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RECOVERY OF Am, Cm AND Pm FROM SHIPPINGPORT
REACTOR FUEL REPROCESSING WASTES BY
SUCCESSIVE TBP AND D2EHPA EXTRACTIONS

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A. L. Boldt and G. L. Ritter

Atlantic Richfield Hanford Company
Richland, WashingtonINTRODUCTION

The production facilities of the USAEC at Hanford are primarily a system of reactors and chemical separations plants constructed and operated for the production of plutonium for military weapons. In recent years, however, with the development of nuclear power and with the development of demands for specific reactor products, the number of production programs aimed at non-weapons objectives has increased. For example, the chemical processing facilities, currently being operated by Atlantic Richfield Hanford Company (ARHCO) for the AEC, have been used to separate radioactive strontium-90 and cesium-137 from irradiated production reactor fuels for beneficial use. Also, they have been used for reprocessing Shippingport power reactor fuel elements, and for the recovery of americium, curium and promethium isotopes as described by this report.

Approximately 13 tons of uranium from the blanket of the PWR-1 (Shippingport) power reactor were reprocessed at the Redox Plant during December, 1966⁽¹⁾. The irradiated Shippingport fuel contained approximately 1.3 Kg of americium and curium isotopes. Because of the potential value of these isotopes in the manufacture of heat sources, the recovery of these isotopes was proposed⁽²⁾.

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The dissolved fuel was reprocessed with a modified Redox solvent extraction flowsheet to segregate the fission products and transplutonium elements into a relatively small volume of aqueous waste for further processing to recover americium, curium and promethium (including the other fission product rare earths). At the conclusion of the reprocessing of Shippingport fuel for uranium, plutonium and neptunium recovery, the americium, curium and promethium isotopes were recovered by using a batch solvent extraction process. The batch extraction process using 50 volume percent tributyl phosphate (TBP) in a normal paraffin hydrocarbon diluent (NPH) had previously been demonstrated on a plant scale with similar feed at the AEC Savannah River Plant⁽³⁾. The americium, curium and promethium were recovered as a "crude" solution and loaded into a 30-ton "bowling ball" cask for shipment to the Semiworks Plant for further purification.

A continuous di-(2-ethylhexyl)phosphoric acid (D2EHPA) solvent extraction process was used to process the americium, curium and promethium at the Semiworks. This process had not been demonstrated previously so that laboratory development work was required prior to plant use. The product from the Semiworks was loaded into a 20-ton "bowling ball" cask and shipped to Pacific Northwest Laboratories of Battelle Memorial Institute for separation of the americium, curium, and promethium isotopes from one another⁽⁴⁾.

This report describes the development and performance of the TBP batch extraction and D2EHPA solvent extraction processes for the recovery of americium, curium and promethium from the Shippingport reactor fuel reprocessing wastes.

SUMMARY

Batch solvent extraction with 50 volume percent TBP followed by continuous D2EHPA solvent extraction was used to recover Am, Cm, and Pm from irradiated Shippingport reactor fuels. The sequence of processing steps utilized was:

1. The Shippingport fuel Zircaloy cladding was penetrated with a Zirflex dissolution ($5.5M$ NH_4F , $0.5M$ NH_4NO_3) and the UO_2 dissolved with a nitric acid leach.
2. The dissolved Shippingport fuel was reprocessed with a modified Redox solvent extraction flowsheet to segregate the fission products and transplutonium elements into a relatively small volume of $Al(NO_3)_3$ waste solution.
3. The $Al(NO_3)_3$ waste solution was adjusted to a TBP batch extraction feed solution by reduction of dichromate with sodium nitrite and adjusting the pH in the range of 0.0 to 0.5.
4. The Am, Cm and rare earths were batch extracted from the adjusted solution with an approximate equal volume of 50 vol. % TBP in NPH. The product-containing organic extractant was re-contacted with succeeding feed batches until high aqueous raffinate waste losses required the reduction of product concentration in the organic phase by back-extraction.
5. The Am, Cm and rare earths were back-extracted from the organic phase by batch contact with a half volume of $1.0M$ HNO_3 .

6. The dilute aqueous product solutions were washed with NPH diluent by batch contact to remove dissolved and entrained TBP prior to product concentration by evaporation.
7. The dilute product solution was concentrated by evaporation and transferred to a "bowling ball" cask. The product "crude" cask was shipped to the Semiworks for further purification.
8. At the conclusion of the campaign the organic extractant was batch contacted with a series of wash solutions to remove residual fission products prior to disposal of the organic extractant.
9. The Redox product in the cask was transferred into the Semiworks processing vessels. Since relatively insoluble solids were found in the cask solution, an acid leach was performed on the solids to recover additional Am, Cm, and rare earths associated with the solids.
10. Organic complexing agents were added to the clarified solution and the pH was adjusted to 4.0 to prepare the feed for D2EHPA processing. The Am, Cm and rare earths were extracted into D2EHPA in the first of two pulse columns and were then back-extracted with nitric acid in the second pulse column.
11. The resulting purified product was concentrated by evaporation and excess acid destroyed by denitration with sugar. The product was then loaded into a 20-ton "bowling ball" cask for shipment to the Pacific Northwest Laboratory of Battelle Memorial Institute.

A total of about 1100 grams of Am, 60 grams of Cm, and 200 grams of Pm was successfully recovered with net recovery efficiencies for each of these isotopes of about 85 percent.

DISCUSSION

I. Processing Facilities

The chemical processing facilities at Hanford were originally constructed and have been operated primarily to receive and chemically process irradiated Hanford reactor fuels, producing plutonium metal and uranium oxide powder, both of which are shipped off-site for further processing. In February 1964, the first fuel loading was discharged from the Shippingport Reactor. At that time, the Hanford Redox Separations plant had been in operation for 12 years; it was also being equipped to chemically reprocess the fuels from the Hanford N-Reactor. From the standpoint of the important chemical reprocessing requirements, the Shippingport blanket fuels are similar to N-Reactor core fuels. Thus, during the planning stages for the Shippingport Reactor refueling, the Redox Plant was selected for reprocessing the discharged blanket fuels.

Compared to Hanford production reactors, the longer residence times, i.e., "exposure times", for fuels in power reactors result in important differences in the end products of the nuclear reactions. In the Shippingport blanket fuel, the continuing nuclear reactions involving plutonium produced from the original uranium fuel result in the production of relatively large amounts of by-product americium, curium and promethium isotopes. These isotopes have potential value for use as

isotopic heat sources in relatively small, packaged power units. Recognizing the possible availability of an appreciable inventory of americium and curium in the Shippingport blanket fuel, it was proposed that an effort to recover these by-products be coupled to the scheduled reprocessing program at the Redox Plant⁽²⁾. Further purification of the "crude" product solution produced at the Redox Plant at the Semi-works facility was also proposed⁽⁵⁾. A brief description of the Redox and Semiworks Plants is presented below.

- A. Redox Plant Description - The basic function of the Redox Plant in the Hanford chemical processing complex is the separations processing function. The Redox Plant utilizes solvent extraction technology to separate uranium, plutonium and neptunium from fission products and other impurities present in the original irradiated fuel. The process is based on the relative extractability of uranium and plutonium into hexone (methyl isobutyl ketone) and the relatively inextractability of fission products and most inert contaminants.

Because of the radioactivity associated with the chemical separations processing operations, the Hanford facilities used for this type of production are built with massive concrete shielding walls separating the processing equipment from the operating personnel. Processes are operated remotely; i.e., the basic operations, including maintenance, are operated from relatively remote stations, with no direct visual or other contact between personnel and equipment. The Redox Plant is typical of this type construction. Figure 1 is an aerial view of the Redox Plant Building, 202-S, with the Building 222-S Laboratory in the background.

Principal features shown in this view include: a) the "Silo", on the right (west) end of the building, in which the tall solvent extraction columns are situated; b) the railroad "cut" and entrance at the left (east) end of the building, through which irradiated fuels are transported by railroad cask cars; and c) the 200-foot ventilation stack, in the lower left corner of the picture, through which the treated process and ventilation air from the building is discharged.

The shielded processing areas of the building are arranged for remote operations and for remote maintenance by means of a traveling crane. The processing cells are located in the concrete-walled structure below shielding cover blocks. Physical access to the processing cells, for maintenance purposes, is accomplished only by removal of the massive cover blocks, using the remotely operated crane. Figure 2 shows the Redox Plant canyon and the remotely operated crane lifting a cell cover block in the background. The crane operator is stationed in a heavily shielded crane cab at a remote control console and uses a system of optics for viewing the crane hook and the necessary features within the canyon. Figure 3 is a view of a typical canyon processing cell after the cover blocks have been removed. Plant operating personnel and the auxiliary processing equipment are located in building spaces that are shielded from the radioactivity of the canyon processing cells.

B. Semiworks Plant Description - The Semiworks Plant was used for a variety of purposes over the past several years. The plant was originally built to develop the Purex process for processing irradiated fuel elements. It was subsequently used to purify several fission products, including strontium-90, technetium-99, cerium-144, and promethium-147. Recently, it was operated to demonstrate new technology for use in Hanford's radioactive waste processing plant (B-Plant).

The Semiworks facility is shown in Figure 4. The large building in the center of the picture contains three heavily-shielded, canyon-type cells in which the process equipment is located. The non-radioactive aqueous makeup and operating control area is located in the front portion of this building. Immediately to its left is located a cask loading or unloading cell and a product storage cell. The tall building on the right is a non-radioactive solvent handling and storage building. A stack is shown at the left and at its base is located a fan house and filter building for treating the off-gases from the process building. Other buildings in the picture are service facilities such as offices, shops, and a change house. The Purex Plant is shown in the upper left corner of the view.

The processing operations within the cells are controlled remotely through the use of appropriate instrumentation. Unlike the Redox facility, however, the Semiworks is not equipped for remote maintenance. Consequently, when maintenance is necessary within the cells, extensive decontamination is necessary to reduce radioactive exposure to tolerable levels prior to entry of personnel.

II. Shippingport Fuel

- A. Fuel Description - The Shippingport blanket fuel load scheduled for reprocessing at the Redox Plant included 113 irradiated fuel assemblies containing approximately 13.5 tons of uranium, as uranium dioxide (UO_2), and containing a total of 63 kilograms of plutonium, which was produced during the irradiation period in the reactor.

Figure 5 illustrates the principal features of a blanket fuel assembly. The total assembly is made up of seven fuel bundles arranged end-to-end and contained in a stainless-steel sheath; it measures approximately nine feet long and is approximately 5-1/2 inches x 5-1/2 inches square in cross-section. Each fuel bundle has 120 parallel, small-diameter, Zircaloy-clad rods containing uranium in the form of UO_2 pellets. The rods are attached at each end to a zircaloy tube sheet. The tube sheets have over 100 small-diameter holes provided for the purpose of orificing the flow of coolant to the rods in the reactor. Table I, Shippingport Blanket Fuel Data, gives a description of the fuel bundle and its composition at 1,040 days aging from discharge.

TABLE I
SHIPPINGPORT BLANKET FUEL DATA

Tube Sheets	Zircaloy-2 0.24 in. thick x 5.2 in. sq. 120 - 0.19 in. dia. to 0.23 in. dia. holes
Rods	120 - 0.411 in. dia. rods, 27 mil thick zircaloy-2 cladding
Nominal size of subassembly	5.2 in. sq. x 10.2 in. long
Cladding and tube sheets	13.2 lb. zircaloy-2/subassembly
Core	40.6 lb. natural UO ₂ /subassembly
Ratio of cladding and tube sheets to core	730 lb. zircaloy-2/ton UO ₂ (830 lb. zircaloy-2/ton U)
Plutonium (average)	4740 grams Pu/ton U
Plutonium isotopic (average)	0.52% Pu-238 65.32% Pu-239 23.17% Pu-240 7.62% Pu-241 3.37% Pu-242
Neptunium (average)	45.4 grams Np-237/ton U
Uranium isotopic (average)	0.006% U-234 0.332% U-235 0.063% U-236 99.599% U-238
Americium (average)	72.7 grams Am-241/ton U 21.0 grams Am-243/ton U
Curium (average)	0.039 gram Cm-242/ton U 5.11 grams Cm-244/ton U

- B. Fuel Receipt and Storage - At Hanford, the Shippingport fuel assemblies were received by railroad cask car at the underwater disassembly and storage facility near the Redox Plant. Disassembly of the fuels was

necessary prior to processing at the Redox Plant because the Redox Plant is not equipped to receive and process stainless-steel fuel element components. Figure 6 shows the facility in which the fuels were disassembled and stored while under water. The water provided shielding protection against the radioactivity while operating personnel disassembled the fuels with special tools.

Following fuel disassembly, the fuel bundles were stored until November, 1966, when their transfer to the Redox Plant for reprocessing was commenced.

C. Fuel Dissolution and Processing - Figure 7 gives a simplified flow diagram of the fuel dissolution and product-waste partition which provided the "feed" for the AmCm recovery campaign.

The total of 13.45 tons of uranium was dissolved in 6 charges, each containing approximately 144 fuel bundles⁽¹⁾. The processing steps utilized in the fuel dissolution were:

1. Cladding penetration with 2 consecutive^t Zirflex dissolutions ($5.5M$ NH_4F , $0.5M$ NH_4NO_3).
2. A water flush to remove fluorides, minimizing dissolver corrosion during subsequent UO_2 dissolution.
3. Nitric acid leach of exposed UO_2 core.
4. A third Zirflex dissolution on the same charge.
5. A second water flush.
6. Nitric acid leach of the UO_2 heel.

Aluminum nitrate was also added to the nitric acid solution during dissolution to complex fluoride ion and to minimize dissolver corrosion. The measured loss of uranium and plutonium to the zirflex waste and rinse solution was 0.6 percent. A conversion of 0.9 percent of the UO_2 to UF_4 during Zirflex decladding was measured.

The dissolver solution was then processed through normal Redox flowsheet steps of feed preparation, acid concentration adjustment and dichromate oxidation of plutonium. The resulting feed solution was processed through the Redox HA extraction and HC back-extraction solvent extraction columns using hexone (methyl isobutyl ketone) as the organic solvent. The normal Redox flowsheet was modified to co-extract uranium, plutonium, and neptunium from an aqueous waste containing the fission products, americium, curium, promethium and inextractable salts. The inextractable salts were added to the dissolver solution during fuel dissolution and feed preparation. The solvent extraction processing produced 4340 gallons of this salt waste as a concentrate containing the Shippingport fission products and americium-curium content. The salt waste "feed" inventory was then stored in the normal dissolver solution storage tanks pending the scheduling of Am-Cm recovery after completion of the Shippingport uranium-plutonium processing.

III. Redox TBP Processing

- A. Preliminary Evaluation - Two processes were evaluated for the recovery of Shippingport fuel Am-Cm content as a small volume "crude" that could

be loaded into a "bowling ball" cask for shipment to the Semiworks. The two processes evaluated were co-precipitation with the rare-earth, double-sulfate⁽⁶⁾ and a batch solvent extraction with 50 volume percent TBP as demonstrated at the Savannah River Plant⁽³⁾. Because of equipment and routing limitations for precipitation processes at the Redox Plant, the TBP process, demonstrated on a similar feed, appeared more practical. Further development work for the specific application of the TBP solvent extraction process to the Shippingport waste was performed at Pacific Northwest Laboratory⁽⁷⁾.

- B. Laboratory Development - Application of Savannah River Plant data and laboratory data⁽³⁾ to the development of an Am-Cm recovery flowsheet at Redox was performed by Pacific Northwest Laboratory⁽⁷⁾. Development work was performed with synthetic Shippingport waste, normal Redox concentrated waste, and Redox Shippingport waste. The primary difference between the Savannah River aqueous feed and Redox Shippingport waste is the presence of chromium, dichromate, zirconium and fluoride ion in the Shippingport waste. Items studied to develop the Redox flowsheet were: 1) effect of pH of the aqueous feed on extraction coefficients; 2) effect of nitric acid concentration of the back-extractant solutions on uranium decontamination; and 3) the effect of repeated extraction contacts of the initial organic using fresh aqueous feed with each succeeding extraction contact.

The laboratory studies on Redox Shippingport waste indicated that extraction distribution coefficients, E_a^O , are greater than 100.

for both americium and curium. The effect of pH of the aqueous phase was not studied in detail but maximum distributions appeared to be obtained near pH 1.1. A separation of americium and curium from the coextracted uranium can be effected by stripping the organic with 0.5M or greater nitric acid leaving the uranium in the organic phase. The loading capacity of the organic is high and at least 4 equal volume extraction contacts using fresh aqueous each time can be made without stripping the loaded organic.

Figures 8 and 9, reproduced from reference 3, show the effect of total inextractable nitrate and extractable HNO_3 on americium and europium distribution coefficients, E_a^0 . Figure 8 shows higher concentrations of inextractable nitrate increasing distribution coefficients. Figure 9 shows decreasing distribution coefficients with increasing HNO_3 concentration.

Several studies of tracer-level laboratory experiments were performed to measure the americium distribution as a function of contact time, and with repeated loading of the initial organic. Americium and uranium distribution ratios were also determined as a function of varying nitric acid concentrations. The starting feed solution used in these experiments was a synthetic Redox waste of the predicted composition shown in Table II.

TABLE II
COMPOSITION OF SYNTHETIC REDOX WASTE
(Shippingport) (7)

<u>Constituent</u>	<u>Concentration, Moles/Liter</u>
Al(NO ₃) ₃	1.56
NaNO ₃	1.53
Cr(NO ₃) ₃	0.2
NaF	0.6
Nd(NO ₃) ₃	0.014
Ce(NO ₃) ₃	0.0057
Sr(NO ₃) ₂	0.0037
Ba(NO ₃) ₂	0.0031
Am-241	Trace

The 50 volume percent TBP-NPH was pretreated by batch washing with 0.8 wt.% NaOH - 2.5 wt.% Na₂CO₃. The aqueous pH was adjusted with HNO₃ to the desired initial pH value. The americium values in both the equilibrated organic and aqueous were measured by gamma spectroscopy.

Americium distribution ratios were determined as a function of contact time (Table III). The results indicate that the extraction of americium into TBP is very rapid, with maximum distribution occurring within one minute.

TABLE III

Am DISTRIBUTION AS A FUNCTION OF CONTACT TIME⁽⁷⁾

Organic: 50 vol.% TBP-NPH - washed with 0.8 wt.%
NaOH - 2.5 wt.% Na₂CO₃.

Aqueous: Synthetic Redox Waste (Shippingport)

o/a: 1

Temp: 35 C

Contact Time (min.)	E_a^O Am	Final pH ^(a)	E_a^O Am	Final pH ^(b)
1	256	1.1	259	1.15
2	240	1.1	246	1.1
5	226	1.2	207	1.1
10	202	1.2	206	1.1
60	191	1.2	187	1.1

(a) Initial pH = 0

(b) Initial pH = 0.3

Since there were 4340 gallons of Redox Shippingport waste to process in approximately 1000-gallon batches, an experiment was performed to determine the americium distribution and equilibrated aqueous pH after repeated loadings of the initial organic (Table IV). Except for radiation damage to the organic, this flow scheme appeared to provide the advantages of a five-fold concentration of the Am-Cm, minimum process manipulation, and increased DF from other extractable cations. After loading the organic it was stripped with an equal volume of HNO₃ to determine the americium stripping distribution as a function of acid concentration (Table V). Since some uranium is present from normal Redox solvent extraction losses, uranium distributions were also determined (Table VI) as a function of HNO₃ concentration.

TABLE IVREPEATED LOADING OF THE INITIAL ORGANIC⁽⁷⁾

Organic 50 vol. % TBP-NPH
 Aqueous: Synthetic Redox Waste
 (Shippingport), pH 0.3
 o/a: 1
 Temperature, C: 35
 Contact Time: 10 minutes

<u>Contact</u>	<u>Final pH</u>	<u>E_a^o Am</u>
1	1.2	194
2	1.1	277
3	0.9	231
4	0.8	175

TABLE VAm STRIPPING DISTRIBUTION AS A FUNCTION OF
NITRIC ACID CONCENTRATION⁽⁷⁾

Organic: 50 vol. % TBP-NPH
 (loaded, contact #4,
 Table IV)
 Aqueous: HNO₃
 o/a: 1
 Temperature, C: 35
 Contact Time: 15 minutes

<u>Initial Aqueous Nitric Acid Concentration, M</u>	<u>Stripping E_a^o Am</u>
0.1	0.015
0.2	0.020
0.5	0.034
1	0.049
2	0.070
3	0.075

TABLE VI
URANIUM DISTRIBUTION AS A FUNCTION
OF NITRIC ACID CONCENTRATION⁽⁷⁾

Organic: 50 vol. % TBP-NPH
Aqueous: M HNO₃, 10 g U/l as UNH
o/a: 1
Temperature, C: 35
Contact Time: 15 minutes

<u>Initial Aqueous Nitric Acid Concentration, M</u>	<u>Stripping E_a⁰ U</u>
0.1	0.96
0.2	1.7
0.5	4.8
1	9.9
2	22.5
3	33.3

Two experiments were carried out in the High-Level Radiochemistry Facility using full-level Redox waste to verify the laboratory tests. The first of these (Table VII) used as a starting feed Redox waste (D-9) from Hanford fuel processing. The rare earths and uranium contents were adjusted to be comparable with the expected Shippingport waste composition. The extractable dichromate present in the waste was reduced to the inextractable chromium(III) valence by the addition of NaNO₂ and the pH was adjusted to 0.5 with 50 wt. % NaOH. The adjusted solution was then contacted at 35 C with an equal volume of 50 vol. % TBP-NPH pretreated as before. Both phases were sampled after 10, 60, and 180 minutes and the distribution ratios of americium, curium and cerium were measured as well as the pH of the aqueous phase.

TABLE VII
HOT CELL TEST I(7)

Organic: 50 vol. % TBP-NPH
Aqueous Normal Redox D-9 adjusted
with RE's to 1100 MWd/T
spiked with Am-241
Initial pH: 0.50
o/a: 1
Temperature, C: 35

Contact Time (min.)	E_a^O Am	E_a^O Ce	E_a^O Cm	Final pH
10	240	159	260	1.12
60	267	170		1.12
180	321	270		1.12

Redox waste from Shippingport fuel processing was used as feed for the second full-level test, Hot Cell Test II. This test was designed to determine the americium and curium distribution with actual plant feed as well as the effect on recovery of the actinides by multiple extractions into the organic phase without stripping after each batch contact. The back extraction of the actinides into 1M HNO_3 was also measured. The Redox Shippingport waste was adjusted to contain 0.6M NaNO_2 to destroy the dichromate. The aqueous feed was then contacted with an equal volume of 50 vol. % TBP-NPH for 15 minutes. After disengaging, the aqueous phase was discarded and the organic phase again contacted with a fresh volume of adjusted feed. This was repeated for 4 cycles. The organic phase was then contacted with an equal volume of 1M HNO_3 strip solution. After each batch contact both phases were sampled and the americium and curium distributions measured. No pH adjustment was required other than that

resulting from the addition of the NaNO_2 . The results are shown in Table VIII. It was observed that phase disengaging times varied greatly. In two of the extraction cycles (#1 and 3) the disengaging times were less than 15 seconds while in the other two, 15-20 minutes were required. Solids found in the feed solution may have caused this variance. The solids, presumably ZrO_2 , carried all of the ZrNb-95 activity and little or none of the CePr-144 or Eu-154 activity. The distribution of the cerium and europium activity was followed qualitatively with the in-cell gamma spectrometer.

TABLE VIII
HOT CELL TEST II (7)

Organic: 50 vol. % TBP-NPH
 Aqueous: Redox Shippingport Waste (with
 the addition of 0.6M/l NaNO_2)
 Initial pH: 0.96 (before addition of
 NaNO_2 , pH < 0)
 Contact Time: 15 minutes
 Temperature, C: 35
 o/a: 1

Extraction Cycle	Isotope	Concentration Organic Phase (g/l)	Concentration Aqueous Phase (g/l)	Distribution E_a^0	Final pH
1	Am-241	0.059	9.6×10^{-4}	61	1.2
	Cm-244	4.2×10^{-3}	6.0×10^{-5}	69.2	
	Cm-242	3.8×10^{-5}	5.3×10^{-7}	71.3	
2	Am-241	0.132	7.3×10^{-4}	181	0.7
	Cm-244	9.1×10^{-3}	4.5×10^{-5}	203	
	Cm-242	8.3×10^{-5}	4.6×10^{-7}	181	
3	Am-241	0.172	1.3×10^{-3}	130	0.8
	Cm-244	1.2×10^{-2}	8.6×10^{-5}	137	
	Cm-242	1.1×10^{-4}	7.6×10^{-7}	143	
4	Am-241	0.249	1.9×10^{-3}	132	1.0
	Cm-244	1.7×10^{-2}	1.2×10^{-4}	137	
	Cm-242	1.6×10^{-4}	1.1×10^{-6}	136	

Strip Cycle

(1M HNO_3)	Am-241	1.3×10^{-3}	0.245	5.3×10^{-3}
	Cm-244	7.7×10^{-5}	1.7×10^{-2}	4.5×10^{-3}
	Cm-242	6.8×10^{-7}	1.6×10^{-4}	4.3×10^{-3}
	Ce-144	-	34 Ci/l	-
	Eu-154	-	2.2 Ci/l	-

C. Flowsheet - On the basis of laboratory development⁽⁷⁾, an Am-Cm recovery flowsheet was proposed for Redox Plant application⁽²⁾.

The principal processing steps shown in Figures 10 and 11 included the following:

- 1) Feed Treatment - Sodium nitrite was added to the salt waste to reduce Cr^{+6} to Cr^{+3} , and the pH was adjusted to a range of 0.0 to 0.5.
- 2) Am-Cm Batch Extraction - The Am-Cm was extracted using an equal-volume contact with solvent (50 vol. % TBP in normal paraffin hydrocarbon diluent). The raffinate was decanted from the extract, and succeeding feed batches were contacted using the product-containing organic phase as the extractant.
- 3) Am-Cm Batch Back-Extraction - Back-extraction (by batch contact) of the Am-Cm with a 1/2-volume of 1M HNO_3 solution was made when excessive aqueous raffinate waste losses indicated the need to reduce the product loading in the organic extractant.
- 4) NPH Scrub - The dilute aqueous product solutions were washed with NPH diluent (by batch contact) to remove dissolved and entrained TBP to prevent formation of explosive organo-uranium compounds during product evaporation and to guard against possible process problems during subsequent processing steps.
- 5) Organic Treatment - Organic treatment was performed at the end of the Am-Cm campaign prior to organic disposal. Following the Am-Cm back-extraction, uranium and plutonium were stripped with

batch contacts of water into the aqueous phase. Following the U-Pu batch strip, the organic was contacted with 0.8 wt. % NaOH and 2.5 wt. % Na_2CO_3 solution to remove dibutyl phosphate (DBP) and residual fission products. This proposed organic treatment step required modification due to severe emulsification problems experienced during the plant campaign, as discussed later in the Plant Performance section.

- D. Equipment Requirements - The Am-Cm recovery campaign was performed in the normal dissolver solution storage and feed treatment vessels concurrently with processing activities in the Redox Plant.

Process routings were installed to utilize the following tanks in their listed functions.

<u>Tank</u>	<u>Process Function</u>
H-7	Feed treatment and storage
H-8	Batch extraction
H-9	Batch back-extraction, make-up solvent inventory and organic treatment
H-10, D-8	Waste neutralization
H-1	NPH scrub
H-4	Preliminary product evaporation and interim storage
F-5	Product evaporation and load-out tank

Batch contacts of organic and aqueous phases were made in the H-1, H-8 and H-9 tanks. These are baffled, 2400-gallon tanks. Agitation was provided with 300 RPM, 10-blade, 4-inch x 22-inch diameter, turbine

agitators. Instrumentation for control of the batch contacts and solution decanting was provided by the normal air-purge differential-pressure dip tubes for weight factor and specific gravity. In addition, interface and organic specific gravity dip tubes were installed in the H-8 batch extraction tank.

The Am-Cm product was loaded into a "bowling ball" cask from the F-5 concentrator by vacuum transfer. The 350-gallon, 30-ton cask supplied by Pacific Northwest Laboratories, provided 9 inches of lead shielding. This product cask was designed for "contact" hookup of process lines for solution transfers. The Am-Cm campaign required loading of the cask in the normal F-4 position in F-Cell of the Redox Canyon. An adapter was designed to permit cask installation and to provide cask routings and instrumentation by use of the canyon crane and remotely installed jumpers. The function of the cask adapter was to permit use of remote connectors without modifying the existing cask "Swagelok"* connectors. The cask adapter was a kick-plate flange with male remote connectors supported approximately two feet above the top of the cask by dunnage bolted to the cask. Polyethylene tubing was used to make the transition between the remote connectors and the existing cask connectors. The connector flange was equipped with a lip on the outer circumference and a drain tube to direct radioactive solution spills to the cell floor, minimizing cask contamination. Services provided by the cask adapter for remote jumper installation were:

*Trademark of Crawford Fitting Co.

1. Loading connector;
2. Suction connector for remote evacuation jet;
3. Cooling water inlet connector;
4. Cooling water outlet connector;
5. Electrical connector for liquid level probe and cask temperature;
6. Unloading connector, normally blanked.

Figure 12 shows the cask and adapter being prepared for transfer into the Redox Canyon. Figure 13 shows the Redox crane transferring the cask in the canyon.

E. Plant Performance - A total of 4340 gallons of Shippingport waste was processed with 50 vol. % TBP-NPH in 5 batches with a net Am-Cm recovery of 92 percent. The batch extraction and strip contacts were made with from 1400 to 2000 gallons total of aqueous and organic phases by agitation in baffled tanks. Am-Cm extraction distribution coefficients, E_a^O , of 85, decreasing to 45, were observed during the campaign. Following an extraction or back-extraction strip contact, the two phases were decanted by settling and jet transfer of either the organic or aqueous phase to another tank leaving approximately a 100-gallon heel of the transferred phase. Organic losses during decanting of an aqueous phase to another tank were approximately 10 gallons for each transfer.

During the series of extraction contacts in the Redox Plant, phase disengaging time increased from 6 hours to 20 hours. Laboratory investigation of this problem during the plant campaign indicated that residual zirconium, originating during the ~~Zir~~flex dissolving of the Shippingport fuels, could be responsible for the emulsification problem. The effect

of soluble zirconium causing an emulsion was qualitatively demonstrated in the laboratory by adding zirconium nitrate to synthetic Shippingport waste and making an extraction contact with 50 vol. % TEP-NPH. Plant instrumentation during decanting operations also indicated that a stable emulsion from 5 to 10 inches thick remained above the interface after settling an extraction contact. Portions of the interfacial emulsions were transferred with the organic phase to the back-extraction tank. It is believed that this interfacial emulsion was caused by the high solids content of the Shippingport waste. The solids are from ZrO_2 fines left in the dissolver after the decladding step and are formed during evaporation of the Shippingport waste to a high salt concentration. The belief that solids caused interfacial emulsions was supported by the visual observation of ZrO_2 solids in the loaded-out cask solution and by the low decontamination factor obtained for inextractable cations (approximately 80).

A severe problem of emulsification was experienced also in the third (and last) back-extraction contact. In this case, a stable aqueous-phase emulsion was formed that was resistant to (a) increasing the back-extractant HNO_3 concentration from 1M to 3.4M; (b) increasing the temperature from 25 C to 65C; and (c) the addition of 100 ppm of a coalescing agent, $MgSiO_3$. The emulsion finally broke after 45 hours of settling time.

During the Redox Plant campaign, process control of the 50 vol. % TBP-NPH processing was followed by analysis of the aqueous extraction raffinate and the aqueous back-extracted strip solution for total alpha activity. Samples of the organic phase for analytical determinations were not taken. Redox Plant performance, calculated by material balance from weight factor recorder charts and the analytical data available, are presented in Table IX, Redox Plant Am-Cm Product Distribution, and Table X, Redox Plant TBP-NPH Solvent Extraction Performance.

TABLE IX
REDOX PLANT Am-Cm PRODUCT DISTRIBUTION

	<u>%</u>
Dissolver decladding solution waste loss	0.6
Hexone, U, Pu, Np partition from Am-Cm, loss	0.4
Terminal organic Am-Cm inventory, organic treatment waste loss	3.3
Product tank and evaporator heels	1.8
Product load-out	92

TABLE X

REDOX PLANT TEP-MPH SOLVENT EXTRACTION PERFORMANCE

Feed Alpha Activity, 5.80×10^{12} d/min/gal (4340 gallons)

Feed Batch Extraction Contact	Back Extraction Strip Contact	Aqueous Volume, Gallons	Organic Volume, Gallons	a/o	Aqueous Alpha Activity, d/m/gal	Am-Cm E_a^0	Aqueous Volume to Waste Neutralization, Gallons	Am-Cm to Waste, %	Aqueous Volume to Product Evaporator, Gallons	Am-Cm to Product Evaporator %	Solvent Exposure at Start of Contact, Watt-hours/Liter	Settling Time Required for Disengaging, Hours
1	-	1050	1000	1.05	6.54×10^{10}	86(a)	850	0.22	-	-	0	6
2	-	960	990	0.97	1.95×10^{11}	51	860	0.66	-	-	1	15
3; 1st ext'n	-	940	980	0.96	2.02×10^{11}	73(b)	-	-	-	-	6	16
	1	720	1070	0.67	1.82×10^{13}	0.05	-	-	660	47.6	12	-
3; 2nd ext'n + interface displace- ment with feed sol'n	-	1230	760	1.62	6.86×10^{10}	52	1170	0.32	-	-	13	15
4	-	960	950	1.01	1.46×10^{11} 2.28×10^{11}	56	200	0.12	-	-	14	15
5; 1st ext'n	-	1230	740	1.66	1.98×10^{11}	65	-	-	-	-	17	20
	2	700	1030	0.68	1.48×10^{13}	0.05	-	-	640	37.6	21	-
5; 2nd ext'n	-	1230	680	1.81	7.70×10^{10}	45	1230	0.38	-	-	22	18
	3	560	830	0.67	5.3×10^{12}	0.06	-	-	560	9.6	23	30-45
									Flushes	0.8		
									1.9	95.6		

(a) Aqueous pH after extraction = 1.05

(b) Aqueous pH after extraction = 0.85

(Evaporator feed
sample 93.8)

The composition of the final product solution loaded into the "bowling ball" cask is presented in Table XI.

TABLE XI
PRODUCT COMPOSITION
(Estimated volume - 275 gal)

UNH	0.06 <u>M</u>
Al(NO ₃) ₃	0.3 <u>M</u>
HNO ₃	2.2 <u>M</u>
NaNO ₃	0.2 <u>M</u>
Cr(NO ₃) ₃	0.1 <u>M</u>
Fe(NO ₃) ₃	0.08 <u>M</u>
F ⁻	0.06 <u>M</u>
Pu	0.06 g/gal
Np	0.09 g/gal
Am-241	3.3 g/gal
Am-243	0.94 g/gal
Cm-242	0.0017 g/gal
Cm-244	0.23 g/gal
RuRh-106	21 Ci/gal
Cs-137	8.0 Ci/gal
CePr-144	890 Ci/gal
Pm-147	900 Ci/gal
Pm-146/Pm-147	3.5 x 10 ⁻⁶ Ci/Ci
Pm-148/Pm-147	1.6 x 10 ⁻⁶ Ci/Ci
Eu-154	46 Ci/gal
Total Rare Earths (as oxides)	131 g/gal

Based on analytical results, an arithmetic decontamination factor (DF) of 80 was attained for total salts, and a DF of 2 for uranium. Contamination of the product with salts resulted from poor disengaging characteristics of the extraction contact and transfer of interfacial emulsion with the organic phase to the strip contactor during decanting.

Solvent washing was required prior to solvent discard to avoid personnel exposure problems that would otherwise be caused by the high radiation levels. Laboratory work during the campaign with plant solvent indicated that solvent treatment with dilute NaOH - Na_2CO_3 solutions as specified in the flowsheet⁽²⁾ would form a stable emulsion. The laboratory work indicated that an unidentified constituent was building up in the organic phase as the solvent was re-used giving the solvent a white, cloudy appearance and causing the formation of increasingly stable emulsions. A satisfactory alternate flowsheet was developed in which a dilution solution of diethylenetriaminepentaacetic acid (DTPA), buffered with hydroxyacetic acid to minimize emulsion formation, was used to strip out the cloudy constituent and emulsifying agent. This was followed with dilute NaOH - Na_2CO_3 washing. The washing contact sequence used in the plant per 1 volume of solvent was:

2 washes	1/2 volume 1M HNO_3
1 wash	1/2 volume 0.05M DTPA, 0.5M hydroxyacetic acid
1 wash	1/2 volume 0.05M DTPA
2 washes	1/2 volume H_2O
2 washes	1/2 volume 0.8 wt.% NaOH, 2.5 wt.% Na_2CO_3
2 washes	1/2 volume H_2O

The series of washes resulted in decontamination factors and activity levels as follows:

TABLE XII
PLANT SOLVENT TREATMENT SUMMARY

<u>Component</u>	<u>Decontamination Factor (DF)</u>	<u>Concentration in Washed Solvent</u>
ZrNb-95	$>4 \times 10^4$	$3 \times 10^4 \mu\text{Ci/gal}$
Ru-103, RuRh-106	$>4 \times 10^4$	$1.7 \times 10^4 \mu\text{Ci/gal}$
Total Alpha	-	$6.2 \mu\text{Ci/gal}$
Total Beta	-	Non-detectable

IV. Semiworks D2EHPA Processing

A. Laboratory Development - The reaction mechanism for extraction of a metal ion by D2EHPA at trace loading is generally accepted to be

where $(\text{HR})_2$ = dimerized D2EHPA

M^{+z} = metal ion of valence z

aq = aqueous phase

org = organic phase

H^+ = hydrogen ion

R = di-(2-ethylhexyl)phosphate ion

It can be seen from the above equation that extraction of a metal ion is dependent upon the hydrogen ion concentration of the aqueous phase so that extraction and back-extraction (stripping) can usually be

accomplished by suitable adjustments of the aqueous phase pH. Organic complexing agents are often added to the aqueous phase to prevent extraction of certain undesirable metal ions and to provide buffering for pH control.

Laboratory studies were initiated⁽⁸⁾ to determine process conditions for extracting Am, Cm, and Pm from a synthetically prepared feed shown in Table XIII to approximate the Redox crude product. Figure 14 shows the extraction distribution ratio results as a function of pH for several metal ions of interest. Nitrilotriacetic acid (NTA) was used for complexing selected metal ions to inhibit their extraction. Radioactive tracers were used to follow the behavior of the cations shown. Table XIV shows results of laboratory studies for back-extracting (stripping) the Am, Ce, and Al from the solvent. Note that short contact times are desirable to maximize aluminum decontamination.

TABLE XIII
SYNTHETIC SHIPPINGPORT SEMIWORKS FEED⁽⁸⁾

<u>Constituent</u>	<u>Concentration, Moles/liter</u> <u>HAF-HAS*</u>
Al(NO ₃) ₃	0.031
NaNO ₃	1 - 2
Nd(NO ₃) ₃	0.021
UO ₂ (NO ₃) ₂	0.0037
Fe(NO ₃) ₃	0.0076
HC ₂ H ₃ O ₃	0.35
NTA	0.13

*For terminology see page 34.

TABLE XIV
STRIPPING AS A FUNCTION OF CONTACT TIME⁽⁸⁾

Aqueous: 1.5M HNO₃
 a/o: 1

Contact Time (Minutes)	Stripping Distribution Ratio, E_a^0		
	<u>Am⁺³</u>	<u>Ce⁺³</u>	<u>Al⁺³</u>
1	0.028	0.038	13.2
2	0.027	0.038	-
5	0.026	0.038	10.0
15	0.025	0.038	8.4

A gallon sample of the Redox crude product was removed from the 30-ton "bowling ball" cask and processed in a "hot" cell at the 325-A High-Level Radiochemical Facility to demonstrate the synthetically developed technology. The sample was found to contain from 10-30 volume percent solids. A simple acid wash of the solids was found to remove the associated rare earths, americium, and curium. A batch solvent extraction test using 0.4M D2EHPA, 0.3M TBP diluted with NPH yielded extraction distribution ratios of 90, 79, 20, 19, and 40 for Ce⁺³, Eu⁺³, Am⁺³, and Pm⁺³, respectively.

- B. Flowsheet - Using the laboratory data presented in the previous section, a flowsheet was written to further purify the Am, Cm and rare earths in pulse column equipment. The simplified flowsheet is shown in Figure 15. The product solution containing some solids was unloaded from the cask into the Semiworks. After flushing the cask, the supernate was decanted and the solids leached with 2 molar nitric acid to recover the Am, Cm and rare earths associated with the solids. The acid leach and supernate

were combined, sodium nitrilotriacetate (Na_3NTA), hydroxyacetic acid and water were added, and the pH was adjusted to 4.0 with sodium hydroxide to prepare solvent extraction feed.

The adjusted feed (HAF) was pumped at 0.21 gal/min to the first pulse column (HA) where the Am, Cm, and rare earths were extracted into the solvent (0.4M D2EHPA - 0.3M TBP-NPH diluent). A scrub solution (HAS) flowing at 0.11 gal/min and containing 0.5M $\text{HC}_2\text{H}_3\text{O}_3$ and 0.01M NTA was used to reduce the sodium content of the solvent prior to its entering the second pulse column (HC). Nitric acid at 2.0 molar was added to the second pulse column at 0.11 gal/min to strip the Am, Cm, and rare earths from the solvent. The solvent was then washed with acid followed by water and adjusted with sodium hydroxide prior to recycling the solvent to the HA Column. The aqueous product from the HC Column was steam stripped and concentrated. Excess acid was destroyed by denitration with sugar. The concentrated product was loaded into a 20-ton "bowling ball" cask and shipped to Pacific Northwest Laboratories.

- C. Equipment Requirements - Two pulse column contactors, batch contactors, concentrators, pumps, tanks and associated equipment were used to further purify the americium, curium and rare earths recovered at Redox. The 4-inch OD pulse columns contained nozzle plate cartridges at 4-inch spacing. The first column cartridge was 25 feet in height divided into a 10-foot extraction section with 23% free area plates and a 15-foot scrub section with 10% free area plates. The second column

cartridge was 16 feet in height with 10% free area plates.

Pulses were supplied by motor driven piston in a cylinder arrangement. The pulse frequencies for both columns generally varied between 50 and 60 cycles per minute at an amplitude of approximately one inch. The batch contactors were simply tanks containing agitators for mixing the aqueous and organic phases. The concentrators were steam heated to minimize organic flammability problems.

- D. Plant Performance - The Semiworks run performance was excellent with no major flowsheet or equipment problems encountered. Extraction waste losses for Am-Cm and Ce were approximately 4 percent. The losses increased slightly near the end of the run due to accumulation of uranium in the solvent. In addition, about 1 percent of the Am-Cm and Ce was not stripped from the solvent in the HC Column.

The composition of the crude feed and final product with appropriate decontamination factors (DF) and recovery efficiencies are presented in Table XV. Material balances were very good and the product quality exceeded expectations. The product was loaded into a 20-ton "bowling ball" cask and shipped to Pacific Northwest Laboratories where the Am, Cm and Pm were successfully separated from one another and further purified⁽⁴⁾.

TABLE XV

SHIPPINGPORT SEMIWORKS FEED AND PRODUCT
COMPOSITION, DECONTAMINATION FACTORS AND RECOVERY EFFICIENCIES

<u>Constituent</u>	<u>Concentration</u>		<u>DF</u>	<u>Recovery Efficiency, %</u>
	<u>Feed (HAF)*</u>	<u>Product</u>		
Am-241	0.53 g/gal	8.0 g/gal	-	95
Am-243	0.15 g/gal	2.3 g/gal	-	95
Cm-242	0.38 Ci/gal	5.8 Ci/gal	-	95
Cm-244	0.037 g/gal	0.56 g/gal	-	95
Pm-147	129 Ci/gal	1900 Ci/gal	-	93
CePr-144	91 Ci/gal	1360 Ci/gal	-	94
Eu-154	7.5 Ci/gal	110 Ci/gal	-	93
Cs-137	1.3 Ci/gal	0.19 Ci/gal	110	-
RuRh-106	2.6 Ci/gal	1.9 Ci/gal	20	-
Rare Earths	0.034M	0.50M	-	93
Al	0.051M	<0.0011M	>740	-
Fe	0.012M	0.0016M	120	-
U	0.012M	2 x 10 ⁻⁵ M	9500	-
Na	1.1M	0.39M	45	-
H	-	0.53M	-	-
NTA	0.10M	-	-	-
HC ₂ H ₃ O ₃	0.40M	-	-	-
Volume	1690 gal	106 gal	-	-

*The feed concentration of Cm-242, Pm-147, CePr-144 and RuRh-106 differ from those in Table XI due to radioactive decay during the time interval between processing at Redox and Semiworks.

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Fig. 1

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Fig. 2

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Fig. 3

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Fig. 4

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32993-C

SHIPPINGPORT BLANKET FUEL ASSEMBLY

FUEL UO_2 ----- 284 lb.
ZIRCONIUM ALLOY _ 186 lb.
STAINLESS STEEL _ _ 40 lb.

LATCH ASSEMBLY

COOLANT FLOW PASSAGE

TOP BUNDLE SPACER

SHELL ASSEMBLY

FUEL BUNDLE (7)

COOLANT FLOW HOLES

TUBE SHEET

FUEL RODS
(120)

5.186"

10 1/4"

Fig. 5

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Fig. 6

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FIGURE 7

FUEL DISSOLUTION AND PRODUCT-WASTE PARTITION FLOW DIAGRAM

FIGURE 8
EFFECT OF NITRATE ON EXTRACTION OF AMERICIUM AND EUROPIUM (3)

FIGURE 9
 EFFECT OF ACID ON EXTRACTION OF AMERICIUM AND EUROPIUM (3)

PRODUCT EXTRACTION

ORGANIC DECANT & PRODUCT STRIP

FIGURE 10

Am-Cm EXTRACTION, DECANT AND STRIP FLOW DIAGRAM

PRODUCT DECANT & NPH SCRUB

PRODUCT DECANT, CONCENTRATION & CASK LOAD-OUT

FIGURE 11

Am-Cm SCRUB, EVAPORATION AND CASK LOAD-OUT FLOW DIAGRAM

Fig. 12

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Fig. 13

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FIGURE 14
EXTRACTION DISTRIBUTION RATIOS AS A FUNCTION OF pH USING NTA (8)

FIGURE 15

SEMIWORKS SIMPLIFIED

FLWSHEET FOR PROCESSING

SHIPPINGPORT Am, Cm, AND RE

PRODUCT CONCENTRATION

AND CASK LOAD-OUT