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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA ○ LOS ALAMOS NEW MEXICO

AQUEOUS DECONTAMINATION OF PLUTONIUM FROM FISSION PRODUCT ELEMENTS

(Project status report as of March 31, 1957)

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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA LOS ALAMOS NEW MEXICO

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By

R. S. Winchester

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ABSTRACT

A survey of plutonium recovery and purification methods as applied to plutonium-rich reactor fuels has been made. These methods consist of precipitation of plutonium peroxide, oxalate and fluoride; sorption by anion exchange resins; and solvent extraction with tri-*n*-butyl phosphate and amines.

The precipitation methods gave poor decontamination from typical fission products. Decontamination by anion exchange was fair. Of the solvent extractants tested,(1,2) secondary amines appeared to give satisfactory decontamination.

Plutonium containing typical fission products (zirconium, molybdenum, ruthenium and cerium) has been recovered and purified from pyrometallurgical and analytical wastes.

ACKNOWLEDGMENTS

All chemical analyses were done by members of Group CMB-1 under the direction of C. F. Metz. The advice of W. J. Maraman and J. A. Leary on all phases of the project is also gratefully acknowledged.

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INTRODUCTION

Pyrometallurgical studies on the separation of plutonium from typical fission product elements have resulted in the production of residue materials.

In order to develop a process for recovering and purifying plutonium from these residues, a survey of the following methods was conducted.

1. Precipitation of plutonium as the peroxide, oxalate and fluoride.
2. Selective sorption of plutonium on anion exchange resins.
3. Solvent extraction with tri-n-butyl phosphate and with amines.

Of the methods tested, extraction of plutonium from 8 M nitric acid solution using a secondary aliphatic amine solvent appears to be the most promising.

SEPARATION OF PLUTONIUM FROM FISSION PRODUCT ELEMENTS BY PRECIPITATION METHODS

Materials

A. Stock Solution

An alloy ingot containing cobalt, cerium, molybdenum, ruthenium, zirconium and plutonium was dissolved in boiling 16 M nitric acid-0.1 M hydrofluoric acid. Undissolved molybdenum and ruthenium were separated by filtration and treated with 6 M sodium hydroxide to dissolve the molybdenum. This was followed by the addition of sodium hypochlorite (6 per cent available free chlorine) to dissolve the ruthenium. These solutions were combined with the original filtrate, the volume adjusted to give a plutonium concentration of 60.58 g./l. and the solution sampled for chemical analysis. This stock solution was used throughout this work and was designated as stock solution A. It had the following composition:

Element	Concentration (g./l.)
Fe	1.26
Co	1.86
Zr	0.34
Mo	0.17
Ru	0.98
Ce	0.36
Pu	60.58

B. Reagents and Equipment

Standard reagent grade chemicals and conventional laboratory equipment were used in this work.

Procedure

A. Plutonium Peroxide Precipitation

Ten milliliters of stock solution A were treated with sodium hydroxide to give a final acidity of 2 M. Sufficient sodium sulfate was added to give a solution 0.15 M in sulfate, and hydroxylamine nitrate was added to give 0.005 M solution. The plutonium peroxide was precipitated by the dropwise addition of 30 per cent hydrogen peroxide and the slurry was stirred for 30 min. at room temperature. The precipitate was removed by filtration and washed on the filter with three 20 ml. portions of 15 per cent hydrogen peroxide. The washes and filtrate were combined and sampled. The precipitate was dissolved with concentrated nitric acid and sampled.

B. Plutonium(III) Oxalate Precipitation

Twenty milliliters of stock solution A were adjusted to 2.5 M acidity with sodium hydroxide and the plutonium reduced to the trivalent state with hydroxylamine nitrate. Ten grams of oxalic acid were added and the plutonium oxalate slurry was stirred 30 min. at room temperature. The precipitate was removed by filtration and washed with three 10 ml. volumes of water. The filtrate and washes were combined.

The precipitate was dissolved by refluxing with concentrated nitric acid, then cooled and sampled.

C. Plutonium(IV) Oxalate Precipitation

Twenty milliliters of stock solution A were adjusted to 0.1 M acidity with sodium hydroxide, and hydroxylamine nitrate was added. Sufficient sodium nitrite was added to oxidize the plutonium(III) to the plutonium(IV) valence state. The acidity of the solution was adjusted to 3 M with nitric acid, and 25 g. of crystalline oxalic acid were added. The slurry was stirred for 30 min. at room temperature and filtered. The precipitate was washed with three 10 ml. volumes of water. The filtrate and washes were combined and sampled. The precipitate was dissolved by refluxing with concentrated nitric acid, then cooled and sampled.

D. Plutonium(III) Fluoride Precipitation

Twenty milliliters of stock solution A were adjusted to 0.1 M acidity with sodium hydroxide and sufficient hydroxylamine nitrate added to give a 0.05 M solution of this reagent. Concentrated nitric acid was added to give an acidity of 3 M. To this solution 0.05 mole of aqueous 50 per cent hydrofluoric acid was added. The slurry was agitated for 30 min., filtered and the precipitate washed with three 10 ml. portions of water. The wash and filtrate were combined and sampled. The precipitate was dissolved by boiling with concentrated nitric acid, and this solution was then cooled and sampled.

Results and Discussion

The precipitation studies involving the treatment of the fissium alloy solution with peroxide, oxalic and fluoride ions gave decontamination factors shown in Table 1. The decontamination factor is expressed as total weight of element per gram of plutonium in the beginning material divided by the total weight of element per gram of plutonium in the product material.

Table 1

Decontamination Factors of Plutonium by Precipitation Methods

Element	Plutonium Peroxide	Plutonium(III) Oxalate	Plutonium(IV) Oxalate	Plutonium(III) Fluoride
Fe	50	33	10	1.4
Co	30	47	>95	8.6
Zr	1	3.5	>44	1.1
Mo	>140	>13	>15	1.1
Ru	>14	>38	33	36
Ce	6	1	1	1.1

A. Plutonium Peroxide Precipitation

This precipitation method gave good to poor decontamination of plutonium. Zirconium and cerium were not adequately separated. Acceptable decontamination from iron, cobalt and molybdenum is apparent.

B. Plutonium(III) Oxalate Precipitation

The precipitation of plutonium(III) oxalate gave poor decontamination of the plutonium from the elements iron, zirconium and cerium. Fair decontamination was achieved from the elements cobalt, molybdenum and ruthenium.

C. Plutonium(IV) Oxalate Precipitation

The plutonium(IV) oxalate precipitation method gave poor decontamination of the plutonium from iron and cerium. Fair decontamination was achieved from cobalt, zirconium and ruthenium.

D. Plutonium(III) Fluoride Precipitation

This method gave poor decontamination of the plutonium from cerium, iron, cobalt, zirconium and molybdenum. The decontamination from ruthenium was fair.

From these data it is evident that the precipitation methods for decontamination of plutonium from these elements are not acceptable. Such processes could be used as methods for concentrating purified plutonium from some other purification system or they could be used to effect "head end" treatment.

SORPTION ON ANION RESINS

Materials

A. Stock Solution

The plutonium stock solution A, described in the first section, was also used for these studies.

B. Reagents

Standard C.P. or A.R. grade reagents were used.

C. Anion Exchange Resins

1. The Dow Chemical Company Dowex A-1 anion exchange resin (250-500 mesh U.S. sieve size) was received as the chloride form and converted to the nitrate form by treating the resin with sodium nitrate-nitric acid solution. The resin was assumed to be chloride-free after the final equilibrium solution gave a negative test with silver nitrate solution. The resin was then washed three times with water and air-dried.

2. Rohm and Haas Company Amberlite IR4B anion exchange resin was also converted from the chloride to the nitrate form as described above.

Procedure

Anion Resin Extraction

Twenty milliliters of stock solution A were stabilized using the following procedure. The solution was treated with sodium hydroxide to reduce the acidity to 1 M. Hydroxylamine nitrate was added and the solution was stirred for 30 min. at room temperature. Sufficient sodium nitrite was then added to oxidize the plutonium to the tetravalent state and to destroy the residual hydroxylamine nitrate. Only the plutonium(IV) oxidation state was detectable when the absorption spectra of the solution was determined with a Beckman Model DU spectrophotometer. Next, the solution was made 8.3 M in acid using

16 M nitric acid containing 0.01 M urea, after which it was equilibrated with 10.0 g. of anion resin for a period of 1 hr. at room temperature. The solution was removed from the resin by vacuum filtration and the resin was washed three times with 8 M nitric acid containing 0.01 M urea, followed by one water wash. The washes were combined with the filtrate and the resultant solution sampled.

The plutonium-bearing resin was then stripped by three successive 15 min. equilibrations with 1 M hydroxylamine nitrate solution. The strip solutions were removed by vacuum filtration, combined and sampled.

Results and Conclusions

The decontamination of plutonium from 8 M in nitric acid solution by anion exchange methods gave the values shown in Table 2.

Table 2

Decontamination Factors of Plutonium
by Anion Exchange

Element	Dowex A-1	Amberlite IR4B
Fe	111	2.9
Co	300	1.6
Zr	52	11
Mo	47	>7
Ru	>16	1.02
Ce	44	1.2

Anion exchange decontamination of plutonium appears to be relatively good for the Dowex A-1 resin, but poor for the Amberlite IR4B. However, the extraction and stripping rates are slow with both resins.

SEPARATION OF PLUTONIUM FROM FISSION PRODUCT ELEMENTS BY SOLVENT EXTRACTION

Materials

A. Stock Solutions

1. The plutonium stock solution A was also used in solvent extraction studies.

2. A plutonium-depleted stock solution, designated as stock solution B, was obtained as the residue from the recovery of ~700 g. of plutonium by anion resin exchange. Plutonium(IV) was stabilized by treatment with hydroxylamine nitrate and sodium nitrite. The acidity of this solution was adjusted to 8.0 M with concentrated nitric acid. The final volume of the solution was 4.32 l., and 1.080 l. were used for each experiment described.

3. A purified plutonium nitrate solution was prepared by dissolving plutonium peroxide with nitric acid and adjusting the volume of solution with water to give an acidity of 8.0 M and a plutonium concentration of 50 g./l.

4. Stock solutions of plutonium nitrate in nitric acid were prepared by dissolving plutonium peroxide in concentrated nitric acid and adjusting the volume of the solutions with water to give a plutonium concentration of 50 g./l. and acid concentrations of 6.3, 7.2, 8.0 and 11.0 M.

5. Stock solutions of thorium nitrate and uranyl nitrate were prepared by dissolving reagent grade chemicals in 8.0 M nitric acid and adjusting the volume of solution with 8.0 M nitric acid to give 10.8 g./l. thorium and 50 g./l. uranium.

B. Reagents and Equipment

Thirty-five volume per cent (v/o) tri-*n*-butyl phosphate and primary, secondary, tertiary and quaternary amines were prepared for this work using organic diluents to give the following composition:

a. 35 v/o tri-n-butyl phosphate-65 v/o Gulf Solvent BT

b. 35 v/o primary amine-65 v/o Gulf Solvent BT

c. 35 v/o secondary amine-10 v/o decyl alcohol-55 v/o Gulf Solvent BT

d. 35 v/o tertiary amine-10 v/o decyl alcohol-55 v/o Gulf Solvent BT

e. 35 v/o quaternary amine-10 v/o decyl alcohol-55 v/o Gulf Solvent BT

Standard laboratory reagents and equipment were used. Descriptions of the solvents and organic diluents are contained in the Appendix.

Procedure

A. Tri-n-Butyl Phosphate Solvent Extraction

Equal volumes of the tri-n-butyl phosphate solvent and stock solution A (2.8 M nitric acid) were equilibrated for 10 min., the phases separated and the aqueous raffinate recontacted with fresh extractant. This equilibration procedure was repeated twice and the aqueous raffinate was then sampled. The solvent solutions were combined, stripped three times with a one-third volume ratio of 0.1 M hydroxylamine nitrate and sampled. The strip solutions were also combined and sampled.

B. Amine Solvent Extraction

1. Stock Solution A

Equal volumes of each amine diluted with organic solvent were equilibrated with aliquot portions of stock solution A for a period of 10 min.; the phases were separated and the aqueous raffinates recontacted with fresh solvent. The extraction procedure was performed three times, after which the aqueous raffinates were then sampled. The solvents were then stripped three times with 0.1 M hydroxylamine nitrate, each stripping portion being one-third of the volume of the organic phase. The combined solvent strip solutions and the combined solvents were then sampled. The acidities of all the feed solutions were 8 M in nitric acid except in the case of the primary amine, where an acidity of 6 M was used.

2. Stock Solution B

Ten minute equilibrations of primary, secondary and tertiary amines diluted with organic solvent were made with stock solution B using 1:4 volume ratio of organic to aqueous. The solvent and aqueous phases were separated; the aqueous phase was sampled and the solvent stripped with 0.2 M hydroxylamine nitrate. Only one pass of fresh solvent against the feed material was made for each amine extraction.

C. Effect of Acidity on the Extraction of Plutonium by Amine Solvents

Ten minute equilibrations of the diluted primary and secondary amines with the varying nitric acid plutonium solutions were made using 1:1 volume ratio of organic to aqueous. The phases were separated and sampled for plutonium and acid analyses.

D. Purified Plutonium, Thorium and Uranium Solutions

Equilibrations of primary, secondary, tertiary and quaternary amines diluted with organic solvent with 8 M nitric acid solutions of plutonium, thorium and uranium were made using an equal volume ratio of organic to aqueous and a 10 min. extraction period. The phases were separated and sampled.

E. Stabilization of Plutonium Oxidation State

Stock solution A, stock solution B and the purified plutonium stock solutions were treated in the same manner previously described in order to stabilize the plutonium in the tetravalent state.

Results and Discussion

A. Tri-*n*-butyl Phosphate Extraction

The results shown in Table 3 indicate poor separation of plutonium from ruthenium and cerium with this extractant.

Table 3

Decontamination Factors of Plutonium in Plutonium(IV) Stabilized Stock Solution A by Tri-n-Butyl Phosphate Extraction

Element	Decontamination Factor
Fe	74
Co	>300
Zr	44
Mo	>100
Ru	1.3
Ce	2.1

B. Amine Extraction

1. Stock Solution A

Equilibration of the diluted amines with stabilized stock solution A gave the decontamination values shown in Table 4.

Table 4

Decontamination Factors of Plutonium in Plutonium(IV) Stabilized Stock Solution A by Amine Extraction

Element	Primary Amine	Secondary Amine	Tertiary Amine	Quaternary Amine
Fe	>41.6	120	...	...
Co	>31.4	>80	>60	>52
Zr	>22	92	31	>52
Mo	>8	>78	>18	>9
Ru	12	38	16	13
Ce	>25	>67	>32	>29

The decontamination of plutonium from this solution appears on an over-all basis to be best when the secondary amine system is used.

2. Stock Solution B

Decontamination factors obtained by equilibrating the organic diluted amines with stabilized stock solution B are shown in Table 5. Secondary amine extraction appears to be significantly better than any of the other methods studied. This amine is currently being used to recover plutonium from the pyrometallurgical residues.

Table 5

Decontamination Factors of Plutonium in Plutonium(IV) Stabilized Stock Solution B by Amine Extraction

Element	Primary Amine	Secondary Amine	Tertiary Amine
Fe	168	2410	>20
Co	231	>1060	>204
Zr	161	455	219
Mo	69	>2050	>152
Ru	49	>815	>70
Ce	34	>224	>70

3. Effect of Acidity on Plutonium Distribution Coefficients

The distribution coefficients of primary and secondary amines diluted with organic solvent, and aqueous solutions of plutonium(IV) nitrate in varying nitric acid concentrations are shown in Table 6.

Table 6

Distribution Coefficients of Plutonium(IV) in Nitric Acid Solutions by Amine Extraction

Nitric Acid, M	Primary Amine	Secondary Amine
6.3	1.6	3.9
7.2	2.1	4.2
8.0	2.6	4.0
9.8	2.7	4.5
11.0	2.9	4.4

These data indicate that variations in nitric acid concentrations do not seriously affect the plutonium distribution coefficient, provided the acidity is approximately 8 M.

4. Purified Plutonium, Thorium and Uranium Nitrate Solutions

Equilibration of diluted primary, secondary, tertiary and quaternary amines with 8 M nitric acid solutions of plutonium(IV), uranium(VI) and thorium(IV) nitrates gave the distribution coefficients shown in Table 7.

Table 7

Distribution Coefficients of Plutonium(IV), Uranium(VI) and Thorium(IV) from Nitric Acid Solution by Amine Extraction

Amine	Distribution Coefficient		
	Plutonium	Uranium	Thorium
Primary	2.4	0.14	0.30
Secondary	4.1	0.62	1.15
Tertiary	16.1	1.22	7.64
Quaternary	564	1.78	17.5

The distribution coefficients for the extraction of plutonium(IV), uranium(VI) and thorium(IV) appear to increase proceeding from the primary amine through the quaternary amine. Potential separation processes are indicated.

PLUTONIUM RECOVERY FROM PYROMETALLURGICAL WASTES

Pyrometallurgical wastes containing typical fission product elements consist of alloys, magnesia and tantalum crucibles. Analytical wastes consist of miscellaneous solid residues and solutions containing organic chemicals and various acids and salts. Plutonium is recovered from these residues on a 300 g. scale by the amine extraction and oxalate precipitation method discussed here and outlined in Figures 1, 2 and 3.

A. Feed Preparation

The plutonium-rich solid alloys are dissolved in boiling 16 M nitric-0.05 M hydrofluoric acid. The magnesia and tantalum crucibles are leached with a similar solution. If plutonium is no longer present, the crucibles are discarded and the leach solution is evaporated and combined with the dissolved alloy solution.

Solutions of analytical residues and recycled oxalate filtrate from the terminal plutonium concentration step are evaporated to a tenfold volume reduction and then combined with the dissolved alloy solution. The combined solution is treated with sodium hydroxide to lower the acidity to approximately 1 M and made 0.05 M in hydroxylamine nitrate. Sodium nitrite is then added to destroy any remaining hydroxylamine and to stabilize the plutonium in the tetravalent state. After stabilization, the acid concentration is increased to 8 M with concentrated nitric acid and this solution is used as feed for the amine solvent extraction step.

B. Plutonium Extraction and Concentration

Six successive batch equilibrations of equal volumes of the feed solution and 35 v/o secondary amine in 10 v/o decyl alcohol and 55 v/o Gulf Solvent BT are made, with fresh solvent being used for each extraction. The aqueous raffinate is discarded. The solvent phases are combined and batch-stripped, first with a single pass at a 1:5 volume ratio of water to solvent (to reduce the acid content of the solvent), then with three successive portions of 0.1 M hydroxylamine nitrate at the same aqueous to solvent ratio. The stripped solvent is then acidified by an 8 M nitric acid equilibration and recycled.

The aqueous strip solutions are combined and oxalic acid is added to precipitate plutonium(III) oxalate. This precipitate is removed by filtration, washed with water and transferred to a stainless steel container. The oxalate filtrate is recycled to the feed material evaporator. The plutonium(III) oxalate is then converted to plutonium dioxide by heating to 500°F. in air. The plutonium dioxide is dissolved by refluxing with 16 M nitric and 0.05 M hydrofluoric acid. After volume adjustment and filtration, the resulting plutonium nitrate solution is sampled and sent to the plutonium metal preparation operation.

Results and Conclusions

The recovery and purification of plutonium from pyrometallurgical and analytical wastes has been demonstrated on a 300 g. scale.

A typical analysis of the feed material and of the purified plutonium product is listed in Table 8.

Table 8

Chemical Analysis of Plutonium Recovered by the Secondary Amine Extraction-Oxalate Precipitation Process

Element	Concentration, p.p.m.(a)	
	Feed	Product
Co	37,300	<500
Zr	15,400	<30
Mo	8,220	<100
Ru	11,600	<100
Ce	8,770	<100

(a) Concentration expressed as grams of element per 10^6 grams of plutonium.

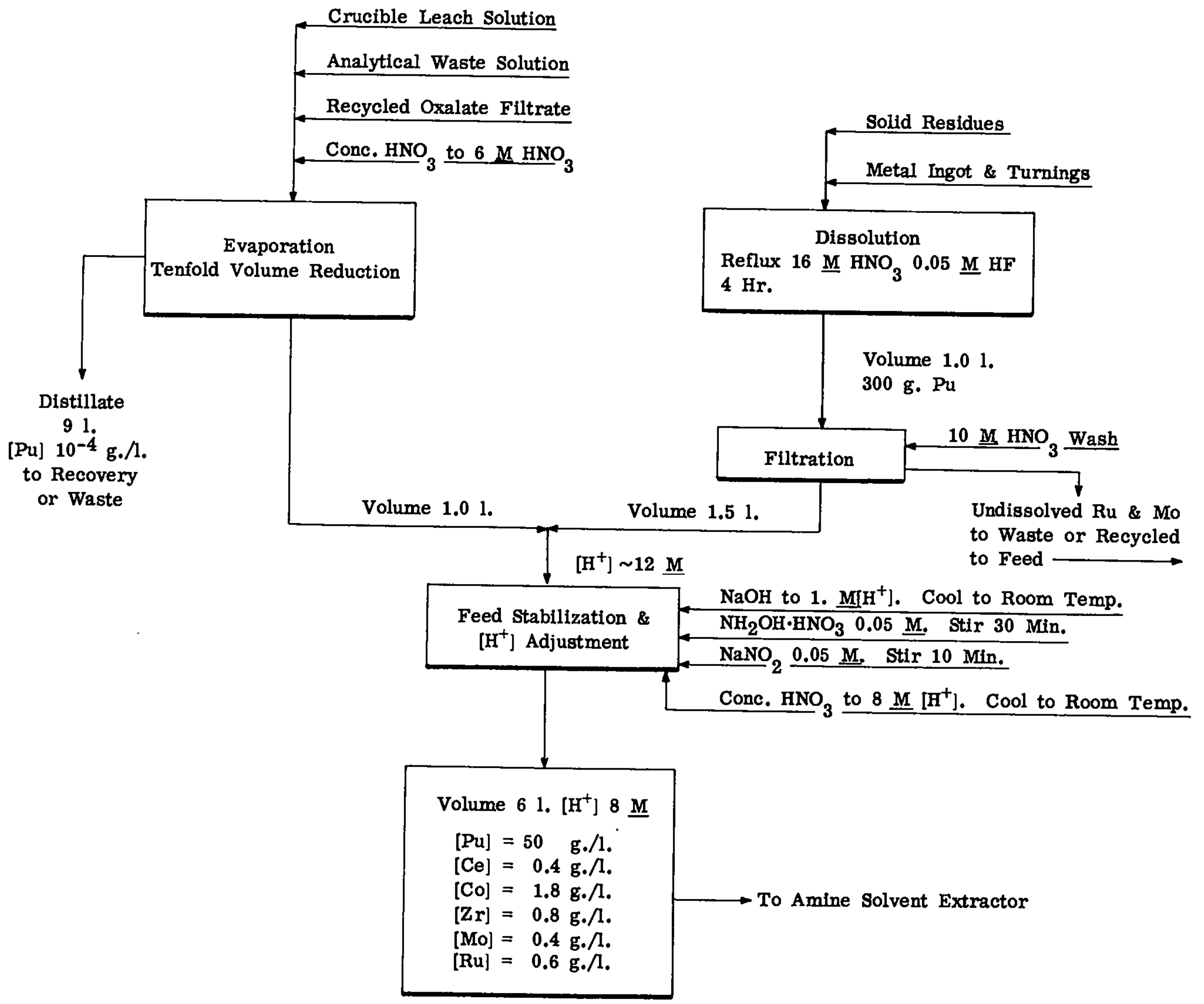

Fig. 1. Flowsheet for the recovery of plutonium from pyrometallurgical wastes. (a) Feed preparation.

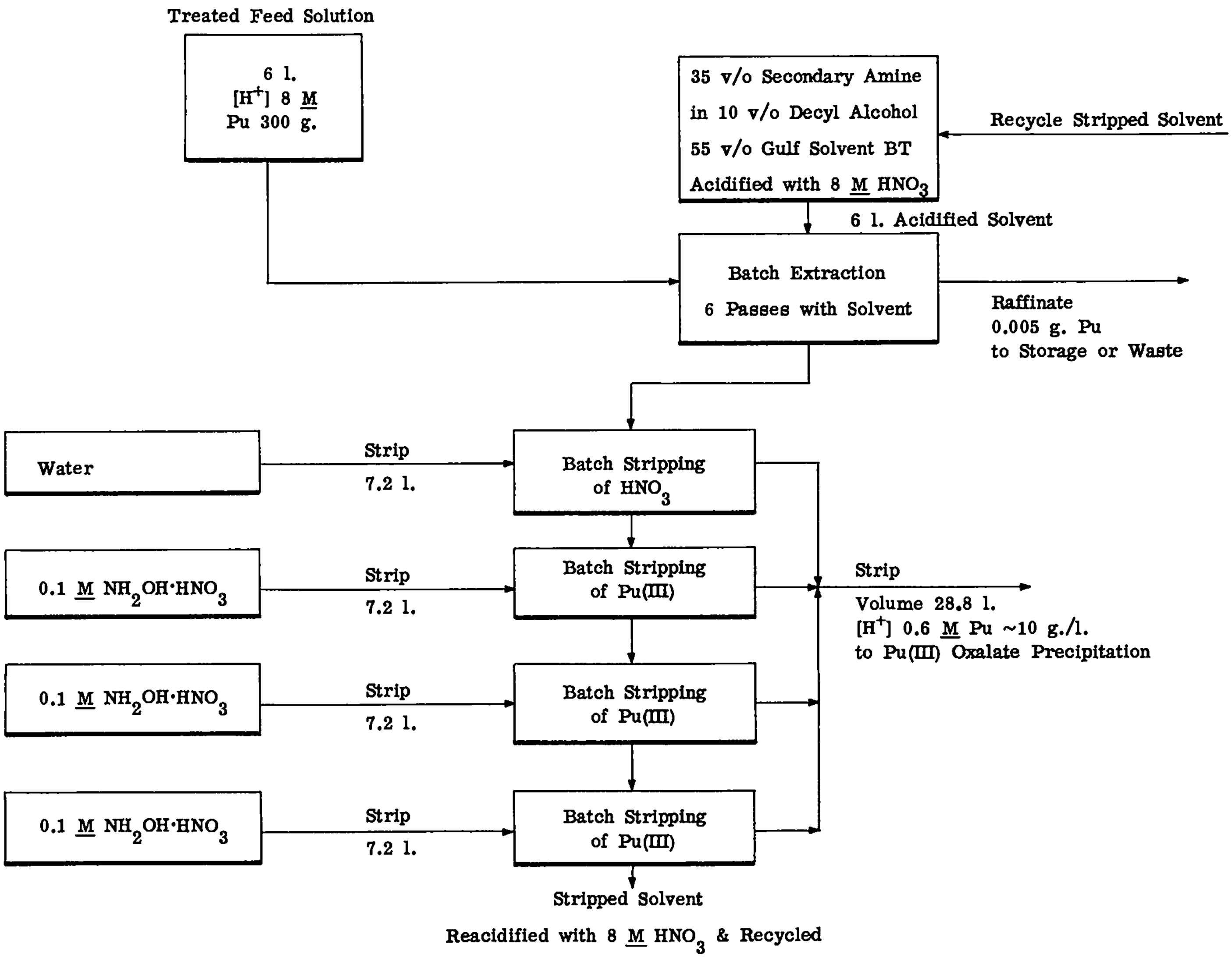

Fig. 2. Flowsheet for the recovery of plutonium from pyrometallurgical wastes. (b) Amine solvent extraction.

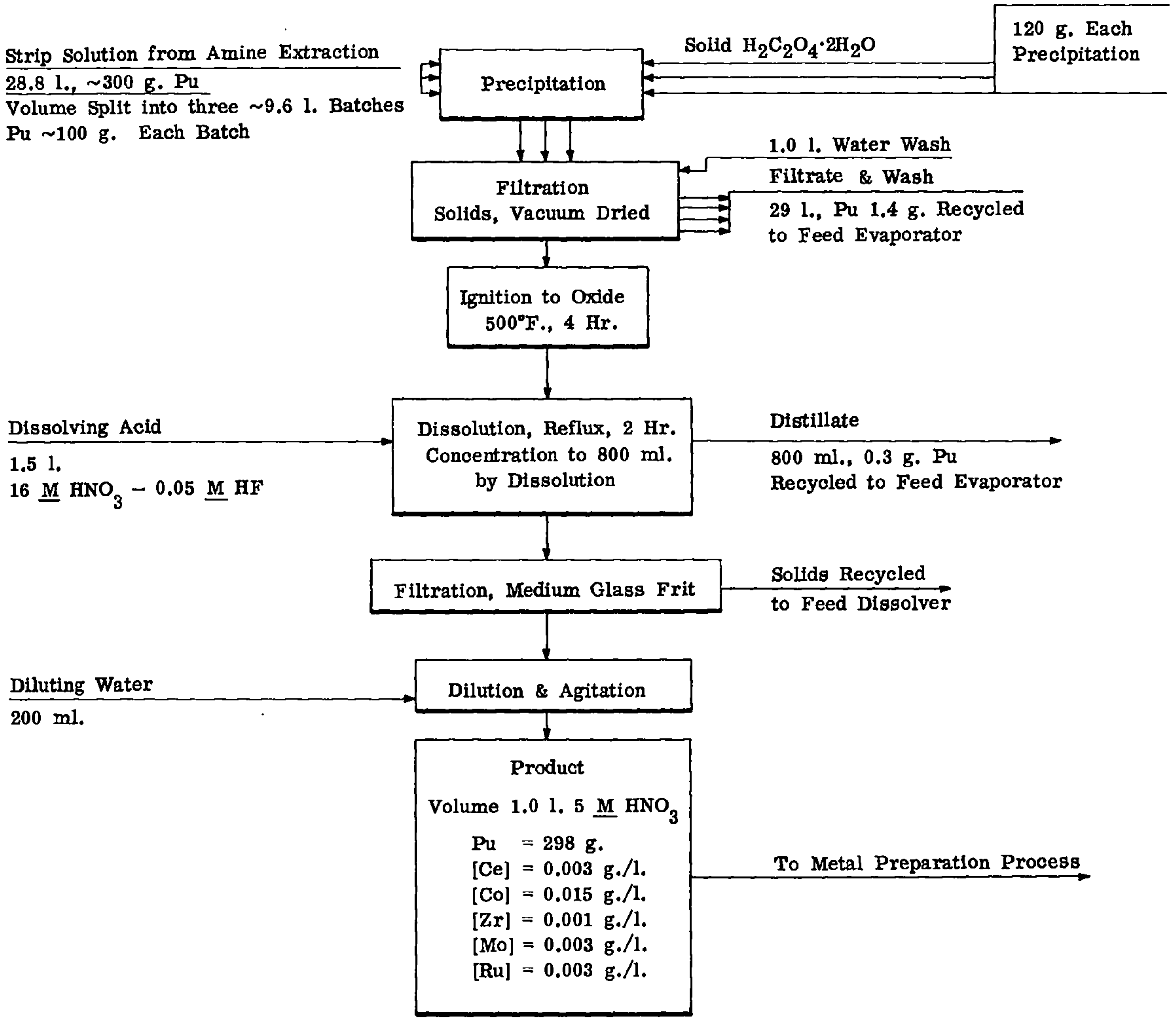

Fig. 3. Flowsheet for the recovery of plutonium from pyrometallurgical wastes.
(c) Plutonium(III) oxalate precipitation.

SUMMARY

A study has been made of the use of plutonium precipitation methods, anion exchange resins and solvent extractants for the separation of plutonium from typical fission product elements. The principal conclusions to be drawn from the results are:

1. The oxalate, fluoride and peroxide precipitation methods for the purification of plutonium did not show promise.

2. Decontamination of plutonium was more effective with Dowex A-1 resin than with Amberlite IR4B at 8 M nitric acid concentration. The slow extraction and stripping experienced with these resins was considered to be an operational disadvantage.

3. Solvent extraction appears to be a usable separation method.

4. A process has been demonstrated for the recovery and purification of plutonium from pyrometallurgical and analytical wastes using a secondary amine extraction-oxalate precipitation method.

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1. Brown, K. B., Coleman, C. F., Crouse, D. J., Denis, J. O., and Moore, J. G., The Use of Amines as Extractants for Uranium from Acidic Sulfate Liquors: A Preliminary Report, Oak Ridge National Laboratory, report AECD-4142, May 27, 1954.

2. Preuss, Al., and Saunders, Jean, The Solvent-Solvent Extraction of Uranium from Sulfuric Acid Solutions with Oil Soluble Amines, Rohm and Haas Company report RMO-2533, April 14, 1955.

APPENDIX

Organic Solvent Compounds

1. Commercial Solvents Corporation tri-*n*-butyl phosphate

The commercial product was scrubbed with two volumes of demineralized water prior to diluting with Gulf Solvent BT.

2. Rohm and Haas Company "Primene JM-T" *t*-alkyl primary amine

One volume of primary amine was acidified with 10 volumes of 6 *M* nitric acid, then neutralized with 6 *M* sodium hydroxide and given a final scrub with demineralized water prior to dilution.

3. Rohm and Haas Company "Amine 9D-178" *t*-alkyl secondary amine

One volume of amine was acidified with 10 volumes of 7 *M* nitric acid, then neutralized with 6 *M* sodium hydroxide and given a final scrub with demineralized water prior to dilution.

4. Carbide and Carbon Chemicals Company tri-iso-octylamine

One volume of amine was acidified with 10 volumes of 7 *M* nitric acid, then neutralized with 6 *M* sodium hydroxide and given a final scrub with demineralized water prior to dilution.

5. Sterwin Chemical Company "Roccal," alkyl dimethyl benzyl ammonium chloride

As supplied, this chloride consists of 10 per cent quaternary amine chloride in 90 per cent inert diluent. It was necessary to separate the quaternary amine from the inert diluent as an initial step for this work. Equilibration of the Roccal with 8 *M* nitric acid caused separation of the amine from the diluent. By successive equilibrations of the quaternary amine chloride with fresh 8 *M* nitric acid the compound was converted to the nitrate form. The chloride content of the amine was checked by testing with silver nitrate solution after each nitric acid equilibration until the compound gave a negative test for chloride.

One volume of quaternary amine nitrate was then washed with 10 volumes of demineralized water prior to dilution.

6. Gulf Oil Company Gulf Solvent BT

   This diluent was used as received from the manufacturer.

7. Eastman Kodak Company n-decyl alcohol (practical)

   The physical properties of the organic reagents were determined and are listed in Tables 9 through 14.

Table 9

Physical Properties of Mixtures of Primary Amine[a] and Gulf Solvent BT at 16°C.

Material, v/o	Specific Gravity	Viscosity, cp.	Refractive Index
100 Amine	0.846	18.86	1.4598
100 Gulf Solvent BT	0.764	0.99	1.4269
10 Amine 90 Gulf Solvent BT	0.733	1.17	1.4268
20 Amine 80 Gulf Solvent BT	0.780	1.38	1.4330
30 Amine 70 Gulf Solvent BT	0.788	1.69	1.4363
40 Amine 60 Gulf Solvent BT	0.796	2.49	1.4390

(a) Rohm and Hass, Primene JM-T, average molecular weight 315, 265°F. flash point, described in bulletin "Tertiary-Alkyl Primary Amine," November, 1955.

Table 10

Physical Properties of Acidified[a] Mixture of Primary Amine and Gulf Solvent BT at 16°C.

Material, v/o	Specific Gravity	Viscosity, cp.	Refractive Index	Acid-Sorbed, Moles per Eq. Wt.
10 Amine 90 Gulf Solvent BT	0.805	1.37	1.4310	4.0
20 Amine 80 Gulf Solvent BT	0.817	2.11	1.4362	3.5
30 Amine 70 Gulf Solvent BT	0.831	3.17	1.4410	3.5
40 Amine 60 Gulf Solvent BT	0.853	6.74	1.4450	3.8
30 Amine 70 Gulf Solvent BT[b]	0.831	3.17	1.4410	3.5

[a] Equal volumes of mixtures and 7.09 M nitric acid equilibrated once.

[b] Life cycle testing of this mixture with 7.09 M nitric acid caused no change in the physical properties or acid sorption value. Irradiation of the primary amine with a cobalt-60 gamma source at 60,000 roentgens per hour for 120 hr. caused no change in physical properties or acid sorption value.

Table 11

Physical Properties of Mixtures of Secondary Amine,(a) Decyl Alcohol and Gulf Solvent BT at 16°C.

Material, v/o	Specific Gravity	Viscosity, cp.	Refractive Index
100 Amine	0.848	105.51	1.4690
100 Gulf Solvent BT	0.764	0.99	1.4269
100 Decyl Alcohol	0.836	11.74	1.4390
10 Amine 90 Gulf Solvent BT	0.772	1.53	1.4309
20 Amine 5 Decyl Alcohol 75 Gulf Solvent BT	0.780	1.98	1.4355
30 Amine 7.5 Decyl Alcohol 62.5 Gulf Solvent BT	0.789	2.64	1.4396
40 Amine 7.5 Decyl Alcohol 52.5 Gulf Solvent BT	0.797	5.78	1.4441

(a) Rohm and Haas, Amine 9D-178, t-alkyl secondary amine, average molecular weight 372, 225°F. flash point, described in bulletin "Amine 9D0178-2S," July, 1956.

Table 12

Physical Properties of Acidified[a] Mixtures of Secondary Amine, Decyl Alcohol and Gulf Solvent BT at 16°C.

Material, v/o	Specific Gravity	Viscosity, cp.	Refractive Index	Acid-Sorbed, Moles per Eq. Wt.
10 Amine 5 Decyl Alcohol 85 Gulf Solvent BT	0.793	1.79	1.4321	3.8
20 Amine 5 Decyl Alcohol 75 Gulf Solvent BT	0.816	5.93	1.4369	4.0
30 Amine[b] 7.5 Decyl Alcohol 62.5 Gulf Solvent BT	0.842	7.42	1.4411	4.1
40 Amine 7.5 Decyl Alcohol 52.5 Gulf Solvent BT	0.864	17.47	1.4460	3.9

[a] Equal volumes of mixtures and 7.6 M nitric acid equilibrated once.

[b] Life cycle testing of this mixture with 7.6 M nitric acid caused no change in the physical properties or acid sorption value. Exposure to cobalt-60 gamma radiation of 60,000 roentgens per hour for 120 hr. caused no measurable change in physical properties or acid sorption value.

Table 13

Physical Properties of Mixtures of Tertiary Amine,[a] Decyl Alcohol and Gulf Solvent BT at 15°C.

Material, v/o	Specific Gravity	Viscosity, cp.	Refractive Index
100 Amine	0.825	9.40	1.4531
10 Amine 10 Decyl Alcohol 80 Gulf Solvent BT	0.777	1.06	1.4305
20 Amine 10 Decyl Alcohol 70 Gulf Solvent BT	0.783	1.27	1.4339
30 Amine 10 Decyl Alcohol 60 Gulf Solvent BT	0.790	1.58	1.4362
40 Amine 10 Decyl Alcohol 50 Gulf Solvent BT	0.796	2.06	1.4390

(a)Carbide and Carbon Chemicals Company, tri-iso-octylamine, boiling range, 0.1 mm. Hg, 123-137°C., typical analysis letter from C. R. O'Rourke, Carbide and Carbon Chemicals Company.

Table 14

Physical Properties of Acidified[(a)] Mixtures of Tertiary Amine, Decyl Alcohol and Gulf Solvent BT at 15°C.

Material, v/o	Specific Gravity	Viscosity, cp.	Refractive Index
10 Amine 10 Decyl Alcohol 80 Gulf Solvent BT	0.800	1.64	1.4318
20 Amine 10 Decyl Alcohol 70 Gulf Solvent BT	0.821	3.82	1.4354
30 Amine 10 Decyl Alcohol 60 Gulf Solvent BT	0.836	7.46	1.4429
40 Amine 10 Decyl Alcohol 50 Gulf Solvent BT	0.860	10.52	1.4440

(a) Equal volumes of mixtures and 8.0 M nitric acid equilibrated once.