UNITED STATES ATOMIC ENERGY COMMISSION
RICHLAND OPERATIONS OFFICE

This document consists of

14 pages, No. 1

Project, Series, Date

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TO: H. E. Parker
DIRECTOR - DEPUTY DIRECTOR
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DATE July 14, 1966

HAN-9385 DEL
Report No. 6
June 1966

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CHEMICAL OPERATIONS BRANCH
CHEM. PROC. DIVISION - RLOO

SUBJECT: 200 AREA MONTHLY REPORT FOR

June 1966

DC: AWL/EAA/MJR

PROCESSING STATISTICS

PRODUCTION

PRODUCT				PERCENT EFFICIENCY		PERCENT WASTE LOSSES		ATM RELEASE	
PUREX REDOX URANIUM REDUCTION	URANIUM				OP	MECH	PU	U	CURIES / DAY
	NORMAL		ENRICHED		U-233	Prod.	144.5 kg		
	TONS	PERCENT FORECAST	TONS	PERCENT FORECAST					
	130.2	Th							
	---		240.8	112					
	---		206	103					
PUREX REDOX	BRANDY OR NEPTUNIUM								
	KGS		PERCENT FORECAST						
	---		---						
	---		---						
PLUTONIUM REDUCTION	PLUTONIUM								
	KGS		PERCENT FORECAST						
	204.4		136		71	99	---	---	---
	70		---		---	---	---	---	---

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INVENTORIES

PLUTONIUM			URANIUM	
			NORMAL	ENRICHED
SCRAP BACKLOG 817.4 KGS			HANFORD CODE C-65	
SCRAP COMPOSITION:	PLUTONIUM PROCESSING	35.1	UNH 36 TONS	44 TONS
	FABRICATION	11.9	UO ₃ 11,723.4 TONS	40.4 TONS
	RECOVERY	0.1	RESTRICTED DATA	
	BNW LABORATORY	16.7	DECLASSIFIED	
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	SUPPORTING LABORATORIES	4.6	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.	
	INCINERATOR GROUP 1	26.3		
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PUREX

Thorium processing continued at rates ranging from 5.4 to 6.0 tons of thorium per day with several interruptions. On June 7 the plant was shut down when the HS column flooded and an unsuccessful attempt was made at that time to install a spare LC column cartridge. The plant was restarted the same day, using the regular HS column cartridge, but was forced down again on June 10 by a power outage. After special flushing to clean a gelatinous organic breakdown material from the HS column cartridge plates, the plant resumed processing on June 12. A modified spare cartridge was later installed in the HS column during a brief outage on June 23. A final shutdown for the month was required on June 28 to replace the HAF pump and DOV jumper to restore flow.

The U-233 quality has been excellent. The U-238 impurity was mostly in the range of .6 to 1.5 percent, as compared with the specification limit of 3.9%. The thorium nitrate product was high in Zr-Nb activity, but acceptable on other aspects. The expected six to nine-month storage period will permit decay of the Zr-Nb activity to acceptable levels.

The HS column interface, F6 concentrator, and "A" dissolver weight factor jumpers were replaced.

The "B" dissolver metal temperature, LCU jet discharge and "C" dissolver thermohm and jetout jumpers were repaired.

A special LBXT jet jumper was removed from service.

An old LC column cartridge was modified and installed in the HS column.

The warped "A" dissolver lid was replaced by a new lid.

The HAF pump and DOV jumper were replaced to restore flow.

The sulfuric acid tank in building 211-A was replaced with a tank from 100-H Area. The tank failed after about ten years of service.

The building service hot water tank which has been in use for eleven years was replaced with a code-approved vessel.

The number 2 organic wash waste tank agitator failed, and was repaired, and then returned to service.

The reclaimed nitric acid pump failed and was replaced with a reconditioned spare.

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PUREX (continued)

The East crane's ten-ton rotating hook failed due to a damaged drive shaft and was replaced with a reconditioned, contaminated spare.

A small equipment burial was completed without spreading contamination.

Iodine emission:

Total for the month	0.12 curie
Average daily	0.004 curie
Maximum daily	.06 curie

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REDOX

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Production of enriched UNH was 241 tons, which was 112% of forecast. The process performed very satisfactorily during the time operated.

Process Experience

Processing outages during the month were associated with minor processing difficulties, except one brief outage which was required to replace the D-14 backcycle concentrator pump and one unscheduled power outage which occurred on June 10, 1966 caused by lightning striking the lines.

Approximately 12 tons of uranium above specification in gamma ratio and 30 kg of plutonium high in gamma ratio and uranium content, were blended within specifications. No out-of-specification material remains in product storage at month end and both product streams are running well with shipping specification.

Maintenance Experience

Three pumps failed during the month. The H-5 (off-gas scrubber) pump which has operated since November 26, 1961 was replaced due to a leak at the shaft annulus. The D-14 (backcycle concentrator) pump failed and was replaced. The 2AF (column feed) pump was unable to keep up at the 105% rates even though it worked satisfactorily at somewhat lower rates. Also the No. 4 pump in 222-S failed when a connecting rod broke as a result of a sheared bolt. Parts were replaced and the vacuum pump has been restored to service at month end.

A quartz iodine light was installed on the trolley of the 60 ton crane. Improved lighting benefits were such that it was decided to replace all four trolley lights with the quartz iodine type. It appears that the degree of illumination achieved will allow the elimination of the ten bridge lights.

Two new fire detection systems have been installed in 202-S and the smoke detection equipment in 233-S has been made more reliable and less susceptible to false alarms.

Radiation Experience

Iodine emission for the month is as follows:

Daily Average	0.41 ci
Daily Maximum	1.04 ci
Month Total	12.19 ci

UO₃

Production of enriched UNH was 103% of forecast. The last of the NPR material was processed by the first of the month and two calciners were on standard 94 material during the operating period which lasted until June 10 when lack of feed forced an outage until June 14. Operations were then continuous until June 24 when the plant was shutdown for vacations. The plant will remain down until July 18 for the vacation period, during which time general cleanup and maintenance will be performed. There were four carloads of enriched UO₃ shipped to Fernald during the month. No natural uranium was processed during the month because Purex plant continued thorium processing.

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Plutonium metal production was 136% of forecast.

Processing Experience

The button line operated only 12 days as determined by availability of Pu nitrate feed. There were no significant maintenance problems. The average rate while operating was 1.40 kg/hr. Reduction yields were excellent at 98.3% recovery.

Product Quality

Metal button quality continued good with total metallic impurities averaging 883 ppm. One button was rejected for impurity, and none rejected for low density. Average metal density was 19.47 g/cc.

Non Weapons Plutonium

The Pu Buyback equipment was tested and altered slightly by addition of a valve and a drain pipe to facilitate better Pu measurements. Transloading of Pu for SEFOR in this equipment was not started.

A Euratom representative witnessed preparation for shipment of 8 kg of Pu metal and 20.3 kg as oxide. Other shipments included 800 g in two shipments of Pu nitrate to GE-Vallecitos; 32 g of metal to Parkwell Laboratory at Croton, Ohio, 1 kg of metal to NUMEC and various items to Battelle Northwest.

PLUTONIUM RECLAMATION

Plutonium recovered and returned to the production line totaled 70 kg. There were 53.2 grams of americium-241 shipped. The scrap backlog is now 817 kg of Pu.

Plutonium Reclamation

Operation of slag and crucible dissolvers recovered 31.5 kg of Pu from 1662 cans of scrap containing 3.3 tons of material. Dissolvers 0-4 and 0-8 operated all month except for minor interruptions. Dissolver 0-6 is ready when needed. There were 35 pumps which failed in service; 31 were repaired and 4 were buried. The desirable mechanical seals were not available in adequate number; therefore, substitute parts were used.

Battelle Northwest scrap was campaigned through miscellaneous scrap dissolvers. Less plutonium was found than listed by BNW. An adjustment is indicated.

Waste Treatment

There were 12,215 liters of aqueous waste treated from which 264 grams of Pu were recovered.

Crude americium recovered was 17.4 grams. There were 53.2 grams of purified americium-241 shipped to Oak Ridge National Laboratory. This had been processed from crude solution in the Redox Laboratory.

Incineration Building

Repairs to the furnace continued. There were 17 waste boxes received; 3 were processed. The backlog is now 617 boxes containing approximately 70 kg of Pu.

Miscellaneous Recovery Activities

The liquid portion of the mixed scrap contracted to Nuclear Fuel Services for recovery is packaged ready for shipment.

The treatment of 76 cans (12% of total) of machining oil recovered 181 grams of Pu.

The ports into the Recuplex equipment are closed and no further activity is scheduled.

Receipts of non-Isochem scrap were 60.4 kg of Pu from Battelle Northwest.

Magnesia crucible scrap from fabrication activities is being ground in the button line reduction crucible grinder.

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FISSION PRODUCTS

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Purex head end fission product recovery did not operate during the month as the equipment was utilized for continued thorium processing.

At B-Plant, product crude was received from Tk-003 CR for oxalate processing. Approximately 60 Kci of strontium-90 and 460 Kci of Pm-147 were separated.

All currently scheduled promethium processing at the Strontium Semiworks has been completed, and equipment flushing and maintenance has been initiated. The remaining strontium nitrate inventory (930 Kci) is presently stored and holding in Tk-E-4.

Following the equipment flushes, the first of seven process tests was completed. These tests are designed to demonstrate and optimize B-Plant Phase III flowsheets. The first test demonstrated the use of an HEDTA (hydroxyethylethylenediaminetriacetic acid) complexant on Zirflex acid waste (ZAW). Product recoveries for Pm, Ce, and Sr were 75%, 93%, and 95%, respectively. Americium and curium were also followed during the test; however, product recoveries were low, approximately 55%. The following table shows the major metal decontamination factors for metal impurities achieved during the test:

Decontamination Fractions

<u>Metal Impurity</u>	<u>Sr Fraction</u>	<u>RE Fraction</u>
Fe	>100,000	4,000
Pb	50	20
Al	>13,000	120
U	>120	36
Pu	>320	>100
Na	>450	>150
Ca	5.4	1.2
Cr	>4,000	1,200
Ce	11,000	---
Pm	800	---
Sr	---	100
Zr-Nb	125	7.5

Since actual B-Plant equipment is expected to be more efficient than the Semiworks facility, the above recoveries and decontamination factors are considered satisfactory.

Cesium Loadout

Loading the STT containers with thermally hot feed to prevent precipitation and screen plugging resulted in low loadings. It has been demonstrated on the most recent cask loading that a one-to-one dilution of the feed with water will allow better

FISSION PRODUCTS (continued)

Cesium Loadout (continued)

loading at reduced temperatures without precipitation of plugging problems. Water dilution is considered and interim solution in view of the undesirable increase in nonboiling waste volume.

Cask Loadings and Shipments

The HAPO IB-3 cask, containing 390 Kci of Sr-90, was shipped to ORNL June 10, 1966. This was the final strontium shipment for FY-1966. A total of 2.83 megacuries of Sr-90 was shipped to ORNL and Quehanna during the fiscal year. STT casks 45-A and 46 were loaded with 74 Kci of Cs-147 and shipped to ORNL on June 13, 1966. A third STT cask was loaded with 60 Kci by month end and will be shipped along with one other STT early in July.

Fission Product Inventory (Kci)

	<u>CR Vault</u>	<u>B-Plant</u>	<u>SSW*</u>	<u>Casks</u>	<u>Totals</u>
Sr-90	2,670	1,000	1,005		4,675
Pm-147 (unaged)		16,560	240		16,800
Cs-147				60	60

*All SWW inventories except 930 Kci Sr-90 are being used for the Phase III process tests.

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Waste Storage Tank Status

Following is the month-end status of the high level waste storage tanks that have leaked or are suspected leakers:

<u>Tank</u>	<u>Lateral</u>	<u>Radiation C/M</u>	<u>Liquid Depth(in)</u>	<u>Sludge Depth(in)</u>	<u>Temp oC</u>
107 SX	S68°15'	1,620,000	183.75	26	138.9
108 SX	S77°15'E	885,000	237.75	28	137
	S59°18'E	930,000			
	S41°50'E	1,020,000			
109 SX	S69°15'W	180,000	214.25	72	116.1
	S45° 0'W	660,000			
	S20°15'W	40,000			
105 A	No. 3	225,000	312	24	110
Test Well No. 29 SX		420,000			

Individual tank inventories and tank farm inventory measurements indicate no measurable loss of material.

The preheating of Tank 104-AX for receipt of boiling waste is now being augmented with a steam heater. The prototype electric heater increased the temperature of the 400,000 gallons of water from 70° F to 130° F in 45 days.

On June 2, 1966, a solution backup occurred from the 103-AX waste storage tank by way of the No. 10 circulator air line to the 801-A instrument building. Removal of a block valve caused a sudden venting of the air line directly from the tank bottom. About 20 gallons of waste solution were expelled resulting in 400-500 rad/hr radiation levels in the building. Decontamination efforts have reduced the radiation 20-fold, but not sufficiently to permit entry. Tank monitoring is being achieved from the tank dome. Data since the incident indicate the Tank 103-AX sludge temperatures have remained constant or have decreased slightly.

In-Tank Solidification

The in-tank solidification unit continued operation, with daily water flushes of the main de-entrainer and of the circulator. The Teflon pad demister has been flushed with a chemical cleaning solution approximately every two weeks; with hot water flushes twice weekly. Late in the month a weld failure occurred in the 10-inch stainless steel pipe carrying air to the main circulator about 6 feet below pit level. To keep from having to repair the leak, the 18-inch pipe sleeve around the 10-inch line was extended and sealed so as to hold the air pressure. Also, two heater banks of the main air heater have partially failed.

WASTE MANAGEMENT ACTIVITIES (continued)

In-Tank Solidification (continued)

Installation of the prototype system for pumping of Tank 101-BY supernatant solution at terminal concentration to Tank 105-BY is nearing completion. The ITS unit recovered 132,000 gallons of storage space during the month for a total to date of 2,117,000 gallons.

242-T Waste Evaporator

The 242-T evaporator unit was down about two weeks to install a cooler to the bottoms stream before the supernatant solution is recycled and mixed with fresh feed. It was found that as experience and improvements allowed increases in the evaporator throughput rates, the heat dissipation capability of the underground waste tanks became capacity limiting. The underground waste tank cooler system contains two underground waste concentrate tanks which act as cooling ponds. Air is drawn through the vapor space of each tank at 2400 cubic feet per minute. The system consists simply of a series arrangement of a blower, steam heater, and a bank of CWS filters; the steam heater prevents condensation in the filters. The unit has been operating since startup June 14, 1966. Heat and water removal rates from one tank have been averaging 7.5×10^5 B.T.U./hr and 1.1 gpm, respectively. Heat and water removal from the other tank have been oscillating between 2 and 6×10^5 B.T.U./hr and between 0.25 and 0.9 gpm, respectively. After two weeks' operation, radiation activity levels throughout the duct work, heater box, and filter unit are only slightly above background. This activity has been determined to be Cs-137.

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CHEMICAL RESEARCH AND DEVELOPMENT

Thoria and U-233

Three denitration runs were made in the 4-inch diameter agitated trough calciner. Thorium nitrate solution was fed into the calciner which had bed temperatures of $400 \pm 25^\circ\text{C}$. The calciner, the off-gas filtration system, and the product transfer line all operated very well. Samples of the ThO_2 taken from the product receiver had dispersibility values which ranged from 75-95% when treated by the standard Sol-Gel process.

Dissolution rate tests were made on samples of thorium oxide pellets compacted to different theoretical density. A pellet compacted to 75% theoretical density disintegrated to a finely divided powder. A pellet compacted to 85% theoretical density did not disintegrate, but was shown to dissolve at a rate equivalent to 400-500 pounds of thorium per hour, assuming a 3-ton charge with excess of dissolvent to all surface areas.

Miniature mixer-settler runs were made to demonstrate solvent extraction de-daughterizing of U-233 product solution using di-sec-butyl phenolphosphonate as solvent. Decontamination factors were good and uranium loss to the raffinate was less than 0.1%.

Conversion of thorium nitrate to thorium oxide by a modified Sol-Gel process has been conducted during the past several months on a test basis in one of the agitated, electrically heated batch denitration pots at the 224-U Building in 200-West Area. Results of the five runs demonstrate that satisfactory Sols at 95% dispersion were accomplished when the steam-denitrated oxide was transferred hot from the denitration pot to the Sol tank. The tests included the addition of various amounts of Purex solvent. Some foaming occurred, but steam denitration proceeded without difficulty. Agitation during drying operation was varied, resulting in variations in the particle size of the resulting powders. In summary, it was indicated that the batch denitration pots can be used to prepare dry gel suitable for conversion to dense oxide.

The above study was continued by diversion of 100 gallons of thorium nitrate from the present Purex separations run for further process demonstration. Completion of the test was deferred, awaiting decay of radiation from protactinium.

Extraction of Np and Pu from Purex 1WW

After extraction of Np and Pu from Purex 1WW waste with 25% DBBP-NPH, it is necessary to strip the Np and Pu from the solvent using 0.1 M oxalic acid. Destruction of excess oxalic

CHEMICAL RESEARCH AND DEVELOPMENT (continued)

Extraction of Np and Pu from Purex 1WW (continued)

acid is then required. Initial experiments indicated a first order reaction when destroying the oxalic acid in boiling hydrogen peroxide-nitric acid solution.

Zircaloy Decladding Studies

Studies to improve or replace the Zirflex process for decladding Zircaloy-clad uranium fuels have been directed to anodic dissolution techniques. Electric current could not be made to flow until a good share of the oxide had been physically removed from the surface. Use of 60-cycle alternating current at 5-6 volts enhanced the oxide dissolution and achieved uniform penetration of the oxide coating. If this approach proves successful Zircaloy decladding times can be reduced considerably.

Insertion of Transfer Pumps into Waste Storage Tanks

A reduced scale model of a sluicing device for opening a path when installing pumps in waste storage tanks was successfully modified and tested. The modified version was demonstrated to be capable of penetrating into two feet of consolidated sludge in 5-10 minutes of operation.

Neptunium Metal Preparation

For demonstration of capability and as a service to Battelle Northwest, approximately 100 grams of neptunium were converted from nitrate solution via Np (IV) oxalate precipitate filtration, calcination to NpO_2 , hydrofluorination to NpF_4 and bomb reduction to the metal with calcium. The metal yield was 93% of the neptunium in the original solution. Impurities were low. After vacuum melting by Battelle, the impurities (expressed in ppm) were as follows: Pu, 400; U, 275; C, 164; Fe+Cr+Ni 53; Al+Si, 51.

Americium Recovery

As reported elsewhere, 54.2 grams of Am-241 were shipped to ORNL on June 29. This material had been purified in the Redox Laboratory.

Recovery of crude americium solution was continued in the waste treatment facilities. In a process test, thirty-eight liters of crude feed containing 8 grams of americium were concentrated by a factor of 10. Solution foaming was overcome by addition of Antifoam B, with sparging of the boiling

CHEMICAL RESEARCH AND DEVELOPMENT (continued)

Americium Recovery (continued)

solution with air. The americium concentrate was found to be compatible with the americium purification flowsheet planned for installation in the plant. A small-scale flowsheet test was successful with the chemical purity of the americium oxide product exceeding 99%.

Detailed design of prototype facilities to be installed in the plant is in progress in the Vitro organization.

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