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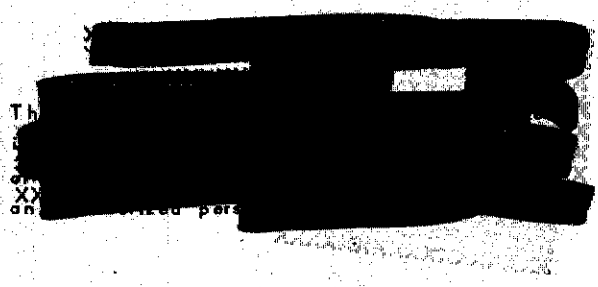
PURIFICATION OF NEPTUNIUM RECOVERED FROM PUREX WASTE

by

G. A. Burney and C. A. Prohaska
Separations Chemistry Division

September 1962



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DP-741, PURIFICATION OF NEPTUNIUM RECOVERED FROM PUREX WASTE

by G. A. Burney and C. A. Prohaska

The attached report describes the laboratory demonstration of the process that is presently operated in Building 221-F to purify neptunium recovered from the high activity waste concentrate of the Purex process. This ion exchange process is operated in the frame designated as Frame IIF.

Our primary concern during the development of the process was to achieve adequate separation from fission products and to demonstrate a satisfactory operation of the cation exchange cycle for the removal of thorium. As a result, the emphasis in the studies described in this report was on the behavior of fission products in tests with actual plant solutions and on the performance of the cation exchange cycle. After the successful laboratory demonstration of the process, it was expected that no unusual problems would be encountered in the plant process.

Contrary to expectations, serious difficulties developed in the early stages of operation in the Plant. The difficulties were completely unexpected because they were associated with the elution of neptunium from the anion resin. It appeared that neptunium was retained on the resin under conditions that should have given complete elution. Because this incomplete elution could not be duplicated in the laboratory with actual plant solutions, it was not possible to identify the cause of the apparent incomplete elution. Following

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careful evaluation of operations in the Plant and some changes in procedures, successful performance of the entire process was achieved. The cause of the original difficulty was not established.

Excellent separation from fission products has been achieved, and the neptunium product has been well within the specifications for gamma activity. The thorium content of the product has been well below the specified limit. The recovery of neptunium has been somewhat erratic, but in general has been above 95%. Any unusually high losses are recycled for recovery.

C. H. Ice

C. H. Ice, Research Manager
Separations Chemistry Division

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PURIFICATION OF NEPTUNIUM RECOVERED FROM PUREX WASTE

by

Glenn A. Burney and Charles A. Prohaska

September 1962

[redacted]

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ABSTRACT

An ion exchange process was demonstrated to be satisfactory for the purification of Np^{237} recovered from Purex process wastes at Savannah River Plant. Two cycles of anion exchange and one cycle of cation exchange gave the required separation from the major impurities - plutonium, thorium, and the fission products.

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PURIFICATION OF NEPTUNIUM RECOVERED FROM PUREX WASTE

INTRODUCTION

Np^{237} is produced as a byproduct when natural uranium is irradiated with neutrons to produce Pu^{239}. The neptunium is present at levels of 2 to 4 ppm in the irradiated uranium after normal reactor exposure. In the Purex solvent extraction process for the recovery and purification of uranium and plutonium, the neptunium is diverted primarily to the high activity aqueous waste stream from the first solvent extraction contactor. The recovery of neptunium from the concentrated waste, by a single cycle of anion exchange on an agitated bed of resin, is described in another report[1].

The product from the recovery step is a dilute solution of neptunium in nitric acid, free of gross amounts of fission products. A process was required to purify the neptunium further and to recover it in high yield. The major contaminants anticipated were Pu^{239}, Th^{232}, Th^{234}, Zr^{95}, Nb^{95}, Ru^{103}, and Ru^{106}. It was necessary that the process be operable in remote ion exchange equipment of the type that has been previously described.[2]

Sufficient data were already available on the anion exchange behavior of neptunium[3,4,5,6] to indicate that the required separation from plutonium and fission products could be obtained in two repetitive anion exchange cycles in which the anionic nitrate complex of Np(IV) was absorbed. In addition, information was available to indicate that the required separation from thorium could be obtained by cation exchange absorption of Th(IV) in the presence of the weakly absorbed NpO_2^+. This report describes the laboratory tests of a proposed purification process consisting of two fixed-bed anion exchange cycles followed by one fixed-bed cation cycle. Particular attention was given to evaluating the purity and the yield of neptunium attainable in this process.

SUMMARY

A process was demonstrated for the purification of neptunium recovered from the high activity waste concentrate of the Purex process at the Savannah River Plant. The major impurities to be removed in the process were Pu^{239}, Th^{232}, Th^{234}, Ru^{103}, Ru^{106}, Zr^{95}, and Nb^{95}. Plutonium and the fission products were separated from the neptunium by two cycles of anion exchange from a concentrated nitrate solution, and thorium was separated by a single cycle of cation exchange from a dilute nitrate solution.

In both anion exchange cycles, Np(IV), Pu(IV), and Th(IV) were absorbed on the resin as the anionic nitrate complexes. The fission products were washed from the resin with 8M HNO_3 - 0.005M KF, and plutonium was removed by washing with 6M HNO_3 - 0.05M N_2H_4 - 0.05M $Fe(NH_2SO_3)_2$. Finally, neptunium and thorium were eluted from the resin with dilute

nitric acid. In the cation exchange cycle neptunium was oxidized to weakly absorbable NpO_2^+. The NpO_2^+ that was absorbed along with the thorium was washed from the resin with 1M HNO_3. Thorium was eluted from the resin with sodium bisulfate when the column was loaded to the point of breakthrough.

After the three cycles of ion exchange, neptunium was separated from plutonium by a factor >200, from thorium by a factor >170, and from fission products by a factor $>1.5 \times 10^5$. The over-all recovery of neptunium was greater than 96%.

DISCUSSION

The concentrated high activity waste from the Purex process contains 10 to 20 mg/l of Np^{237}, 1 to 20 mg/l each of Pu^{239} and Th^{232}, and 100 to 1000 γ curies/l of fission product activity. Actual plant waste was processed in the laboratory to recover neptunium by a single cycle of anion exchange on an agitated bed of resin[1]. The neptunium solution from this operation was then used in the demonstration of the purification process. A typical composition of the solution produced from the single cycle of anion exchange is shown in Table I.

TABLE I

Composition of Feed to Purification Process

Constituent	Concentration
Nitric acid	2.2M
Neptunium	0.1-0.2 g/l
Plutonium	0.1-0.2 g/l
Thorium	<0.05 g/l
Fission products	1-10 γ curies/l

The specifications for the Np^{237} product from the purification process were:

Fission product activity	$<3 \times 10^{-4}$ γ curies/g Np
Plutonium	<1 wt %
Thorium	<1 wt %

From these specifications and the composition of the feed solution it was estimated that separation from plutonium by a factor of 100, from thorium by a factor of 50, and from fission products by a factor of $\sim 1.5 \times 10^5$ would be required in the three ion exchange cycles of the purification process. A further goal of the process was that neptunium should be recovered in yields greater than 95%.

Prior work[3,4,5,7,8] has shown that Np(IV), Pu(IV), and Th(IV) form anionic nitrate complexes that are strongly absorbed by anion resins from solutions 6 to 10M in total nitrate. Under these conditions the fission products are absorbed to only a very slight extent, and residual amounts may be washed from the resin with 8M HNO_3. Plutonium may be separated from neptunium and thorium by washing the resin with 6M HNO_3 containing ferrous sulfamate and hydrazine[6]. Neptunium and thorium are eluted readily from the anion resin with dilute nitric acid.

A cation exchange cycle was designed to separate neptunium from thorium. In this cycle neptunium is present as the NpO_2^+ ion and is absorbed much less strongly than Th(IV). Because the conditions for this separation were not well defined, the various steps in the cycle were investigated to establish suitable conditions for the process.

EXPERIMENTAL

The initial studies of the process were made with solutions containing only neptunium, plutonium, and thorium, to establish the conditions required for adequate recovery of neptunium and for adequate separation from plutonium and thorium. A solution prepared from actual plant waste was used in the final demonstration of the process. The ion exchange columns used in the demonstration were 13 inches high and 0.62 cm^2 in cross-sectional area. This height was approximately the same as that of the plant columns, and the area was about 1/1250 of the plant columns. "Dowex" 1-X3 and 1-X4, 40-60 mesh anion resins and "Dowex" 50W-X8 cation resin were used in the tests. The resins were washed with nitric acid to convert the anion resin to the nitrate form and the cation resin to the hydrogen form. This wash also removed iron from the cation resin.

Ferrous sulfamate was prepared by dissolving iron powder in a slight excess of sulfamic acid. The stock solution of hydrazine was either a 3M aqueous solution of hydrazine nitrate from the Fairmount Chemical Co., Inc. or "Hyzeen", a 30% aqueous solution of hydrazine from the Betz Laboratories, Inc.

DESCRIPTION OF ANION EXCHANGE CYCLES

The crude neptunium recovered from the concentrated waste of the Purex process was processed through three cycles of fixed-bed ion exchange for purification (Figure 1). Two cycles of anion exchange were required for removal of plutonium and fission products. These cycles differed only in the order of the wash steps. The feed was adjusted to 8M HNO_3 and ferrous sulfamate was added; Np(IV) and Pu(III) were produced almost instantaneously. The solution was then heated to 55°C for 30 minutes to oxidize the excess Fe(II) to Fe(III) and the Pu(III) to Pu(IV). The anionic nitrate complexes of Np(IV), Pu(IV), and Th(IV) formed in solution were absorbed on the anion resin, and the resin was washed with 10 to 20 bed volumes of 8M HNO_3 - 0.005M KF to remove the fission products. Plutonium was washed from the resin with 15 bed volumes of 6M HNO_3 - 0.05M N_2H_4 - 0.05M $Fe(NH_2SO_3)_2$; this wash removed 85 to 100%

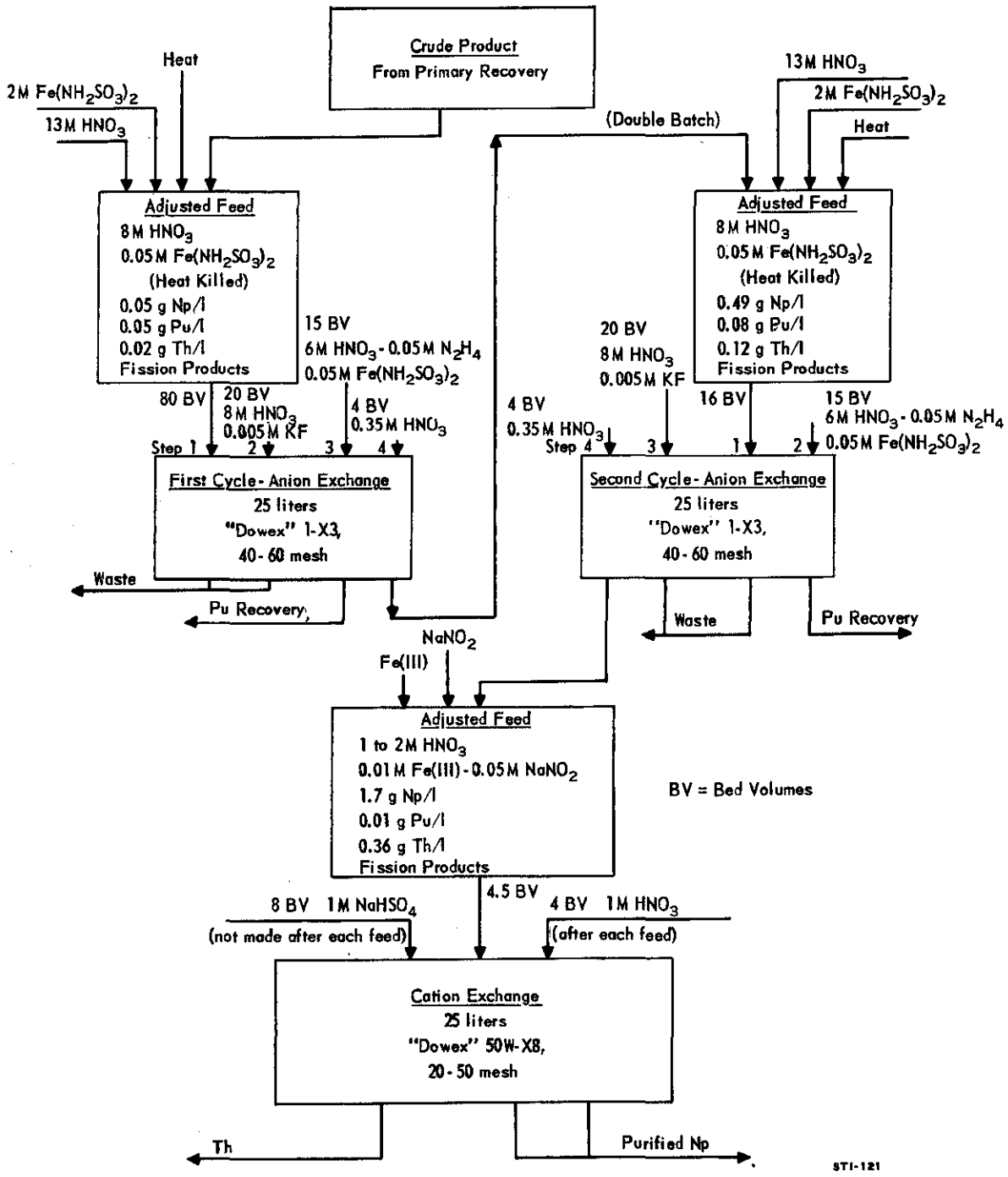

FIG. 1 FLOWSHEET

of the plutonium. In the second anion exchange cycle the loaded resin was washed first to remove plutonium and then to remove fission products. The wash steps were reversed even though the removal of fission products was somewhat less effective with this sequence, because the presence of hydrazine in the neptunium product solution from this cycle would interfere with the adjustment of the valence of neptunium in the following cation exchange cycle.

The process was designed so that plutonium that was separated from neptunium in the anion exchange cycles could be returned to the second cycle of the Purex process.

PERFORMANCE OF ANION EXCHANGE CYCLES

Laboratory tests with "Dowex" 1-X4, 40-60 mesh resin demonstrated the necessity for two cycles of anion exchange on fixed beds of resin. Two cycles were necessary to attain adequate separation of plutonium when the Np/Pu ratio was less than 3 or 4[6]; they were also required to ensure adequate removal of fission products. When "Dowex" 1-X3, 40-60 mesh resin was used, only one cycle was required to separate plutonium even with Np/Pu ratios as low as 0.2; however, two cycles were required to remove fission products.

The results given in Table II are typical of those obtained with feed solutions prepared from actual plant waste. These results show that both the recovery and purification of neptunium were quite adequate in the two anion exchange cycles.

TABLE II

Performance of Anion Exchange Cycles

Over-all factor for separation from Th: ~2
Over-all factor for separation from Pu: >100
Factors for separation from fission products
First cycle: >500
Second cycle: >100
Over-all: >5x10⁴
Over-all recovery of Np: >98%

Cycle Step	Volume, bed volumes	Flow, ml/(min)(cm²)	Composition HNO_3, M	$Fe(NH_2SO_3)_2$, M	KF, M	N_2H_4, M	Np, g/l	Pu, g/l	Th, g/l	Effluent Composition, % of Total Amount Fed to 1st Cycle Np	Pu	Th
1st Cycle of Anion Exchange												
Feed absorption	80	5	8	0.05	-	-	0.05	0.05	0.02	0.5	0.01	5
Fission product wash	20	5	8	-	0.005	-	-	-	-	0.2	0.01	30
Plutonium wash	15	2	6.0	0.05	-	0.05	-	-	-	0.05	>85	5
Elution	4	0.5	0.35	-	-	-	-	-	-	>99	<15	<60
2nd Cycle of Anion Exchange[a]												
Feed absorption	16	3	8	0.05	-	-	0.49	0.08	0.12	<0.1	0.01	1
Plutonium wash	15	2	6.0	0.05	-	0.05	-	-	-	<0.1	<15	3
Decontamination wash	20	5	8	-	0.005	-	-	-	-	<0.4	<1	~5
Elution	4	0.5	0.35	-	-	-	-	-	-	>98	<1	~50

(a) The products from two first cycle runs were combined for processing through the second cycle.

THORIUM SEPARATION BY CATION EXCHANGE CYCLE

The separation of thorium and neptunium and further purification of neptunium from fission products was accomplished in the cation exchange cycle with "Dowex" 50W-X8 resin. Neptunium in the 1 to 2M HNO_3 solution from the second anion exchange cycle was oxidized to Np(V). The NpO_2^+ ion was absorbed only weakly by the cation resin, while the Th(IV) was strongly absorbed. Fe(III) was added to the feed to increase the rate of oxidation and to complete conversion of Np(IV) to Np(V) when the solution was heated to 70°C. The effect of Fe(III) on the oxidation of Np(IV) is shown by the data in Table III.

TABLE III

Oxidation of Np(IV) to Np(V)

Composition of Solution	Temp, °C	Time, hr	Np(IV) Oxidized, %
2M HNO_3 - 1 g Np/l	23	24	<5[a]
2M HNO_3 - 1 g Np/l	70	0.5	10-50[a]
2M HNO_3 - 0.05M $NaNO_2$ - 1 g Np/l	23	0.5	<5[a]
2M HNO_3 - 0.05M $NaNO_2$ - 1 g Np/l	70	0.5	<25[a]
1M HNO_3 - 0.01M Fe(III) - 1 g Np/l	23	5	>98
1M HNO_3 - 0.01M Fe(III) - 1 g Np/l	70	<0.5	>98
2M HNO_3 - 0.01M Fe(III) - 1 g Np/l	23	24	70-80
2M HNO_3 - 0.01M Fe(III) - 1 g Np/l	70	<0.5	>98

(a) Observed oxidation possibly due to traces of Fe(III) or other cationic impurities.

Although sodium nitrite did not appreciably affect the rate of oxidation of Np(IV) to Np(V), it was added to the feed after cooling to stabilize Np(V) in the presence of the resin. In the absence of nitrite, approximately 5% of the Np(V) was reduced to Np(IV) and was retained on the resin[9] with the thorium. The addition of nitrite to the feed decreased the amount retained on the resin to 1 to 2%.

"Dowex" 50W-X8, 20-50 mesh resin was used to obtain the desired hydraulic characteristics. The use of 20-50 mesh resin limited the flow during the absorption step to 3 ml/(min)(cm^2) and the number of batches processed to 4 or 5 before the breakthrough of thorium occurred and elution was required (Figure 2). The volume of solution that could be processed before the breakthrough occurred decreased as the concentrations of thorium and nitric acid increased (Figures 3 and 4). The absorption flow was increased to 5 ml/(min)(cm^2) and the number of batches to 10 by using 50-100 mesh resin; however, hydraulic properties of the smaller resin particles were less favorable. The variation in particle size of resin did not alter the effectiveness of the separation of thorium from neptunium.

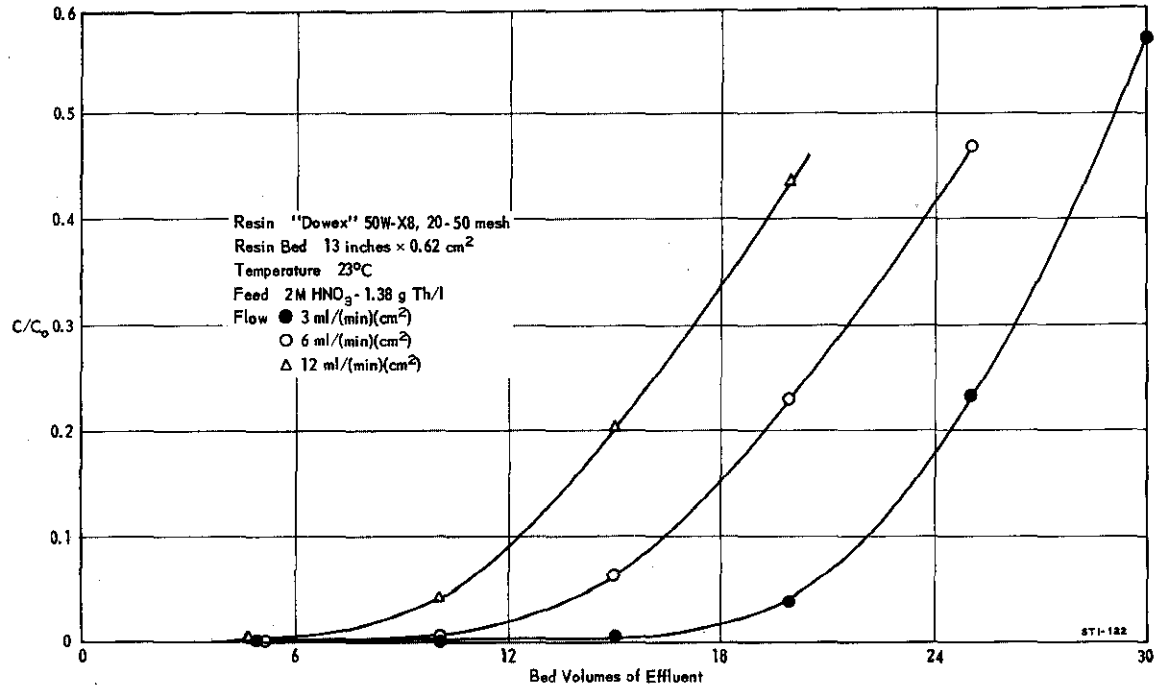

FIG. 2 EFFECT OF FLOW ON ABSORPTION OF THORIUM BY CATION RESIN

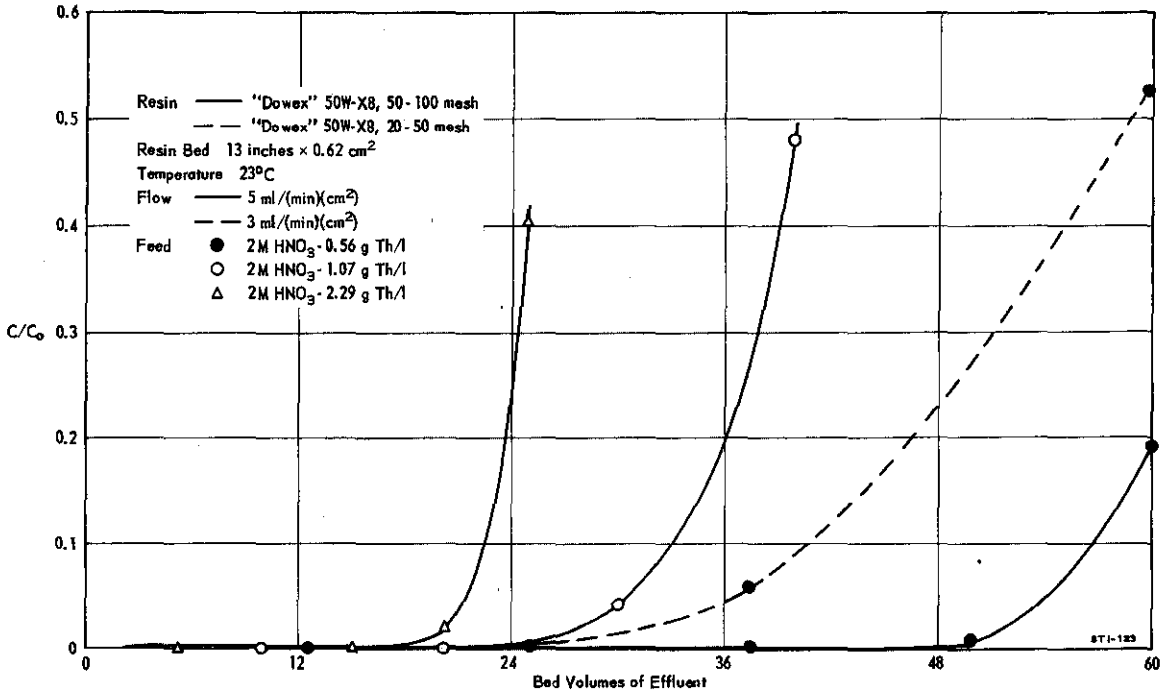

FIG. 3 EFFECT OF THORIUM CONCENTRATION ON ABSORPTION OF THORIUM BY CATION RESIN

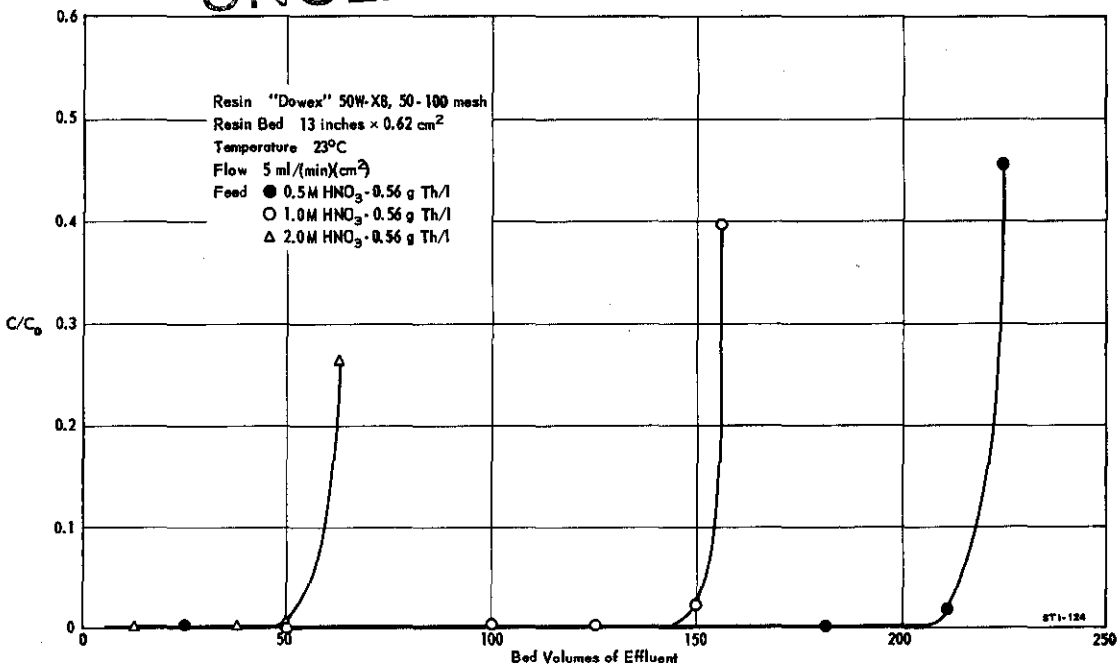

FIG. 4 EFFECT OF ACID CONCENTRATION ON ABSORPTION OF THORIUM BY CATION RESIN

At the completion of the absorption step approximately 25% of the Np(V) was uniformly distributed over the resin, and the remainder was in the effluent (Figure 5). Immediately after passage of the feed, the resin was washed with 4 to 5 bed volumes of 0.5 to 1M HNO_3 to elute the neptunium because when Np(V) remained in contact with the resin it was reduced to Np(IV). Following this wash, only 1 to 2% of the neptunium remained on the resin. If the resin was not washed immediately but was allowed to stand for 72 hours, 25 to 50% of the neptunium on the resin was reduced to Np(IV) and was not removed by the wash with 1M HNO_3.

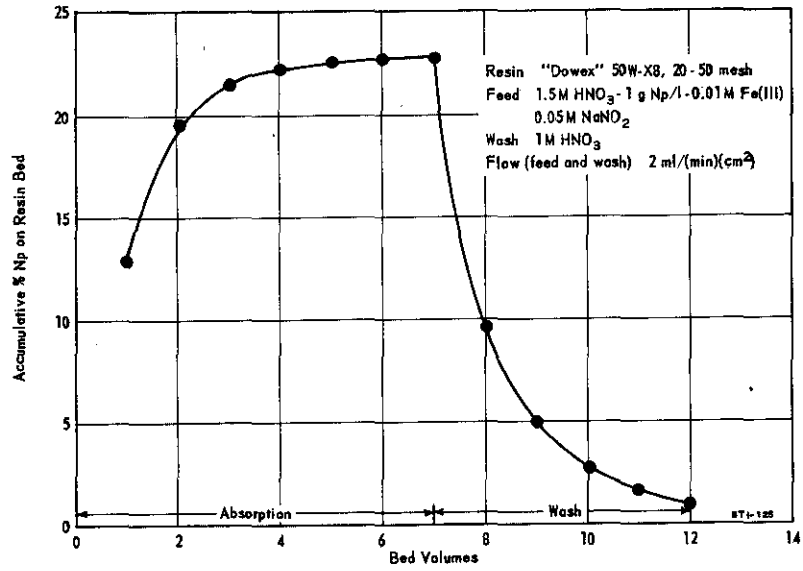

FIG. 5 SEPARATION OF THORIUM AND NEPTUNIUM BY CATION EXCHANGE

The satisfactory performance of the cation exchange cycle is demonstrated by the typical results given in Table IV. In addition to the satisfactory separation from thorium and the adequate recovery of neptunium, an appreciable separation from Zr^{95}-Nb^{95} was achieved.

TABLE IV

Performance of Cation Exchange Cycle

Factor for separation from fission products: 3 to 10

Cycle Step	Volume, bed volumes	Flow, ml/(min)(cm²)	Composition HNO_3, M	Fe(III), M	$NaNO_2$, M	Np, g/l	Pu, g/l	Th, g/l	$NaHSO_4$, M	Effluent Composition, % of Amount Fed to Cycle Np	Pu	Th
Feed absorption	4.5	2	1-2	0.01	0.05	1.7	0.01	0.36	-	75	10-75	<0.5
Neptunium wash	4-5	2	0.5-1	-	-	-	-	-	-	23-25	<1	<0.5
Thorium elution(a)	8	0.5	-	-	-	-	-	-	1	1-2	25-90	>99

(a) As required

Thorium was eluted satisfactorily from "Dowex" 50W-X8 resin with 1M $NaHSO_4$. About 60% more elutriant was required with 20-50 mesh resin than with 50-100 mesh (Figure 6). Elution with 4.5M HNO_3 was much less effective (Figure 7). Increasing the concentration of nitric acid to 6M or the temperature of elution to 55°C did not improve the efficiency.

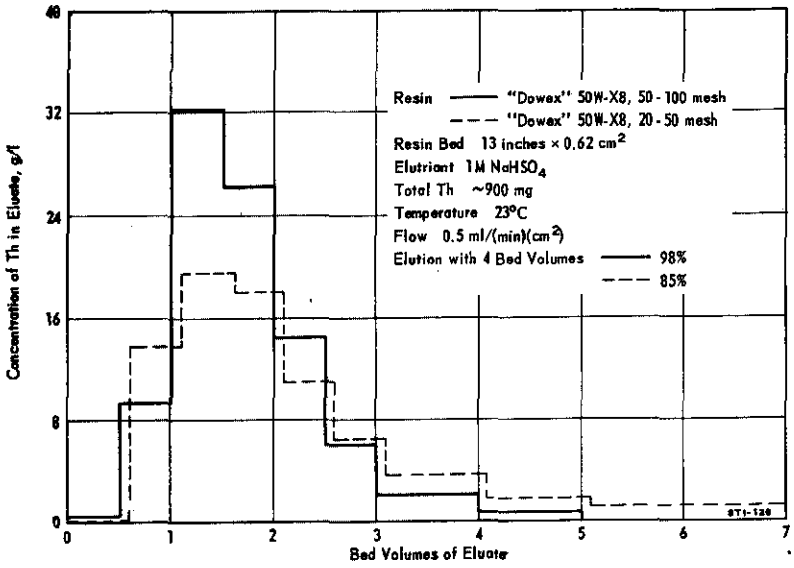

FIG. 6 ELUTION OF Th(IV) FROM CATION RESIN WITH SODIUM BISULFATE

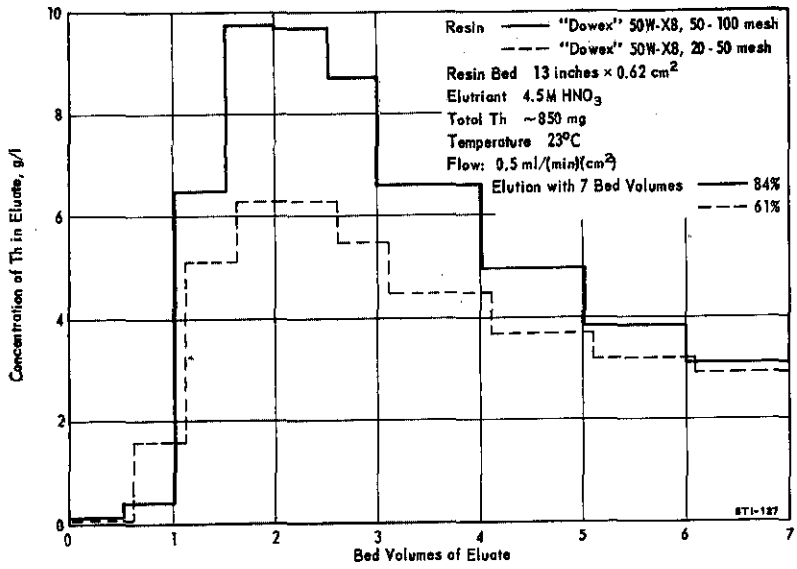

FIG. 7 ELUTION OF Th(IV) FROM CATION RESIN WITH NITRIC ACID

OVER-ALL PERFORMANCE OF PROCESS

The recovery of neptunium and the separations achieved over the three cycles of ion exchange were as follows:

Recovery of neptunium: >96%
Factor for separation from thorium: >170
Factor for separation from plutonium: >200
Factor for separation from fission products: $>1.5 \times 10^5$

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