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**CHEMISTRY-SEPARATION PROCESSES
FOR PLUTONIUM AND URANIUM**

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**CONCENTRATION OF PLUTONIUM BY CATION EXCHANGE
PART I - NEW ELUTRIANTS**

by

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December 1956

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CONCENTRATION OF PLUTONIUM BY CATION EXCHANGE PART I - NEW ELUTRIANTS

INTRODUCTION

Plutonium in the trivalent state can be conveniently concentrated by absorption on a bed of cation exchange resin followed by elution with a small volume of concentrated nitric acid. The nitric acid used as the elutriant is stabilized with sulfamic acid to prevent oxidation of the plutonium with the evolution of gas. This elutriant removes only 90 per cent of the plutonium from the bed at a concentration greater than 50 grams per liter, and on rare occasions the sulfamic acid fails to repress the reaction between the nitric acid and the trivalent plutonium.

The ideal elutriant would remove essentially all the plutonium from the bed and would not react with trivalent plutonium. A scouting study was made in an effort to find a more nearly ideal elutriant which would elute at least 99.5 per cent of the plutonium from the bed at an acceptable average concentration and would not oxidize the plutonium with evolution of gas.

The approach in this study was to select on the basis of known properties a number of candidate elutriants, to screen these by laboratory tests to find the most promising, and finally to scout the effects of operating variables on the performance of the candidate chosen.

SUMMARY

The concentration of plutonium by cation exchange was significantly improved by using a solution of ammonium lactate and sulfamate as the elutriant instead of nitric and sulfamic acids. The new elutriant permitted essentially complete elution of the plutonium and also avoided formation of gases in the resin bed during elution. At 45°C, 2.8M ammonium lactate plus 0.15M ammonium sulfamate eluted 99.3 per cent of the total plutonium at an average concentration of 43.8 grams per liter.

It was found necessary to elute hydrogen and hydroxylammonium ions from the resin beds before elution of plutonium. The hydroxylammonium ions decompose to form gases in the resin bed at the desired temperature for plutonium elution. The hydrogen ions exchange with ammonium ions of the elutriant and decrease the concentration of plutonium in the eluate.

DISCUSSION

GENERAL BACKGROUND

Trivalent plutonium in nitric acid solution can be conveniently concentrated from about 0.3 g/l to about 50 g/l by cation exchange on resin beds:

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Because this reaction is reversible, the plutonium can be absorbed from dilute (0.5M) nitric acid, and eluted by strong (5.7M) nitric acid. A typical process cycle for concentration and purification consists of four steps:

- (1) Feed Absorption - The plutonium (III) is absorbed from a dilute solution, which contains a reductant such as hydroxylamine sulfate (0.05M).
- (2) Uranium Elution - Uranium and some fission products are eluted with 0.25M sulfuric acid; this treatment does not elute plutonium (III), but can spread the absorbed plutonium over a larger volume of the resin.
- (3) Product Elution - The plutonium is eluted with strong acid (5.7M), which is prevented from oxidizing the plutonium (III) to (IV) by a stabilizer such as sulfamic acid (0.3M); most of the plutonium can be removed as a concentrated solution, but the concentration falls off rapidly as complete elution is approached.
- (4) Reconditioning - The column is prepared for next process cycle by flushing with dilute nitric acid (0.1M).

This procedure is satisfactory, but operation of the process would be easier if all of the absorbed plutonium were eluted during each cycle and if the retention of the plutonium in the trivalent state during elution did not depend on a stabilizer.

All the absorbed plutonium is not eluted. About 10 per cent of the total load must be left on the resin bed as a "heel" in order to maintain the concentration of plutonium in the composite eluate at 50 g/l or greater. The total amount of plutonium on the bed is difficult to control in cyclic operation of the process. An unrecognized cumulative buildup in the size of the heel can gradually increase the total amount of plutonium on the resin bed at the end of the absorption step, until the bed is overloaded and plutonium is lost in the effluent stream.

If the stabilizer fails to prevent oxidation during the elution, gases, which interfere with the process, are evolved in the resin bed. This gassing can be sufficiently vigorous to disrupt and channelize the bed. The gas is probably chiefly nitrogen. The generation of the gas is believed to result from the oxidation by nitric acid of either trivalent plutonium or hydroxylamine. The latter is added to the feed stream to maintain the plutonium in the trivalent state and is absorbed by the resin along with the plutonium. If the sulfamic acid, which is used in the elutriant to serve as a holding reductant for the plutonium and hydroxylamine during the elution step, were inadvertently omitted, gases would be generated rapidly. This is a potential hazard of the process.

The objective of the scouting study was to find an elutriant which would eliminate the cumulative heel problem by eluting at least 99.5 per cent of the plutonium at the required composite concentration of 50 g/l or greater and could not generate a gas under the proposed operating conditions. The work consisted of two parts: (1) a screening of various types of elutriant which had promising chemical properties, and (2) a preliminary attempt to develop a process based on the use of ammonium lactate, which was the most promising candidate found in the screening tests.

SCREENING TESTS

Background

Any solution containing cations that will exchange easily for absorbed plutonium is potentially usable as an elutriant. Many such cations can, however, be rejected on the basis of what is known of their chemical properties and behavior in ion exchange processes. The following elutriants were selected as being worthy of screening tests:

- (1) 7M nitric acid stabilized with sulfamic acid
- (2) mixtures of nitric and sulfuric acid stabilized with sulfamic acid
- (3) 12M nitric acid, which elutes Pu(IV) as an anionic complex
- (4) ammonium acetate and ammonium lactate

An elutriant more concentrated in nitric acid than 5.7M should elute the plutonium more completely and at a higher concentration because a high concentration of the exchangeable ion in the elutriant usually favors the desorption of the absorbed ions. Unfortunately, an increase in the concentration of nitric acid also increases the oxidizing power, and consequently the gassing tendency, of the elutriant. A higher concentration of the sulfamic acid would counteract the increase in oxidizing power, but the solubility of sulfamic acid in nitric acid is limited (0.3M in 7M nitric acid).

The use of mixtures of nitric and sulfuric acid could provide an elutriant with a high concentration of exchangeable cations that would, however, have less oxidizing power than the equivalent concentration of nitric acid alone. Sulfuric acid cannot be used alone because the solubility of Pu(III) in sulfate solutions is low⁽³⁾.

If the plutonium were absorbed on the resin as Pu(IV) instead of as Pu(III), 12M nitric acid would act as a complex-forming elutriant. When the concentration of nitric acid is greater than 8M an appreciable fraction of the Pu(IV) in a solution is converted to an anionic complex⁽⁴⁾⁽⁵⁾, probably $\text{Pu}(\text{NO}_3)_6^-$. In 12M nitric acid the plutonium is almost completely converted to this complex. Since Pu(IV) is not oxidized at a significant rate by nitric acid, no appreciable gas formation can occur if other oxidizable ions, such as hydroxylamine, are absent.

The anions in solutions of ammonium acetate or ammonium lactate can form complexes with the heavy metals, thus removing the metal ions, as such, from solution. This decrease in the concentration of the metal ions shifts the exchange equilibrium in favor of desorption and permits elution of the metal as concentrated solutions of the complexes. Ion exchange processes for concentrating uranium, using elution with mixtures of ammonium acetate and acetic acid, have worked very well⁽¹⁾⁽⁶⁾. This system was also used at KAPL to elute Pu(III)⁽⁷⁾. Good results were obtained, but an inert atmosphere was necessary to prevent oxidation of the plutonium and the subsequent precipitation of double acetates of ammonium and Pu(IV). Lactate elutriants have been used to separate actinide elements produced in cyclotron targets.⁽⁸⁾⁽⁹⁾

Experimental Method

The screening tests were made in glass columns 8 mm in diameter, each containing 10 ml of resin. The resin used was 12 per cent cross-linked "Dowex" 50, 50 to 100 mesh, with an exchange capacity of 2.5 milliequivalents per milliliter, measured in water⁽²⁾. For the test runs at high temperatures the columns were heated by an external heating tape. Approximately 0.5 gram of plutonium was used for each run, and the composition of the feed solution was 0.6 gram of plutonium per liter, 0.4M nitric acid, and 0.05M hydroxylamine sulfate. The hydroxylamine sulfate was not used in the runs with Pu(IV). An attempt was made to use reasonable rates of flow in each step, but the control of the rates was only approximate. The runs made in this small column are useful for screening only; the data obtained are not valid for engineering design.

To serve as a basis for comparing the new elutriants, a run was made with a standard elutrient, 5.7M nitric acid plus 0.3M sulfamic acid. In the "base" run 75 per cent of the total plutonium was eluted at an average concentration of 23.0 g/l.

Nitric Acid

An unsuccessful attempt was made to elute the plutonium more completely at a high concentration by increasing the concentration of nitric acid. The elutrient consisted of 7M nitric acid stabilized with 0.3M sulfamic acid used at 25°C. As soon as this elutrient entered the bed, a large amount of gas formed and disrupted the bed. The gas pockets prevented the flow of solution through the column. Higher concentrations of sulfamic acid, that might prevent the formation of gas, cannot be used because the solubility of sulfamic acid in 7M nitric acid is only slightly more than 0.3M.

Mixtures of Nitric and Sulfuric Acids

The substitution of sulfuric acid for part of the nitric acid in an effort to reduce the oxidizing (gas generating) potential of the elutrient without decreasing its ability to elute plutonium was not promising. The data are summarized in Table I. For Run 1, the elutrient was 5.0M nitric acid, 1.6M sulfuric acid stabilized with 0.3M sulfamic acid. With this elutrient there was excessive formation of gas, and flow of liquid through the column was blocked. For Run 2 the concentrations in the elutrient were decreased to 4M nitric acid, 0.9M sulfuric acid, stabilized with 0.3M sulfamic acid. The concentration of exchangeable hydrogen ions in this elutrient was approximately equal to that of the present standard elutrient. The product cut contained 75 per cent of the plutonium at an average concentration of 21.7 g/l. There appeared to be no advantage in the use of mixtures of nitric and sulfuric acids.

Anionic Complex of Pu(IV) with Nitric Acid

Absorption of the plutonium as Pu(IV) and subsequent elution as the anionic complex was not promising. The elutrient consisted of 12M nitric acid used at 25°C. Throughout the elution the eluate was pale green, which is the color of the anionic complex, but the intensity of the color indicated that the concentration of plutonium was low. The light green color persisted for an appreciable volume of eluate, i.e., the elution "tailed" excessively. The elutrient was left in the column overnight, and a much darker green eluate was obtained in the morning. These results indicate that at practical rates of flow, acceptable concentrations of

plutonium cannot be obtained, and that complete elution would be difficult. Better results might be obtained at higher temperatures, or with resin of lower cross-linkage.

The Pu(IV) for the feed was prepared by oxidizing the stock solution with 12M nitric acid and then diluting to feed composition.

Organic Elutriants

A scouting run with ammonium acetate was promising; 98 per cent of the plutonium was removed at an average concentration of 21.9 g/l, compared to 75 per cent at 23.0 g/l for the "base" run. To avoid the necessity for an inert atmosphere, sulfamic acid was added to the elutriant to prevent precipitation of plutonium. This run was made at 60°C with an elutriant containing 2.9M ammonium acetate, 0.40M acetic acid, and 0.30M ammonium sulfamate. The pH of the elutriant was 5.0 at 25°C. The results of this run indicated that complete elution of the plutonium with ammonium acetate was likely and that, in this range of plutonium concentrations, an inert atmosphere may not be necessary. The sulfamate did not prevent oxidation of the plutonium, but only inhibited the formation of a precipitate. Although the fresh eluate was brown, which usually indicates the presence of Pu(IV), precipitation did not occur until several hours after the elution was completed. Since there was a possibility that, with more concentrated solutions of plutonium, double acetates of ammonium and Pu(IV) might be precipitated by air oxidation, further effort, at this time, was limited to the ammonium lactate system.

The runs with ammonium lactate were very promising, and a study was made to establish favorable conditions for better evaluation of the process in the larger resin column. The data for these runs are summarized in Table II. The best results were obtained in Run 4; 98 per cent of the plutonium was removed at an average concentration of 22.6 g/l, compared to 75 per cent at 23.0 g/l in the "base" run. This run was made at 65°C with an elutriant containing 3.2M ammonium lactate and 0.30M ammonium sulfamate. The pH of the elutriant was 5.0 at 25°C.

The data in Table II reveal several important points about the use of ammonium lactate as an elutriant. Runs 1 and 2 were made at 25°C, and both the quantities of plutonium eluted and the average concentrations were appreciably below the values obtained in the "base" run. In Run 2, the pH value of the elutriant was increased to 4.8 and the elution results were much better than in Run 1. Runs 3 to 5 were made at 65°C, and the elution results were greatly improved compared to the runs at 25°C. Comparing Runs 3 and 4, for which the pH of the elutriant was increased from 4.8 to 5.0 by neutralizing all the lactic acid, the per cent of the total plutonium eluted increased from 90 to 98 per cent while the average concentration of plutonium remained at 22.6 g/l. For Run 5, the total ionic strength of the elutriant was increased to 6.0M, and only 75 per cent of the plutonium was eluted at 17.7 g/l. These results and similar results reported from KAPL for ammonium acetate⁽⁷⁾ indicate that there is an optimum range of elutriant composition for each elution temperature. This is probably the result of a balance between the advantages of having a higher concentration of exchangeable ions and the disadvantages of greater resin shrinkage and increased solution viscosity with the more concentrated elutriants.

Conclusions

The most promising results in the screening tests were obtained with the ammonium lactate - ammonium sulfamate elutriant, and therefore, the

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second phase of this study was made using this elutriant. Additional significant information developed in the screening tests was that the average concentration of plutonium in the product is greatly increased if the elution is made at an elevated temperature and the optimum composition of the elutriant is a function of the elution temperature.

EVALUATION OF LACTATE ELUTRIANT

A short series of runs was made with the ammonium lactate - ammonium sulfamate elutriant on a column of larger diameter to obtain data that could be used for process design. Since in this series several variables were changed simultaneously in order to arrive rapidly at an acceptable flowsheet, the results obtained are merely indicative of what could be accomplished with a more thorough study.

Background

The chemistry of the process must be clarified to permit evaluation of the experimental data. The following discussion is based on this study and on the literature.

For the best operation of the process, the hydrogen and hydroxylammonium ions absorbed from the feed must be removed from the column before the plutonium elution step. These ions must be removed without spreading the plutonium over a larger volume of resin because the concentration of plutonium in the eluate depends on the maximum concentration in the resin.

The hydrogen ions must be removed to avoid a side reaction which decreases the concentration of plutonium in the eluate. The need for removing the absorbed hydrogen ions from the resin bed before the plutonium is eluted was first reported in KAPL-1153 for the ammonium acetate elutriants, and in DP-98 for the elution of uranium. If the hydrogen ions are not removed, ammonium ions from the elutriant are exchanged for the absorbed hydrogen ions, and these hydrogen ions react to produce un-ionized lactic or acetic acid. This deactivation of the elutriant occurs during the initial stage of the elution when, normally, the highest concentrations would be attained, and consequently, the average concentration of the eluate is significantly decreased.

The hydroxylammonium ions must be removed to prevent the formation of gas in the resin bed. When a solution of hydroxylamine sulfate and ammonium lactate is heated, rapid evolution of gas occurs.

Experimental Method

In these runs the feed was absorbed downflow, and a simulated uranium elution was carried out, also downflow. The latter step was included, although the feed contained no uranium, to simulate operation of the process in a complete cycle. The plutonium was eluted upflow.

The runs were made in a glass column containing a bed of "Dowex" 50-12X, 50 to 100 mesh. The exchange capacity of the resin was 2.3 milliequivalents per milliliter, measured in water.⁽²⁾ The bed was 19 mm in diameter and 356 mm in length (volume, 104 ml). For high-temperature runs the bed was heated by a heating tape wrapped around the body of the column. The temperature of the bed was a function of the voltage applied to the tape, and could be maintained within $\pm 5^{\circ}\text{C}$. The conditions and the data for all the runs are summarized in Tables III and IV.

Summary of Results

In order to arrive at an acceptable cycle as rapidly as possible the conditions for the uranium and plutonium elutions were varied simultaneously. The data thus obtained do not permit a full analysis of the effects of all variables. The optimum conditions were, however, fairly well defined by the concentration profiles obtained, by the observed tendencies to gas during plutonium elution and by visual observation of the extent of plutonium band spread during uranium elution.

The results of Run 3F were the best attained and approached the objective of the project, i.e., elution of 99.5 per cent of the plutonium at 50 g/l or higher. In this run 99.3 per cent was eluted at 43.8 g/l. These results gave promise that with further work more nearly optimum conditions would be developed and the goal attained. The uranium elutriant was essentially free of both hydrogen and hydroxylammonium ions and consisted of 0.1M ammonium sulfate and 0.05M sulfamic acid. The plutonium elution was carried out at 45°C with 2.8M ammonium lactate and 0.15M ammonium sulfamate at a pH of 4.6. There was no danger of gas formation in the bed. The hydroxylamine absorbed from the feed was removed from the column during the uranium elution, and the elutriant cannot be oxidized under run conditions.

Elution of Hydrogen and Hydroxylammonium Ions

To attain best operation of the process, hydrogen and hydroxylammonium ions must be eluted from the bed before the plutonium elution step. The series of runs summarized in Tables III and IV show that 0.10M ammonium sulfate stabilized with 0.05M sulfamic acid is a reasonably satisfactory elutriant for hydrogen and hydroxylammonium ions as well as uranium ions. In Run 1F, 1285 ml of 0.25M sulfuric acid, the usual elutriant, did not measurably spread the plutonium band. In Run 2F, 600 ml of 0.15M ammonium sulfate spread the plutonium band 56 per cent of the original length. In Run 3F, the ammonium sulfate concentration was decreased to 0.10M, and 1000 ml of the elutriant spread the plutonium band only 18 per cent. Data obtained in other studies indicate that the elutriant used in Run 3F would remove most of the absorbed uranium.

The decrease in plutonium concentrations of the product cuts when hydrogen ions are not desorbed during the uranium elution step is shown by the data for Runs 1F and 3F, in Table IV. In Run 1F, no attempt was made to remove hydrogen ions before the plutonium elution; but, in Run 3F, the hydrogen ions were essentially completely replaced with ammonium ions. In the latter run, the concentrations of plutonium were substantially higher in the early stages of the elution even though the processing temperature was only 45°C instead of 65°C. Part of the increase in concentration was due, however, to the slower elutriant flow rate, 0.2 instead of 0.3 ml/(min-cm²).

The need for eluting the hydroxylamine before heating the resin and the ability of the proposed uranium elutriant to do this was also demonstrated in Runs 1F and 3F. In Run 1F, in which no attempt was made to elute the hydroxylamine, an appreciable volume of gas accumulated in the resin bed during the plutonium elution step although no nitric acid or other oxidizing agent was present. This gas was probably the decomposition product of the hydroxylamine which was left on the resin bed. In Run 3F, the hydroxylamine in the uranium elutriant was replaced with sulfamic acid. When the plutonium was eluted, only a small amount of gas accumulated in the resin bed. This gas was probably dissolved air which was evolved from the elutriant at the higher temperature.

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Conclusions

With further development, the lactate - sulfamate elution process could probably attain elution of at least 99.5 per cent of the plutonium at a concentration of 50 g/l. Significant information developed during this scouting phase of the program indicated that (1) to prevent formation of large quantities of gas in the resin bed all hydroxylammonium ions must be eluted from the column before heat is applied, and that (2) all of the resin not occupied by plutonium ions must be converted to the ammonium form prior to elution of the plutonium, since any hydrogen ions that remain on the bed deactivate the elutriant and thereby decrease the concentration of plutonium in the composite eluate.

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TABLE I
SCREENING TESTS

ELUTION WITH MIXTURES OF NITRIC AND SULFURIC ACIDS

Resin: "Dowex" 50-12X, 50-100 mesh

Elution Temperature: 25°C

All solutions were 0.3M in $\text{NH}_2\text{SO}_3\text{H}$

Run No.	Elutriant Composition		Plutonium in Product Cut	
	HNO_3 M	H_2SO_4 M	% of Total	Avg., g/l
"Base"*	5.7	-	75	23.0
1	5.0	1.6	(Vigorous gassing)	
2	4.0	0.9	75	21.7

* Present, standard elutriant.

TABLE II

SCREENING TESTS - ELUTION WITH AMMONIUM LACTATE

Resin: "Dowex" 50-12X, 50-100 mesh

All solutions were 0.30M in $\text{NH}_4\text{SO}_3\text{NH}_2$

Run No.	Elutriant Composition				Elution Temp., °C	Total Pu, g	Plutonium in Product Cut	
	NH_4 Lac,* M	H Lac,** M	Total M	pH (25°C)			% of Total	Avg., g/l
1	2.3	1.22	3.8	2.8	25	0.35	23	8.7
2	3.1	0.20	3.6	4.8	25	0.30	57	14.7
3	3.1	0.20	3.6	4.8	65	0.35	90	22.6
4	3.2	-	3.5	5.0	65	0.28	98	22.6
5	5.7	-	6.0	5.0	65	0.32	75	17.7

* Ammonium lactate

** Lactic acid

TABLE III

EVALUATION OF LACTATE ELUTRIANTS - RUN CONDITIONS

Feed Absorption

Feed Composition: 0.3 g Pu/l, 0.35M HNO₃, 0.05M (NH₂OH) · $\frac{1}{2}$ H₂SO₄

Flow rate: 15 ml/(min-cm²)

Temperature: 25°C

Uranium Elution

Flow rate: 2 ml/(min-cm²)

Temperature: 25°C

Elutriant Composition:	<u>Run 1F</u>	<u>Run 2F</u>	<u>Run 3F</u>
H ₂ SO ₄ , M	0.25	-	-
(NH ₄) ₂ SO ₄ , M	-	0.15	0.10
NH ₂ OH · $\frac{1}{2}$ H ₂ SO ₄ , M	0.05	0.05	-
NH ₂ SO ₃ H, M	-	-	0.05
Volume of elutriant, ml	1285	600	1000
Spread of plutonium band, %	~0	56	18

Plutonium Elution

Elutriant Composition: 2.8M NH₄Lac*, 0.15M NH₄SO₂NH₂, pH 4.6 (25°C)

	<u>Run 1F</u>	<u>Run 2F</u>	<u>Run 3F</u>
Flow rate, ml/(min-cm ²)	0.3	0.2	0.2
Temperature, °C	65	25	45
Total Pu on column, g	5.8	4.8	5.3

* Ammonium lactate

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TABLE IV

EVALUATION OF LACTATE ELUTRIANTS - PRODUCT CUT DATA

(For elution conditions see Table III)

<u>Fraction Eluted, % of Total Pu</u>	<u>Average* Concentration of Plutonium in Product Cut, g/l</u>		
	<u>Run 1F</u>	<u>Run 2F</u>	<u>Run 3F</u>
20	65.3	81.7	101.0
30	79.4	82.8	104.8
40	85.6	79.0	104.9
50	88.2	72.0	101.4
60	88.1	64.2	96.3
70	85.8	54.5	90.0
80	80.4	43.6	82.9
90	68.6	30.9	72.9
95	58.0	-	67.2
96	55.4	-	65.8
97	51.6	-	64.2
98	47.2	-	61.0
99	-	-	51.5
99.3	-	-	43.8

* Cumulative average for total eluate

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