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AEC Research and Development Report

# RECOVERY OF PLUTONIUM FROM METALLURGICAL REDUCTION RESIDUES

by

E. K. Dukes and W. E. Prout
Separations Chemistry Division

June 1961

E. I. du Pont de Nemours & Co.
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# ABSTRACT

A semicontinuous process was developed for dissolving the solid residues that are produced in the calcium reduction process for converting plutonium fluoride to plutonium metal. The slag and crucible residues are charged to a column-type dissolver, and are there exposed to a flowing solution of hot nitric acid followed by a nitric acid - aluminum nitrate solution. The resultant solution is suitable as feed for an ion exchange process for recovering plutonium.

A method was demonstrated for recovering plutonium from the dissolver solution by anion exchange in agitated beds of resin. The use of agitated beds of resin eliminates the need for filtering the feed.

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# RECOVERY OF PLUTONIUM FROM METALLURGICAL REDUCTION RESIDUES

## INTRODUCTION

Plutonium metal is produced by reducing plutonium trifluoride or plutonium tetrafluoride with calcium in a bomb process(1-3). Recoverable amounts of plutonium are retained in the solid residues from the reduction process, and this plutonium is recovered as a routine operation. The reduction process generates about 2.5 kilograms of solid residue for each kilogram of plutonium reduced. The residue contains MgO sand and crucible, $CaF_2$, CaO, excess Ca, and a mixture of plutonium fluorides, oxide, and metal. The total plutonium content of the residue is from 2 to 5% of the plutonium charged in the reduction process.

The first step in the recovery of plutonium is the dissolving of the solid residues(4,5). The current process at the Savannah River Plant involves batchwise dissolution in a stainless steel vessel that does not have critically safe dimensions. Although this procedure has been used successfully for some time, it has the disadvantages: (1) the time cycle for dissolving is quite long; (2) corrosion of the dissolver is severe; and (3) the large dimensions of the dissolver necessitate a very careful control of operations to avoid criticality incidents.

The second step in the recovery is the anion exchange absorption of plutonium from the filtered solution produced by dissolving the reduction residues. The ion exchange step has also been used successfully for some time and has no serious disadvantages; however, use of agitated beds of resin would permit elimination of the filtration step.

The objectives of this study were to obtain basic data for the design of a small, semicontinuous dissolver of critically favorable dimensions as a replacement for batch dissolving equipment, and to determine the feasibility of recovering plutonium from dissolver solution by an agitated bed of anion exchange resin. This report summarizes the results of laboratory studies of the effects of temperature, composition of dissolvent, and particle size of reduction residue on the dissolution of plutonium, slag, sand, and crucible. The results of laboratory studies are also presented for the recovery of plutonium from unfiltered dissolver solution with a series of agitated beds of anion exchange resin.

## SUMMARY

The feasibility of semicontinuous dissolution of reduction residues was demonstrated in laboratory tests. The most satisfactory procedure consisted of a two-step dissolution in which the residue was treated first with hot nitric acid and then with hot nitric acid - aluminum nitrate solution. In this procedure 99% of the plutonium and about

95% of the residue were dissolved with a solution-to-solid ratio of about 15 ml/g. A one-step dissolution with either hot nitric acid or hot nitric acid - aluminum nitrate solution dissolved 98% of the plutonium, but these dissolvents left 15 to 25% of the residue undissolved and required liquid-to-solid ratios of 18 to 23 ml/g.

The plutonium was recovered from the dissolver solution in greater than 99% yield by absorption on a series of three small, agitated beds of anion exchange resin. Plutonium loss during the washing of the loaded resin was less than 0.01% per bed volume of wash solution. The absorbed plutonium was eluted at a high flow with the agitators on, to produce a dilute product solution. Alternatively, the stages can be eluted separately with the agitators off, to produce a concentrated product solution.

## DISCUSSION

### DISSOLUTION OF REDUCTION RESIDUES

The dissolution of reduction residues was tested on samples of residue taken from the bomb reduction process in the plant. The composition of a typical batch of reduction residue is presented in Table I.

TABLE I

Composition of Reduction Residues

| | Crucible, g | Sand, g | Slag, g | Total, g |
|---|---|---|---|---|
| MgO | 2200 | 1300 | - | 3500 |
| $SiO_2$ | 2 | 1 | - | 3 |
| $CaF_2$ | - | - | 1200 | 1200 |
| CaO | - | - | 180 | 180 |
| Ca | - | - | 220 | 220 |
| Pu(a) | - | - | 110 | 110 |
| | | | | 5213 |

---

(a) Plutonium is present as plutonium metal, plutonium oxide, and plutonium fluorides.

To recover plutonium from the reduction residues and to minimize waste handling, it is desirable to dissolve completely the plutonium and the solids. Prior to the bomb reduction step the plutonium is precipitated as plutonium trifluoride, dried, and partially oxidized by roasting in oxygen. The plutonium in the slag and crucible can exist as oxide, fluoride, or metal. The dissolvent should be capable of dissolving plutonium in any or all of these forms.

The laboratory experiments consisted of batch and semicontinuous dissolving tests. In the batch tests, weighed samples of reduction residue were contacted with various dissolvents, and observations were made of the effect of temperature, composition of dissolvent, and particle size of residue on the dissolving of the plutonium, slag, sand, and crucible.

Two types of experiments were performed in the semicontinuous dissolving tests. In the first type, semicontinuous dissolving was simulated by adding and removing increments of hot dissolvent. In the second type, a heated column dissolver was operated and the dissolvents were fed continuously to the column containing the residues.

## BATCH DISSOLUTION STUDIES

A number of batch tests were made to define suitable conditions for semicontinuous dissolving runs. The batch tests indicated the advantage of using two dissolvents in sequence, nitric acid followed by nitric acid - aluminum nitrate. Hot nitric acid alone dissolved most of the components of the residue, but left a residue of $CaF_2$ that contained 10 to 30% of the plutonium, presumably as plutonium fluorides. Hot nitric acid - aluminum nitrate solution readily dissolved most of the solids, including $CaF_2$, but left a small heel that contained as much as 40% of the plutonium, presumably as metal. With the two dissolvents used in sequence, essentially complete dissolution of the plutonium and the solids was obtained.

Other batch tests showed the advantages of crushing the solids (especially the crucible material) and dissolving at elevated temperatures. In one test, reduction residues reduced in size to 100 mesh dissolved in hot nitric acid - aluminum nitrate solution in 1 hour; particles about 1/8 inch in diameter dissolved in 1.5 hours; and 1/2-inch pieces were only 98% dissolved in 3 hours. In another test, under conditions that gave essentially complete dissolution of plutonium and solids at 95°C, only 50% was dissolved at 25 or 50°C.

## SEMICONTINUOUS DISSOLUTION STUDIES

In the simulated semicontinuous dissolutions, 1-milliliter increments of dissolvent were added and removed from a 2-gram sample of solid. The results of the simulated runs are shown graphically in Figure 1. Three dissolvents were compared: 13 molar nitric acid alone, 10 molar nitric acid - 0.8 molar aluminum alone, and 13 molar nitric acid followed by 10 molar acid - 0.8 molar aluminum. The data show that in order to minimize the size of the equipment and the dissolving time cycle a two-step dissolution is the most satisfactory procedure. With two dissolvents used in sequence, nitric acid and nitric acid - aluminum nitrate, all of the MgO sand, 98% of the slag, 95% of the plutonium, and about 90% of the crucible dissolved in 70 minutes. At a higher temperature and longer dissolving time, dissolution was complete.

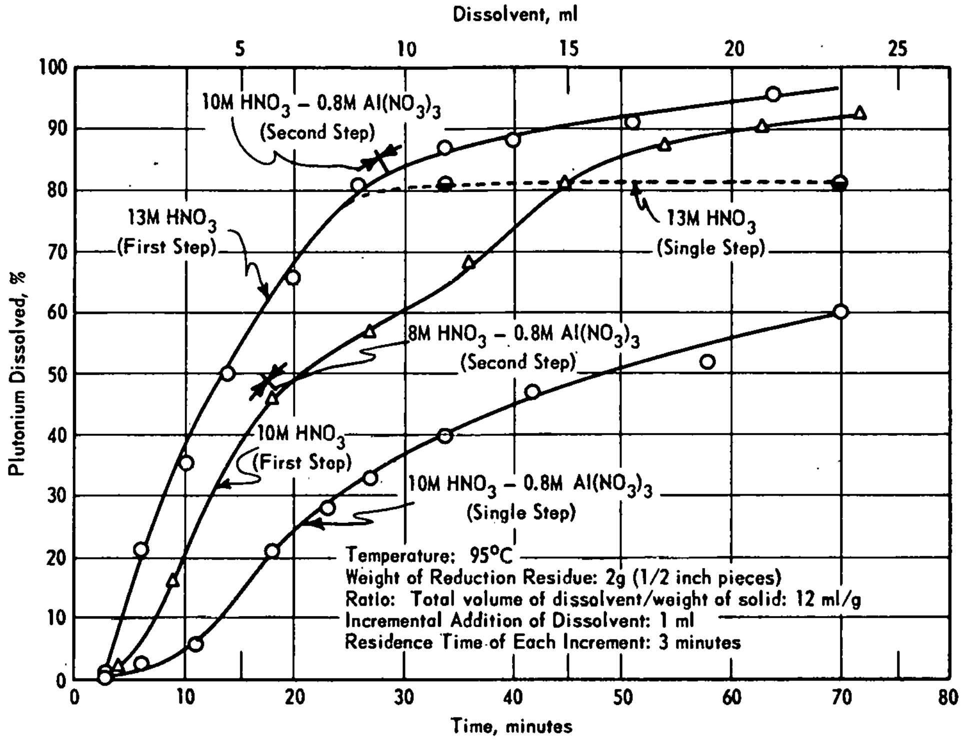

FIG. 1 INCREMENTAL DISSOLUTION OF PLUTONIUM FROM REDUCTION RESIDUE

In a second series of experiments, the feasibility of semicontinuously dissolving reduction residues was demonstrated with a small column dissolver. The dissolver was constructed of 16-mm-ID glass tubing, and a 10-gram charge of solids was retained at the base of the tubing by a coarse glass frit. Dissolvents were fed upflow to the dissolver from feed tanks by displacement with kerosene. The flow of displacement liquid was controlled by an adjustable Lapp "Pulsafeeder" pump. A diagram of the apparatus is shown in Figure 2.

In these tests, the dissolvent was initially fed to the dissolver at room temperature to minimize the bumping and foaming that result from the rapid reaction of calcium metal in the early stages of dissolving. After 5 minutes the bulk of the calcium was dissolved and the temperature was raised to 100°C for the remainder of the test. To prevent the accumulation of explosive mixtures of hydrogen and air, a stream of nitrogen was maintained above the surface of the liquid. Solution leaving the dissolver was collected in a vessel that contained sufficient 2 molar aluminum nitrate to provide a final concentration of 0.6 molar aluminum nitrate. This amount of aluminum is required to complex the fluoride ion present in the dissolver solution. The collected solution was maintained at 50°C to assist in dissolving the small quantity of suspended solids that were carried from the dissolver. At the end of the dissolving run the solution was filtered and the small quantity of solids (principally silica) was washed with nitric acid. Analyses were then made to determine the amount of plutonium in the solution, the amount remaining on the silica, and the amount remaining in the solid heel in the dissolver.

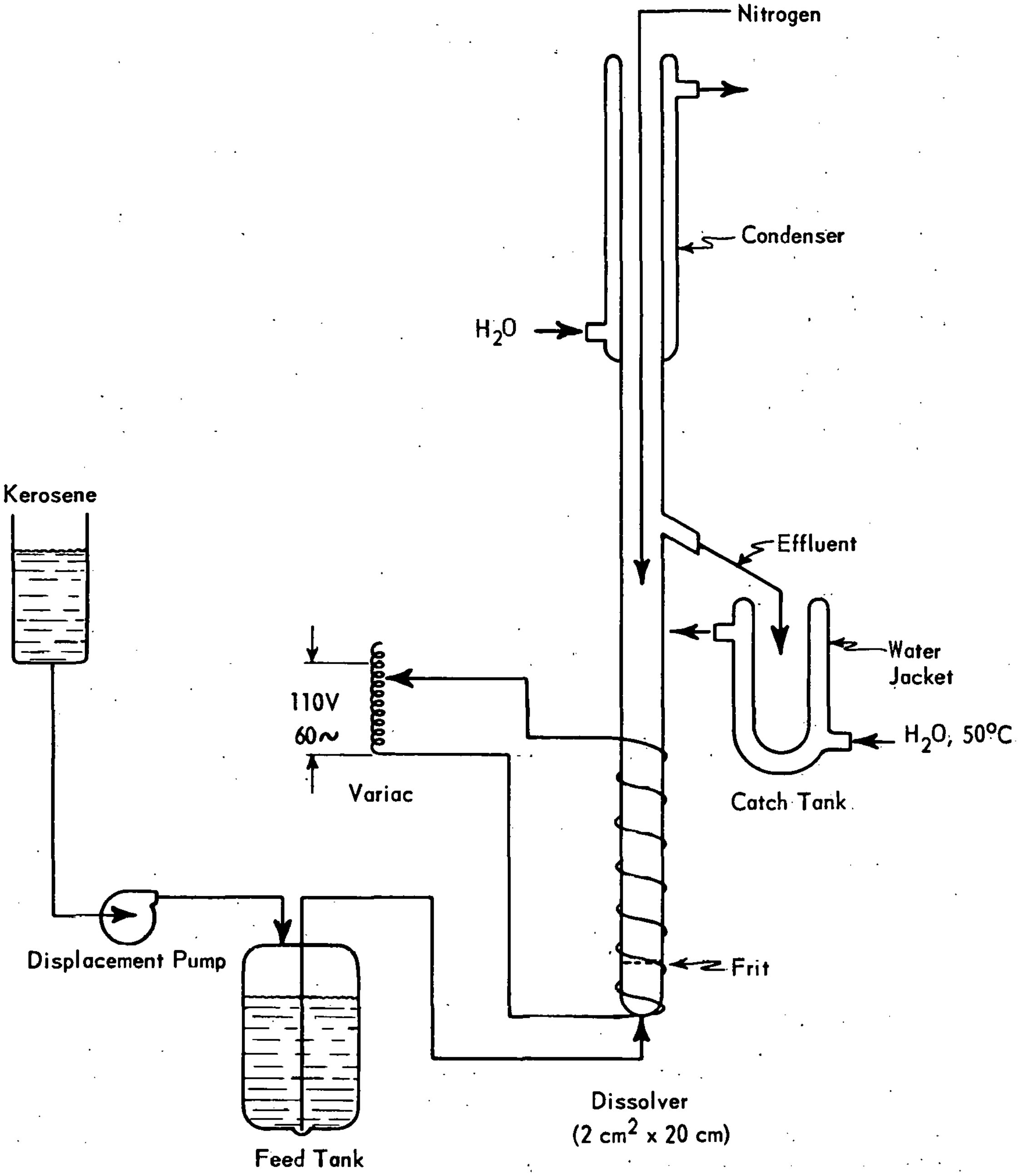

FIG. 2 SEMICONTINUOUS DISSOLVER-FLOW DIAGRAM

The results of ten tests are shown in Table II. The data from tests 1, 2, and 3 show that a temperature near 100°C is required for a suitable rate of dissolving. Tests 4, 5, and 6 were made consecutively without removing the solid heel from the dissolver; the results show that 99.7% of the total plutonium was dissolved. The final heel was only 1.5 grams, or 5% of the total solids charged. Tests 7 through 10 were made with a single dissolvent in a one-step procedure; the results indicate that the one-step procedure is slightly less effective than the two-step procedure that was used in the first six tests.

TABLE II

Semicontinuous Dissolution of Reduction Residues

Charge: 10-g reduction residues; mostly 1/2-inch pieces (bulk density 1.45)
Dissolvent Flow: 2 ml per minute, upflow

| | Dissolvent | | | | Pu Distribution, % | | | |
|---|---|---|---|---|---|---|---|---|
| | First Step | Second Step | | | | | | Final |
| Test | 10M $HNO_3$, ml | 10M $HNO_3$ - 0.5M $Al(NO_3)_3$, ml | Temp, °C | Heel[a], % | Heel | $SiO_2$[b] | Soln | Soln, ml[c] |
| 1 | 45 | 95 | 23 | 50 | 62 | - | 38 | 136 |
| 2 | 46 | 108 | 50 | 40 | 46 | - | 54 | 143 |
| 3 | 46 | 110 | 100 | 5 | 0.3 | 0.1 | 99.6 | 154 |
| 4 | 42 | 144 | 100 | 8 | - | 0.2 | - | 119 |
| 5 | 42 | 124 | 100 | 10 | - | 0.2 | - | 141 |
| 6 | 45 | 100 | 100 | 15[d] | 0.5 | 0.1 | 99.7 | 129 |
| | Dissolvent | Volume, ml | | | | | | |
| 7 | 12.4M $HNO_3$ - 0.1M $Al(NO_3)_3$ | 160 | 100 | 25 | 0.8 | 0.2 | 99.0 | 183 |
| 8 | 12.4M $HNO_3$ - 0.1M $Al(NO_3)_3$ | 164 | 100 | 15 | 1.0 | 0.2 | 98.8 | 193 |
| 9 | 12.4M $HNO_3$ - 0.1M $Al(NO_3)_3$ | 160 | 100 | 25 | 1.4 | 0.6 | 98.0 | 139 |
| 10 | 13M $HNO_3$ | 164 | 100 | 20 | 1.0 | 0.5 | 98.5 | 177 |

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(a) Undissolved residue
(b) Filtered solids (washed), presumably $SiO_2$
(c) 2M $Al(NO_3)_3$ added to provide a final concentration of 0.6M aluminum
(d) Tests 4, 5, and 6 made consecutively. Final heel was 1.5 grams or 15% of a single charge.

The specific gravities of the final product solutions were 1.38, and the solutions were stable at 23°C for several days and were suitable for subsequent recovery of plutonium by an anion exchange.

When one full-size crucible and the associated slag and sand (Table I) are dissolved by the two-step process described above, the filtered product solution is a highly salted solution with the approximate composition shown in Table III.

## TABLE III

### Composition of Dissolver Solution

Basis: One crucible and associated sand and slag dissolved in 23 liters 10M $HNO_3$ plus 57 liters 10M $HNO_3$ - 0.5M $Al(NO_3)_3$ - 16 liters 2M $Al(NO_3)_3$ added to give an Al/F ratio of 2.

| Component | Concentration, molar |
|---|---|
| $Ca^{++}$ | 0.25 |
| $Mg^{++}$ | 0.90 |
| $Al^{+++}$ | 0.64 |
| $F^-$ (complexed) | 0.32 |
| $H^+$ | 6.25 |
| $NO_3^-$ | ~10 |
| Pu | 1.1 g/l |

Specific gravity ~1.38

## ABSORPTION OF PLUTONIUM BY ANION EXCHANGE

Several methods of recovering plutonium from slag and crucible solutions have been used in plant applications. Plutonium has been recovered from filtered solutions by solvent extraction in mixer-settlers and by absorption on fixed beds of anion exchange resin. The recent development and application of agitated beds of resin(6-9) offers an attractive method for processing feeds that contain solids.

This section of the report describes the chemical performance of a series of agitated beds for recovering plutonium from unfiltered dissolver solutions. The aim of this study was to produce an eluate solution that contained 1 gram of plutonium per liter free of solids and cationic impurities.

## EXPERIMENTAL PROCEDURE

The performance of the ion exchange process was evaluated by operating three agitated beds in series. The miniature ion exchange beds were constructed of "Pyrex" glass and were jacketed for work at constant temperature. The resin, "Dowex" 1-X4, 40-60 mesh, was retained at the base of the beds by a 100-mesh screen, and was agitated by a motor-driven paddle that rotated just above the screen. The height of liquid in each bed was controlled by an adjustable weir on the outlet line. Solutions were fed to the beds by displacement with kerosene. The flow of displacement liquid was controlled by an adjustable Lapp "Pulsafeeder" pump. A flow diagram is shown in Figure 3.

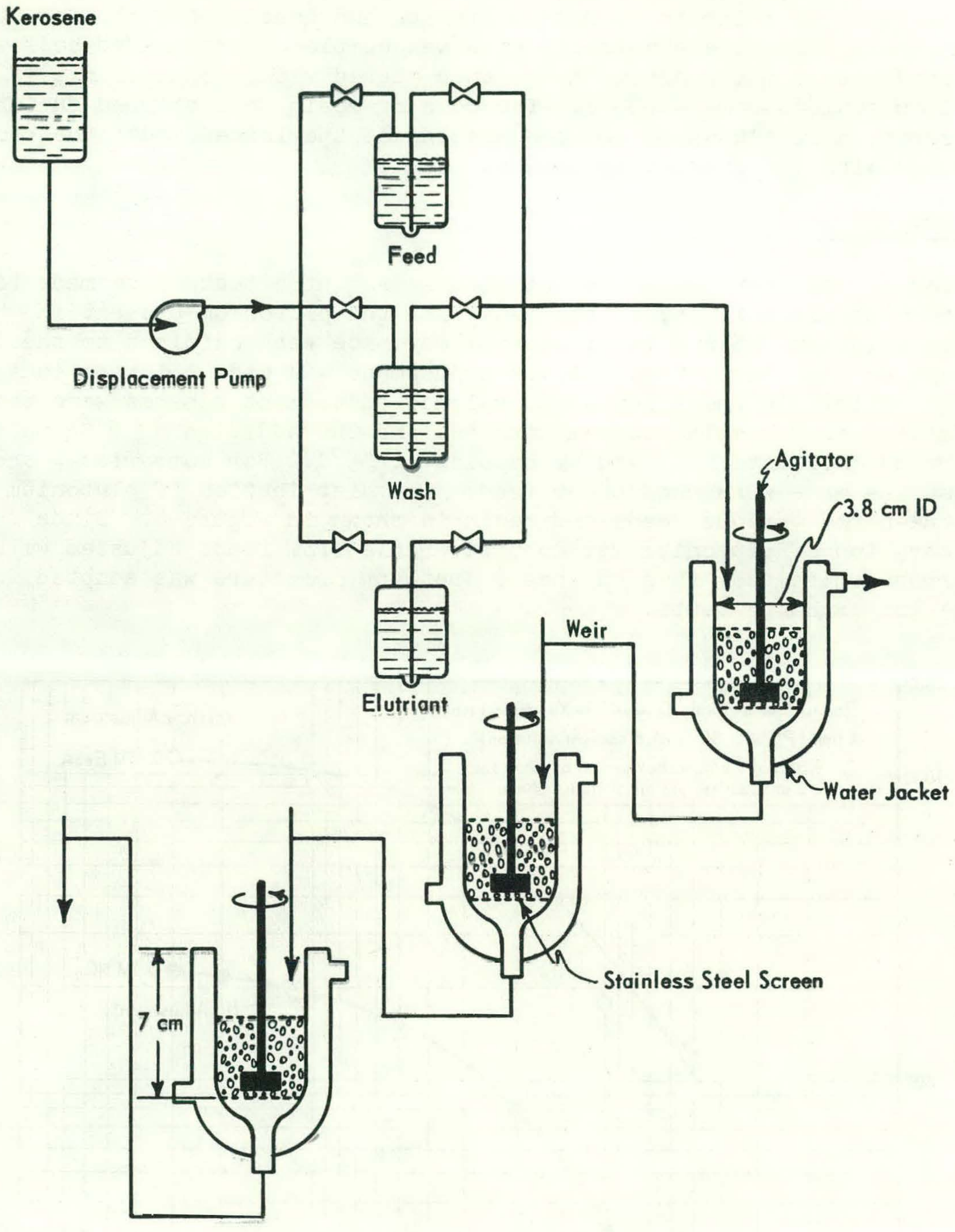


FIG. 3 AGITATED BEDS – FLOW DIAGRAM

The solution produced in the dissolving tests (cf. Table III) was used as feed for the ion exchange tests. The feed contained 0.9 grams per liter of plutonium, which was adjusted to plutonium(IV) by making the solution 0.05 molar in ferrous sulfamate and heating at 55°C for 30 minutes. After the absorption step was complete, the loaded beds were washed with 8 molar nitric acid, then eluted with 0.3 molar nitric acid; all operations were at 35°C. The beds of resin were stirred during the absorption of plutonium and the washing of the loaded beds; the beds were eluted with and without agitation.

## EXPERIMENTAL RESULTS

Prior to the tests with the agitated beds, batch tests were made to determine the affinity of the resin for the plutonium present in the feed solution. Since the plutonium may have been oxidized to the (VI) state during dissolving, valence adjustment was made prior to contact with resin. In these tests two valence adjustment schemes were tried; addition of 0.1 molar sodium nitrite, and the addition of 0.05 molar ferrous sulfamate followed by heating at 55°C. For comparison, one test was made with unadjusted feed. The distribution of plutonium between the various feeds and resin is shown in Figure 4. Since the absorption of plutonium was most favorable from feeds adjusted with ferrous sulfamate, this valence adjustment procedure was adopted for the ion exchange tests.

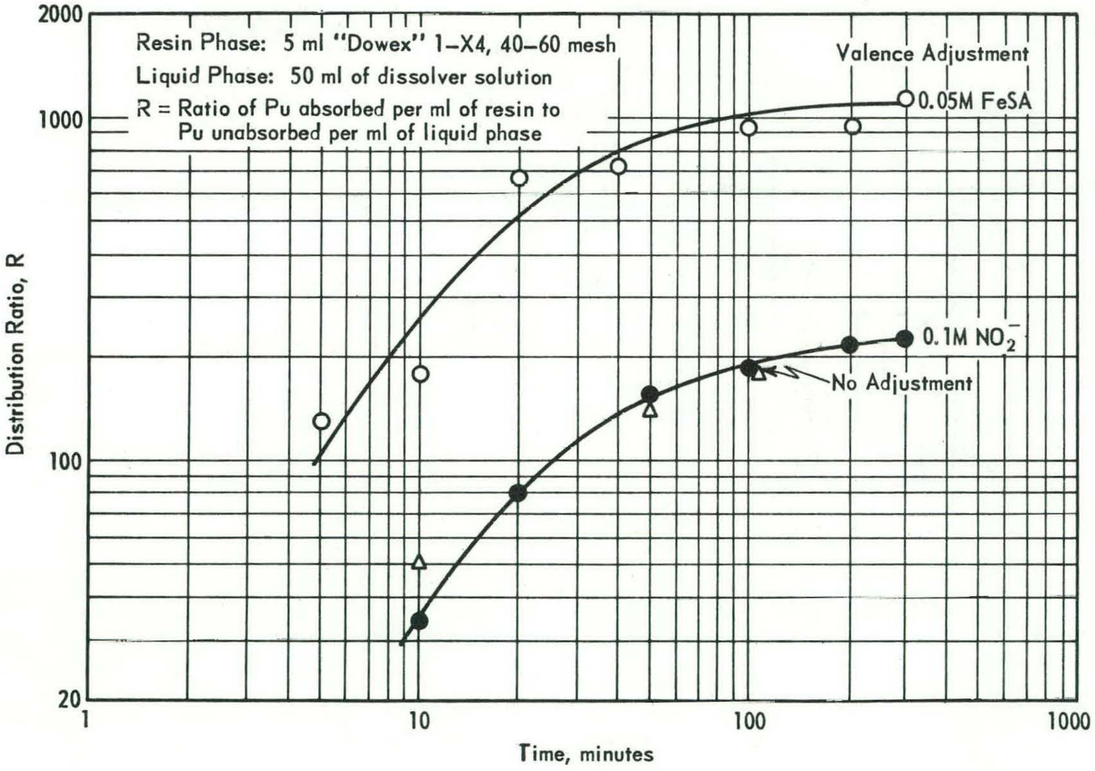


FIG. 4 EFFECT OF VALENCE ADJUSTMENT AND CONTACT TIME ON DISTRIBUTION RATIO, R

Three runs were made on the series of three agitated beds with feeds adjusted with ferrous sulfamate. In these runs the effluent stream from each bed was analyzed for plutonium; and the data pertaining to operation with one, two, and three stages are shown in Table IV. The data indicate that two beds of anion resin are sufficient to absorb plutonium from 40-50 bed volumes of dissolver solution with a loss of about 0.2%. Plutonium loss during the washing of the loaded bed was less than 0.01% per bed volume of wash solution. The plutonium was eluted at a high flow with the agitators on and a dilute product solution was obtained. The beds may be eluted separately with the agitators off if a concentrated product solution is required.

TABLE IV

Recovery of Plutonium from Dissolved Reduction Residues

Feed: One liter of dissolved reduction residue
Temp: 35°C

Feed Absorption Step

| | | | | Pu in Absorption Effluent, % of Pu in feed | | |
|---|---|---|---|---|---|---|
| Test | Flow, ml/min | RCT,(a) minutes | Feed,(b) bed vol | One Bed | Two-Bed Cascade | Three-Bed Cascade |
| 1 | 3.3 | 7.6 | 40 | 1.8 | 0.1 | $<$0.01 |
| 2 | 3.5 | 7.1 | 40 | 1.5 | 0.1 | $<$0.01 |
| 3 | 5.0 | 4.0 | 50 | 4.3 | 0.1 | $<$0.01 |

Wash Step - 8M $HNO_3$

| | | | | Pu in Wash Effluent, % of Pu in feed | | |
|---|---|---|---|---|---|---|
| Test | Flow, ml/min | RCT, minutes | Wash,(b) bed vol | One Bed | Two-Bed Cascade | Three-Bed Cascade |
| 1 | 3.3 | 7.6 | 6.0 | 0.6 | 0.03 | 0.04 |
| 2 | 3.5 | 7.1 | 6.0 | 0.6 | 0.03 | 0.03 |
| 3 | 5.0 | 4.0 | 7.5 | 0.8 | 0.06 | 0.05 |

Elution Step - 0.3M $HNO_3$

| | | | | Elutriant Required to Elute 99% of the Absorbed Pu, bed vol(b) | | |
|---|---|---|---|---|---|---|
| Test | Flow, ml/min | RCT, minutes | Agitators | One Bed | Two-Bed Cascade | Three-Bed Cascade |
| 1 | 0.8 | 30.0 | On | 20 | 24 | 28 |
| 2 | 3.5 | 7.1 | Off | 16 | 20 | 24 |
| 3 | 2.0 | 10.0 | On | 25 | 35 | (c) |

---

(a) Resin Contact Time: Ratio of volume of resin in one stage to volume rate of liquid flow

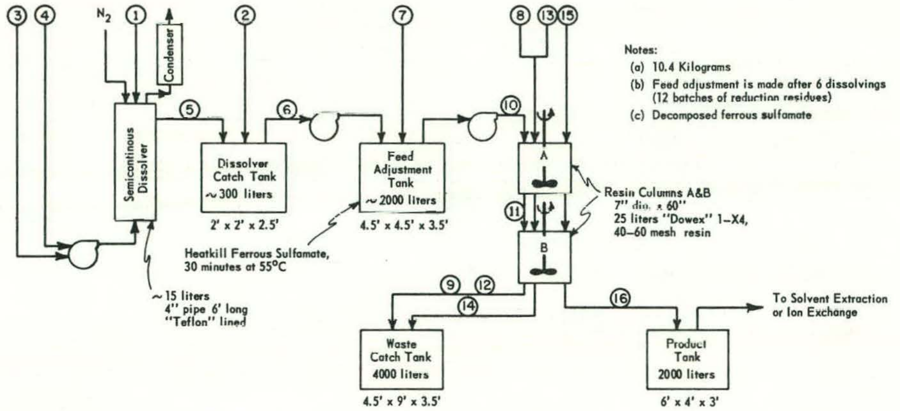
(b) The feed, wash, and elutriant were fed to Bed 1. The bed volumes shown for solutions are multiples of the bulk volume of resin in one bed (25 ml in Tests 1 and 2 and 20 ml in Test 3). In the elution step the plutonium from Bed 1 was progressively displaced through Beds 2 and 3.

(c) Bed 3 was not eluted in the third test.

## RECOMMENDED FLOWSHEET

From the results of the tests described, a tentative process flowsheet was designed for the recovery of plutonium from reduction residues. The flowsheet is shown in Figure 5. This process produces a dilute plutonium solution free of macro contamination and suitable for further processing; the solution is particularly well suited for recycle to a primary solvent extraction system.

| | ① | ② | ③ | ④ | ⑤ | ⑥ | ⑦ | ⑧ | ⑨ | ⑩ | ⑪ | ⑫ | ⑬ | ⑭ | ⑮ | ⑯ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| PROCESS MATERIAL | Two[(a)] Residue Batches | Complexing Solution | Dissolvents | | Dissolver Effluent | Dissolver Solution | Feed[(b)] Adjustment | Column Recondition Wash | Recondition Wash Effluent | Resin Column Feed | Effluent Column A | Effluent Column B | Wash | Wash Effluent | Elutriant | Eluate |
| Flow, liters/min | | | 1.2 | 1.2 | 1.2 | 3.0 | | 5.0 | 5.0 | 3.8 | 3.8 | 3.8 | 5.0 | 5.0 | 2.0 | 2.0 |
| Volume, liters | | 32 | 46 | 114 | 160 | 192 | 30 | 240 | 240 | 1182 | 1182 | 1182 | 180 | 180 | 720 | 720 |
| Temperature, °C | | 50 | 100 | 100 | ~95 | 50 | 25 | 25 | 25 | 25 | 25 | 25 | 25 | 25 | 25 | 25 |
| Specific Gravity | | 1.3 | 1.3 | 1.3 | ~1.5 | 1.38 | 1.25 | 1.25 | 1.13 | 1.30 | 1.30 | 1.30 | 1.25 | 1.25 | 1.01 | 1.06 |
| Plutonium, grams | 220 | | | | 220 | 220 | | | | 1320 | ~53 | ~1 | | < 0.1 | | 1319 |
| Nitric Acid, molar | | | 10 | 10 | 7.5 | 6.25 | | 8.0 | 6.1 | 6.09 | 6.1 | 6.1 | 8.0 | 7.2 | 0.30 | 1.8 |
| Fluoride ($F^-$), molar | | | | | 0.38 | 0.32 | | | | 0.31 | 0.31 | 0.31 | | 0.1 | | |
| Calcium (metal), grams | 446 | | | | | | | | | | | | | | | |
| Calcium Fluoride, grams | 2368 | | | | | | | | | | | | | | | |
| Calcium Oxide, grams | 366 | | | | | | | | | | | | | | | |
| Calcium Nitrate, grams | | | | | 0.30 | 0.25 | | | | 0.24 | 0.24 | 0.24 | | 0.08 | | |
| Magnesium Oxide, grams | 6994 | | | | | | | | | | | | | | | |
| Magnesium Nitrate, molar | | | | | 1.08 | 0.90 | | | | 0.88 | 0.88 | 0.88 | | 0.29 | | |
| Aluminum Nitrate, molar | | 2 | | 0.5 | 0.36 | 0.64 | | | | 0.62 | 0.62 | 0.62 | | ~0.3 | | |
| Ferrous Sulfamate, molar | | | | | | | 2 | | | 0.05[(c)] | 0.05[(c)] | 0.05[(c)] | | 0.02[(c)] | | |
| Silica ($SiO_2$), grams | ~6 | | | | ~6 | ~6 | | | | ~6 | ~6 | ~6 | | ~1 | | |
| Nitrate Ion ($NO_3^-$), molar | | | 10 | 11.5 | 11.4 | ~10 | | | | ~10 | ~10 | ~10 | | | | |
| Time, hours | ← 2.2 → | | | | | | ← 1.5 → | ← 0.8 → | | ← 5.2 → | | | ← 0.6 → | | ← 6.0 → | |



Notes:
(a) 10.4 Kilograms
(b) Feed adjustment is made after 6 dissolvings (12 batches of reduction residues)
(c) Decomposed ferrous sulfamate

FIG. 5 RECOVERY OF PLUTONIUM FROM REDUCTION RESIDUES – DISSOLUTION AND ION EXCHANGE FLOWSHEET

## DISSOLUTION OF RESIDUE

1. Add 32 liters of 2 molar aluminum nitrate solution to the dissolver catch tank to complex the fluoride ion in the dissolver solution. During the dissolving, heat the dissolver catch tank to 50°C.

2. Charge two slag and crucible batches (10.4 kg) to the dissolver. Feed 46 liters of 10 molar nitric acid then 114 liters of 10 molar nitric acid - 0.5 molar aluminum nitrate to the dissolver at a flow of 1.2 liters per minute. Initially, feed the 10 molar nitric acid to the dissolver at ambient temperature until the bulk of the calcium metal dissolves, then heat the dissolver and dissolver feed streams to 100-105°C. To eliminate the hazards of explosive hydrogen - air mixtures feed nitrogen to the dissolver during the dissolution. The dissolver is lined with "Teflon" to minimize corrosion from fluoride ion.

3. After dissolving is complete (2.8 hours), cool the solution in the dissolver catch tank to 30°C and transfer to the feed adjustment tank.

## ABSORPTION OF PLUTONIUM BY ANION EXCHANGE

1. Collect the solution from six dissolvings in the feed adjustment tank. Add 30 liters of 2 molar ferrous sulfamate, agitate the solution for 30 minutes, then heat to 55°C for 30 minutes and cool to 30°C.

2. Feed the adjusted solution from the feed adjustment tank to the two agitated beds connected in series at 3.8 liters per minute (5.2 hours). The beds are agitated during the absorption cycle.

3. Wash the beds with 180 liters of 8 molar nitric acid at 5 liters per minute with the agitators on (0.6 hour).

4. Elute the absorbed plutonium with 720 liters of 0.3 molar nitric acid at 2.0 liters per minute with the agitators on (6 hours).

5. Return the product solution to main line process for plutonium concentration and conversion to metal.

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## BIBLIOGRAPHY

1. Orth, D. A., E. L. Field, and J. H. Radke. (Mainland, E. W., ed.) "Production of Plutonium Metal." *Ind. Eng. Chem*. *53*, 685-94 (1961).

2. Burney, G. A. and F. W. Tober. *Precipitation of Plutonium Trifluoride for Isolation of Metal*. E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, S. C. AEC Research and Development Report DP-322, 47 pp. (November 1959) (Secret).

3. Field, E. L. *Plant Test of the Wet Fluoride Process*. E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, S. C. AEC Research and Development Report DP-292, 26 pp. (August 1958) (Secret).

4. Folger, R. L., et al. *Recovery of Plutonium from Metallurgical Wastes*. Oak Ridge National Laboratory, Oak Ridge, Tenn. AEC Research and Development Report ORNL-1442, 100 pp. (April 1953) (Confidential).

5. Groot, C. and H. H. Hopkins, Jr. *Recovery of Plutonium from Slag and Crucible*. General Electric Co., Nucleonics Division, Hanford Works, Richland, Wash. AEC Research and Development Report HW-23343, 58 pp. (December 1951) (Secret).

6. Bartels, C. R., et al. "A Novel Ion-Exchange Method for the Isolation of Streptomycin". *Chem. Eng. Progr*. *54*, No. 8, pp. 49-51 (1958).

7. Prout, W. E. and L. P. Fernandez. "Performance of Anion Resins in Agitated Beds". *Ind. Eng. Chem*. *53*, No. 6, pp. 449-52 (1961).

8. Marchello, J. M. and M. W. Davis, Jr. "Theoretical Investigation of Agitated Ion Exchange Beds." (to be presented at the A.I.Ch.E. National Meeting, Los Angeles, February 4-7, 1962).

9. Davis, M. W., Jr., M. R. Klingler, and J. F. Ward, Jr. "Operating and Design Variables of Agitated Ion Exchange Variables", (to be presented at *First Inter-American Congress of Chemical Engineering*, San Juan, Puerto Rico, October 23-28, 1961).