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AEC-GE RICHLAND, WASH.

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DOCUMENT NO.: HAN-93855
Report #3
March 1966

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RL-337 (REV. 4-65) AEC-RLOO RICHLAND WASH.

UNITED STATES ATOMIC ENERGY COMMISSION
RICHLAND OPERATIONS OFFICE

This document consists of 17 pages, No. 1 of 2 Copies, Series A

TO: H. E. Parker
DIRECTOR - DEPUTY DIRECTOR
CHEM. PROC. DIVISION - RLOO

DATE April 12, 1966

FROM: J. T. Christy, CHIEF,
CHEMICAL OPERATIONS BRANCH
CHEM. PROC. DIVISION - RLOO

HAN-93855
Report #3
March 1966

SUBJECT: 200 AREA MONTHLY REPORT FOR March

DC:AWL/MJR/EAA/EB

PROCESSING STATISTICS

PRODUCTION

PRODUCT	URANIUM NORMAL TONS	URANIUM NORMAL PERCENT FORECAST	URANIUM ENRICHED TONS	URANIUM ENRICHED PERCENT FORECAST	PERCENT EFFICIENCY OP	PERCENT EFFICIENCY MECH	PERCENT WASTE LOSSES PU	PERCENT WASTE LOSSES U	ATM RELEASE CURIES / DAY
PUREX	371.9	66.4	259.7	103.9	88.3	95	.62	.21	0.19
REDOX	--	--	45.9	145.9	39	100	0.71	0.99	0.025
URANIUM REDUCTION	321.2	79.4	262.7	105.1					

PRODUCT	BRANDY OR NEPTUNIUM KGS	BRANDY OR NEPTUNIUM PERCENT FORECAST
PUREX	0	--
REDOX	--	--

PRODUCT	PLUTONIUM KGS	PLUTONIUM PERCENT FORECAST	PERCENT EFFICIENCY OP	PERCENT EFFICIENCY MECH	PERCENT WASTE LOSSES PU
PLUTONIUM REDUCTION	409.1	136.4	80	78	0.02
RECOVERY	92.6	--	--	--	--

INVENTORIES

PLUTONIUM

SCRAP BACKLOG 904.8 KGS

SCRAP COMPOSITION:

PLUTONIUM PROCESSING	44.5
FABRICATION	12.3
RECOVERY	0.1
BNW LABORATORY	11.4
OFF-SITE	5.8
SUPPORTING LABORATORIES	2.3
INCINERATOR	23.6
	100 %

EXCLUDED FROM AUTOMATIC DOWNGRADING AND DECLASSIFICATION GROUP 1

URANIUM

	NORMAL	ENRICHED
HANFORD CODE C-65		
UNH	77.8 TONS	49.2 TONS
UO_3	11368.2 TONS	-- TONS

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PUREX

The plant operated at 23 TU/D through March 10, 1966 on 94 uranium metal. The plant was then shut down for inventory and return to normal uranium processing. Operation was resumed on March 14, 1966 at 23 TU/D, and the rate was increased to 25 TU/D on March 15, 1966. Processing continued at that rate during the remainder of the month. The production of UNH was 66.4 % of forecast for normal and 103.9% enriched.

Both the uranium and plutonium products were within specifications during the month; however, a small amount of dilute plutonium was produced at startup. Neptunium runs 2-66, 3-66, and 4-66 were purified and shipped to the Plutonium Chemistry Laboratory for processing to the oxide for N-Reactor. Approximately 3,150 grams of Np were purified.

The new NPH diluent continues to give excellent performance. The performance is much better than with Soltrol-170.

During March, the fission product activity discharged to the A-36 crib by "A" and "B" dissolvers averaged 20 and 2 microcuries per ton of uranium, respectively. The amounts discharged show great improvement over previous operation. Part of the trouble has been due to foaming, so a sonic defoamer has been installed in the "A" dissolver off-gas line. This may have contributed to the lower discharges.

Pump experience during March included:

1. F-13 pump failed and was replaced by a new spare.
2. G5-2 (No. 1 organic system) pump failed and will be replaced next shutdown.
3. R5-2 (No. 2 organic system) pump was replaced with one of larger power to meet pumping requirements.
4. Nitric acid pump 13-1 failed and was replaced by the pump removed from R5-2.
5. Two failed pumps were sent to T-Plant for repair.

Jumper installation and replacements:

1. The F-5 to F-3 overflow jumper leaked and was replaced.
2. F-3 pump discharge jumper leaked and was replaced.

PUREX (continued)

3. C-3 to D-4 jet jumper plugged and was replaced.

4. A prototype sonic defoamer jumper was installed on the "A" dissolver vapor line.

The obsolete G-G3 centrifuge was sent to T-Plant and four of the G-3 and G-3 centrifuge support blocks were removed from the canyon and buried to obtain in-cell storage space.

A temporary venting system was installed on "A" dissolver ammonia scrubber catch tank to minimize contamination escaping to A-36 crib, and a new crib line sampler was installed.

The regulated spare filter, which is stored in the burial grounds, was tested and found to be in good operating condition for use in F-Cell or dissolver off-gas systems.

A new agitator was installed in D-1 tank in preparing for the thorium run.

A program of replacing lifting bails on cell cover blocks was completed with the replacement of bails on eleven blocks this month.

The electrical lighting on the East Canyon Crane was improved by installing 1500 watt quartz high efficiency lights.

The sound system on the West Canyon Crane was overhauled.

Iodine emission was:

Month's total	5.83 curie
Daily Average	.19 curie
Maximum Daily	.35 curie

REDOX

Production of enriched UNH was 145.9 % of forecast. During the month a neptunium isolation and decontamination campaign was completed. In addition, the first full production campaign on NPR fuels was initiated.

Processing Experience

The neptunium isolation campaign was concluded on March 6, 1966, with 1,900 grams being transferred to 233-S for final concentration. A line plug gage problem and a leaking L-6 (product sampler tank) pump resulted in the loss of 600 grams back into the process inventory, leaving 1,300 grams to be shipped to Purex for final purification.

The remainder of the month was used for the processing of NPR fuels. Dissolution of the first production campaign of Zircaloy-clad 0.947 percent enriched N-Reactor fuel was started on February 23, 1966. The basic flowsheet includes three parts:

1. Dissolution of the aluminum canister in sodium hydroxide--sodium nitrate.

2. Zircaloy cladding dissolution using ammonium fluoride--ammonium nitrate (NH_4F - HN_4NO_3).

3. Dissolution of uranium slugs in nitric acid using aluminum nitrate as fluoride complexant for corrosion control.

Process performance and experience to date are summarized as follows:

1. Over-all dissolution capacity has been about one-third lower than expected due to increased fuel handling and Zircaloy dissolution time. The increased fuel handling time is the result of N-Reactor experiences of failure of the aluminum fuel canisters which allowed individual fuel elements to escape into the N-Area basin. To preclude such a failure during charging operations at the Redox Plant, a stainless steel bucket was designed and placed into service to transport the aluminum canisters. These buckets prevent the storage of fuels in the J-1 storage vessel at the Redox Plant, which necessitates charging directly from the cask cars.

   The increased Zircaloy decladding time is apparently caused by a refractory oxide coating which is difficult to penetrate with the NH_4F - NH_4NO_3 dissolvent. On at least one occasion, undissolved Zircaloy was observed in a dissolver after an

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REDOX (continued)

Processing Experience (continued)

18-hour boiling digest using 5.5 M NH_4F - 0.5 NH_4NO_3. Currently, the remaining Zircaloy heels are dissolved during the decladding operation on the next charge.

2. Losses to canister and decladding wastes on the first fifteen charges have been as follows:

	Waste losses, percent	
	Pu	U
Canister dissolution	0.04	0.05
Zircaloy decladding	0.48	0.62

3. Original development data indicated neutralized Zircaloy waste contains 20 to 35 volume percent solids. On two occasions, the waste transfer line from Redox Plant to the waste storage tank plugged. Laboratory investigations indicated that the addition of excess caustic causes the formation of a more mobile slurry. This addition is currently being used on coating wastes.

4. Hydrogen and ammonia control in the diluted off-gases during Zircaloy decladding has been good, although flowsheet modifications were required to control hydrogen during special Zircaloy heel-removal operations.

5. Fluoride analyses of the dissolved uranium solutions show conversion of uranium to UF_4 during the decladding step has been approximately 3.9 percent. Aluminum nitrate is added at a ratio of 3 moles of Al per mole of fluoride as a complexant to minimize dissolver corrosion. Iron and chromium analyses of waste and uranium solutions indicate corrosion rates no greater than expected; that is, 4 mils/month for ordinary dissolver surfaces and 8 mils/month on heat transfer surfaces.

6. Although this N-Reactor fuel contains nearly three times more fission products than aluminum-clad enriched fuels at Redox, no difficulty has been experienced in meeting fission product specifications on the products. Both products have, however, been consistently borderline or above specifications with regard to metallic impurities. The plutonium has been above specifications with respect to uranium concentration

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caused by uranium solids left in L-6 during the neptunium run. Extensive flushing has eliminated these solids, and the product is now meeting specification. The uranium product is out of specification with respect to sodium and iron content. The high sodium is due to lack of sufficient scrub at the low rates. The excessive iron is due to the prolonged ozonation at low rates. Both impurities are being brought into control by flowsheet and ozonation adjustments.

Maintenance Experience

There were no canyon pump failures during the month. The failed L-6 (sampler tank) pump was replaced with a spare unit. A new helical type tube bundle was installed on the left side of the F-2 concentrator. The bundle has the new type retainer cups for C-clamp installation. A 1-inch flex line was installed to replace the 2-inch cooling water supply for the F-1 (2D column feed tank) external cooler. This installation has reduced the water buildup in the sump.

Radiation Experience

Iodine emission:

Average Daily	0.025 Ci
Daily Maximum	0.19 Ci
Total for Month	0.79 Ci

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UO_3

Processing of normal and enriched oxide was 79.4 % and 105.1 % of forecast, respectively. There were 430 drums loaded with depleted uranium oxide during the month for a total of 14,879. This constitutes approximately 11,365 tons of normal depleted uranium. There were five carloads of enriched oxide shipped to Fernald.

Processing was continuous throughout the month, with only minor mechanical and process problems. Operations were down two days due to a shortage of shipping containers that were delayed in transit due to the severe weather conditions in the mid-west.

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PLUTONIUM FINISHING--234-5

The production of unfabricated plutonium metal was 136.4 % of forecast.

Processing Experience

Button line operations proceeded normally, with maintenance being performed during periods when no nitrate solution feed was available. The filter housing on the calciner was changed to accept standard rather than custom built filters. The production line operated 62 % of the available time on a five-day week schedule. The average rate while operating was 1.30 kg per hour. Reduction yields were excellent, averaging 98.5% recovery. Renovation of hood 7A (Pu solution handling) was deferred while men were assigned to modify the CX column in the plutonium reclamation facility. There were four process operators transferred to work in the plutonium reclamation activities.

Product Quality

Plutonium metal quality was high with impurities, averaging only 622 ppm. There were no buttons rejected for low density and five buttons rejected due to high impurities. The average metal density was 19. 44 g/cc.

Nonweapons Plutonium

The following plutonium shipments were made for peaceful purposes programs: United Nuclear Corporation, 0.7 kg as oxide at 8% Pu-240; General Electric Company--Vallecitos, .2 kg as nitrate containing approximately 8% Pu-240; Argonne National Laboratory, 25 kg of shattered metal and .7 kg of plutonium fluoride, both containing approximately 8% Pu-240; Oak Ridge National Laboratory, 2 kg of plutonium fluoride at 8% Pu-240; Battelle Memorial Institute at Ohio, .3 kg of metal and .9 kg as oxide both containing 8.6% Pu-240; Cadarache in France, 78 grams of metal containing 22% Pu-240; Euratom, 79 kg of metal ingots containing 8.4% Pu-240. The Euratom plutonium was released to a commercial trucking firm on March 25 and was trucked to Seattle. The material left Seattle by Flying Tiger on Monday, March 28, via San Francisco to New York. It left the U. S. A. via boat shortly thereafter.

PLUTONIUM RECLAMATION

There were 92.6 kg of plutonium recovered and transferred to the button line as feed. The scrap backlog at month end was 904.8 kg of plutonium.

Plutonium Reclamation Facility

The slag and crucible dissolvers and the solvent extraction system were accelerated to a seven-day week schedule starting March 7. This was accomplished by shifting manpower from other parts of 234-5 Building, including some who had worked at mothballing of idle portions of the building.

Dissolving of 1,528 cans of slag and crucible material yielded 49 kg of plutonium. There were 140 hours of dissolver down time during the month, with 40 hours due to pluggage of a portion of tank 5 and 100 hours due to five dissolver malfunctions. Dissolver 06 experienced ball valve difficulties twice which were repaired in the maintenance cell. Dissolver 04 was removed once to repair a leak and again at month end it plugged with solids. The Teflon liner in 08 dissolver was found to have a hole approximately 1/2 inch wide and 1-1/2 inches long, apparently caused by excessive temperature. There were two melted spots in a nearby solid Teflon plug. A collection of plutonium metal fines burning spontaneously is hypothesized to be the cause of these problems. The 06 and 08 dissolvers were operating at month end.

Increased operating time resulted in increased pump failures. Twenty pumps were removed; 19 were repaired, and one Deanline pump was not repairable.

The flowsheet for depleting uranium from the solvent extraction system was invoked once, resulting in 156 grams of uranium being discarded.

There are 60 boxes of slag and crucible material which was packaged prior to use of a hammer mill. Pulverizing and repackaging of this S&C material was started.

Waste Treatment

There were 129,360 liters of aqueous waste processed in waste treatment facilities which yielded 437 grams of plutonium. Twenty grams of plutonium were recovered from reprocessing a sump tank of solution. Waste treatment also recovered 8.8 grams of americium-241.

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PLUTONIUM RECLAMATION (continued)

Incinerator Building

Fabrication and installation of parts for the incinerator furnace will not be completed prior to mid-June. Leaching activities resulted in .7 kg of plutonium in recovered solutions. The backlog of dry waste is now 573 boxes, containing about 65 kg of plutonium.

Plutonium Scrap Received

The plutonium scrap arriving from Lawrence Radiation Laboratory, Battelle Memorial Institute--Ohio, Battelle-Northwest Laboratory, and from the Purex Plant added 15.7 kg of plutonium to the scrap backlog. The backlog was 904.8 kg of plutonium compared to 939.1 kg one month ago.

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FISSION PRODUCTS

Purex head end fission product recovery operated during the month to recover 1,620 kci of Pm-147 and 360 kci of Sr-90. The same amounts were transferred from 003 CR crude feed storage to B-Plant for oxalate processing.

At the Strontium Semiworks, promethium processing was started. By month end, two runs had been completed with 730 kci Pm-147 recovered in the first run and 1,250 kci Pm-147 recovered in the second run for a total of 1,980 kci of Pm-147 and associated rare earths recovered from Purex acid waste and aged over 2.5 years. Extraction was accomplished using D2EHPA (Di-2-ethylhexylphosphoric acid) at a pH of 1.5 with hydroxyacetic acid as a buffer and HEDTA (hydroxyethylethylenediaminetriacetic acid) as a complexant to prevent iron and lead extraction. After stripping with 1.5 to 2.0 molar HNO_3, the rare earth nitrate solution was continuously concentrated and denitrated with sugar. Promethium-147 losses were less than one percent. Lead and sodium decontamination factors were 2,000 and 2,500, respectively. Greater than 95% of the americium-241 was also recovered. The composition of the purified rare earth product solution is as follows:

	Ci/liter	Molarity
Pm-147	1,700	
Ce-144	1,350	
Zr-Nb-95	4	
Ru-106	<1	
Am-241	0.13	
Pu-239		1.7×10^{-4}
RE (excluding Ce-144)		4.0×10^{-5}
Pb		0.40
Fe		0.0027
Na		0.02
Acidity		0.42

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FISSION PRODUCTS (continued)

Flowsheet and equipment performance of this promethium processing campaign were excellent. The new transfer line and the comprehensive Semiworks flushing prior to the promethium processing assured the absence of green promethium contamination.

Cask Loadings and Shipments

A 200-gallon bowling ball cask loaded with 730 kci of greater than + 2.5-year aged Pm-147 was shipped to PNL on March 18, 1966. A second bowling ball cask loaded with 1,250 kci of + 2.5-year aged Pm-147 has been loaded and will be shipped to PNL early in April. The HAPO II-1 cask was loaded with 180 kci of Sr-90 and shipped to Quehanna on March 18, 1966. The HAPO IB-1 cask was loaded with 340 kci of Sr-90 and is undergoing final calorimeter checking at B-Plant. STT casks 44 and 45-A were loaded with 60 kci of Cs-137 each and were shipped to ORNL on March 4, 1966. STT casks 47 and 48 are being loaded with Cs-137 for shipment to ORNL early in April.

Fission Product Inventory (kci)

	Head End	CR Vault	B-Plant	SSW	Casks	Total
Sr-90	20	1,600	2,190(1)	1,470	340	5,620
Pm-147 (unaged)	90	2,720	15,400(2)	--	--	18,210
Pm-147 (aged)	--	--	--	1,110(3)	--	1,110

(1) Includes 370 kci in process. Waste loss of 300 kci.
(2) Adjusted downward 530 kci based on new analysis.
(3) Decay adjustment of 40 kci.

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WASTE MANAGEMENT ACTIVITIES

The table below shows the March 31, 1966, status of the waste storage tanks that have leaked or are suspected leakers.

Tank	Lateral	Radiation c/m	Liquid Depth (in.)	Sludge Depth (in.)	Average Temp (°C)
107 SX	S68°15'E	1.7×10^6	193.25	14	150
108 SX*	S77°15'E	930×10^3	242	17	140.6
	S59°15'E	705×10^3			
	S41°50'E	945×10^3			
109 SX	S20°45'W	555×10^2	228	72	115.6
	S45° 0'W	160×10^3			
	S69°15'W	22×10^3			
105A	No. 3	225×10^3	312	24	110
Test Well	No. 29 SX	410×10^3			

* Note that for the first time, significant activity was detected in lateral S41°51'E under this tank.

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

In-Tank Solidification

The ITS unit has operated the entire month with a deentrainment unit on the condenser outlet which contains a four-inch thick Teflon pad of 20-micron fibers packed to a density of 18.9 pounds per cubic foot. The deentrainer has effectively stopped the excessive loading of the CWS filters on the exhauster outlet. Excessive radiation fields around the CWS filters have required a few filter changes. Decreasing radiation levels on the inter-connecting piping indicate that this problem may diminish as the previously accumulated activity is purged from the system. Gradual increases in the deentrainer pad have required periodic water flushing. At month's end, it has been necessary to increase the frequency of flushing to maintain an operable pressure drop. During March, 124,000 gallons of underground waste storage space was recovered by the in-tank solidification unit. Total space recovered to date by this unit: 1,817,000 gallons.

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WASTE MANAGEMENT ACTIVITIES (continued)

In-Tank Solidification (continued)

The GE low-power density prototype immersion heater was removed from tank 101 BY for inspection after the heater failed. Examination revealed that 5 of the 12 elements had failed. The heater circulator draft tube was found to be plugged with salt cake and the Inconel sheathed heater elements were severely corroded. Subsequent sludge level measurements indicate that the sludge level under the draft had reached the lower end of the draft tube, thus plugging it. When salt cake is present around the heaters, the salt would melt and severe corrosion would be expected. Studies have been initiated to define Inconel corrosion rates in various waste solutions including molten salt. Also, studies have been started to develop methods for detecting loss of draft tube circulation since it has been indicated that sludge buildup can occur when circulation stops.

242-T Evaporator

The 242-T evaporator was shut down on February 28, 1966, due to excessive bottoms tank temperatures. The unit was restarted on March 3, 1966, at reduced rates of 4 gpm. Lower than design rates will be necessary until cooling facilities in the bottoms tanks are in operation. During March, this unit recovered 136,125 gallons of storage space for a total to date of 1,022,313 gallons.

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Alternate Reductants for Plutonium

The use of ferrous sulfamate as a reductant for plutonium in Purex solvent extraction processes results in undesirable quantities of iron in the waste. Alternate reductants for use in the aqueous phase have been extensively investigated, whereas reductants which could function in the organic phase are now being examined. Tests were made with ethyl and methyl alcohol, formaldehyde, urea, and tributyl phosphite. Only tributyl phosphite in the presence of ultraviolet light reduced U(VI) to U(IV); experiments are continuing.

Conversion of Thorium Nitrate to Thorium Oxide

The agitated electric pot and auxilliary equipment in the 224-U Building were used to make four test runs in evaluating this conversion of thorium nitrate to thorium oxide by the Sol-Gel technique. These few tests indicated that steam denitration in the pots is feasible and that excellent sols can be prepared. Preliminary data indicated that pot drying of the sols was controlled satisfactorily to permit the ultimate production of a dense oxide to meet target specifications. Samples of the dried sols were sent to DUN for high temperature firing and evaluation of the dense oxide. Two more test runs are scheduled, which will include emphasis on contact of the steam-denitrated oxide with the atmosphere, which reportedly affects sol formation.

Nuclear Safety

In order to facilitate criticality calculations, cross sections for fluorine were added to the GAMTEC-II library tape. Fluorine cross sections were also added to the Battelle Master Library. The data were checked in the critical mass laboratory using 2 wt.% enriched uranium and UF_4-paraffin mixtures; good agreement was found.

Bismuth-Polonium Separation

Recent miniature mixer-settler runs testing a study flowsheet for solvent extraction recovery of polonium from irradiated bismuth show good recovery and separation of polonium without excessive reflux of polonium in the scrub column as observed previously.

Neptunium Chemistry

Precise spectrophotometric measurement of the equilibrium constant for NpO_2^+/NpO_2^{++} was found to disagree significantly with past values. The discrepancy is being investigated further with emphasis on the effect of organic constituents present.

CHEMICAL RESEARCH AND DEVELOPMENT (continued)

Rhodium and Palladium Recovery

The hot cell experiment was completed showing the once-through capacity of IRA-401 resin to be 3.0 g/l for Pd and 2.1 g/l for Rh at 100% breakthrough. These loading values apparently are too low for economical recovery by burning of the resin, so further work is being performed on eluting these products.

Recovery of Neptunium and Plutonium from Purex Waste Sludge

Batch solvent extraction studies were continued on recovering Np and Pu from acidified Purex sludge solution. Extractions using D2EHPA as solvent on sludge dissolved from Tank 04-A yielded greater than 95% of the plutonium and 99% of the neptunium.

Zirflex Waste Processing

Pulse column solvent extraction studies with D2EHPA were aimed at demonstrating quantitative extraction of strontium and rare earths from Zirflex-type Purex waste. The test indicated that processing of Zirflex wastes was feasible. Both tartaric acid and HEDTA were tested as complexing agents for the high iron and aluminum found in these wastes. The HEDTA, as expected, was the most effective, but even when used in excess, up to 1% of the aluminum in the feed carried into the rare earth product.

Americium Recovery

Americium recovery in the plutonium waste treatment facility was studied to show that in the December-January period, the system recovered 75% of the americium, and in February it recovered 89%.

Promethium feed concentrate, recently sent to Battelle-Northwest, and containing 1.5 to 2 kg of well aged Pm-147, is estimated to contain 40 grams of Am-241; it is planned to recover this material. Since development of a batch D2EHPA solvent extraction process for the plutonium recovery facilities, the research effort was shifted to pulse column operation for recovery of americium from Purex wastes associated with recovered strontium and rare earth concentrates. The work included study of the behavior of Eu-152, a highly radioactive contaminant.

MAJOR PROJECT ACTIVITY

CAC-981 - B-Plant, Phase II. Design is 100% and construction 97%.

CGC-124 - Increased Processing Flexibility--Purex. Design is 100% and construction 80%. Revised schedule completion date is November 1, 1966.

CAC-144 - Waste Fractionization--B-Plant. Design is 97% and construction 30%.

CAC-169 - Sludge Removal and Waste Transfer--200-E Area. Design was completed to 90%. Bid invitation for the second lump sum contract, to be received on June 2, was issued.

CAC-176 - In-Tank Solidification Unit #2. Title II design was completed to 15%.

CAC-180 - Plutonium Buy-Back Facility. Design is 100% and construction 87%.

CAC-181 - B-Plant--FPCE Integration. Interim design funds were made available on February 9, 1966. Design of the initial phase was started.

CAC-183 - Purex 4.0 Capacity Factor Expansion. Design funds were requested on January 12, 1966.