K_2S_2O_8 - 0.02 \text{ M } AgNO_3 - 2.0 \text{ M } HNO_3$  media and extraction of the cerium with an equal volume of  $0.4 \text{ M } D_2EHPA - 0.2 \text{ M } TBP$  in Soltrol-170 solvent. About 10 per cent of the rare earths based on promethium analysis was also extracted.

A successful batch run was also made to demonstrate the separation of the rare earths from the sulfate and silver by D2EHPA extraction. A silver df greater than  $1 \times 10^5$  was achieved and the over-all rare earth waste loss based on promethium was about two per cent.

X. Fission Product Recovery - W. C. Schmidt

Approximately 1300 gallons of carbonate product was shipped to 003. Sr-89/Sr-90 ratio, calculated by the cesium to strontium ratio, was 6.6 on September 4.

This document consists  
of No. 4 of 11 copies.  
Series A.

-132-

DECLASSIFIED

HW-76912

Distribution:  
1 - CF Beaulieu  
2 - WS Frank  
3 - AM Platt  
4 - BF Judson  
5 - RW McCullugh  
6 - WM Harty  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 9/16/63 TO 2400 ON 9/22/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 2.6 (actual 2.5) CF during the report period. Rework of hydrolyzed waste solution continued at infrequent intervals until September 22 when attempts were suspended because of jet plugging difficulties. The flow restriction in the 2DF system and two dissolver operations continued to limit the plant production rate. The Neptunium Recovery Cycle was switched from Phase I to Phase II operation on September 16 and from Phase II to Phase III operation on September 18. Phase I operation was resumed on September 19.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.9	4.1			.002	0.11	5.3	73%
Final	3.4	3.4	6.0	1.87	.002		4.8	-210% *
Ion Exch.				.035				
Overall	7.3	8.6	6.0	1.9	.004	0.11	10.1	-137% *

B. Production

1. HAF = 149.2 T
2. U Production = 145.0 T
3. Pu Production = 88.1 Kg
4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	605 Lbs	0.21
2. Pu	260	0.29
3. Np, F16, F18	15	4.0
K6	16	4.2
4. Solvent	805 Gal	0.08

\*Phase III.

D. Rework

	<u>Units</u>	<u>Total Production</u>
1. U	1259 *	0.43
2. Pu	590	0.67
3. Np	49 *	12.9

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	54	104-A
OWW	216	106-A
CD	21	105-A
FP	141	105-A
IWW	8	105-A
CW	300	109-B

F. IWW Flows

6.5 Avg. HNO<sub>3</sub> Conc. - 3.75 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-Off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	2.08	121
102-A	.84	90
103-A	.25	89
104-A	2.65	135
105-A	8.4	93
106-A	7.7	126

III. Flowsheet - G. A. Nicholson

- (a) On September 16, the 3A pulse frequency was reduced from 67 to 50 cycles per minute. It was increased back to 60 cycles per minute on September 17.
- (b) On September 16, during transition to Phase II conditions, the 3AF acidity dropped about 25 per cent. It was increased back to normal on September 17.
- (c) Commencing on September 19, the 2EX-H<sub>2</sub>O was reduced from 392 to 343 flows over the following four days.
- (d) On September 21, the 2DF uranium concentration was reduced from 2.2 to 2.0 M.
- (e) On September 22, the IOBW (carbonate-permanganate) change schedule was changed from twelve to eight hours. Also, the 20BW schedule was changed from a 24 to a 12-hour period.
- (f) During the report period, the 3WB nitric acid concentration averaged 9.0 molar - about 12 per cent high.

DECLASSIFIED

HW-76912

IV. Feed Preparation - G. J. Raab

Dissolvers A3 and B3 maintained the plant rate. Three 15-bucket charges were made to maintain normal cycle times. On September 20 and 21, the samples of TK-D2 (coating waste) showed high uranium by the Cs method. An inspection of the charts showed nothing to indicate any metal solution was jetted into TK-D2 or that the rinse cycle was abnormal. TK-D2 was given a 15 per cent caustic flush and all samples since the flush have been normal. The metal age varied from 125 to 181 days. The I<sub>131</sub> emission was 1.65 curies for the period.

V. Solvent Extraction - G. A. Nicholson

Process performance during the report period continued at the excellent level typical of recent weeks. Rework of the hydrolyzed waste solution in E-F11 produced a noticeably adverse effect on both HAW losses and HSP activity. Except for the brief periods of rework, however, the losses have averaged less than 0.04 per cent and the activity of the First Cycle product streams has continued at a low level.

Final Plutonium Cycle performance has been excellent with the 2BP activity typically only three-fold above shipping limits. A temporary loss of sodium nitrite flow to the 2AF on two occasions resulted in high 2AW losses to the Backcycle Waste System for brief periods.

Final Uranium Cycle performance also continued at an excellent level during the report period, with Zr-Nb DF's averaging about 2700. The Ru DF's have averaged nearly two-fold higher since the 2EX temperature was reduced during the previous report period. The plutonium contamination in the final product averaged only slightly above 3 ppb for the report period and it was possible to blend to shipping limits all of the off-specification uranium product. The saturation in the 2D Column was below the normal on several occasions, resulting in increased neptunium losses via the final product. The under-saturation in the 2D Column was caused by the erratic and frequently low 2DF flow.

A process test was initiated to determine the minimum aqueous-organic flow ratio in the 2E Column consistent with good performance. While results to date are clouded by questionable instrument calibrations and analyses, indications are that flow ratios at or very near the theoretical minimum values are required for good performance.

Neptunium accumulation continued at a relatively high level during the report period, but losses were about 60 per cent above the previous week. About 870 units of neptunium which was accumulated in the J-Cell Package was decontaminated to a low level and diverted to the Neptunium Purification Cycle for final clean-up. An instability problem in the 3A Column was encountered during transition from Phase I to Phase II. While laboratory analysis of the "cold" streams indicated correct acidities, the 3AW nitric analysis was low and addition of fresh acid to the 3AF Tank alleviated the problem.

VI. Product TreatmentA. Plutonium Purification - N-Cell - R. W. Lambert

The plutonium ion exchange unit operated very smoothly during the report period. The activity of the final product remained extremely low in spite of using no fluoride in the XAF feed stream.

An additional five gallons of resin was changed out during the report period on a gallon-at-a-time basis.

B. Neptunium Purification - S. M. Nielson

On September 18, J-Cell Package was switched to Phase III. After 16 hours, the J-Cell Package was shut down and the flow to Q-Cell stopped. The E-Q2 concentrator was shut down, allowed to cool and jetted to Tank Q3. Samples of Tank Q3 indicated that between 700 and 900 units had been transferred from the package. Purification of this material will begin during the next report period.

VII. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $1.14 \times 10^3$  uc/gal and  $2.28 \times 10^3$  uc/gal Zr-Nb and Ru, respectively. The over-all loss from the two systems was 935 gallons. During this period, the TK-R1 carbonate changes were returned to a 24-hour change cycle from a 36-hour change period and the TK-G1 carbonate changes were returned to 8 hours from a 12-hour cycle. These moves were prompted by the increased activity in the solvent. The No. 2 solvent system activity was 2 uc/gal and 27 uc/gal Zr-Nb and Ru, respectively.

VIII. Acid Recovery and Waste Concentration - R. W. LambertF-Cell Acid Recovery

Operation of the F-Cell acid recovery system was somewhat erratic during the report period. On several occasions, the acid absorber (E-F5) was flooded and the E-F6 concentrator specific gravity was abnormally high. The erratic operation resulted in wide fluctuations in the activity level of the recovered acid with both Zr-Nb and Ru exceeding normal operating levels.

IX. Fission Product Recovery - W. C. SchmidtStrontium

Approximately 1500 gallons of carbonate product were shipped to 003. Sr-89/Sr-90 ratio, calculated by the cesium to strontium ratio, was 7.2 on September 12.

**DECLASSIFIED**

Distribution:  
1 - CF Beaulieu .  
2 - WS Frank  
3 - AM Platt  
4 - BF Judson  
5 - RW McCullugh  
6 - WM Harty  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - ME Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 9/23/63 TO 2400 ON 9/29/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 2.6 (actual 2.5) CF during the report period. Rework of hydrolyzed waste (F8) solution was commenced on September 23 and on September 28.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	4.1	4.4			.002	.03	2.0	-46%
Final	3.2	3.4	6.0	2.2	.002		2.5	+162%
Ion Exch.				0.03				
Overall	7.3	8.7	6.0	2.23	.004	.03	4.5	116%

B. Production

1. HAF = 143.7 T
2. U Production = 143.8 T
3. Pu Production = 80.9 Kg
4. Np Production = 1184 (Prelim. Est. - Final Conc. Data not avail.)

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	583 Lbs	0.2
2. Pu	255	0.3
3. Np, F16, F18	12	3.1 *
K6	9	2.3 *
4. Solvent	724 Gal	0.07

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	3289	1.14
2. Pu	3052 -From MF; 487 -From F8	4.0 -Total
3. Np	77	19.5 *

\*Based on Total Input.

DECLASSIFIED

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	201	106-A
OWW	50	104-A
OD	48	105-A
FP	116	105-A
IWW	9	105-A
OW	300	109-F

F. IWW Flows6.9 Avg. HNO<sub>3</sub> Conc. - 5.7 MG. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	2.1	120
102-A	0.8	91
103-A	0.3	91
104-A	2.7	136
105-A	8.4	93
106-A	7.7	126

III. Flowsheet - G. A. Nicholson

- (a) During the period from September 23 through September 24, the 2EX-H<sub>2</sub>O was reduced from 343 to 320 flows. It was increased back to 340 flows on September 26.
- (b) During the period from September 26 through September 29, the 2E pulse frequency was increased from 60 to 75 cycles per minute.

IV. Feed Preparation - J. J. Raab

Dissolvers were shut down for ~ 10 hours for the President's visit on September 26. The feed supply has been low since. This was aggravated by allowing scrubber water to flow from a dissolver on coating removal to a dissolver on a cut. The time cycles were improving by end of period and the metal solution supply was better. Four 15-bucket charges were made to improve the time cycle. The metal age varied from 132 to 187 days. The I131 emission was 0.78 curies for the period.

V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance during the report period continued to be excellent, with the quality of both product streams near all-time low and with waste losses at minimal levels. The tonic effect of the hydrolyzed waste (F8) rework solution was considerably more pronounced than normal and the First Cycle product stream activities were at record low levels for processing of "hot" feed. The solution which was reworked contained the uranium recovered by caustic precipitation from the "sludge" removed from the I131 Concentrator early in August.

Final Plutonium Cycle performance was generally at the same high level of the previous week. A significant decrease in decontamination performance was noted during the last two days of the report period. Also, on September 28, nitrite flow to the 2AF Tank was lost temporarily and the 2AX rate was increased substantially for a short time. As soon as the nitrite flow was restored, the 2AW losses returned to normal and the 2AX was reduced. It is possible, however, that the sudden changes, while not of great magnitude, were sufficient to throw the 2A Column into a cyclic instability condition.

Final Uranium Cycle performance has also been excellent, with Zr-Nb DF's averaging about 3000 and recycle losses much more constant (at about six per cent) than during the preceding week. The plutonium contamination in the uranium product decreased by about two-fold to an average of ~ 7.5 ppb. The 2DF flow restriction continued to limit the processing rate.

The process test in the 2E Column, discussed briefly in the previous summary, was concluded. The minimum A/O ratio obtained without a significant increase in waste loss was about 0.82. This is very close to the theoretical minimum A/O for the temperature and IBP concentration existing during the test (45° C and 28 per cent, respectively). At the conclusion of the above test, the 2DX flow was increased by five per cent and a test commenced to determine the maximum stable operating frequency. The frequency test was terminated when a frequency of 75 cycles per minute was attained without any indication of instability.

Neptunium accumulation in the plant was improved over the previous report period as a result of lower HAW losses and improved control of the 2DW losses. It is interesting to note also that average instantaneous losses have agreed quite closely with total batch losses during the past few weeks. Neptunium accumulation in the Neptunium Recovery Cycle has continued at a fast rate since start-up of the "E-Cell Package". The hydrolyzed waste solution which was reworked apparently contained a substantial quantity of neptunium.

## VI. Product Treatment

### A. Plutonium Purification - N-Cell - R. W. Lambert

By the end of the report period, N-Cell completed 45 days of operation without a complete resin changeout. The decrease in resin pushing problems can primarily be attributed to intermittently changing out small fractions of the resin (~ 10 per cent).

The over-all operation of N-Cell was excellent during the report period. On September 28, ANN (no fluoride) was started into the XAF stream as a convenient means of providing a complexant for the fluoride introduced in MR material via the HA Column.

### B. Neptunium Purification - S. M. Nielson

On September 2, seven cans of Redox neptunium were transferred to Tank Q3 which contained 847 units of Purex neptunium. A sample taken after the transfer showed 1252 units in Tank Q3.



Q3 was butted and the resin loading step started on September 25. By September 28, all scrubs were completed. Regular scrub volumes were used as approximately 60 units of plutonium were received with the Redox material. The elution step was completed on September 29. Valence adjustment was accomplished in the second attempt using a one minute heating period with no vacuum.

No major problems were encountered during the run. With only TMI and thorium analyses unreported, the product is well within shipping specifications. The following tables show the losses for the individual steps and the over-all DF's attained for the run:

<u>Step</u>	<u>Losses for Run #6</u>			<u>Elution</u>
	<u>Loading</u>	<u>Pu Scrub</u>	<u>FP &amp; F1 Scrubs</u>	<u>Forecut</u>
Loss (% of Feed)	1.0	1.5	1.8	12.2

\*DF's For Run #6

<u>Pu</u>	<u>Zr-Nb</u>	<u>Ru</u>	<u>U</u>
265	1323	89,500	12,500

VII. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $1.26 \times 10^3$  and  $2.11 \times 10^3$  uc/gal Zr-Nb and Ru, respectively. The over-all loss from the two systems was 774 gallons. During the period the R-Cell organic inventory was butted up from 28.1 per cent TBF to 29.1 per cent with fresh solvent. The number two system activity levels were 4 uc/gal and 27 uc/gal Zr-Nb and Ru, respectively.

VIII. Fission Product Recovery - W. C. Schmidt

Strontium

New jumpers for routing waste from E3 to UGS vice the old method of E5 to F18 to UGS was installed and the new disposal procedure initiated. No process difficulties have appeared except the total process time cycle has been increased by 3 hours.

An evaluation of the waste loss as a function of centrifuge radiation and heat load was initiated. This evaluation will be accomplished by centrifuging the precipitated strontium, cerium and rare earths supernatant by turning off the feed tank agitator. Waste samples will be taken, the entire supernatant returned to the centrifuge feed tank, and the solution recentrifuged with the feed tank agitator rotating. Waste samples will again be taken and compared with the previous samples.

Approximately 1300 gallons of carbonate product were shipped to 003. Sr-89/Sr-90 ratio, calculated by the cesium to strontium ratio, was 5.9 on September 23, 1963.

\*Based on a per gram neptunium basis.

**DECLASSIFIED**IX. Miscellaneous - R. W. LambertMR (Metal Recovery) Rework

On September 28, rework of MR material from Z-Plant was begun with plans calling for the rework program to continue for several weeks. The rework solution contains fluoride ion up to 0.3 M, complexed at a one to one mole ratio with aluminum. Expected plutonium content is 100 - 150 grams/liter with a nitric acid concentration of 12 - 15 M.

As an added protection against introducing insoluble plutonium into the plant, a special 10 micron screened slurr wand was installed in the PR cage. By the end of the report period, 3800 units had been reworked via the HA Column.

**DECLASSIFIED**

Distribution:  
1 - OF Beaulieu  
2 - WS Frank  
3 - AM Platt  
4 - BF Judson  
5 - RW McCullugh  
6 - WM Harty  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 9/30/63 TO 2400 ON 10/6/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 2.6 (actual 2.36) CF during the report period. Rework of waste solution which was hydrolyzed in the spare IWW concentrator was commenced on September 30 and completed on October 5. Rework of an acid flush of the spare IWW concentrator was commenced on October 5.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.9	4.3			.004	.03	7.0	-5.4%
Final	3.6	3.0	7.0	2.05	.002		3.5	+74.4%
Ion Exch.		1.1		.04				
Overall	7.5	8.4	7.0	2.09	.006	.03	10.5	69.0% *

B. Production

1. HAF = 133.8 T
2. U Production = 130.7 T
3. Pu Production = 104 Kg
4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	571 #	.22
2. Pu	288 Gr	.28
3. Np, F16, F18	9	1.4
K6	10	1.6
4. Solvent	2177	0.23

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	4.3 T	3.2
2. Pu	14,986 Gr (MR) 1088 Gr (F11)	15.0
3. Np	335	53 % *

\*Based on Input.

DECLASSIFIED

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	251	106-A
OWW	63	104-A
CD	38	105-A
FP	184	105-A
IWW	14	105-A
CW	300	109-B

F. IWW Flows

6.0

Avg. HNO<sub>3</sub> Conc. - 5.7 MG. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.02	122
102-A	1.86	90
103-A	0.1	90
104-A	2.13	136
105-A	10.0	94
106-A	9.0	126

III. Flowsheet - G. A. Nicholson

- On September 30, the 2E pulse frequency was reduced from 75 to 68 cycles per minute.
- On October 2, addition of sulfamic acid to the 20S at a concentration of 0.03 M was commenced.
- On October 4, the 2A pulse frequency was increased from 85 to 88 cycles per minute.
- During the period from September 29 through October 6, the TCM-H<sub>2</sub>O was increased from about 440 to about 475 flows.

IV. Feed Preparation - G. J. Raab

Dissolver rates were greater than the plant rate. As a result, the metal supply inventory was returned to normal following the 10-hour dissolver shutdown on September 26. Two 15-bucket charges were made. The metal age varied from 120 to 180 days. I131 emission was 1.24 curies for the period.

V. Solvent Extraction - G. A. Nicholson

The level of performance of the solvent-extraction system, while somewhat poorer than recent weeks, was still excellent. All product was well within specifications and losses averaged less than 0.04 per cent via the HAW stream. A nearly two-fold increase in the activity of the IBP stream was noted after the F8 rework was terminated on September 28. Rework of the second batch of hydrolyzed waste solution (from the spare IWW Concentrator) had no beneficial effect on the IBP activity. The IBU activity, on the other hand, increased

sharply after termination of the F8 rework, but decreased following rework of the second batch of hydrolyzed waste solution. Performance of the IC Column has gradually deteriorated since start-up of the plant. The ICW losses have gradually increased in spite of increased ICX-H<sub>2</sub>O flow. Also, some instability has been noted even at the relatively mild conditions at which the column is operating (ICX temperature of 65° C, pulse frequency of 34 cycles per minute, and volume velocity of 1000 gal/hr/sq ft).

Final Plutonium Cycle performance during the report period was substantially lower than previous weeks. An approximate two-fold decrease in DF occurred during the report period in addition to the two to three-fold increase which occurred on September 28 when the nitrite flow to the 2AF was lost. As a result, the 2BP activity is the highest it has been since the last start-up of the plant. The increase in frequency of the 2A Column did not appear to have a significant effect on the decontamination performance.

Final Uranium Cycle performance has continued at an excellent level, with the Zr-Nb DF averaging about 4000. Plutonium contamination in the final product increased slightly (to about four ppb) during the report period. Sulfamic acid was added to the 20S during most of the report period with no apparent effect on plutonium DF.

Neptunium accumulation in the Solvent Extraction System has continued at a high rate, indicating a substantial amount of neptunium recovered in the rework material. Accumulation in the Neptunium Recovery Cycle has also continued at a satisfactory rate, with transition to Phase II scheduled to commence at the conclusion of the rework of hydrolyzed waste. The neptunium material balance for the past week has shown a much lower accumulation than indicated by the losses. This is due to an inaccurate estimate of the amount of neptunium in the two batches of hydrolyzed waste. The previous report period balance was high by about 20 per cent so an average for the two weeks is in good agreement with the figures for the past eight months.

VI. Product Treatment - R. W. Lambert

Plutonium Purification - N-Cell

Operation of the Ion Exchange Unit during the report period was excellent. Toward the end of the report period, push pressures and push times began increasing and the program of changing out small amounts of resin intermittently was resumed. The DF for the unit remained about 10 with no fluoride being used during the period.

During the week, aluminum was added to TK-N1 as part of the process for reworking MR material.

V. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $1.63 \times 10^3$  uc/gal and  $2.53 \times 10^3$  uc/gal Zr-Nb and Ru, respectively. The over-all loss from the two systems was 2217 gallons. Part of this loss can be attributed to a caustic changeout in TK-G7 which was required when the G5 activity rose above normal. A reducing agent test is being conducted in R-Cell to determine the effect on Pu levels in K-Cell. Sulfamic acid is the reducing agent presently being evaluated. No changes have been noted in K-Cell operations. The No. 2 solvent system activity averaged 4 uc/gal and 29 uc/gal Zr-Nb and Ru, respectively.

DECLASSIFIED

VI. Fission Product Recovery - W. C. Schmidt

No difference in waste losses could be found when the centrifuge feed tank agitator was idle or rotating. The centrifuge Beckman radiation recorder indicated that a large percentage of the precipitated solids were transferred to the centrifuge bowl without any agitation. From this observation, it appears that the precipitate is light rather than the dense precipitate normally reported in laboratory experiments.

Approximately 1600 gallons of carbonate product were shipped to 003. Sr-89/Sr-90 ratio, calculated by the cesium to strontium ratio, was 8.8 on September 30.

VII. Miscellaneous - R. W. LambertMR Rework

During the report period, 15.5 Kgs of MR plutonium were reworked via the HA Column without incident. Abnormally long times (>1 hr) were required to slurp most cans using the finely screened wand which may indicate a partial plugging of the screen.

**DECLASSIFIED**

Distribution:  
1 - OF Beaulieu  
2 - WS Frank  
3 - AM Platt  
4 - BF Judson  
5 - RW McCullugh  
6 - WM Harty  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 10/7/63 TO 2400 ON 10/13/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 2.6 (actual 2.35) CF during the report period. Rework of the acid flush solution from the spare IWW Concentrator commenced October 5 and was completed on October 8. Transition of the Neptunium Recovery Cycle from Phase I to Phase II was completed on October 7; from Phase II to Phase III on October 9; and from Phase III back to Phase I on October 10. Processing of feed material of a nonrepresentative irradiation history was commenced on October 12 after recycle of two batches of "cold" uranium.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.6	3.7			.004	.035	2.5	15.2%
Final	3.5	2.9	7.6	1.8	.002		3.2	-239% *
Ion Exch.		1.5		.04				
Overall	7.1	8.1	7.6	1.84	.006	.035	5.7	-224%

B. Production

1. HAF = 125.6 T
2. U Production = 139.2 T
3. Pu Production = 75.2 Kg
4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	548 #	0.2
2. Pu	261 Gr	0.3
3. Np, F16, F18	20.1 Gr	5.7
K6	11.1 Gr	3.1
4. Solvent	1378	0.15

\*3BN trans. to Q-Cell - ~ 1200 units.

-146-

**DECLASSIFIED**D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	14.5 T	10.4
2. Pu	3.8 Kg	4.9
3. Np	44 Gr	12.4 (Based on Input)

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	243	106-A
OWW	65	104-A
CD	35	105-A
FP	109	105-A
IWW	12.0	105-A
CW	300	109-B

F. IWW Flows5.0 Avg. HNO<sub>3</sub> Conc. - 5.7 MG. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.0	122
102-A	1.9	98
103-A	0.1	88
104-A	2.13	136
105-A	7.6	94
106-A	7.9	125

III. Flowsheet - G. A. Nicholson

- (a) On October 9, addition of sulfamic acid to the 20S was terminated and addition of ferrous sulfamate was started at a concentration of 0.01 M.
- (b) On October 8, the 3WB was increased by about 50 per cent after transition of the Neptunium Recovery Cycle to Phase II operation. The 3WB was reduced back to about 20 per cent above normal on October 9 during Phase III operation and to normal 32 flows on October 10 when the Neptunium Recovery Cycle was switched to Phase I operation.
- (c) On October 11, the 2A pulse frequency was decreased temporarily from 89 to 79 cycles per minute. It was increased back to 90 cycles per minute later the same day.

IV. Feed Preparation - G. J. Raab

The dissolver heels were removed for transition to nonrepresentative metal on October 11. The heel removal from dissolver B3 appeared to be incomplete or the heel cut was very fast. The representative metal charged was generally 140 to 180 days old. The nonrepresentative metal was all 180-day old except one Key 6836-D. This key started at 119 days and this key contributed 99 per cent of the I<sub>131</sub> charged to the plant. The I<sub>131</sub> emission was 1.58 curies of which half was emitted in the last three days.



DECLASSIFIED

-147-

V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance deteriorated markedly with termination of the hydrolyzed waste rework. The activity of the First Cycle product streams increased more than five-fold. Waste (HAW) losses, however, remained low (averaging less than 0.03 per cent). The high activity problem was due to termination of rework and to the high acidity in the HA Column during the Phase II Neptunium Recovery Operation.

Performance of the Final Plutonium Cycle continued to deteriorate during the report period from a DF of about 1000 to about 700. On the possibility that the 2A Column was partially flooded or emulsified, the pulse frequency was decreased rapidly ten cycles per minute, then brought back up slowly. No effect on performance was detected, however.

Final Uranium Cycle performance remained generally excellent, with the Zr-Nb DF typically 3000 to 4000. Periodic upsets caused by attempts to increase the 2DF flow rate resulted in brief bursts of activity in the product. This, together with the generally poor First Cycle performance, necessitated segregation of the uranium product for processing through the Silica Gel Facility. As with the sulfamic acid addition to the 20S, addition of ferrous sulfamate to the 20S did not affect the 2D Column plutonium DF appreciably.

A neptunium recovery run was completed successfully in the Neptunium Recovery Cycle, with about 1250 units transferred to the Neptunium Purification Cycle. Losses via both the 3AW and 3BW streams were higher than normal during Phase II operation, but were reduced by minor flowsheet adjustments. Neptunium accumulation in the Backcycle Waste System during the report period was good, with losses averaging about three per cent via the HAW and four per cent via the 2EU stream. Since the transition to Phase I operation on October 10, accumulation in the J-Cell Package has been normal.

VI. Product TreatmentA. Plutonium Purification - N-Cell - R. W. Lambert

Operation of N-Cell during the report period was satisfactory although pushing was sluggish and waste losses increased. On October 9, the sodium fluoride was started to keep the product within specifications as the activity level in the 2BP stream increased a factor of five. N-Cell DF's subsequently increased from about 10 to about 30.

On October 12, the transition from representative to nonrepresentative metal was made in N-Cell with flushes of E-N6 and TK-N7 being made before the nonrepresentative metal reached N-Cell. Weight factor problems in both E-N6 and TK-N7 have caused some processing difficulties including a product leak at the E-N6 weight factor flange.

B. Uranium Silica Gel - G. A. Nicholson

The Silica Gel Unit was started up on October 11, but was shut down for regeneration when product samples indicated poor performance.

DECLASSIFIED

C. Neptunium Purification - S. M. Nielson

Phase III was started at 1510 on October 9. Approximately 1050 units were transferred to Q-Cell and combined with the 150 units left from the previous run.

Q4 was pretreated on October 11, but was again pretreated on October 12 with the solution going to Tank Q3. This was done in order to make certain the column was filled with liquid and also to increase the feed volume so that a reasonable feed rate to Column Q4 could be established.

On starting the feed on October 12, there was no pressure built up in Column Q4. It was discovered that the rupture disc on Column Q4 had been broken due to a closed manual valve on the column effluent stream allowing the pressure in the column to exceed 80 pounds per square inch.

VII. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $5.64 \times 10^3$  and  $2.97 \times 10^3$  uc/gal Zr-Nb and Ru, respectively. The over-all loss from the two systems was 1378 gallons. Following rework of the F8 material, the solvent activity rose to a high of  $1 \times 10^4$  uc/gal Zr-Nb and has now fallen to an average of  $5 \times 10^3$  uc/gal. A test of ferrous sulfamate as a reductant in the 20S wash tank was made. The No. 2 solvent system activity has averaged 10 uc/gal and 36 uc/gal Zr-Nb and Ru, respectively.

VIII. Acid Recovery and Waste Concentration - R. W. LambertF-Cell Acid Recovery

On October 10, a test was started to determine the feasibility of replacing sodium nitrite with sugar as a ruthenium suppressant in the IWW Concentrator (E-F6). For several weeks prior to the test, the activity levels in the AAA (TK-F3) recovered acid had been typical of single cycle acid recovery with Zr-Nb at about  $1.5 \times 10^4$  uq/gal and Ru at about  $1 \times 10^4$  uq/gal. IWW flows had consistently been about 6 flows.

The test consists of adding enough sugar to TK-F12 to make the IWF stream 0.001 M while still adding the customary 0.013 M  $\text{NaNO}_2$ . After demonstrating the controlled sugar addition, the nitrite flow will gradually be reduced with the goal of the test being complete delimitation of the nitrite addition.

By October 13, test results were encouraging but inconclusive. Following the test start-up, the ruthenium level decreased, but because of flow control problems during the 12th and 13th, the sugar addition was erratic and the ruthenium activity in the acid returned to near normal. By October 13, the nitrite flow had been decreased 30 per cent.

IX. Fission Product Recovery - W. C. SchmidtStrontium

Evaluation of the effects of radiation on solution pH was completed. The pH of the solution just prior to centrifugation and after centrifugation was completed varied by only 0.1 pH unit.

DECLASSIFIED

Evaluation of the waste loss as a function of the amount of cake in the centrifuge was completed. The concentration of strontium in the centrifuge supernatant was obtained periodically throughout a centrifugation period. No differences in concentrations could be found.

Approximately 300 gallons of carbonate product were shipped to 003. Sr-89/Sr-90 ratio, calculated by the cesium to strontium ratio, was 7.8 on October 5.

Cesium and Technetium

An STT cask loaded with Decalso plus the extra STT cask loaded with IRA-401 were placed in series and 103-C supernatant passed through the beds. Preliminary numbers indicate the cesium to be loading routinely and very little technetium being adsorbed on the IRA-401.

X. Miscellaneous - R. W. Lambert

Water Treatment

On October 9, the main demineralized water filters were replaced. These filters had been in service for 30 days which is a factor of three greater than the average service obtained during the May-June run period. The reason for the change in service has not been determined.

Because of a shortage of filter elements, the filters were bypassed for the five days prior to the change.

**DECLASSIFIED**

Distribution:  
1 - OF Beaulieu  
2 - WS Frank  
3 - AM Platt  
4 - BF Judson  
5 - RW McCullugh  
6 - WM Harty  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 10/14/63 TO 2400 ON 10/20/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 2.5 (actual 2.4) CF during the report period. Processing of feed material of an unclassified irradiation history continued.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.6	4.0			.002	.08	3.0	13.3% }*
Final	3.3	2.8	6.0	1.7	.002		2.5	85 % }
Ion Exch.		1.4		.3				
Overall	6.9	8.2	6.0	2.0	.004	.08	5.5	98.3%

B. Production

- 1. HAF = 125.7 T
- 2. U Production = 139.0 T
- 3. Pu Production = 110.3 Kg
- 4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	548 #	0.20
2. Pu	266 Gr	0.24
3. Np, F16, F18	12 Gr	3.0 )**
K6	10 Gr	2.5 )
4. Solvent	1037	0.11 ***

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	None	-
2. Pu	595	0.5
3. Np	None	-

\*Based on virgin input and recycle from Q-Cell → J1.

\*\*Based on input from virgin feed.

\*\*\*Based on total gal. pumped at nominal CF 2.4.

**DECLASSIFIED****E. Volumes to UGS, Gal/Ton**

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	205	104-A
OWW	110	106-A
CD	35	105-A
FP	217	105-A
IWW	-0-	
CW	300	109-B

**F. IWW Flows**

~ 5.0

Avg. HNO<sub>3</sub> Conc. - 5.5 M**G. Tank Farm**

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.02	123
102-A	1.86	111
103-A	0.1	87
104-A	2.13	137
105-A	7.55	94
106-A	7.9	125

**III. Flowsheet - G. A. Nicholson**

- On October 15, ferrous sulfamate addition to the 20S was terminated.
- On October 15, the carbonate-permanganate wash solution (IOB) changeout schedule was changed from eight to four hours and the volume per change-out was reduced by half.
- On October 17, the 2A pulse frequency was temporarily reduced from 88 to 60 cycles per minute. It was increased back to 94 cycles per minute later the same day and to 98 cycles per minute on October 18.
- On October 17, the HA pulse frequency was increased from 79 to 87 cycles per minute. It was reduced back to 83 cycles per minute later the same day.
- From October 17 through October 20, the 3WB nitric acid concentration was about 15 per cent above normal.

**IV. Feed Preparation - G. J. Raab**

The dissolvers have been able to maintain the plant rate without difficulty. The coating waste jet to UGS has continued to perform well since the jet air blow was increased to one hour. The I<sub>131</sub> emission for the period was 2.42 curies. Key 6836-D continued to be the only significant I<sub>131</sub> contributor. Mercuric nitrate was added to metal solution on October 18 and the week-end emission rate was less than half the previous day. For this reason, it does not appear the I<sub>131</sub> emissions are coming from the dissolvers.

DECLASSIFIED

V. Solvent Extraction - G. A. Nicholson

A substantial improvement was noted in the performance of the Solvent Extraction System during the report period. Waste losses were near nondetectable levels during the first half of the period, but increased during the last half after the HA Column pulse frequency was decreased. The problem appeared to result from a change in control points caused by the change in pulse frequency. Also, the acidity in the HA Column, which would alter the organic saturation, was reduced at about the same time. In spite of these minor problems, the losses averaged less than 0.07 per cent. The activity of the First Cycle product streams remained essentially constant during the report period at a level about 30 per cent lower than during the latter half of the previous report period.

Performance of the Final Plutonium Cycle, which had been on a sharp downward trend until the pulse frequency adjustment, improved dramatically after the frequency was increased back to greater than 90 cycles per minute. Prior to the adjustment, the dial indicator showed a frequency of about 88 cycles per minute, which in the past has been sufficient for good scrubbing efficiency. On the assumption that a mild flood was occurring in the scrub section and limiting the scrubbing efficiency, the pulse frequency was reduced a nominal 20 cycles per minute. A check after the reduction, however, showed the indicator to be reading 10 cpm high and the actual reduction was ~ 28 cycles per minute. The frequency was increased back to 94 cycles per minute in about eight hours and the decontamination performance improved immediately. All plutonium product was within limits for processing in the Plutonium Anion Exchange Cycle.

Performance of the Final Uranium Cycle continued to be very good. Ruthenium contamination was excessive (two to three-fold above normal), but the Zr-Nb contamination was low and all product was within shipment limits without silica gel treatment. The addition of ferrous sulfamate to the 20S was terminated after six days, as no beneficial effect was apparent. Plutonium contamination in the final product trended downward during the report period to an average of 2 ppb.

Neptunium accumulation in the Solvent Extraction System was excellent during the report period, with total losses averaging less than six per cent. Moreover, the integrated instantaneous losses were in excellent agreement with the total batch losses. Also, the calculated accumulation was in excellent agreement with input plus loss figures. Accumulation in the Neptunium Recovery Cycle has continued at a good rate, with all virgin input rapidly accumulating in the J-Cell Package and the inventory in the Backcycle Waste System remaining essentially constant at about 500 units.

VI. Product TreatmentA. Plutonium Purification - N-Cell - R. W. Lambert

Operation of N-Cell was satisfactory for the report period although incremental resin changeouts were necessary to prevent pushing problems. A leak around the E-N6 thermowell necessitated blanking off the well. This will not be repaired until shutdown.

B. Uranium Silica Gel - G. A. Nicholson

The Silica Gel Facility was shut down after it was regenerated due to lack of feed. The material intended for silica gel treatment was high in plutonium. For this reason it was segregated for eventual blending or rework.

C. Neptunium Purification - S. M. Nielson

On October 13, the Resin Loading Step of Run #7 was completed and the Plutonium Scrub Step started. The Plutonium Scrub Step was interrupted for several shifts and upon completion of this step, it was found that ~ 220 units of neptunium had been lost to the Waste Tank Q5. This neptunium loss is attributed to a loss of reductant in the Plutonium Scrub causing the loaded neptunium to oxidize to Np-V and elute. The waste containing the 220 units was returned to the Backcycle System.

High waste losses continued when the Fission Product Scrub was started indicating that the neptunium had been smeared on the resin during the latter part of the Plutonium Scrub. Approximately 75 units of neptunium were lost to the Backcycle System before the Fission Product Scrub Step was terminated.

The Q4 resin was eluted to Tank Q3 with 100 liters of eluant and the resin pretreated. Tank Q3 contents were then butted to feed specifications.

Before starting the feed step again, it was decided that the resin level indicator probe should be put back in service as the equipment was available. Upon calibration, the resin probe indicated that Q4 contained little or no resin. Two gallons of resin were added to the column to see if the probe would respond but it did not. The new probe was put in the column and it indicated only 9 liters of resin instead of the required 28. Three more gallons of resin were added to check the probe calibrations.

A sample was taken on October 20 of Tank Q5. The sample contained ~ 20 per cent resin, thus explaining the whereabouts of the Q4 resin. Efforts will continue during the next report period to clear Q5 of the resin and to determine the manner in which the resin was lost.

VII. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $5.8 \times 10^3$  uc/gal and  $4.8 \times 10^3$  uc/gal Zr-Nb and Ru, respectively. The over-all loss from the two systems was 1037 gallons. The high activity in the G-Cell organic continued. The change-out of TK-G1 wash was changed from a 700 gallon, 8 hour change to a change of ~ 400 gallons every 4 hours. The No. 2 solvent system inventory was butted from 28.2 per cent TBP to 29.3 per cent. The No. 2 solvent system activity has averaged 2.2 uc/gal and 4.2 uc/gal Zr-Nb and Ru, respectively.

IX. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

Sugar addition to the IWW Concentrator (E-F6) via TK-F12 continued during the report period with encouraging results. During the period, the sugar

concentration in the IWF was controlled at 0.001 M while the sodium nitrite concentration was gradually decreased from 0.009 M to 0.003 M (about 23 per cent of original). The sugar addition continued to be smooth with no signs of foaming or instability in the concentrator.

The ruthenium activity level in the AAA recovered acid continued to show unexpected improvement and averaged about  $4 \times 10^3$  uq/gal which represented a reduction of about 60 per cent over the pre-test activity. Zirconium-niobium activity remained unchanged during the test.

X. Waste Treatment and Storage - R. C. Forsman

Denitration of Purex acid waste (PAW) with sugar coupled with improved control of the IWW Concentrator reduced the over-all sodium hydroxide consumption 25 per cent. The IWW Concentrator is currently controlled by maintaining a constant steam flow to the tube bundles in E-F6 and maintaining a relatively constant IWW overflow by adjusting the feed rate to the concentrator. This type operation results in far smoother operation of the waste system and reduces the amount of acid lost to the IWW stream. Sugar denitration has reduced IWW waste from 6.0 M in free acid analysis to about 2.0 M in free acid. The pH measurements used for fission product recovery operations indicate that the acid concentration of sugar treated waste is actually about 0.5 M to 1 M rather than the reported 2.0 M.

XI. Fission Product Recovery - W. C. Schmidt

Strontium

Approximately 1600 gallons of carbonate product were shipped to 003. Sr-89/Sr-90 ratio, calculated by the cesium to strontium ratio, was 4.5 on October 17.

Cesium and Technetium

Approximately 67 bed volumes of 103 supernatant were passed through six Decalso loaded STT's and one IRA-401 anion loaded STT for cesium and technetium recovery. The cesium loaded routinely. Technetium analyses appear to be very erratic but indicate approximately a 35 per cent loss. Elution with 6 M nitric for technetium recovery will be performed at SSW.



**DECLASSIFIED**

Distribution:  
1 - OF Beaulieu  
2 - WS Frank  
3 - AM Platt  
4 - BF Judson  
5 - RW McCullugh  
6 - WM Harty  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 10/21/63 TO 2400 ON 10/27/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 2.5 (actual 2.4) CF during the report period. After recycling two batches of "cold" uranium from TK-P2 to purge the system of unclassified plutonium, the plant was shut down on October 27 with the HAF off at 1430.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.7	4.1			.002	.03	2.5	- 4%
Final	3.3	3.2	6.0	.95	.002		2.5	+86%
Ion Exch.		1.3		.80				
Overall	7.0	8.6	6.0	1.75	.004	.03	5.0	82%

B. Production

1. HAF = 111.6
2. U Production = 136.7
3. Pu Production = 101.5 Kg
4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	460 #	0.21
2. Pu	270 Gr	0.27
3. Np, F16, F18	11 Gr	3.0
K6	9 Gr	2.5
R8 - Est. Loss Q8 → R8	40 Gr	11.2
4. Solvent	1628	0.18

)\*Based on Input

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	11.2 T	8.2
2. Pu	49 Gr	.05

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	294	104-A
CD	28.0	105-A
FP	142	105-A
IWW	15	105-A
CW	300	109-B

F. IWW Flows

6.5 Avg. HNO<sub>3</sub> Conc. - 5.0 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.0	123
102-A	1.9	109
103-A	0.1	87
104-A	2.1	135
105-A	7.6	95
106-A	7.9	124

III. Flowsheet - G. A. Nicholson

- (a) On October 22, the 2DF ferrous sulfamate addition was reduced from 0.36 to 0.30 flows.
- (b) On October 22, the IOB carbonate-permanganate wash solution change schedule was returned to eight hours.
- (c) On October 22, the 2D Column pulse frequency was reduced from 74 to 54 cycles per minute, the 2DF was shut off, and the column stripped out. The column was started up after about two hours and the frequency increased back to 66 cycles per minute. It was increased to 74 cycles per minute on October 23.
- (d) On October 24, the HAF uranium concentration was increased by ~ 15 per cent and the nitric acid concentration was reduced from 1.1 molar to 0.5 molar due to elimination of the feed adjustment step.
- (e) On October 26, the 3WB-HA flow was increased by about 50 per cent after transition to Phase II operation in the Neptunium Recovery Cycle.
- (f) On October 27, the Neptunium Recovery Cycle was shut down at equilibrium.

IV. Feed Preparation - G. J. Raab

The last charge for the run period was made on October 23. The heels were cut out for inventory and converting production to representative metal. The I131 emission was 0.61 curies for the period. Mercuric nitrate was used during all of the period. The lower bed on silver reactor C-2 was regenerated in preparation to return Dissolver C-3 to operation the next run period. The acid butt was omitted from TK-E6 starting October 24 to test the effect on column operation.

V. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance during the report period remained at about the same level as during the previous week. The activity of the First Cycle product streams showed a slight downward trend and the waste losses improved somewhat. Except for two brief periods, the losses averaged about 0.03 per cent.

Performance of the Final Plutonium Cycle continued to improve with the DF averaging about 1700 during the report period. Recycle losses fluctuated considerably, but were generally between 0.5 and 0.8 per cent. All product was well below the limit for processing in the Plutonium Anion Exchange Cycle.

Performance of the Final Uranium Cycle received a severe setback early in the report period when a faulty instrument caused a misinterpretation of conditions in the 2D Column. The column was thought to be flooding when a leaky sensing line on the extraction DP instrument caused a false indication. The pulse frequency was reduced drastically and the column stripped out. This action resulted in a severe blast of activity and plutonium that necessitated segregating five batches of product for reworking. Within two days, however, the product was within shipping limits without silica gel treatment.

Neptunium accumulation in the Solvent Extraction System continued to be excellent, with low losses and good agreement between instantaneous and batch values. Accumulation in the Neptunium Recovery Cycle continued at a good rate and transition to Phase II conditions was made on October 26. A flood in the 3A Column (probably precipitated by a low 3AF temperature) resulted in high losses via the 3AW Stream. Also, the 3BW losses were higher than normal during Phase II operation and an estimated 20 per cent of the accumulated neptunium was lost back to the Backcycle Waste System. The Neptunium Recovery Cycle was shut down at equilibrium on October 27 after analyses showed the product to be within limits for processing in the Purification Cycle and losses continued high.

VI. Product Treatment

A. Plutonium Purification (N-Cell) - R. W. Lambert

Operation of the Plutonium Ion Exchange unit was only marginal during the report period although all product produced was well within specifications. In spite of making incremental resin changes, pushing was erratic and at times nearly impossible. As a result, losses to the backcycle were greater than desired. On October 27, stripping of the unit along with preparations for shutdown maintenance work were begun.

During the 10 week run period, N-Cell operated continuously without a total resin change. This is the longest continuous run on a single resin charge since 1960. The long run was made possible by intermittently changing out about two gallons of resin at a time throughout the run. A total of about 65 gallons of resin were changed out using this technique.

B. Neptunium Purification - S. M. Nielson

The cause of the resin loss from the Q4 Column to the Q5 Waste Tank mentioned in last week's report is as yet unknown. A sight glass and screen installed in the 3XW line showed that no resin passes through the bottom screen of the Q4 Column. There are certain valving sequences that could have been made allowing resin to reach Q5.

The resin in the column was eluted, then removed to Tank R8 via Tank Q8. The column was filled with new resin and the resin probe calibrated.

On October 24, the resin was pretreated and the neptunium in Q3 was butted to feed specifications. Resin loading of this material started on October 25, however, a sample of the 3XW showed high neptunium losses. The acid concentration in the feed was slightly low and the feed rate was 20 per cent high due to a faulty flow reading. A resin pretreatment step was made with the solution going to Q3. Tank Q3 material was again butted and then loaded on the column.

The problem of resin removal from the Q5 Waste Tank was complicated by the presence of neptunium in Q5. The material could not be jetted to R8 for the neptunium would be lost and it could not be jetted to the Backcycle System because of the organic resin. As one of several alternatives, the following plan was decided upon:

1. Transfer the neptunium-bearing liquid from Tank Q5 to Tank Q3 through a filter.
2. Concentrate this liquid in E-Q2 and store in Q3.
3. Elute the neptunium on the Q5 resin with water and transfer this water to Q3.
4. Jet the resin from Q5 to R8 and flush Q5.

All routings were available except a line from Q5 to Q3 containing a filter. A temporary line containing a filter was connected to the Q5 recirculation line and the suction leg of the degassing jet in the maintenance hood.

Transfer of Q5 to Q3 started on October 26, but was very slow due to constant plugging of the filter which required backflushing. Because of water added from backflushing the line and filter, very little had been gained in Q5 by the end of the report period.

VII. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $3.3 \times 10^3$   $\mu\text{c/gal}$  and  $5.2 \times 10^3$   $\mu\text{c/gal}$  Zr-Nb and Ru, respectively. The TK-G1 carbonate wash cycle was returned to an eight-hour, 700 gallon changeout. The overall loss from both systems was

DECLASSIFIED

DECLASSIFIED

HW-76912

-159-

1195 gallons. The No. 2 solvent system activity has averaged 2 uc/gal and 36 uc/gal Zr-Nb and Ru, respectively.

VIII. Acid Recovery and Waste Concentration - R. W. Lambert

Sugar addition to the IWW concentrator, E-F6, was continued during the report period and from data obtained, it appears that the test is a complete success and that sugar can replace sodium nitrite as a means of controlling ruthenium volatilization.

Early on October 21, the nitrite flow was completely stopped and the sugar left at 0.001 M in the IWF. For the following seven days, the ruthenium level in the AAA recovered acid averaged about  $2 \times 10^3$  ug/gal while the zirconium-niobium level remained at its pre-test level of  $1 \times 10^4$  ug/gal. It is believed that the current ruthenium activity level, which is a factor of five below its pre-test levels, is nearing the limit determined by entrainment and that very little ruthenium volatilization is now taking place.

Another possible advantage of using sugar rather than sodium nitrite is that the speed with which the sugar reacts and forms the nitrite ion is such that inconsistencies in sugar flow do not heavily influence the amount of ruthenium volatilization. This appears to be because residual sugar in the concentrator acts as a buffer and continues to react, whereas with straight nitrite addition, the life of the nitrite ion is short and as soon as the nitrite flow stops, the suppression effect is immediately lost. This characteristic was postulated after the sugar flow had been unintentionally shut off for about two shifts and only one rise in ruthenium activity was observed and it did not occur until the shift the sugar was restarted.

IX. Fission Product Recovery - W. C. Schmidt

Strontium

Approximately 1400 gallons of carbonate product were shipped to 003. Sr-89/Sr-90 ratio, calculated by the cesium to strontium ratio, was 5.6 on October 23.

Although sugar kill of nitric acid in IWW has maintained the acidity at one pound per gallon, centrifuge cakes have been difficult to remove. Both the pre-precipitation cake and the strontium-cerium-rare earth cakes have been difficult.

**DECLASSIFIED**

Distribution:  
1 - OF Beaulieu  
2 - WS Frank  
3 - AM Platt  
4 - BF Judson  
5 - RW McCullugh  
6 - WM Harty  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 10/28/63 TO 2400 ON 11/3/63

I. General - G. A. Nicholson

The plant was shut down from October 28 through November 3. Preparations for start-up commenced with the Plutonium Anion Exchange Cycle "spinning". Early on November, the IBX and IES Columns were started at about 2200.

II. Feed Preparation - G. J. Raab

The dissolvers were down most of the period. The ammonia scrubber lines were flushed to remove any possible restrictions. Dissolver A-3 and B-3 were started with coat removal on November 1. The 5 per cent caustic solution used to hold Dissolver C-3 metal while the metal aged was jetted to TK-F10, neutralized and concentrated in E-24.

III. Solvent Extraction - G. A. Nicholson

During the shutdown, the following solvent-extraction equipment was flushed and/or inspected:

- (1) IC Column was flushed with hot 5 per cent caustic followed by hot 10 per cent nitric to remove crud accumulations.
- (2) The 2DF Tank was emptied to the HAF Make-up Tank and inspected. It appeared to be relatively clean. About 2,000 gallons of water was added to the tank and the agitator was turned on, revealing a considerable quantity of relatively coarse particulate matter.
- (3) The 2DF pump and jumper were removed, inspected, and transferred to the Decontamination Facilities for testing. No flow restriction was noted during the test.
- (4) The 2D Column was filled with water with a blank on the 2DF nozzle. The blank was then removed and the 2DF distributor backflushed. No flow restriction was apparent.

It was concluded that the 2DF flow restriction was caused by an accumulation of the relatively large particles against the narrow opening (< one inch) between the bottom of the tank and the bottom of the pump housing. A new pump was installed with a screen around the suction and a preliminary test gave satisfactory performance.

After the shutdown, the HA Column was noted to be leaking slowly to the IWF Tank via the HAW. The organic was displaced to the IBXF Tank to prevent its loss.

#### IV. Product Treatment

##### A. Plutonium Purification (N-Cell) - R. W. Lambert

Following shutdown of the plant on October 27, N-Cell resin was stripped in preparation for making a resin change. During the shutdown, routine maintenance work was performed.

A new change of resin was placed in the unit on November 3 in preparation for start-up.

##### B. Neptunium Purification - S. M. Nielson

Efforts are continuing to remove the resin from Q-Cell waste tank. On October 28, solution and resin were jettted from Q5 to Q3 through the regular transfer jet (no screen) as the Q5 weight factor had increased to overflowing from a leak in a backflush system. Since this added resin to Tank Q3, a filter system had to be placed between Q3 and the E-Q2 concentrator. Two plastic jugs in series, each with a screen in their outlets, were placed between Q3 and pump P-Q3-2 in the maintenance hood.

By October 31, approximately 100 gallons of solution had been worked off through Q5 to Q3 to Q1 to E-Q2; however, very little resin was collected in the plastic jugs in the transfer line.

On November 1, the jugs were replaced by a flex line and a removable filter.

By November 2, all of the liquid in Q5, Q3 and Q8 had been worked off and concentrated in E-Q2. All of the neptunium was stored in E-Q2.

The resin in Q3 was washed with water which was allowed to drain through a filter to Q8 where it was jettted to Q5. An ANN solution of 1.5 specific gravity was added to Q3 in an effort to float out the resin. This mixture was allowed to drain to Q8 where it was jettted to R8.

By the end of the report period, all of the resin was apparently removed from Tanks Q8 and Q3. Efforts will continue during the next report period to remove the resin from Q5.

#### V. Solvent Treatment - G. Ward

Plant shutdown and TK-G1, TK-G2, T-G2 and TK-G7 flushed with standard caustic tartaric and oxalic HNO<sub>3</sub> flushes. Considerable difficulty was encountered with an emulsion in TK-G7 during flushing. Approximately 2,000 gallons of organic were lost to TK-G8 from TK-G7. TK-G1 required more than the normal amount of flushes to cut the solid build up.

TK-R1, T-R2, TK-R2 were also flushed with oxalic-HNO<sub>3</sub> and caustic-tartaric. OWW waste from TK-G8 has been backing up into TK-R8 during jet-outs.

VI. Fission Product Recovery - W. C. Schmidt**DECLASSIFIED**Strontium

Separation of cerium and rare earths from strontium by an oxalate precipitation was completed in B-Plant. No operational difficulties were found except the removal of the cerium oxalate from the centrifuge bowl. Approximately 300 gallons of 4 M nitric plus 100 gallons of water have been required to remove this cake.



**DECLASSIFIED**

Distribution:  
1 - CF Beaulieu  
2 - WS Frank  
3 - AM Platt  
4 - BF Judson  
5 - RW McCullugh  
6 - WM Harty  
7 - SG Smolen  
8 - RE Tomlinson  
9 - AJ Waligura  
10 - MT Walling  
11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 11/4/63 TO 2400 ON 11/10/63

I. General - G. A. Nicholson

Normal processing at a nominal 3.0 CF was commenced on November 4, with HAF on at 0700. After recycling three batches of "cold" uranium, processing of "hot" feed was commenced at 0200 on November 5. The Final Uranium Cycle was started late on November 4, with 2DF on at ~ 2200. The processing rate was increased to a nominal 3.6 CF on November 5, but a restriction in the IBXF Flow System necessitated a reduction to a nominal 3.3 (actual 3.3) CF later the same day.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.7	3.6			.002	.05	3.5	
Final	3.3	3.2	8.0	1.3	.002		5.0	
Ion Exch.		1.3		.2				
Overall	7.0	8.1	8.0	1.5	.004	.05	8.5	

B. Production

1. HAF = 169.6 T  
2. U Production = 170.3 T  
3. Pu Production = 68.6 Kg  
4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	946 #	0.28
2. Pu	423 Gr	0.62
3. Np, F16, F18	73.1*	19.6
K6	17	4.6
4. Solvent	8212	0.7

\*Primarily from F16 batches 1 & 2 - 1.927 - ~ 2150 Gal.

DECLASSIFIED

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	32 T	18.8
2. Pu	6.5 Kg	9.5

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	249	104-A
OWW	396	101-A
CD	142	105-A
FP	79	105-A
IWW	9	105-A
CW	300	109-B

F. IWW Flows

5.0 Avg. HNO<sub>3</sub> Conc. - 3.8 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.5	125
102-A	1.0	91
103-A	0.2	84
104-A	1.0	134
105-A	9.8	95
106-A	6.0	125

III. Flowsheet - G. A. Nicholson

- (1) The plant was started up under the same flowsheet in effect at shutdown except for the following:
  - (a) The 2A pulse frequency was 80 rather than 98 cycles per minute.
  - (b) The 2DF ferrous sulfamate was 0.27 rather than 0.30 flows.
  - (c) The 3WB nitric acid concentration was 6.6 rather than 8.0 molar and the 3WB flow rate was about 38 rather than 45 flows.
- (2) On November 4, the 2A pulse frequency was increased from 80 to 88 cycles per minute. It was further increased to 98 cycles per minute on November 5 when the column was flooded. The frequency was reduced to 63 cycles per minute and the 2DF flow shut off and the column stripped to "break" the flood. The pulse frequency was increased back to 81 cpm later the same day, to 90 cpm by November 6, and to 95 cpm by November 10.
- (3) On November 5, the IBX pulse frequency was reduced from 54 to 49 cycles per minute to dissipate an unstable condition. It was increased back to 54 cycles per minute later the same day.

- (4) On November 5, the 2DF-ferrous sulfamate was increased from 0.27 to 0.36 flows. It was reduced to 0.22 flows on November 9, then increased back to 0.36 flows on November 10.
- (5) On November 5, the 2BP flow was diverted to the "L-Cell Package". The 2BX composition and rate were adjusted accordingly.
- (6) On November 6, the 3WB nitric acid concentration was increased to 8 molar and the rate was reduced to about 32 flows.
- (7) On November 9 and 10, the TBP concentration in the First Solvent System was increased from 29 to 30.5 per cent by additions of 100 per cent TBP.
- (8) On November 8, the HAO was reduced from 77 to 69 flows.

#### IV. Solvent Extraction - G. A. Nicholson

Start-up of the Solvent-Extraction System was marked by numerous minor difficulties and, consequently, a little more time elapsed than normal before all components were operating smoothly and the product streams were within shipping limits. However, the system was operating smoothly by mid-week and all product was within specifications by the end of the fifth day of the report period. Major problems are enumerated below:

1. HAF flow problems caused, undoubtedly, by the transfer during the shut-down of uranium solution in the 2DF Tank to the HAF Make-up Tank.
2. High activity in the Final Uranium Cycle caused initially by the organic (and interface) displacement from the HA Column during the shutdown and propagated by the minor upsets in the IBX and IC Columns during the first day of operation.
3. The 2A Column flood resulting in a severe burst to the Anion Exchange Cycle and necessitating diversion of the 2BP Stream to the "L-Cell Package".
4. A flow restriction in the IBXF Flow System limiting the plant to a 3.4 CF rate.

After about three days, the HAF flow problem diminished and performance improved quickly to normal levels. Except for two brief periods of HAF flow trouble, the losses averaged less than 0.03 per cent.

Performance of the Final Plutonium Cycle, following stabilization from the flood, improved rapidly and the DF was averaging 3000 by the end of the report period. Losses to the Backcycle Waste System averaged less than one per cent.

Performance of the Final Uranium Cycle was generally excellent with the Zr-Nb DF averaging 2000 and the Ru contamination in the product stream reduced to negligible proportions by the end of the report period. Due to the activity burst at start-up, the first 13 batches of UNH required silica-gel treatment. However, by November 9, the activity was within shipment limits without further treatment or blending. A mild increase in plutonium contamination

resulted when the ferrous sulfamate rate was reduced from 225 to 137 per cent of the flowsheet value causing suspicion that the ferrous ion is being oxidized excessively in the 2DF Tank.

Neptunium accumulation in the Solvent Extraction System was generally very good. Except for a brief high (25 per cent) loss during start-up of the HA Column, the total losses (EAW + 2EU) have averaged well under ten per cent.

V. Product Treatment

A. Plutonium Purification (N-Cell) - R. W. Lambert

For the plant start-up, the 2BP was routed directly to N-Cell but, following an L-Cell upset, was diverted to the L-Cell Package for about eight hours. Subsequent operation of N-Cell was satisfactory although the first two loadouts exceeded activity specifications.

Near the end of the report period, the 2BP activity decreased and the NaF flow in N-Cell was stopped. Plutonium throughput in N-Cell was increased appreciably from last run period by the higher plant rate and the high rate of plutonium rework.

B. Uranium Silica Gel - J. A. Nicholson

The Silica Gel Facility was operated for the last four days of the report period. All product from the unit was well below shipment specifications.

C. Neptunium Purification - S. M. Nielson

The resin in Tank Q5 was water washed until analyses of the wash water decreased to  $1 \times 10^7$  count/min/gal alpha. The water was transferred to Tank Q3 through the temporary line containing a screen. Tank Q3 was then water flushed and jetted to Tank R8. A 1.5 specific gravity solution of ANN was added to Q5 and jetted to R8 in an effort to float the resin out of Q5.

The neptunium on the resin in Q4 Column was eluted to Tank Q3 and concentrated (Q2). In an effort to change the valence of the neptunium in Tank Q3 to the +IV state without adding more iron (FeSA), Tank Q3 was buffered to 0.2 M  $N_2H_4$  and heated to 60° C. During the heating step, the recirculating line containing the temperature measuring element of Tank Q3 plugged. In an effort to bring the contents of Q3 to the desired temperature, the steam flow was increased to the coils causing the tank contents to boil. It is believed that the combination of high temperature, 0.2 M  $N_2H_4$  and high acid caused rapid formation of gases (oxide of nitrogen) at the surface of the steam coils. This rapid formation of gases at the bottom of the Tank Q3 caused part of the liquid in the tank to "bump" out into the Hot Cell vent system and into Tanks Q1, Q8 and Q5. Analyses of the contents of these tanks verifies this.

An inspection of the Q3 filter showed quantities of grayish precipitate along with resin. A sample of this material was sent to Separations Chemistry for identification. The material was found to be 50 per cent

degraded resin and 50 per cent unknown organic or silicate. A spec analysis iron in the ratio of 2 to 1 silica plus detectable aluminum was also noted. The solid appeared to have an affinity for plutonium as an alpha count showed 60 per cent plutonium and 40 per cent neptunium while the solution itself was 60 per cent neptunium alpha and 40 per cent plutonium alpha.

VI. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $4.29 \times 10^3$  uc/gal Zr-Nb and  $3.59 \times 10^3$  uc/gal Ra. In an effort to increase plant capacity, the solvent inventory in G-Cell was bumped from 29 per cent up to 30.5 per cent with 55F. The over-all loss from both systems was 1,036 gallons. The No. 2 solvent system activity has averaged 3 uc/gal Zr-Nb and 30 uc/gal Ra. The EK-FI carbonate wash changeout was increased from once/24 hours to once/12 hours in an effort to lower EK-R7 Pu retention which reached a high of  $1.6 \times 10^{10}$  by the end of the period, this had dropped to  $1.05 \times 10^{10}$ .

VII. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

The start-up F-Cell flowsheet was the same as that in use prior to shutdown with the IWF stream being made 0.001 M in sugar. During the shutdown, the IWW concentrator, E-F6, was jetted empty.

An analyses of EK-204 dropout data from the previous run period indicated that, on all occasions when a check could be made, the average sugar flow was only 75 per cent of the recommended flow. Such flow discrepancies are a result of the F & O Gallery flow control system which depends on manual valve adjustments for control.

VIII. Waste Treatment and Storage - R. C. Forsman

Approximately 660,000 gallons of supernatant solution was transferred from EK-102-A in the 241-A Tank Farm to non-boiling storage in 241-C Farm in preparation for sluice-mixing tests in EK-103-A. Installation of special pumps and transfer lines for the test are underway.

IX. Fission Product Recovery - W. S. Schmidt

A. Cerium and Rare Earths

After three days of storing the cerium-rare earth product in EK-6-1, a leak developed in the cooling coil. This tank was originally installed in E-Plant. The leakage of cerium to the cooling water header was detected immediately by the cooling water scintillation counter. Background radiation readings are as high as 5 rad/hr at the north pond.

B. Strontium

Approximately 600 gallons of carbonate product were shipped to 003. Sr-89/Sr-90 ratio, calculated by the cesium to strontium ratio, was 6.0 on October 29.

**DECLASSIFIED****X. Feed Preparation - G. J. Raab**

The metal solution which had been stored in TK-D4 since September 2 to "cool" was started to process on October 5. No excessive I131 emission was noted. Dissolver C3 was also returned to service. The metal solution from C3 was butted with mercuric nitrate as an extra precaution. Total I131 emission for the report period was 0.64 curies.

**XI. Miscellaneous - R. W. Lambert****Water Treatment**

On November 8, the main AMC demineralized water filters were changed. The replacement filter elements were of cellulose acetate construction rather than glass fibers and are being used on a test basis.

**DECLASSIFIED**

- Distribution:
- 1 - GE Beaulieu
  - 2 - WS Frank
  - 3 - AM Platt
  - 4 - BE Judson
  - 5 - RW McCullugh
  - 6 - WM Earty
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MI Walling
  - 11 - EB Fecht

PLANT PROCESS PERFORMANCE SUMMARY  
PERM 0000 ON 11/11/63 BY 2400 ON 11/17/63

I. General - P. A. Nicholson

The plant was maintained at a nominal 3.6 (actual 3.65) CF during the report period. Rework of hydrolyzed waste (75) solution was commenced on November 13 at a rate of 100 gallons per batch of feed and terminated on November 17. Rework of product solution continued during the report period at a rate of about 800 units per batch.

II. Performance Data - L. C. Keyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Fu</u>	<u>U</u>	<u>Fu</u>	<u>U</u>	<u>Fu</u>	<u>Np</u>	
First	3.7	3.7			.003	.06	2.5	
Final	3.1	3.4	6.5	1.15	.004		8.0	
Ion Exch.		1.1		0.8				
Overall	6.5	8.2	6.5	1.95	.007	.06	10.5	

B. Production

- 1. HAF = 211 l
- 2. U Production = 214.6 l
- 3. Pu Production = 129.6 Kg
- 4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	964 #	.22
2. Pu	333	.26
3. Np, F16, F18	10	2.0 )Based on U.F. &
K6	25	4.9 ) Rework
4. Solvent	1531 gal	0.1

DECLASSIFIED

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	-Nil-	
2. Pu	16.7 Kg	12.9%
3. Np	46	10.0%

Based on  
M.F.

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	176	104-A
CD	55	105-A
FP	89	105-A
IWW	6	105-A
Flush (F16)	7	105-A
CW	300	109-B

F. IWW Flows

4.6 Avg. HNO<sub>3</sub> Conc. - 5.2 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.5	126
102-A	0.9	89
103-A	0.2	84
104-A	1.0	131
105-A	9.8	95
106-A	6.0	126

III. Flowsheet - G. A. Nicholson

- (1) On November 11, the Solvent-Extraction System was increased to a nominal 3.6 CF rate, except for the following:
  - (a) The HAO was maintained at a nominal 3.3 CF rate.
  - (b) The HSS was maintained at a nominal 3.0 CF rate.
  - (c) The IBP and IBS Streams and the Final Plutonium Cycle streams were maintained at a nominal 3.3 CF rate.
- (2) On November 12, the 2DF-ferrous sulfamate was reduced from 0.36 to 0.32 flow. It was further reduced to 0.29 flows on November 15.
- (3) On November 11, the 2D pulse frequency was increased from 72 to 76 cycles per minute. It was reduced back to 71 cycles per minute later in the day.
- (4) On November 11, the 2E pulse frequency was reduced from 70 to 68 cycles per minute. It was further reduced to 66 cycles per minute on November 14.



DECLASSIFIED

IV. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance was generally good during the report period. Waste losses were considerably above normal, averaging about 0.08 per cent. The major factor contributing to the high losses appeared to be the F8 rework, but other factors included product rework, rework of product-bearing sump solutions, and malfunction of both the HA Extraction Section D.P. and the HSP gamma monitor instruments. The activity of the uranium (IBU) stream was normal, but the IBP stream activity was about two-fold higher than normal during the report period. The IC Column performance was improved by the chemical flush. Losses were nondetectable until the last of the report period and then responded to increased ICX flow.

Performance of the Final Plutonium Cycle was substantially improved over the last run period with the DF averaging about 3000 in spite of below normal recycle losses (~ 0.5 per cent). All product was within shipment specifications after processing through the Anion Exchange Cycle.

Performance of the Final Uranium Cycle continued to be very good, with the Zr-Nb DF averaging 1300. The ruthenium activity in the final product, which was decreasing sharply at the end of the last report period, reversed and increased about midway in the report period, requiring silica gel treatment of ten batches of UNH to bring the total activity within shipping limits. Plutonium contamination in the UNH decreased quickly after the ferrous sulfamate flow was increased and remained steady at three to five ppb during the remainder of the report period.

Neptunium accumulation in the Solvent Extraction System continued at a good rate. Losses via the HAW averaged only two per cent and were in good agreement with the batch loss figures. Losses via the 2DU stream, however, were about two-fold higher, averaging eight per cent and reflected the increasing neptunium inventory in the system. The Neptunium Recovery Cycle remained shut down awaiting completion of processing of the previous batch of neptunium in the Purification System.

V. Product TreatmentA. Uranium Silica Gel - G. A. Nicholson

The Silica Gel Facility was shut down on November 12 for lack of feed. Though the product quality was excellent at shutdown, the unit was regenerated. It was started up on November 16 with the DF averaging about six.

B. Neptunium Purification - S. M. Nielson

As of the first of the report period, all neptunium (1035 gr) in Q-Cell had been segregated and stored in Tank Q3. Because of the precipitate material mentioned in the last report period, extensive flush was planned for Q-Cell. The general plans were as follows:

1. Adjust the valence of the feed in Q3 by digesting in 0.1 M  $\text{N}_2\text{H}_4$  for two hours.
2. Loading the neptunium on the resin in T-Q4.

DECLASSIFIED

HW-76912

3. Jet loading wastes to J1.
4. Extensively flush vessels E-Q2, Q1, Q3, Q5 and Q8.
5. Displace neptunium in J-Cell Package to Q-Cell.
6. Elute T-Q4 to Q3.
7. Replace resin in T-Q4 and flush column.
8. Adjust valence and acid of Q3 and load on new resin in T-Q4.

On November 14, the contents of Tank Q3 was butted to 0.1 M  $N_2H_4$  and digested for two hours at 60° C. Only 18 per cent of the neptunium was reduced to Np-IV. On November 17, the step was repeated and ninety per cent of the neptunium was reduced. The loading step was started on November 18.

VI. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $2.65 \times 10^3$  uc/gal Zr-Nb and  $6.16 \times 10^3$  uc/gal Ru. The over-all loss from both systems for the period was 1531 gallons. The IOS scrub flow has been less than adequate due to apparent plugging of the IOS DOV. The No. 2 system activity has averaged 2 uc/gal Zr-Nb and 60 uc/gal Ru. The TK-R1 carbonate wash changeout cycle was reduced from once/12 hours to once/24 hours.

VII. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

On November 12, the water flow to the E-F6 demister (~ 2 gpm) was turned off to determine what influence, if any, it has on the amount of entrainment carried over into the acid absorber T-F5. By the end of the report period, a slight increase in AAA acid activity had been noted. During the test, sugar flow to E-F6 remained unchanged.

IX. Fission Product Recovery - W. C. Schmidt

Cerium and Rare Earths

The cooling water in the North Pond was allowed to flow to the first 1000 feet of the overflow ditch. Approximately 30 curies of cerium-144 and .05 curies of strontium-90 were discharged in 1.3 million gallons of water. The ditch was backfilled to prevent contamination spread and bypassed to prevent further discharge of fission products to this soil column.

X. Feed Preparation - G. J. Raab

The past run period non-representative metal was higher in Pu-240 content than most runs. The in-plant inventory, plus starting dissolver operation with metal of near maximum representative specification produced plutonium product which exceeded representative specifications. A program of charging the lowest Pu-240 content metal available was in progress. The I131 emission took a jump for no apparent reason; mercuric nitrate was added to metal solution, but really after the rise had been noted. I131 emission for the period was 2.99 curies.

**DECLASSIFIED**

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - AM Platt
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - WM Harty
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling
  - 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 11/18/63 TO 2400 ON 11/24/63

I. General - G. A. Nicholson

Normal processing continued at a nominal 3.6 (actual 3.6) CF rate during the report period. Processing of feed material of an unclassified irradiation history was commenced on November 24 without the usual segregation.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	Gamma dF		% Recycle		% Loss			Np Accum.
	U	Pu	U	Pu	U	Pu	Np	
First	3.8	3.8			.003	.03	3.5	
Final	3.2	3.4	6.2	1.4	.003		10.0	
Ion Exch.		1.2		1.6				
Overall	7.0	8.4	6.2	3.0	.006	.03	13.5	

B. Production

- 1. HAF = 215 T
- 2. U Production = 212 T
- 3. Pu Production = 126.5 Kg
- 4. Np Production = -0-

C. Overall Loss

	Units	% of Total Production
1. U	867 #	0.2
2. Pu	345 Gr	0.3
3. Np, F16, F18 K6	8 Gr 90 Gr	1.6 18.2
4. Solvent	1041 Gal	0.07

D. Rework

	Units	% of Total Production
1. U	Nil	
2. Pu	3.0 Kg	2.4

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	122	101-A
OWW	77	104-A
CD	26	105-A
FP	89	105-A
IWW	8	105-A
CW	300	109-B

F. IWW Flows

4.7 Avg. HNO<sub>3</sub> Conc. - 4.9 M

G. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.5	127
102-A	1.0	89
103-A	0.2	83
104-A	1.0	181
105-A	9.8	95
106-A	6.0	126

III. Flowsheet - G. A. Nicholson

- (1) On November 19, the 2DF-ferrous sulfamate flow was lost for a short time. It was started at 0.55 flows, then lowered to 0.32 flows later the same day and to 0.29 flows on November 21.
- (2) On November 23, the Neptunium Recovery Cycle was started up under Phase I conditions. It was necessary to reduce the plant rate by about five per cent to accommodate the organic (3BW) flow because of a flow restriction in the IBXF System.

IV. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance was considerably improved over the previous report period, despite numerous minor upsets caused by temporary flow restrictions. The HAW losses were lower, averaging less than 0.06 per cent, and the activities of all streams were on downward trends. Plutonium carry-over into the IBU Stream has been considerably higher at the increased rates, averaging about 0.8 per cent during the report period, compares to less than 0.3 per cent for the previous run period.

Performance of the Final Plutonium Cycle continued to be excellent with an average DF of about 3000 and losses to backcycle averaging less than one per cent. A mild flood occurred in the 2A Column early in the report period because of a temporary loss of then excessive 2AS flow. The condition was corrected easily by temporarily reducing the pulse frequency and flow rates.

Final Uranium Cycle performance has continued to be very good with the Zr-Nb DF averaging 1500 to 2000 and the Ru DF remaining fairly steady at about 30. All product was within specifications without further treatment. The

temporary loss in ferrous sulfamate flow caused a slight increase in plutonium contamination in the product, but not above limits and it quickly returned to normal.

Neptunium accumulation in the Solvent-Extraction System continued at about the same rate as during recent report period, with average losses of two and ten per cent via the HAW and 2EU streams, respectively. Start-up of the Neptunium Recovery Cycle on Phase I conditions commenced after the batch of neptunium which it had been holding was displaced to the Purification Cycle.

V. Product Treatment

A. Plutonium Purification - N-Cell - R. W. Lambert

Operation of N-Cell continued to be good with all product being well within specifications. Rates during the report period were high due to significant amounts of plutonium rework being introduced into the plant and frequent problems arose in maintaining adequate steam supply to the E-N6 concentrator.

During the report period, the SLD sump sampler was overhauled and placed in working order. The SLD sump alarms were also checked for proper operation.

B. Uranium Silica Gel - G. A. Nicholson

The silica-gel unit was operated continuously through November 22, when it was shut down for lack of feed material. Approximately 175 tons were processed and the DF was about 4 at shutdown.

C. Neptunium Purification - S. M. Nielson

Loading of the Q3 material on the resin commenced on November 18 and was completed on November 19. A total of 291 grams (28.3 per cent of original 1035 gr) was lost in the loading step. The loading waste was jetted to J1.

Extensive acid flushing of Q1, E-Q2, Q3, Q5 and Q8 started on November 19 and was completed on November 22. On November 22 and 23, the J-Cell Package contents were displaced to Q-Cell and at the same time, T-Q4 was eluted to Q3. A sample of Q3 showed a total of 1033 grams of which 289 grams were transferred from the J-Cell Package.

On November 23 and 24, the resin was removed from T-Q4 and the column flushed. FRC-Q3-2 had failed during the eluting step and was replaced. The linear polyethylene valve seats of HOV-Q4-9, 10 and 11 were replaced with Teflon and plans made to replace all others to protect against failure because of high temperatures.

VI. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

On November 22, the water to the E-F6 demister was turned back on. Test data indicated that the activity in the AAA recovered acid (TK-F3) increased almost by a factor of two while the water was off.

DECLASSIFIED

VII. Solvent Treatment - G. Ward

The No. 1 solvent system activity averaged  $1.81 \times 10^3$  uc/gal Zr-Nb and  $6.43 \times 10^3$  uc/gal Ru. The over-all loss from both systems was 1041 gallons. The IOS scrub flow was returned to normal when the organic heel in TK-G2 was removed. The No. 2 solvent system activity has averaged 6 uc/gal Zr-Nb and 60 uc/gal Ru.

VIII. Fission Product Recovery - W. C. SchmidtStrontium

Approximately 1500 gallons of carbonate product was shipped to 003. Sr-89/Sr-90 ratio, calculated by cesium to strontium ratio, was 7.2 on November 15.

IX. Fission Product Purification - P. W. SmithTechnetium Recovery

During the Cs-137 loading in October, the cesium cask effluent (23,500 gallons) was passed through the technetium cask at nominal rates of 3 gpm. It was later estimated by Frank Roberts after analysis of 100 ml of the loaded resin that assuming about 400 gallons of resin, the cask should be loaded with about 1100 grams of technetium. Analysis of the effluent solution from the cask further indicated that the resin was approaching approximately 50 per cent breakthrough at the end of the cycle.

The loaded cask was moved to the Strontium Semiworks and the 1500 liter resin bed was washed with 6000 liters of dilute sodium nitrate followed by 2400 liters of dilute (0.25 M) nitric acid.

The technetium was then eluted to Tank 55 with approximately 8 column volumes or 12000 liters of 6 M nitric acid which was continuously concentrated and steam stripped for nitric acid removal. Finally the bed was purged of residual nitric with 2400 liters of water. The contents of Tank 55 were reduced to final volume of 350 liters, moved to Tank 6 where it was further reduced to 250 liters, and then moved to Tank 64 for final concentration to 80 liters. The conventional A-Cell - D-Cell loadout routings were used to transfer the technetium into a 40-gallon Hanford Laboratories cask at a final volume including flushes of about 100 liters.

No unusual problems were encountered although analysis of the final product indicated that the iron concentration was several fold above expected, but still relatively insignificant as to corrosion damage to the coil or tank. Less than 1 per cent of the technetium was found in the condensate.

Analytical data for the loading cycle, elution cycle and loadout are summarized below. All analytical data are those of Frank Roberts. The analyses determined by the Purex Laboratory and Milt Campbell are inconsistent.

| **DECLASSIFIED**Loading Cycle

103-C Supernate Feed:	0.066 grams Tc per gallon
Throughput, Gallons	Conc. Tc in effluent; g/gal
5 000	0.022
7 800	0.017
9 800	0.018
15 000	0.031
16 000	0.032
23 000	0.040

Estimated Cask Load: 1100 grams

Elution Cycle

Eluant, Gallons (6 M HNO <sub>3</sub> )	Gram Tc Eluted, Cumulative
1 500	155
3 900	421
6 300	725
8 700	893
11 100	986
13 100*	1 088

\*Bed was flushed with 2000 liters of process water to push the final 500 liters of acid eluant out and leave about 500 liters of water in the cask.

During transfers from Tank 55 to Tank 6 and finally to Tank 64 and subsequent concentrations, an apparent loss of approximately 50 grams may have occurred although it is felt that this discrepancy is more likely to be a result of manometer inaccuracies.

Loadout Cycle - Tank 64 prior to loading:

80 liters containing - 1025 grams Tc  
54 g/l Fe  
5.3 M HNO<sub>3</sub>

Combined flush of lines, vessels after loading: 120 liters containing  
2.3 grams Tc.

X. Feed Preparation - G. J. Raab

The I<sub>131</sub> emission problem disappeared and mercuric nitrate addition was discontinued. The I<sub>131</sub> emission was 0.85 curies for the period. On November 23, the first charge of non-representative metal was made. No attempt was made to segregate as the plutonium product was still slightly above specification.

**DECLASSIFIED**

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - AM Platt
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - WM Harty
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling
  - 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 11/25/63 TO 2400 ON 12/1/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.6 CF processing rate during the report period. However, a slight flow restriction with IBXF System limited the actual rate to about 3.41 until November 26, when it was increased to 3.6 CF. Processing of unclassified feed material continued.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	4.2	4.0			.002	.03	4.5	
Final	3.1	3.5	7.0	2.3	.002		7.5	
Ion Exch.		1.0		2.8				
Overall	7.3	8.5	7.0	5.1	.004	.03	12.0	

B. Production

- 1. HAF = 180.5 T
- 2. U Production = 195.9 T
- 3. Pu Production = 135 Kg
- 4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	739 #	0.2
2. Pu	306 Gr	0.2
3. Np, F16, F18	28.0	4.8 )Based on Input
K6	38.0	6.6 )from Virg. Feed
4. Solvent	2075 Gal	0.16

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	13.9 T )Primarily From P2	7.1
2. Pu	3 Kg - L11 ; 2.6 Kg - J1	4.1
3. Np	275 Gr - J1 ; 115 Gr - Q5	67.5 )Based on Input from Virg. Feed



**DECLASSIFIED****E. Volumes to UGS, Gal/Ton**

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	245	104-A
CD	13	105-A
FP	76	105-A
IWW	14	105-A
CW	300	109-B

**F. IWW Flows**4.7      Avg.  $\text{HNO}_3$  Conc. - 5.7 M**G. Tank Farm**

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (<math>^{\circ}\text{C}</math>)</u>
101-A	1.5	126
102-A	1.0	89
103-A	0.2	84
104-A	1.0	130
105-A	9.8	96
106-A	6.0	124

**III. Flowsheet - G. A. Nicholson**

- (1) On November 26, for a brief period, the free sulfamic acid concentration in the IBX was two-fold above flowsheet. The  $2\text{AF-NaNO}_2$  flow rate was increased accordingly.
- (2) On November 28 and 29, the  $2\text{DF-ferrous sulfamate}$  flow rate was low because of control difficulties. The problems were corrected and the rate was increased temporarily to 0.44 flows, then reduced back to 0.29 flows on November 29.
- (3) On December 1, the HAX was reduced by about ten per cent because of the recurring flow restriction in the IBX<sup>F</sup> System.
- (4) On November 29, the Neptunium Recovery Cycle was changed to Phase II conditions. Transition to Phase III conditions was made on November 30.

**IV. Solvent Extraction - G. A. Nicholson**

Solvent-extraction performance was generally very good during the report period, with all product within shipping limits without supplemental treatment. Major problems during the latter part of the report period were excessively high plutonium levels in the Backcycle Waste System and flow restrictions in the 3WB Stream. Consequently, the 3WB inventory was at a maximum on December 1 and the normal process control limits were exceeded with respect to plutonium inventory in the 3WB Tank. A batch of cold feed was recycled on December 1 to allow the plutonium level to decline and a substantial gamma burst ensued. Waste losses were also on an upward trend during the latter half of the report period, but the over-all average was less than 0.05 per cent.

Final Plutonium Cycle performance continued at the same high level as during the previous report period, with excellent DF's and low losses to recycle.

Final Uranium Cycle performance has also continued to be excellent with all product within shipping limits without silica gel treatment. The plutonium contamination in the uranium product increased to near limits for a brief period when the ferrous sulfamate addition was lost (because of faulty control system), but quickly returned to the normal 3 to 5 ppb when the flow was restored.

Neptunium accumulation in the Solvent Extraction System continued at about the same rate as during recent weeks. A temporary loss of saturation in the 2D Column caused high losses via the 2EU Stream late in the report period and the losses averaged about ten per cent. Neptunium losses via the HAW Stream averaged about six per cent for the report period. Accumulation in the Neptunium Recovery System was very rapid after the "package" was started up on November 23. Transition to Phase II was made on November 29 and to Phase III on November 30 with approximately 2000 units accumulated. The package was shut down after completion of Phase III because of difficulties with high plutonium inventory in the Backcycle Waste System and 3WB flow difficulties.

V. Product Treatment

A. Plutonium Purification - N-Cell - R. W. Lambert

Operation of N-Cell continued to be excellent with no signs of push problems. To date, the same charge of resin has been in service 30 days with no resin additions or changeouts. This is the longest run on a single resin batch for well over a year. Also satisfying was the fact that plutonium throughput during the period was considerably above normal.

B. Uranium Silica Gel - G. A. Nicholson

The Silica-Gel Unit was shut down during the report period for lack of feed.

C. Neptunium Purification - S. M. Nielson

The neptunium solution in Q3 was butted to 0.1 M FeSA and N<sub>2</sub>H<sub>4</sub>. The column was filled with new resin; however, the sonic probe did not respond. When the resin was pretreated, no pressure could be built up in the column. The rupture disk was found to be failed and was replaced.

The resin loading step was completed on November 27 with a loss of 400 gr (39.7 per cent). The Plutonium Scrub was started on the same day and was completed on November 28. An additional 485 grams (47.8 per cent) were lost during this step. A Fission Product Scrub and Elution step were completed with 32 grams collected in the product tank.

All neptunium except the 32 grams in Q6 and 35 grams in the Elution Forecut (Q3) were returned to the Backcycle System.

On November 30, Phase III was started and a total of 2344 grams were collected in Tank Q3. On December 1, the resin in Q4 was loaded out to check its volume. A full 28 liters was accounted for. Loss of the run is believed to be due to poor valence control caused by crud in the feed.

DECLASSIFIED

VI. Solvent Treatment - G. Ward

The No. 1 solvent system activity averaged  $1.07 \times 10^3$  uc/gal Zr-Nb and  $3.88 \times 10^3$  uc/gal Ru. The over-all loss from both systems was 1837 gallons of organic. The OWW waste back-up into TK-R8 was traced to a faulty valve and 241-A Tank Farm. This has been corrected. The No. 2 solvent activity has averaged 1 uc/gal Zr-Nb and 40 uc/gal Ru.

V. Fission Product Recovery - W. C. Schmidt

Strontium

Approximately 1400 gallons of carbonate product was shipped to 003. Sr-89/Sr-90 ratio, calculated by the cesium to strontium ratio, was 8.2 on November 2.

VI. Feed Preparation - G. J. Raab

The dissolvers have been operating very well and have had periods of 1 to 2 hours slack time and still maintaining feed enough for the 3.6 CF rate. The metal age was 140 to 270 days. The I<sub>131</sub> emission was 0.70 curies for the period.

- Distribution:
- 1 - CF Beaulieu
  - 2 - WS Frank
  - 3 - AM Platt
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - WM Harty
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling
  - 11 - JB Fecht

**DECLASSIFIED**

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 12/2/63 TO 2400 ON 12/9/63

I. General - G. A. Nicholson

The plant was maintained at a nominal 3.6 (actual 3.3) CF until December 3, when it was shut down at equilibrium to change out the 3WB flowmeter and valve jumpers which appeared to be plugged. In addition to 3WB flow problems, the processing rate was limited by a flow restriction in the IBXF system. The plant was started up early on December 6, but another flow restriction in the 3WB jumper necessitated a shutdown later the same day. It was started up early on December 7, but a failed 3WB pump necessitated another shutdown on December 8.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First								
Final								
Ion Exch.								
Overall								

(Start-up -- No Typical Data)

B. Production

- 1. HAF = 92.6 T
- 2. U Production = 105.1 T
- 3. Pu Production = 67.1 Kg
- 4. Np Production = 1875 Gr

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	413 #	0.19
2. Pu	212 Gr	0.31
3. Np, F16, F18	10.0 Gr	4.9
K6	20.0 Gr	9.8
4. Solvent	4157 Gal	0.31

} % of Input

**DECLASSIFIED**D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	8460 #	4.0
2. Pu	2.0 Kg	3.0

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	571	104
CD	28	105
FP	195	105
CW	300	109-B

F. IWW Flows4.1 Avg. HNO<sub>3</sub> Conc. - 5.7 MG. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.8	125
102-A	0.8	89
103-A	0.2	83
104-A	2.9	78
105-A	10.7	96
106-A	5.1	89

III. Flowsheet - G. A. Nicholson

No flowsheet changes of significance were made during the report period.

IV. Solvent Extraction - G. A. Nicholson

Because of the numerous start-ups and shutdowns, steady-state operation was not achieved during the report period. Rework of a mixture of cold uranium from a UNH Storage (P2) Tank and 3WB (50-50) at the end of the last report period, which was necessitated by high plutonium inventory in the Backcycle Waste System, caused a gamma burst throughout the plant. The 2BP was diverted to the Final Plutonium Concentrator to prevent high activity from reaching the Plutonium Anion Exchange Cycle. The activity had just about dissipated when the plant was shutdown on December 3. The two subsequent start-ups were accomplished without any difficulty and only the normal mild activity burst of short duration. No plutonium product was loaded after December 3 and the three batches of uranium at the end of the report period were high in plutonium contamination.

V. Product TreatmentA. Plutonium Purification - N-Cell - R. W. Lambert

Operation of N-Cell during the report period was discontinued because of the unscheduled plant shutdowns. During the down time, a new E-N6

**DECLASSIFIED**

steam DOV was installed and the conductivity recorder (CRC) cleaned and recalibrated. No resin change was made.

**B. Neptunium Purification - S. M. Nielson**

On December 2, the T-Q<sup>4</sup> Column was filled (28.3 liters) with new resin in preparation for Purification Run #7. Minor maintenance work was completed to protect against needlessly breaking any more Q<sup>4</sup> rupture discs. The pressure relief valves on the remote head were set at 56#/in<sup>2</sup>.

Loading of the 23<sup>44</sup> units in Q3 started on December 3 and was completed the following day. A short Plutonium Scrub of 14 column volumes was made followed by an 11 column volume Fission Product Scrub. The SMQ<sup>4</sup> scintillation monitor was used to determine when the ZrNb had essentially been scrubbed from the resin column.

Elution was completed on December 6 on swing shift. The valence adjustment step was made and the product loaded into bottles for shipment on December 7.

The following tables summarize the losses and DF's achieved for the run:

Losses For Run #7

Loading Step	1.26%
Pu Scrub	11.3 %
FP Scrub	5.8 %
Elution Forecut	3.1 %

\*DF's Achieved For Run #7

Pu	3.9
U	7200
Zr	2200
Ru	430
TMI	7.0

The high losses are attributed to the fact that the resin was nearly completely saturated with neptunium. Breakthrough on the loading step was not achieved even though the resin was loaded to 82.8 gr Np/liter of resin. This number is subject to some error because of possible error in resin volume measurement.

A total of 1874 grams of Np was loaded for shipment. All shipping specifications were met.

\*(on a g/g Np or mc/g Np basis).

DECLASSIFIED

VI. Solvent Treatment - G. Ward

The No. 1 solvent system activity averaged  $1.64 \times 10^3$  uc/gal Zr-Nb and  $2.24 \times 10^3$  uc/gal Ru. The overall solvent loss from both systems was 2148 gallons. During the period of shutdown during this week, both solvent systems were spun to clean up organic. The No. 2 solvent system activity averaged 1 uc/gal ZrNb and 30 uc/gal Ru.

VII. Acid Recovery and Waste Concentration - R. W. LambertF-Cell Acid Recovery

During the first half of the report period, the activity in the AA recovered acid, TK-F3, fluctuated widely. The ruthenium was running nearly twice previous norms and there was considerable doubt as to the amount of sugar actually being added via the P & O Gallery flow control system. This problem should be corrected, however, since during one of the unscheduled plant shutdowns, the sugar flow was switched to the old formaldehyde flow control system and can now be controlled from Central Control.

VIII. Waste Treatment and Storage - R. J. Forsman

Tests completed in the 300 Area using the new slow speed Purex agitator indicated excellent mixing of phases with Sp.Gr. of 0.80 to 1.35. Based upon these tests, a slow speed agitator was installed in TK-F13 for use in future trilauryl amine extraction tests. The main differences between the new type agitator and the standard Purex agitator are as follows:

	<u>New</u>	<u>Old</u>
rpm	300	600
Diameter of Blades	14"	9"
Number of Blades	10"	4"
Pitch of Blade	10"	None
Width of Blade	4"	2"
Distance Blades from Tank Bottom	~ 3"	13" and 63"
Normal Amperage while Operating	~ 10-15 amps	~ 27 amps

IX. Fission Product Recovery - W. C. SchmidtStrontium

Approximately 1500 gallons of carbonate product was shipped to 003. Sr-89/Sr-90 ratio, calculated by the cesium to strontium ratio, was 4.4 on December 2.

X. Feed Preparation - G. J. Raab

Dissolver operation good. I<sub>131</sub> emission for the period 0.95 curies.

**DECLASSIFIED**

Distribution:

- 1 - OF Beaulieu
- 2 - WS Frank
- 3 - AM Flatt
- 4 - KE Hudson
- 5 - RW McCullugh
- 6 - WM Harty
- 7 - SG Smolen
- 8 - RE Tomlinson
- 9 - AJ Waligura
- 10 - ME Walling
- 11 - BS Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 12/10/63 TO 2400 ON 12/15/63

I. General - G. A. Nicholson

Normal processing was resumed early on December 10 at a nominal 3.3 CF. (An attempted start-up on December 9 was foiled by failure of the HAF pump.) The rate was increased to a nominal 3.6 CF on December 11. However, because of the continuing problem with the EXF flow, the actual rate averaged only about 3.4 for the remainder of the report period. A batch of cold uranium was recycled to the ZAF on December 13 to lower the high plutonium inventory in the Backcycle Waste System.

II. Performance Data - D. C. Leysen

A. Solvent Extraction Performance by Cycles (Typical)

	Gamma dF		% Recycle		U	Pu	Ru	Np Accum.
	U	Pu	U	Pu				
First	3.7	4.0			.009	.03	2.0	
Final	3.0	3.7	6.0	2.7	.016		10.0	
Ion Exch.		1.1		1.0				
Overall	6.7	8.8	6.0	3.7	.026	.03	12.0	

B. Production

- 1. HAF = 150.2
- 2. U Production = 157.7
- 3. Pu Production = 103 Kg
- 4. Np Production

C. Overall Loss

	Units	% of Total Production
1. U	649 #	0.20
2. Pu	244 Gr	.24
3. Np, F16, F18 K6	12.0 48	2.5 ) % of Virg. 10.0 ) Input
4. Solvent	2711 Gal	0.22 % of Gal Pumped



**DECLASSIFIED****D. Rework**

	<u>Units</u>	<u>% of Total Production</u>
1. U	8.2 M	5.2
2. Pu	Nil	

**E. Volumes to UGS, Gal/Ton**

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	239	101-A
JD	57	105-A
FP	92	105-A
IWW	0	
CW	300	109-B

**F. IWW Flows**

5.0      Avg. HNO<sub>3</sub> Conc. = 4.5 M

**G. Tank Farm**

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.81	125
102-A	.83	89
103-A	.21	84
104-A	2.93	127
105-A	10.7	96
106-A	5.08	89

**III. Flowsheet - G. A. Nicholson**

- (1) The plant was started up on December 10 under the same flowsheet conditions in existence at the shutdown, except that the 2A pulse frequency was 70 rather than 95 cycles per minute. It was increased to 92 cycles per minute later the same day.
- (2) On December 10, the 2DF-ferrous sulfamate was increased from 0.37 to 0.48 flows.
- (3) On December 10, the Neptunium Recovery Cycle was started up at nominal 3.6 CF rates.
- (4) On December 15 (early), the Neptunium Recovery Cycle was converted to Phase II conditions. It was necessary to commence transferring the 3WB to H1 in small increments to compensate for the increased 3WB flow.
- (5) On December 15, the 2DF ferrous sulfamate make-up was changed - the ferrous sulfamate concentration was changed from 2.0 to 1.0 molar and the solution was made 1.0 molar in sulfamic acid. The addition rate was then increased to 0.58 flows, but the total iron added was reduced by 40 per cent. (This was still 80 per cent above flowsheet, however.)

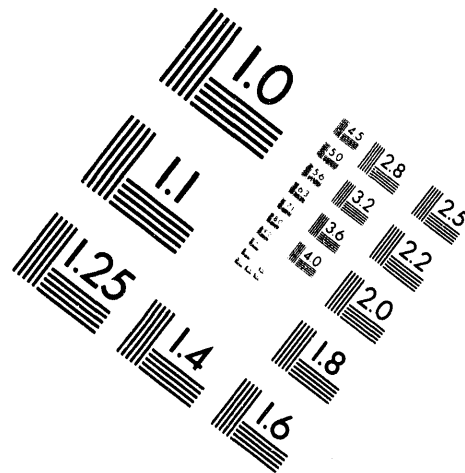
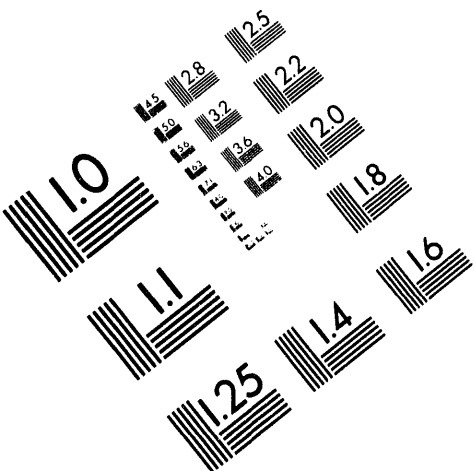


**AIM**

**Association for Information and Image Management**

1100 Wayne Avenue, Suite 1100  
Silver Spring, Maryland 20910

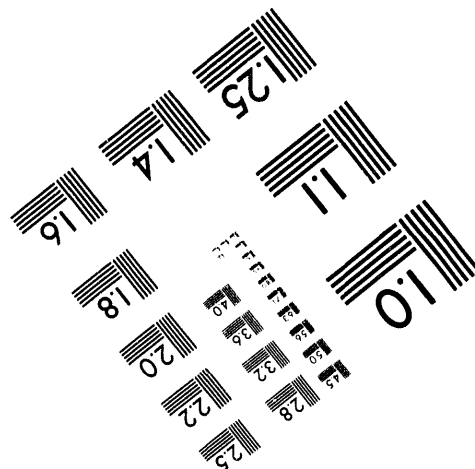
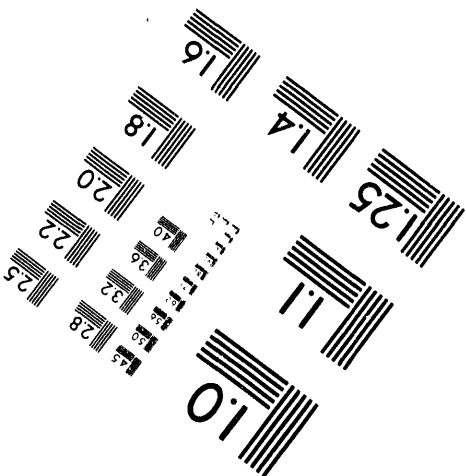
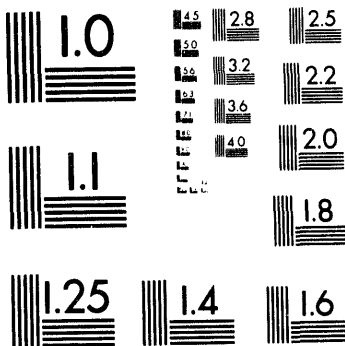
301/587-8202



Centimeter



Inches



MANUFACTURED TO AIM STANDARDS  
BY APPLIED IMAGE, INC.

**3 of 3**

~~184~~ DECLASSIFIED

- (6) On December 15, the solvent wash in the Second Organic System was changed back to a simple carbonate wash in the 20 Column because of failure of the R1 Recirculation Pump.

#### IV. Solvent Extraction - G. A. Nicholson

The Solvent Extraction System was still unsettled because of the continuing flow problems with the 3WB and IBXF Streams. The losses to the HAW remained generally low, averaging less than 0.05 per cent in spite of two periods of temporary high loss because of low 3WB flow.

A sharp decrease in decontamination performance of the Final Uranium Cycle occurred during the week. This was probably caused by poor solvent quality. (Early in the week, the IBF Recirculation Pump appeared to have failed, but nothing was done until December 14 when it became very noisy.) Apparently, there had been no solvent treatment since the plant was started on December 10. As a result, all uranium product required segregation - either for rework or processing through the Silica Gel Facility.

Final Plutonium Cycle performance has been excellent since start-up, with all product well within limits for processing in the Plutonium Anion Exchange Cycle.

Neptunium accumulation has been generally good, but losses have been up because of the frequent upsets, averaging about three and ten per cent via the HAW and 2EU Streams, respectively. Accumulation in the Neptunium Recovery Cycle was rapid and about 1800 units were ready for transfer to the Purification Cycle by the end of the report period.

#### V. Product Treatment

##### A. Plutonium Purification - N-Cell - R. W. Lambert

Operation of N-Cell continued satisfactorily during the report period with the original resin completing its seventh week of continuous use. Plutonium input to N-Cell continued at near capacity levels as a result of plant rates and processing high exposure non-representative metal.

##### B. Neptunium Purification - S. M. Nielson

The product from the Neptunium Purification Run #7 was shipped on December 9. On the same day, an entry was made in the Hot Cell to install a new resin level detector probe in the column.

The waste (402 units Wp) in Tank Q5 from the Plutonium and Fission Product Scrubs from Run #7 was jettied to the Backcycle System on December 13.

The dilute acid in Tank Q3 was concentrated in E-Q2 in preparation for another batch of material from the N-Cell Package which was expected during the next report period.

##### C. Uranium Silica Gel - G. A. Nicholson

The Silica Gel Unit was started up on December 15.

**DECLASSIFIED**VI. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $2.10 \times 10^3$  uc/gal ZrNb and  $2.58 \times 10^3$  uc/gal Ru. The overall solvent loss from both systems was 3888 gallons, approximately 2000 gallons of this solvent is in TK-R8 and will be recovered. The TK-R1 recirculation pump failed on December 10 and was discovered on December 14. While waiting for replacement, a carbonate wash was used in the 20W (TK-R2) in place of the normal nitric acid. The No. 2 solvent plutonium retention reached a high of  $6.6 \times 10^8$  on December 14 prior to putting carbonate in T-R2 and dropped back to  $3 \times 10^6$  within 12 hours after carbonate was introduced. The No. 2 solvent system activity averaged 5 uc/gal ZrNb and 60 uc/gal Ru.

VII. Fission Product Recovery - W. C. SchmidtStrontium

Approximately 1400 gallons of carbonate product was shipped to 003. Sr-89/Sr-90 ratio, calculated by cesium to strontium ratio, was 4.2 on December 10.

VIII. Feed Preparation - G. J. Raab

Dissolver operation has been good. I131 emission was low ( $\sim 0.06$ ) curies/day until December 12 when it started climbing. On December 14, the emission peaked at 2.32 curies per day. The stack sample contained a considerable amount of I132. Hanford Laboratories estimated one to three slugs of 3 to 6 days age had been mixed into a bucket of metal from the ratio of the short-lived isotopes in the stack sample. Total emission for the period was 5.81 curies.

**DECLASSIFIED**

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - AM Platt
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - WM Harty
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling
  - 11 - JB Fecht

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 12/16/63 TO 2400 ON 12/22/63

I. General - G. A. Nicholson

The plant was shutdown at equilibrium on December 16 to permit replacement of a failed 3WB pump and a failed 2OF Recirculation Pump. It was started up early on December 17 at a nominal 3.3 CF and shutdown at equilibrium again on December 18 to permit replacement of a failed 3WF pump. Operation was resumed about 16 hours later at a nominal 3.3 CF. The rate was increased on December 20 and maintained at a nominal 3.6 for the remainder of the report period. About one and one-half batches of cold uranium were recycled from the UNH Storage (P2) Tank to permit reworking of plutonium solution.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.7	3.9			.003	.025	7.0	
Final	3.1	3.2	7.0	2.8	.003		12.0	
Ion Exch.		1.0		0.15				
Overall	6.8	8.1	7.0	2.95	.006	.025	19.0	

B. Production

- 1. HAF = 147.8
- 2. U Production = 162.0
- 3. Pu Production = 108.8 Kg
- 4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>	
1. U	725 #	0.22	
2. Pu	228 Gr	0.21	
3. Np, F16, F18	18 Gr	5.2	)% Input
K6	36 Gr	10.4	)
4. Solvent	2017 Gal (Gain)	0.14	% of Vol Pumped

**DECLASSIFIED****D. Rework**

	<u>Units</u>	<u>% of Total Production</u>
1. U	Nil	-
2. Pu	5959 Gr	5.5

**E. Volumes to UGS. Gal/Ton**

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
WW	217	101-A
	16.3	104-A
	24	105-A
WF	101	-
WW	0	-
OW	300	109-B

**F. LWW Flows**

3.8 Flows      Avg HNO<sub>3</sub> Conc. = 5.5 M

**G. Bank Farm**

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. °F</u>
101-A	1.8	127
102-A	0.8	89
103-A	0.2	89
104-A	2.9	127
105-A	10.7	97
106-A	5.1	90

**III. Flowsheet - S. A. Nicholson**

- (1) On December 16, the 2DF-ferrous sulfamate was reduced from 0.58 to 0.48 flows.
- (2) On December 16, transition from Phase II to Phase III was made in the Neptunium Recovery Cycle. The periodic transfer of 3WF to the HAF Bank was terminated. On December 17, the "package" was started up on Phase I conditions.
- (3) On December 17, the regular solvent wash procedure was reinstated in the Second Organic System.
- (4) On December 17, the 3WE was transferred to H1 in small increments for about four hours while the 3WE Jumper was replaced.
- (5) On December 19, the 2EX temperature was increased from 48 to 53° C. It was reduced to 50° C on December 21.

**IV. Solvent Extraction - S. A. Nicholson**

The solvent extraction performance picture was clouded by the numerous shutdowns during the report period. Despite the resulting upsets,

however, performance was generally improved over the previous several weeks. Waste losses averaged less than 0.03 per cent and were below the normal values during start-up. Decontamination performance of the First Cycle was also excellent during the report period and no significant blasts of activity occurred during any of the start-ups.

Performance of the Final Uranium Cycle was improved considerably over the previous report period. After a brief burst at start-up on December 18, the Zr-Nb activity decreased rapidly and the uranium product was in specifications without silica-gel treatment by the end of the report period. Four batches of uranium product required segregation because of high plutonium contamination resulting from the start-ups.

Performance of the Final Plutonium Cycle continued to be excellent in spite of erratic 2AW losses to the Backcycle Waste System. The problem appeared to be caused, at least partly, by insufficient nitrite flow to the 2AF Tank. All product was within limits for processing through the Plutonium Anion Exchange Cycle.

Neptunium accumulation in the Solvent Extraction System was slightly below normal during the report period, with losses averaging five and twelve per cent via the HAW and 2EU Streams, respectively. The HAW losses were undoubtedly due to the start-ups, etc.

The cause of the continuing high 2EU loss, however, remains obscure. It does not appear to be a function of uranium saturation in the 2D Column. Accumulation in the Neptunium Recovery Cycle has been normal since its transition to Phase I conditions. Numerous adjustments in 3AF flow have been necessary to compensate for varying uranium concentrations in the 3WB Stream.

## V. Product Treatment

### A. Plutonium Purification - N-Cell - R. W. Lambert

N-Cell continued to perform satisfactorily during the report period operating the majority of the time with the sodium fluoride flow off. By the end of the period, plutonium input to N-Cell had noticeably declined reflecting the change back to processing regular metal.

Early in the period, it was noticed that the E-N6 mud leg was plugged and efforts to free it were unsuccessful. Plans are to continue to operate until the scheduled January shutdown before attempting the necessary maintenance.

### B. Neptunium Purification - S. M. Nielson

Neptunium Purification Run #8 was completed during the report period. Phase III was started in the J-Cell Package on December 16 and by graveyard on December 17, a total of 1942 grams was collected in Tank Q3. Losses during the resin loading step were high due to insufficient pretreatment of the resin column with high acid. The resin loading step was completed and the Plutonium Scrub step started on December 19. At the beginning of the Plutonium Scrub step, waste



losses reached the initial feed concentration indicating that break-through was reached just at the end of the resin loading step. The resin was loaded to 65 gr Np/liter of resin. On continuing the Plutonium Scrub, waste losses dropped to normal.

The Plutonium Scrub consisting of 10 column volumes was followed with a Fission Product Scrub of 20 column volumes.

Product elution was completed on December 20 with a total of 1544 grams recovered.

The following tables show the DF's and waste losses for Run #8:

DF's For Run #8

<u>U</u>	<u>Pu</u>	<u>Zr</u>	<u>Ru</u>
38,200	20	260	60

Losses For Run #8

Loading Step	4.4%
Pu Scrub	7.3%
FP Scrub & Fluoride Scrub	12.0%
Forecut	1.0%

The high losses are attributed to the saturation of the resin column with neptunium.

C. Uranium Silica Gel - G. A. Nicholson

The Silica Gel unit was shutdown for regeneration on December 20. Approximately 125 tons were processed since the last regeneration.

VI. Solvent Treatment - G. Ward

The No. 1 solvent system activity has averaged  $1.15 \times 10^3$  uc/gal ZrNb and  $4.42 \times 10^3$  uc/gal Ru. The overall solvent loss from both systems was 1683 gals. The TK-R1 recirculation pump was replaced on December 16 and the No. 2 solvent system's scrub was back to a normal changeout cycle, with acid in the 20S rather than carbonate. The No. 2 solvent system activity averaged  $\underline{1}$  uc/gal ZrNb and 50 uc/gal Ru.

VII. Acid Recovery and Waste Concentration - R. W. Lambert

F-Cell Acid Recovery

During the report period, the ruthenium numbers in the AAA (TK-F3) recovered acid fluctuated widely as a result of low sugar flow. Following to short duration plant shutdowns, the acid recovery system was started up with the

~~SECRET~~ DECLASSIFIED

HW-76912

-194-

sugar flow to TK-F12 omitted. On each occasion, the ruthenium jumped abruptly reaching  $3 \times 10^5$  uc/gal following the second start-up. Again on December 22, the ruthenium activity jumped sharply following a period of about six hours of no sugar flow while the system was being flushed to remove the sugar mold which had accumulated and was plugging the flow control systems.

Following each ruthenium excursion, the activity returned to the normal operating level of about  $3 \times 10^3$  uc/gal soon after sugar flow was reestablished. Zirconium-niobium levels remained near normal ( $\sim 1.5 \times 10^4$  uc/gal) during the entire period.

X. Feed Preparation - G. J. Raab

Third cuts were taken on all dissolvers before starting charging of representative metal on December 16. The last pure non-representative metal solution was moved forward as a block before storing metal solution dissolved from representative metal charges. The  $I_{131}$  emission slowly returned to normal. Total emission for the period was 2.02 curies.

- Distribution:
- 1 - OF Beaulieu
  - 2 - WS Frank
  - 3 - AM Platt
  - 4 - BF Judson
  - 5 - RW McCullugh
  - 6 - WM Harty
  - 7 - SG Smolen
  - 8 - RE Tomlinson
  - 9 - AJ Waligura
  - 10 - MT Walling
  - 11 - JB Fecht

**DECLASSIFIED**

PUREX PROCESS PERFORMANCE SUMMARY  
FROM 0000 ON 12/23/63 TO 2400 ON 12/29/63

I. General - G. A. Nicholson

The plant was shutdown at equilibrium on December 23 when the HAF Pump failed. Normal processing was resumed on December 24 at a nominal 3.6 CF. Rework of hydrolyzed sump (TK-F8) solution commenced on December 27 at a rate of about 150 gallons per batch and proceeded intermittently during the remainder of the report period.

II. Performance Data - D. C. Leyson

A. Solvent Extraction Performance by Cycles (Typical)

	<u>Gamma dF</u>		<u>% Recycle</u>		<u>% Loss</u>			<u>Np Accum.</u>
	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>U</u>	<u>Pu</u>	<u>Np</u>	
First	3.8	3.9			.004	.03	8.0	
Final	3.2	3.6	6.5	2.9	.004		8.0	
Ion Exch.		0.7		0.5				
Overall	7.0	8.2	6.5	2.95	.008	.03	16.0	

B. Production

- 1. HAF = 194.8 T
- 2. U Production = 194.6 T
- 3. Pu Production = 109 Kg
- 4. Np Production = -0-

C. Overall Loss

	<u>Units</u>	<u>% of Total Production</u>
1. U	973 #	.25
2. Pu	442 Gr	.41
3. Np, F16, F18	35 Gr	8.1 (Virg. Input)
K6	30 Gr	7.0 (Virg. Input)
4. Solvent	2074	0.16

D. Rework

	<u>Units</u>	<u>% of Total Production</u>
1. U	587 #	.15
2. Pu	384 Gr	.35
3. Np		

DECLASSIFIED

E. Volumes to UGS, Gal/Ton

<u>Source</u>	<u>Gal/Ton</u>	<u>Waste Tank</u>
OWW	178	104-A
CD	49	105-A
IWW	19	105-A
FP	87	105-A
CW	300	109-B

F. IWW Flows4.2 Avg. HNO<sub>3</sub> Conc. - 5.9 MG. Tank Farm

<u>Waste Tank</u>	<u>Boil-off, Gal/Min</u>	<u>Temp. (°C)</u>
101-A	1.8	125
102-A	0.8	89
103-A	0.2	87
104-A	2.9	125
105-A	10.7	97
106-A	5.1	90

III. Flowsheet - G. A. Nicholson

- (1) The nitric acid concentration in the 2AF was increased by about 15 per cent intermittently until December 27.
- (2) On December 23, the 2AF-NaNO<sub>2</sub> was increased from 2.9 to 3.2 flows.
- (3) On December 25, the 2DF-ferrous sulfamate was increased from 0.48 to 0.60 flows. It was reduced back to 0.48 flows on December 28.
- (4) On December 24, the Neptunium Recovery Cycle was switched to Phase II operating conditions. Transition to Phase III conditions was made on December 25 and transition back to Phase I on December 26.
- (5) On December 25, the 2EX temperature was decreased from 50 to 48° C.
- (6) On December 28, the 2AX was found to be low because of a faulty flow indicator. The flow was increased back to the normal six flows.

IV. Solvent Extraction - G. A. Nicholson

Solvent-extraction performance was generally excellent during the report period. Start-up after the equilibrium shutdown was accomplished without difficulty, with a relatively mild, brief increase in activity of the HSP Stream. Waste losses averaged less than 0.02 per cent until start of the hydrolyzed waste rework. The rework produced losses averaging about 0.05 per cent, apparently because of over-saturation. The IBXF System continued to be a problem, restricting flow and, therefore, production rate. The

3WB flow has been very steady and easily controlled since a screened-suction pump was installed, but there is a distinct flow limitation that causes some problem when the Neptunium Recovery Cycle is on Phase II operation. HAF flow control has been much improved since the HAF butt step was eliminated and, consequently, operational control of the HA Column is much better.

Performance of the Final Uranium Cycle has shown considerable improvement during the report period, with product quality well within shipping limits by the end of the week despite high ruthenium activity. The plutonium contamination in the uranium product remained above normal, however, at about six ppb. Also, for reasons not yet defined, the neptunium contamination in the uranium product has been above normal of late. Analytical problems are suspect (because neptunium analyses throughout the plant have been unusual), but investigation is continuing.

Performance of the Final Plutonium Cycle has continued at an excellent level, with the DF averaging about 4500. Losses were erratic during the first of the week, but were improved after adjustment of the 2AX flow.

Neptunium accumulation in the Solvent-Extraction System has continued slightly below normal, but the recovery for the year has averaged 85 per cent, vs. less than 50 per cent the previous year. Neptunium analyses in all streams have been rather erratic lately, indicating a possible analytical problem. Accumulation in the "package" has continued to be excellent as has decontamination during Phase II, thus easing the load on the Purification Cycle.

## V. Product Treatment

### A. Neptunium Purification - S. M. Nielson

The product collected from Run #8 required six valence adjustment attempts before specifications were met, making it necessary to add distilled water to reduce product concentration to specifications. The product was loaded out on December 24.

Neptunium Purification Run #9 was started on December 25. A total of 1390 grams was transferred from the J-Cell Package to Q-Cell bringing the total neptunium in Tank Q3 to 1552 grams. Radiation levels in the maintenance hood were a factor of 10 high despite the fact that the ZrNb and Ru levels were normal. The laboratory reports that there is Neptunium-239 (half life 2.35 days) in the system possibly from a green slug.

The resin loading step and ten column volumes of Plutonium Scrub were completed by December 28 and the Fission Product Scrub started. During the Fission Product Scrub, the diaphragm in the remote head pump failed and was replaced. Product elution was completed on December 29 with a total of 1544 grams neptunium collected in Tank Q6, bringing the total production for the year to 10,464 grams. Of this year's total, 9380 grams were from Purex and 1084 grams from Redox.

Losses and DF's for Run #9 are listed in the following tables:

DF's For Run #9

<u>U</u>	<u>Pu</u>	<u>ZrNb</u>	<u>Ru</u>
9200	8.2	150	18

Losses For Run #9

Resin Loading Step	2.20%
Pu Scrub Step	6.90%
FP Scrub Step	16.61%
Forecut	8.1 %

**B. Uranium Silica Gel - G. A. Nicholson**

The Silica-Gel unit was started up on December 23 and operated until December 27 when it was shutdown for lack of feed. About 100 tons were processed. Regeneration of the units commenced after shutdown of processing.

**VI. Solvent Treatment - G. Ward**

The No. 1 solvent system activity has averaged  $1.00 \times 10^3$  uc/gal ZrNb and  $5.75 \times 10^3$  uc/gal Ru. The overall solvent loss from both systems was 2074 gallons. It was necessary to water flush the IOF jumper twice during this period to maintain the IOF flow rate at a 3.6 CF. The No. 2 solvent activity averaged 4 uc/gal ZrNb and 80 uc/gal Ru.

**V. Feed Preparation - G. J. Raab**

The C-Cell silver reactor differential pressure has been building up throughout the run period indicating a plug. The metal charged has been 124 to 150 days old, however, the average was nearly 130 days old. The I<sub>131</sub> emission was 2.18 curies for the period. Silver reactors A2 and B2 are overdue for regeneration on a ton's processed basis.

**DATE**

**FILMED**

**10 / 19 / 94**

**END**

