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SPECIAL RECOVERY

MASTER

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ROCKWELL INTERNATIONAL
ATOMICS INTERNATIONAL DIVISION
ROCKY FLATS PLANT
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GOLDEN, COLORADO 80401

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MODIFIED PUREX PROCESS
FOR THE SEPARATION AND RECOVERY
OF PLUTONIUM-URANIUM RESIDUES

James D. Navratil and Robert G. Leebl

Abstract. A modified (one-cycle) Purex process has been developed for the separation and recovery of plutonium and uranium from mixed actinide residues. The process utilizes 30 volume percent tributyl phosphate-dodecane to extract uranium from a 5M nitric acid-plutonium (III)-uranium(VI) feed. After uranium extraction, plutonium in the aqueous feed solution is purified by anion exchange technology. Uranium in the organic is scrubbed and stripped to effectively purify the uranium so that it contains <5,000 ppm plutonium. The process has been used successfully to separate residues consisting of plutonium and uranium oxide.

INTRODUCTION

Chemistry Research and Development was asked to investigate processes for separating small amounts of miscellaneous, mixed actinide residues for recovery. The plutonium residues concerned here contained 0.1 to 54 wt % uranium. The residues were received as oxides.

Processing the variety of plutonium oxide and uranium oxide mixtures required recovery development. The governing criteria for the plutonium-uranium separation were <5,000 ppm of uranium in the plutonium product and <5,000 ppm plutonium in the uranium product. These requirements permit prompt shipment of the plutonium product to the plutonium purification, ion-exchange process at Rocky Flats and the uranium oxide (U_3O_8) product to the Savannah River Plant. Processing methods for separation and recovery included ion exchange, precipitation, and solvent extraction techniques. This report deals with the last technique.

A modification of the Purex process was selected because (1) that method was best suited for a variety of plutonium-uranium ratios, (2) fission product removal was not required, and (3) plutonium purification by ion exchange was available. Solvent extraction evaluation included (1) development of a flow sheet for the process, (2) familiarization and direct experience with solvent extraction as a special recovery tool, and (3) demonstration of the solvent extraction process to include an evaluation of its capability for processing the mixed actinide residues.

EXPERIMENTAL

Materials

Tributyl phosphate (TBP) was supplied by either E. H. Sargent and Company (technical grade) or by Eastman Kodak Company. Eastman practical dodecane was used. Freshly prepared 30 vol % TBP-dodecane was equilibrated twice with equal volumes of 1M Na_2CO_3 (sodium carbonate) to remove residual traces of TBP. Prior to use of the treated organic, it was equilibrated twice with equal volumes of 5M HNO_3(nitric acid)-0.1M $Fe(SO_3NH_2)_2$ (ferrous sulfamate) to acidify the organic. Ferrous sulfamate was supplied as a 3M solution from the Shepherd Chemical Company. All other chemicals were reagent grade and were used without further purification.

The mixed actinide solutions were prepared by dissolving mixed oxides in 12.5M HNO_3-0.1M HF (hydrofluoric acid). After dissolution, the solutions were filtered and a stoichiometric amount of aluminum nitrate was added to complex the fluoride ion. Dilution of the stock mixed actinide solutions usually was made with 0.35M HNO_3 or used scrub solution.

Equipment and Procedure

Batch equilibrium experiments were conducted at ambient temperatures (22-24 °C) using laboratory-scale separatory funnels (250-400 ml). Equal volumes of both phases were mixed, using an air-motor stirrer, for 20 minutes in the funnel. The phases were allowed to separate for 20 minutes and then were sampled in duplicate.

Continuous, countercurrent, solvent extraction studies were performed with a small pilot-plant facility. The facility consisted of four 16-litre glass-storage tanks (15 cm diameter) and three York-Scheibel extraction columns enclosed in a glove box. Scrub and strip solutions were stored in eight-litre glass tanks located on top of the glove box.

The York-Scheibel columns, 2.5 cm in diameter by 122 cm high, contained 11 transfer units each. Each transfer unit consisted of a mixing chamber (stirring is by a 1,000 to 1,200 rpm air motor) and a settling chamber containing stainless steel baffles. Tankage was arranged so as to allow the organic and aqueous solutions to gravity feed through rotameters to the columns. The organic (light phase) entered the column from the bottom and exited at the top; the aqueous (heavy phase) route was the opposite. The columns initially were filled with the organic phase prior to the aqueous flow being started.

Routine processing of the uranium-plutonium residues was performed with the tankage of the small pilot plant facility. One of the 16-litre glass tanks was converted to a batch contactor by placement of a Teflon tapered insert at the bottom of the tank and by the addition of an air sparge used for mixing.[1]

The air-sparge batch contactor (Figure 1) was operated by placing the tank under vacuum and transferring the aqueous and organic phases into that tank from the other 16-litre holding tanks. The valves then were closed, but the vacuum was maintained. This allowed air to enter through a sample tap at the bottom of the tank for mixing the aqueous and organic phases. After five minutes of mixing, the tank was vented, and the phases were allowed to separate for 10 minutes. The phases then were removed via the bottom outlet. The white Teflon insert facilitated good separation of the phases by allowing the interface to pass through a restricted area. The insert also provided good visual observation for interface detection.

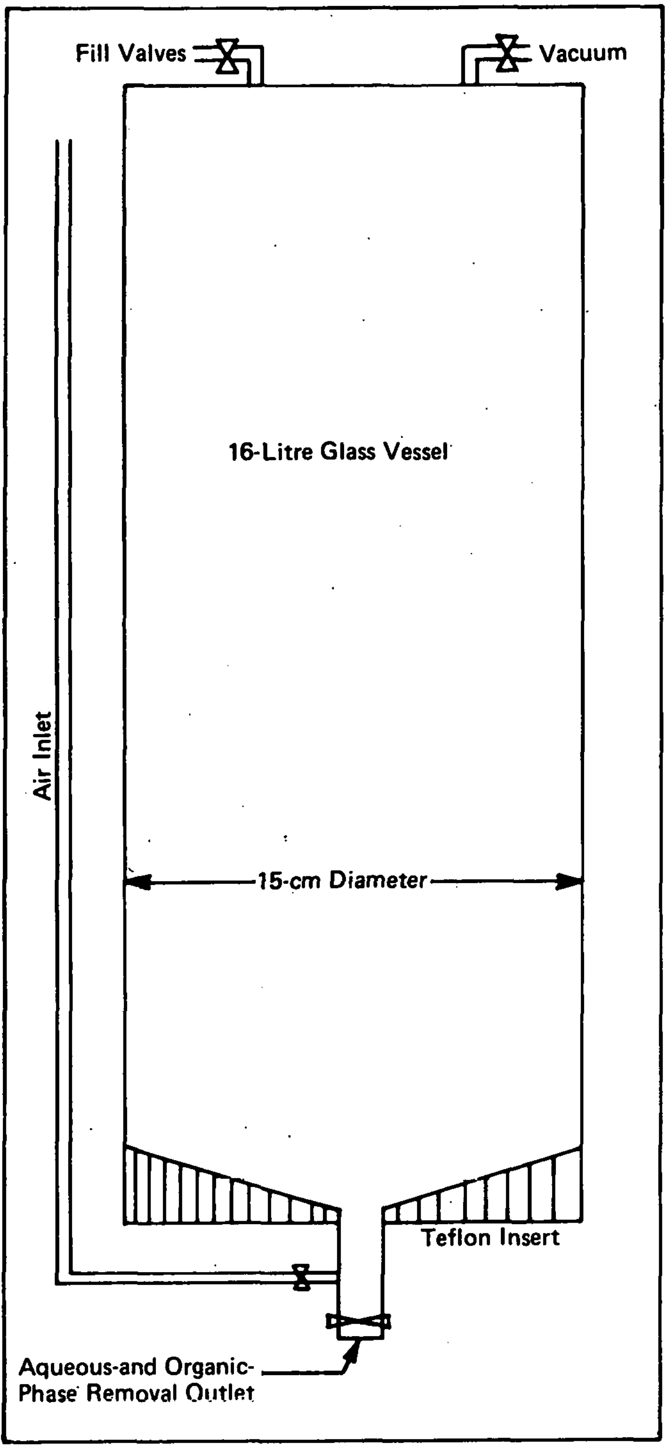

FIGURE 1. Air Sparge Batch Contactor

Analysis

Uranium in <0.1 g/l solutions was determined by fluorometric techniques whereas uranium in >0.1 g/l solutions was determined by X-ray fluorescence (wavelength dispersive method). Plutonium in these samples was removed by a precipitation method prior to analysis. Plutonium was analyzed radiometrically except for >1 g/l plutonium solutions, which were analyzed by X-ray fluorescence (wavelength dispersive method). The oxidation state analysis of the plutonium was accomplished by spectrophotometric techniques.

The iron(II) normality in ferrous sulfamate was determined by volumetric ceric sulfate titration. Trace impurities in the feed solutions were determined by optical emission spectroscopy, whereas macroamounts of anions were determined by ion selective electrodes and wet chemical techniques. Nitric acid concentrations were determined by acid-base titration. Hydrolysis products in the tributyl phosphate (mainly dibutyl phosphate) were estimated by plutonium retention numbers.[2] The percentage of tributyl phosphate in the 30 vol % TBP-dodecane was checked periodically by infrared spectroscopy techniques.

RESULTS AND DISCUSSION

Plutonium and Uranium Separation Methods

Ion exchange, solvent extraction, and precipitation separation techniques were considered for recovering plutonium and uranium from mixed oxide residues. The governing criteria for the plutonium-uranium separation were <5,000 ppm of uranium in the plutonium product and <5,000 ppm of plutonium in the uranium product.

Anion exchange separation of plutonium and uranium, using Dowex* 1-X4 anion exchange resin, has the disadvantage of requiring large volumes of 7M HNO_3 wash to remove the weakly sorbed uranium from the plutonium-loaded resin. Preliminary experiments were performed to compare other resins to Dowex 1-X4 from the standpoint of uranium removal by washing. A few macroreticular resins were tested with disappointing results.[3]

Precipitation methods were considered for feeds with a high uranium content. The separation of uranium from plutonium in a nitric acid medium was tried by precipitating uranium as sodium magnesium uranyl acetate. The results of a preliminary experiment appear adequate, but the work is not yet finished.[4]

During development of the modified Purex process, several extraction systems were considered. Amine extractants have the advantage of extracting plutonium(IV) and leaving uranium(VI) in the feed. This is an especially attractive scheme when uranium is the major constituent in the feed.

Schulz and his co-workers developed a reflux amine flow sheet for recovering plutonium from metallurgical scrap.[5] The secondary amine extractant (Amberlite** LA-2) was an improvement over tributyl phosphate to purify plutonium because of process simplification and reduction in aqueous waste. The amine flow sheet was not tested, however, for purification of plutonium from scrap that contained uranium.

Extraction of plutonium(IV) from 10 g/l plutonium and uranium feed solutions in 7M HNO_3, using 35 vol % Amberlite LA-2 and 10 vol % octyl alcohol-docecane, was evaluated. The plutonium(IV) extraction coefficients were good $[E^{O}_{Pu(IV)} = 5 \text{ to } 15]$, but the extraction coefficients for uranium were too high $[E^{O}_{U(VI)} = 0.4 \text{ to } 1]$ to permit good separation. Residual uranium in the plutonium loaded organic also was found difficult to remove by 7M HNO_3 scrubbing; therefore, work with this extractant was discontinued.

Development of Modified Purex Process

A modified (one-cycle) Purex process using 30 vol % TBP-dodecane as the extractant has shown the best performance for separating plutonium-uranium residues. The process, briefly outlined in Figure 2, will be followed in the separation of plutonium-uranium residues.

In this process, uranium is extracted from 4 to 5M HNO_3-Pu(III)-U(VI) feed solution into 30 vol % TBP with dodecane used as the diluent. The plutonium, reduced to plutonium(III) with ferrous sulfamate $\{[Fe(II)]/[Pu] = 3\}$ is not extracted in a significant quantity, and the amount left remains in the aqueous phase. The aqueous feed solution containing the plutonium and impurities is processed by routine anion-exchange technology. The uranium-loaded organic phase is then washed free of residual plutonium with 0.35M HNO_3 containing 0.1M $Fe(SO_3NH_2)_2$. Finally, a sodium or ammonium sulfate solution is used to strip the uranium from the organic. The uranium product treatment consists of ammonia (NH_3) precipitation and calcination to uranium oxide. An alternate stripping procedure, which combines the stripping and precipitation steps, consists of using ammonia for direct

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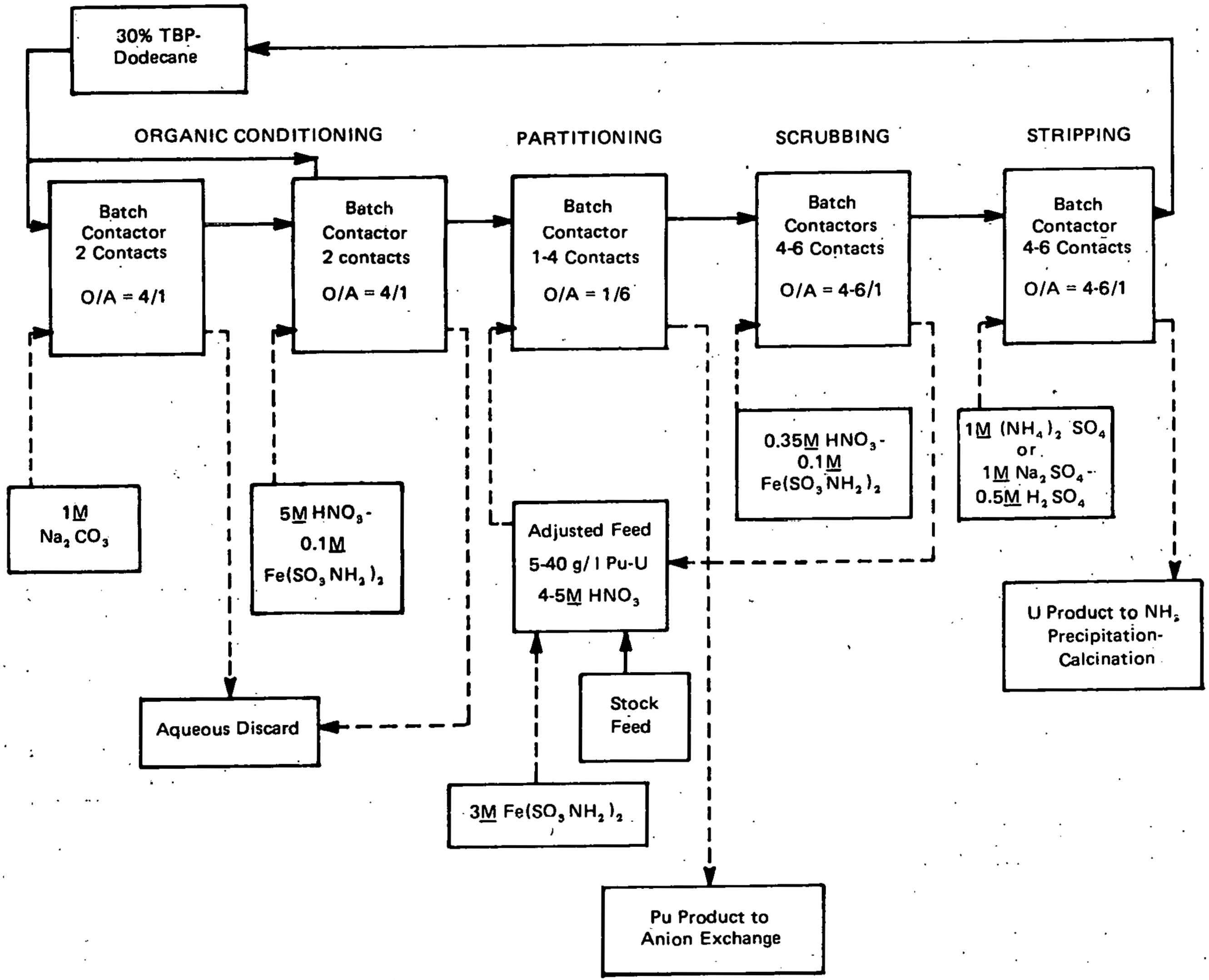
*Trademark of The Dow Chemical Company, Midland. Michigan.

**Manufactured by Rohm and Haas Company, Philadelphia, Pennsylvania.



LEGEND

Aqueous Stream -----
Organic Stream _____
O/A = Organic Aqueous Volume Ratio

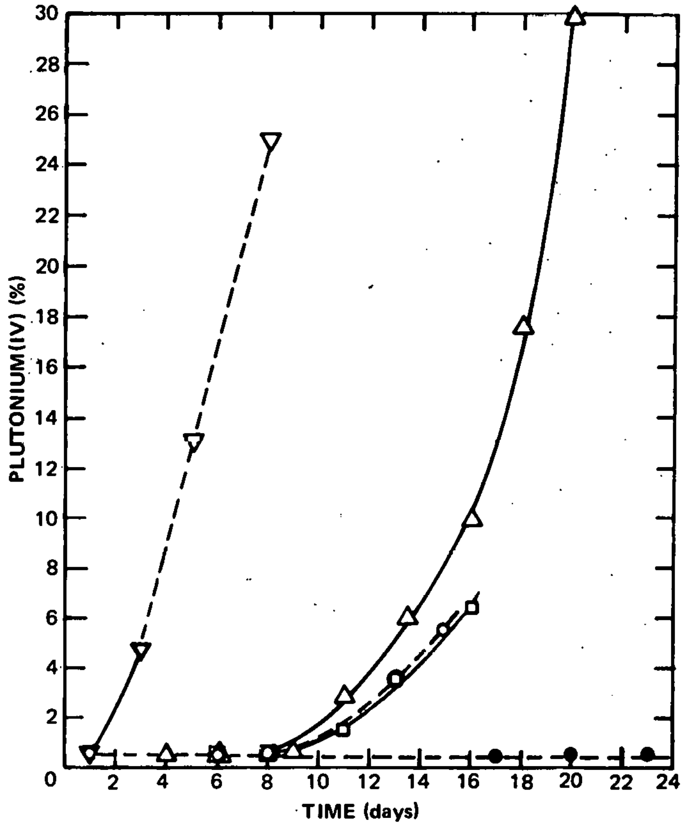
FIGURE 2. Flow Sheet for the Modified (One-Cycle) Purex Process

precipitation of uranium from the scrubbed organic. Sodium carbonate washing of the organic also is used periodically to remove dibutyl phosphate (produced by alpha radiolysis of tributyl phosphate) when large plutonium retention numbers are obtained (about once a month).

Immediately before the partitioning steps, plutonium in the feed is reduced to the aqueous-favoring trivalent species. Ferrous sulfamate, which is more effective than hydroxylamine nitrate, was used in the process to keep the plutonium reduced. Figure 3 shows the time required for plutonium(III) to oxidize to plutonium(IV) in representative feed samples from the batch equilibrium experiments (vide infra). The data show that during the equilibrium experiments, which required no more than one day, the plutonium was maintained in the trivalent oxidation state.

The data in Figure 3 also show increased oxidation rates of trivalent plutonium with increased nitric acid concentrations. Doubling the ratio of iron(II) to total plutonium (mole basis) slowed the rate of oxidation considerably. The increased plutonium(III) oxidation with acidity also is caused by increased oxidation of iron(II) with increased nitric acid concentration.[6]

Laboratory-scale, batch, equilibrium techniques were used to evaluate the one-cycle, modified, Purex process for plutonium-uranium separation. Preliminary extraction, scrubbing, and stripping parameters for the process were determined.



LEGEND

[Fe(II)]/[Pu] = 3 (solid line)	[Fe(II)]/[Pu] = 6 (broken line)
□ = 3.7M HNO_3	● = 3.8M HNO_3
△ = 4.3M HNO_3	○ = 4.7M HNO_3
▽ = 5.4M HNO_3	

FIGURE 3. Oxidation of Plutonium(III) in Adjusted Feed

Extraction coefficients for plutonium (III) and uranium(VI) have been documented.[7,8] The extraction coefficients are dependent on nitric acid and solute concentrations. For example, uranium(VI) extraction coefficients increase with nitric acid concentration to about 5M, then decrease; they increase with a decrease in uranium concentration and decrease with increased plutonium(IV) concentration.

TABLE 1

RESULTS OF LABORATORY SCALE PARTITIONING EXPERIMENTS

	Feed			Organic Phase		Aqueous Phase		Extraction Coefficients[a]	
HNO_3($\underline{M}$)	Pu (g/l)	U (g/l)	Pu/U	Pu (g/l)	U (g/l)	Pu (g/l)	U (g/l)	E^o_{Pu}	E^o_U
2.4	4.6	4.5	1	0.36	4.2	4.2	0.26	0.09	16
3.7	10.0	6.0	2	0.67	5.7	9.5	0.30	0.07	19
3.8	55.0	21.0	3	7.0	20.0	48.0	1.1	0.14	18
4.2	36.0	34.0	1	-	33.0	-	1.2	-	27
4.4	31.0	0.10	300	2.1	0.10	29.0	0.0028	0.07	36
4.7	8.5	9.0	1	0.54	8.7	8.0	0.40	0.07	28

---

a. E^o_M = Concentration of metal ion (M) in the organic phase divided by metal ion concentration in the aqueous at equilibrium.

Nitric acid concentrations between 4$\underline{M}$ and 5$\underline{M}$ were chosen for feed solutions. The nitric acid concentration was kept below 5$\underline{M}$ to avoid rapid plutonium(III) oxidation, but above 4$\underline{M}$ for maximum extraction.

Results of partitioning various plutonium (III)-uranium(VI) feed solutions in different nitric acid concentrations are shown in Table 1. The extraction coefficients for plutonium(III) and uranium (VI) are in fair agreement with reported values.[7,8] Results showed the partitioning of plutonium and uranium was effective for the separation scheme.

The next step in the process was to scrub the uranium-loaded organic free of residual plutonium with 0.35$\underline{M}$ HNO_3-0.1$\underline{M}$ $Fe(SO_3NH_2)_2$. This scrub solution was selected on the basis of improved scrubbing with lower nitric acid concentration (see Table 2). Subsequent experiments showed 0.1$\underline{M}$ $Fe(SO_3NH_2)_2$ was sufficient to maintain plutonium in the trivalent state; 0.35$\underline{M}$ HNO_3 was used since it was available in bulk. The desired Pu/U ratio (<0.005) in the organic was achieved by scrubbing with this solution. Because of the high plutonium and uranium concentrations (3 to 5 g/l) in the resultant aqueous solutions, the used scrub solution was concentrated by evaporation and used for feed makeup.

The final step in the solvent extraction process was the stripping of uranium from the organic phase. Water or dilute acid usually was used as the uranium stripping agent.[7] Since a more efficient stripping agent would decrease the contact time and volume of stripping solution, several stripping agents were compared.

TABLE 2

RESULTS OF PLUTONIUM-URANIUM LOADED, ORGANIC-SCRUBBING EXPERIMENTS

Scrub Solution	Loaded Organic No. 1 (Pu = 8.2 g/l) Pu/U (org)	 (U = 23 g/l) U/Pu (aq)
$5\underline{M}\ HNO_3-0.5\underline{M}\ Fe(SO_3NH_2)_2$	0.03[a]	0.08
	0.002[b]	0.6
$3\underline{M}\ HNO_3-0.5\underline{M}\ Fe(SO_3NH_2)_2$	0.02	0.07
	0.002	12
$1\underline{M}\ HNO_3-0.5\underline{M}\ Fe(SO_3NH_2)_2$	0.0008	0.13
	0.0005	29
$1\underline{M}\ HNO_3-0.5\underline{M}\ Fe(SO_3NH_2)_2-$	0.05	0.04
$0.5\underline{M}\ Al(NO_3)_3$	0.01	0.8

Scrub Solution	Loaded Organic No. 2 (Pu = 0.58 g/l) Pu/U (org)	 (U = 4.2 g/l) U/Pu (aq)
$5\underline{M}\ HNO_3-0.5\underline{M}\ Fe(SO_3NH_2)_2$	0.01	0.2
	0.0006	4
$3\underline{M}\ HNO_3-0.5\underline{M}\ Fe(SO_3NH_2)_2$	0.01	0.8
	0.0009	4
$1\underline{M}\ HNO_3-0.5\underline{M}\ Fe(SO_3NH_2)_2$	0.001	3
	0.0002	27
$1\underline{M}\ HNO_3-0.5\underline{M}\ Fe(SO_3NH_2)_2-$	0.04	1
$0.5\underline{M}\ Al(NO_3)_3$	0.01	3

---

a. Pu/U ratio after first contact with scrub solution.

b. Pu/U ratio after second contact with scrub solution.

TABLE 3

RESULTS OF TESTS WITH VARIOUS STRIPPING AGENTS

	36.6 g/l U Loaded Organic		6.86 g/l U Loaded Organic	
Stripping Agent	U (g/l) in Organic*	S_o^a**	U (g/l) in Organic*	S_o^a**
Water	10.1	0.72	1.80	2.8
1M Ammonium Sulfate	0.39	93	0.018	380
1M Sodium Sulfate	0.39	93	0.054	126
7M Aqueous Ammonia***	0.018	2033	0.0027	2540

---

* Concentration after one contact, aqueous to organic ratio of unity.

** S_o^a (stripping coefficient) is the concentration of uranium in the aqueous phase divided by the concentration in the organic phase at equilibrium.

*** Yellow precipitate formation.

Results of the stripping experiments, including the uranium concentrations of the organic phase (after contacting with various stripping agents) and the stripping coefficients, are shown in Table 3. Of the stripping agents tried, aqueous ammonia removed the most uranium as a precipitate; it also combined the precipitation and strip steps. Sulfate solutions were selected as the most practical stripping agents (vide infra). Figure 4 shows a plot of sodium sulfate concentration in the stripping solution versus uranium concentration (after stripping) in two organics. The data show that uranium removal increases with an increase in sulfate concentration. The slopes of both plots change at a sodium sulfate concentration of about 1.5M, which indicates a decrease in stripping efficiency. Based on slope change, reagent makeup problems, and increased sulfate contamination in the uranium oxide (U_3O_8) product with high sulfate concentrations, 1M $(NH_4)_2SO_4$ or 1M Na_2SO_4 was selected as the aqueous stripping agent.

An alternative stripping scheme was devised using gaseous or aqueous ammonia (14M) as the stripping agent. Use of ammonia as a precipitative stripping agent combined the uranium(VI) strip and precipitation steps, eliminated the sodium carbonate step to remove dibutyl phosphate, and reduced aqueous waste. The process consisted of bubbling ammonia gas through the uranium-loaded organic phase instead of stripping with

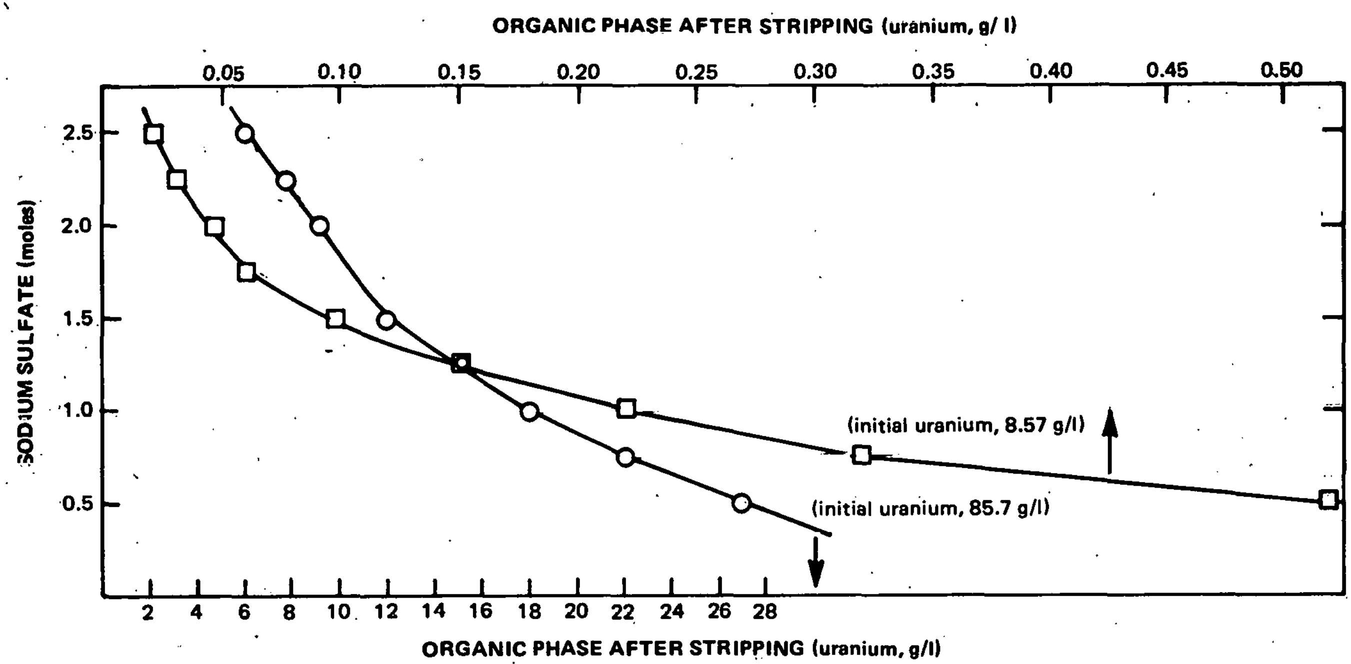


FIGURE 4. Stripping of Two Uranium-Loaded Organic Solutions With Various Concentrations of Sodium Sulfate

aqueous sulfate solution. Uranium stripping coefficients were a factor of 20 greater than sulfate stripping.

Ammonia stripping eliminated approximately 0.1 litre of aqueous waste per gram of uranium processed, and it was effective in removing dibutyl phosphate. The resultant uranium oxide contained less than one weight percent phosphorus, which was acceptable for subsequent processing. The filtration rate will have to be improved or centrifugation techniques will have to be used before the process can be implemented for processing plutonium-uranium residues.

Demonstration of Process

The extraction facility, which uses continuous, countercurrent extraction columns, was evaluated for the partition, scrub, and strip operations of the modified Purex process. Flow rates and organic-to-aqueous ratios were varied for the three operations to determine operating conditions for the column. Stirring was maximized (to the point just below column flooding) at each set of operating conditions.

During the column partition runs, organic was passed countercurrent to the feed at various flow rates. Stirring in mixing chambers varied from 1,000 rpm if the flow rates were slow to 1,200 if the flow rates were fast. After steady state conditions were achieved (greater than one total column-volume change of both phases) the exit aqueous and organic streams were sampled. The sampling was repeated 15 to 30 minutes later before the run was terminated.

TABLE 4

RESULTS OF PARTITION RUNS USING YORK-SCHEIBEL COLUMN

Feed: 13.4 g/l Pu and 19.5 g/l U in 4.4$\underline{M}$ HNO_3

Operating Conditions		Pu Product		
O/A[a]	Feed Flow Rate (l/hr)	U (g/l)	Pu (g/l)	U/Pu (ppm)
1	0.25	0.032	10.4	3080
1	1.0	0.016	12.9	1240
1	1.5	0.0042	11.9	353
0.5	0.5	0.023	9.1	2530
0.5	1.0	0.022	13.1	1680
0.5	1.5	0.026	13.4	1940
0.33	0.75	0.022	11.8	1860

---

a. O/A = Organic-to-aqueous ratio.

At termination, the amount of aqueous phase entrained in the organic phase was measured. First, the interface was positioned at a reference point near the bottom of the column. The flow of both phases and the stirring were then stopped. After five minutes of settling, the interface was lowered to the reference mark, and the discharge aqueous volume measured. The aqueous entrained in the organic (28 to 30 ml) did not change significantly between runs.

Results of varying the organic-to-aqueous ratio and flow rates during the partition runs are shown in Table 4. For the desired <5,000 ppm uranium in the plutonium product, all operating conditions tried were acceptable.

Scrub experiments were conducted in a manner similar to the partition runs except that loaded organic was passed countercurrent to the 0.35$\underline{M}$ HNO_3-0.1$\underline{M}$ $Fe(SO_3NH_2)_2$ scrub solution. The results, shown in Table 5, indicate all organic flow rates tried [organic-to-aqueous (O/A) = 1 and 2] yielded <5,000 ppm plutonium in the scrubbed organic.

Flow rates for the stripping operation were varied between 0.5 and 3 litres per hour where an O/A of unity was maintained; the results in Table 6 indicate effective stripping (<0.1 g/l uranium) up to 2.0 litres per hour of 1$\underline{M}$ Na_2SO_4.

In summary, it appears that York-Scheibel type columns are an acceptable contactor

TABLE 5

RESULTS OF SCRUB RUNS USING YORK-SCHEIBEL COLUMN

Starting Organic: 19.2 g/l U, 2.7 g/l Pu, Pu/U = 140,000 ppm

Operating Conditions		Analysis of Organic Phase[b]		
O/A[a]	Organic Flow Rate (l/hr)	U (g/l)	Pu (g/l)	Pu/U (ppm)
1	0.50	12.6	0.020	1600
1	0.75	14.2	0.019	1300
1	1.0	15.0	0.025	1700
1	1.5	12.1	0.056	4600
2	0.5	19.0	0.020	1000
2	1.0	19.4	0.045	2300
2	1.6	19.6	0.057	2900

---

a. O/A = Organic-to-aqueous ratio.

b. Analysis of organic phase prior to scrubbing.

TABLE 6

RESULTS OF STRIP RUNS USING YORK-SCHEIBEL COLUMN

Starting Organic: 14.1 g/l U
Organic-to-Aqueous Ratio = 1

	Analysis of Organic Phase
Flow Rate (l/hr)	U (g/l)
0.5	0.0088
1.0	0.042
1.5	0.078
2.0	0.12
3.0	0.48

TABLE 7

PROCESS DECONTAMINATION RESULTS
MONTHLY AVERAGES

Month	Plutonium Product U/Pu (ppm)	Uranium Product Pu/U (ppm)
March 1974	3000	1370
April 1974	1300	1050
May 1974	720	230
June 1974	920	2220
July 1974	680	3600

for a solvent-extraction facility using the modified Purex process. Studies have shown that the transfer unit efficiency[7] was about 55 percent; the partition, scrub, and strip operations could be performed within the operating limits of this size column; and that the column design and operation are advantageous for a remote-control, continuous operation.

Because of the limited amount of mixed actinide residues to be processed, and because of the involved operating requirements of the York-Scheibel columns, the 11 transfer unit columns were not used for processing the residues. The glove-box facility was stripped of equipment except for the 16-litre glass tanks that were used as storage tanks and air-sparge batch contactors.

Processing of the plutonium-uranium residues was initiated using a simple air-sparge batch contactor (Figure 1) and following the process flow sheet (Figure 2). Decontamination results of the plutonium and uranium products were at acceptable levels (<5,000 ppm U and <5,000 ppm Pu, respectively) as shown in Table 7. The amount of uranium in the plutonium product was dependent on the number of organic contacts made during the partitioning step; more contacts would decrease the amount of uranium. Similarly the plutonium content in the uranium product could be reduced simply by more plutonium scrubbing contacts. Unnecessary contacts, however, would decrease the efficiency of the process.

Material balance studies of both the uranium and the plutonium appeared to be good (>98 percent) and were within the error of analytical measurements. No significant losses of the organic were experienced.

SUMMARY

Solvent extraction, ion exchange, and precipitation techniques were considered for the separation of plutonium and uranium from mixed actinide oxide residues. A modified (one-cycle) Purex process was selected and developed for the plutonium-uranium separation. The governing criteria for the process were prompt recovery of the plutonium after removal of >5,000 ppm uranium and a uranium product to contain <5,000 ppm plutonium.

In the process, plutonium in the plutonium-uranium feed was reduced to the trivalent state with ferrous sulfamate to extract only the uranium(VI) into the organic phase (30 vol % TBP-dodecane). After uranium removal (<5,000 ppm uranium), plutonium in the aqueous phase was purified in an anion-exchange facility. The uranium-loaded organic phase was scrubbed with $0.35\underline{M}$ HNO_3-$Fe(SO_3NH_2)_2$ to remove residual plutonium. Uranium was removed from the organic by either aqueous sulfate stripping or direct precipitation of the uranium with ammonia. The uranium was effectively purified except for <5,000 ppm plutonium.

An experimental, solvent-extraction, pilot facility using York-Scheibel columns and a 16-litre batch contactor was used in the evaluation study. The batch contactor, using an air sparge for mixing, was selected to process the residues. The process has been used successfully to separate the plutonium-uranium oxide residues and allow prompt recovery of the plutonium.

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