950 34/6

AMERICIUM RECOVERY AND PURIFICATION AT ROCKY FLATS

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ROCKWELL INTERNATIONAL

Abstract

Americium processing at Rocky Flats Plant is described.
The americium is recovered from plutonium metal by a pyrochemical
process; the resulting molten salt extraction residues must be
processed to recover americium. At present the process uses cation
exchange with Dowex 50W-X8 resin. This work shows that a macroporous
resin, BioRad AG MP-50, has 30 to 50% more capacity and better elution
kinetics than Dowex 50W-X8. Two extraction methods, solvent extraction
and extraction chromatography with an organophoshorus bidentate,
are also described. Americium is effectively decontaminated from
magnesium and aluminum, and recovered by these methods.

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INTRODUCTION

Rocky Flats has a large recovery facility to recover plutonium from miscellaneous scraps and residues; a by-product in the plutonium recovery stream is americium from the decay of plutonium-241. Currently a $NaCl-KCl-MgCl_2$ eutectic salt is used at Rocky Flats to separate americium from plutonium in a molten salt extraction (MSE) process.

As a consequence of experimental runs and changes in process salt mixtures, a variety of waste salts and alloys have been produced, and much of this material is in storage. These waste products contain varying quantities of magnesium, sodium, potassium, calcium, and aluminum as well as plutonium and americium.

Although alloys produced during metallothermic reduction clean-up of waste salts are not being processed, processing of waste salts (except those containing aluminum) is being done by the process shown in Figure 1. The process includes (1) dilute hydrochloric acid dissolution of residues; (2) cation exchange to convert from the chloride to the nitrate system and to remove gross amounts of monovalent impurities; (3) anion
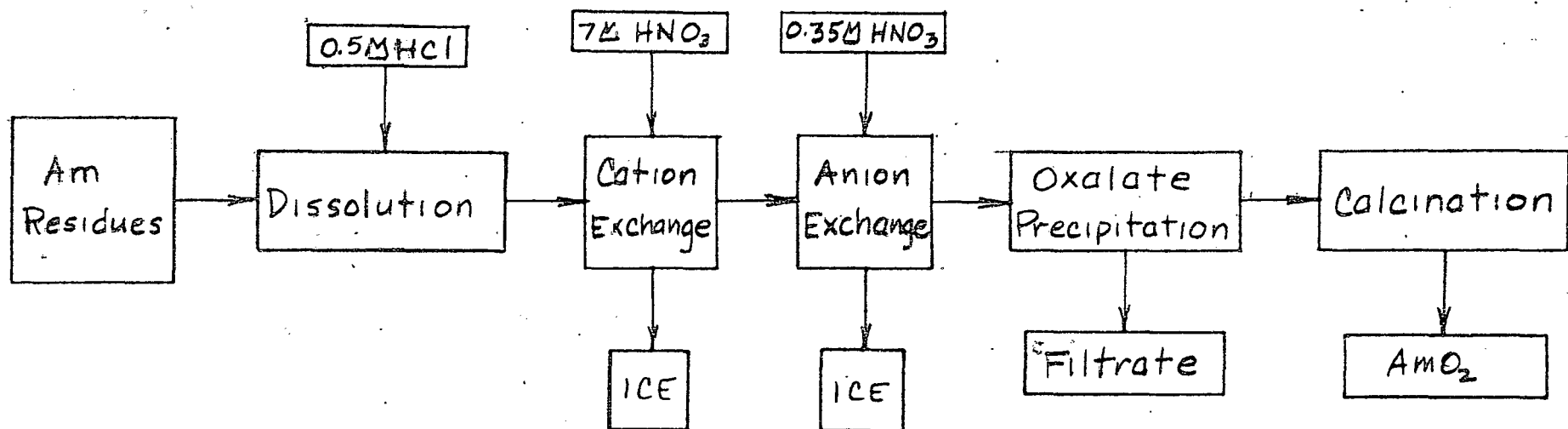
Fig. 1. Rocky Flats Americium Recovery Process

exchange removal of plutonium; (4) conversion to solid by oxalate precipitation; and (5) calcination at 600°C to yield AmO_2. AmO_2 which meets specifications ($\geq$95% AmO_2, <1% individual contaminant elements) is sent to the Department of Energy Isotope Pool.

Americium recovery development at Rocky Flats comprises work to improve the existing process as well as to introduce new methods, especially those that can partition americium and aluminum since the present cation exchange process can not do this.

This report describes attempts to improve the recovery of americium. The first part of the report deals with evaluation of cation exchange resins used for the concentration of americium from low level solutions and for separating americium from major impurities. The second part of the report describes development of a process that will recovery americium from residues containing aluminum as well as other common impurities. Results of laboratory demonstration tests of both approaches will be described.

Ion Exchange

In the cation exchange process plutonium and americium are cosorbed on the resin with mono- and divalent cations from the MSE process.[1] After actinide breakthrough the column is washed with water to remove nonadsorbed ions remaining (e.g. chloride), then eluted with 7$\underline{M}$ HNO_3. Plutonium is oxidized to Pu(IV) and forms the hexanitrato complex $Pu(NO_3)_6^{2-}$. Although this anionic complex is not adsorbed on the resin it still does not elute very rapidly. We attribute this slow recovery to the difficulty the bulky complex has in diffusing through the resin matrix, as well as the high

selectivity of the resin for Pu(IV). The process should be made easier by using a gel-type resin of lower crosslinkage or a macroporous resin. The more open or porous structure of these resins decrease the distance an ion must travel through the matrix until an open channel is reached. In addition it should also be easier to selectively elute contaminant ions from more porous resins by using acid washes or chelating agents for the same reason.

Gel-type cation exchange resins were therefore compared with a macroporous resin for the recovery of plutonium and americium from molten salt residues. The effects on actinide recovery of loading and eluting in the downflow mode, and loading in the upflow, eluting in the downflow mode were also determined.

Experimental

Materials. Two MSE residues were used. These were Na-K-Mg chloride and Ca-K-Mg chloride waste salts containing Pu and Am. The procedure for preparing the solutions is to solubilize the salt in 0.5$\underline{M}$ HCl at 90°C and filter to remove insolubles. The solutions are diluted to a chloride concentration of 50 g/l. Analysis of these solutions gave the results listed in Tables I and II.

Three resins were tested during this preliminary program. The first was the conventional gel-type resin currently used at Rocky Flats Plant, Dowex^R 50W-X8. The second was also a gel-type resin, but with only 4% crosslinkage, Dowex 50W-X4. The third resin was BioRad^R AG MP-50, a macroporous resin. All three cation exchange resins were in the 50 to 100 mesh particle size range.

Chemicals used were reagent grade.

TABLE I

Composition of Na-K-Mg Chloride Waste Solution

$H^+ = 0.5M$ $Cl^- = 1.4M$ (50 g/1)

Component	Concentration mg/1	Analytical[a] Method
Al	< 10	AA
Ca	5	AA
Fe	7	AA
K	17700	AA
Mg	1600	AA
Na	12100	AA
Pb	< 1	AA
Pu	3190	R
Am	170	R

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a   AA - Atomic Absorption; R - Radiometric

TABLE II

Composition of Ca-K-Mg Chloride Waste Solution

$H^+ = 0.44M$ $Cl^- = 1.4M$ (50 g/l)

Component	Concentration mg/l	Analytical[a] Method
Al	20	AA
Ca	20000	AA
Fe	10	AA
K	6200	AA
Mg	650	AA
Na	420	AA
Pb	10	AA
Pu	10420	R
Am	430	R

a   AA - Atomic Absorption; R - Radiometric

Procedure. Plutonium was adjusted to Pu(III) by addition of
2.5 g/l hydroxylamine hydrochloride one-half hour before the column
test. Seven tests were made in the downflow loading and elution mode.
Solutions were fed to the column (3.0 cm dia., 150 ml column volume)
at 6 ml/min. The column effluent was collected in fractions and
sampled to determine actinide content. After actinide breakthrough
(0.1 g actinide/l determined by radiometric monitoring of the column
effluent) loading was stopped and the column was washed with water
at 13 ml/min (total volume = 1 liter). Eultion with $7\underline{M}$ HNO_3 followed.
In the first two tests 4.2 column volumes of ion column effluent
were collected. This volume is the average volume required for
product recovery in production columns; it should be noted, however,
that in production runs loading is upflow, elution is downflow, so
this comparison is not appropriate. In the subsequent five tests,
product fractions were collected to determine the rate of elution
and elution was continued until recovery was more complete. After
the product fractions were collected the column was washed with water
to remove the $7\underline{M}$ HNO_3.

Four additional tests were made in the upflow loading, down-
flow elution mode. There were no changes in procedure, flow rates,
etc., other than this change in direction of flows. Only the
Dowex 50W-X8 and BioRad AG MP-50 resins were tested in this test
series.

When it became evident that not all plutonium was being removed
from the resins, additional washes with $6\underline{M}$ HCl or $0.1\underline{M}$ diethylene-
triaminepentaacetic acid (pH = 8.0) were used to strip the column

before the next test.

Samples of all ion column effluent were submitted for analysis.

Results and Discussion

Seven ion exchange tests were made in the downflow loading and elution mode. The amount of actinides fed to the column and the percent actinide recovery obtained with 4.2 column volumes of 7M HNO$_3$ elutriant are shown in Table III.

The capacity of the resins for actinide is less when calcium is present; this is because the divalent calcium competes more effectively for ion exchange sites than does monovalent sodium. The capacities of the resins for individual actinides were not determined in these experiments. The relative capacities for both Pu and Am from the MSE residues are discernible from the actinide adsorbed (the actinide in the feed).

The ideal resin would have high actinide capacity and give complete recovery of plutonium and americium in a small volume of elutriant. Each of the resins tested has apparent advantages and disadvantages, which can best be seen by referring to Table III and Figures 2 and 3. (Figures 2 and 3 show the percent recovery as a function of column volumes for plutonium and americium, respectively.)

Dowex 50W-X4 gives the best recovery of both plutonium and americium, 99.6 and 99.4% respectively in 3.9 column volumes of elutriant. However, the capacity of this resin is inadequate, being only 48% of that of Dowex 50W-X8 resin.

The problem with Dowex 50W-X8 is the slow recovery of

TABLE III

ACTINIDE ELUTION FROM CATION EXCHANGE RESIN - DOWNFLOW MODE

Column Volume (CV): 150 ml
Elutriant: 7M HNO_3
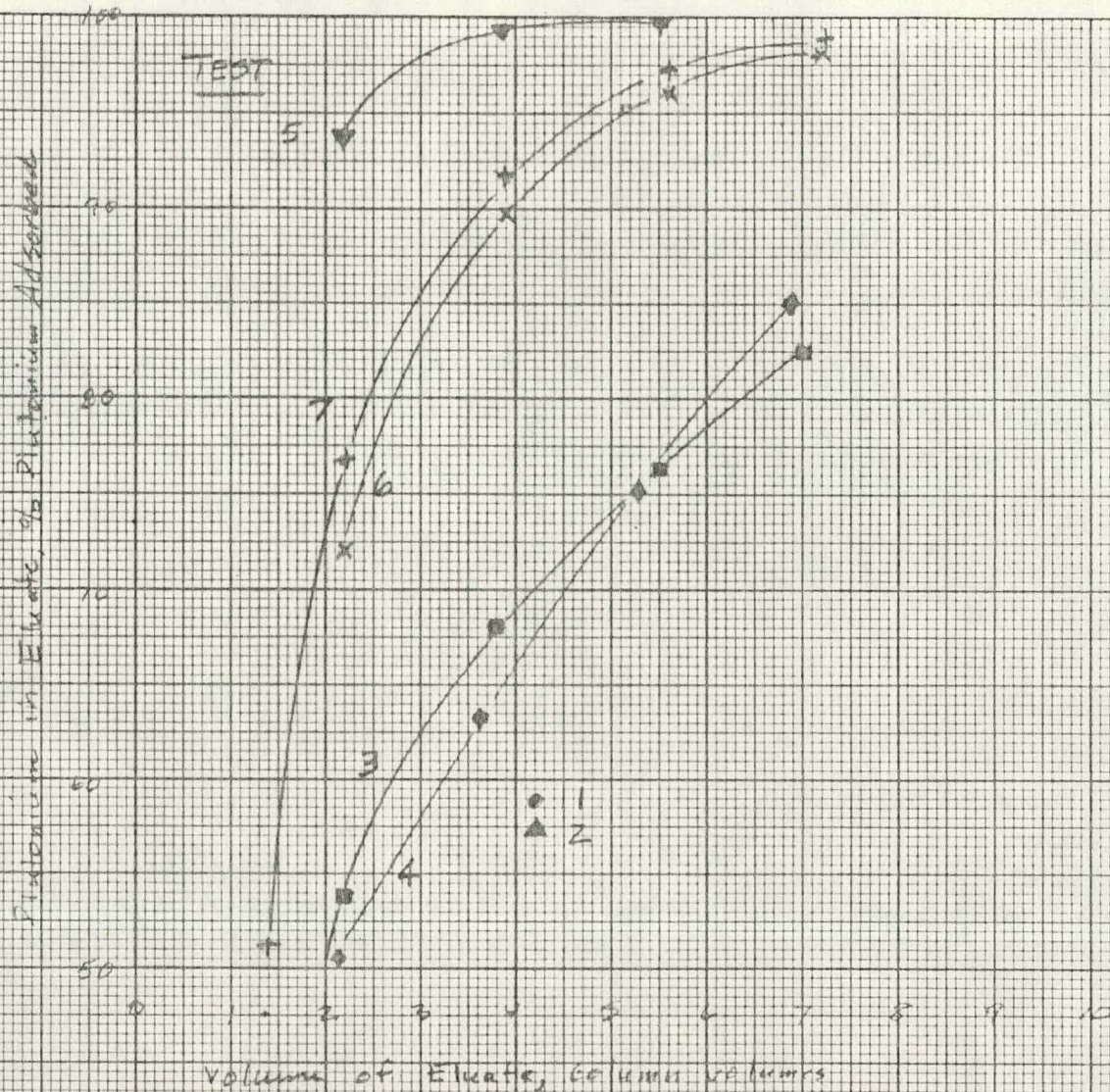Flow Rate: 0.85 ml/min-cm^2 (6 ml/min)

Volume of Elutriant: 630 ml (4.2 CV)
Temperature: 23°C
Mode: Downflow for loading and elution

Resin*	Type of MSE Waste**	Actinide in Feed		Actinide Recovery+	
		Pu, g	Am, g	Pu, %	Am, %
Dowex 50W-X8	Na-K-Mg	4.60	0.220	58.2+1.5	94.6+0.1
Dowex 50W-X8	Ca-K-Mg	3.44	0.143	68.8+1.1	88.7+0.1
Dowex 50W-X4	Na-K-Mg	2.19	0.105	99.6	99.4
Bio-Rad AG MP-50	Na-K-Mg	5.94	0.284	91	66
Bio-Rad AG MP-50	Ca-K-Mg	4.21	0.217	93	80

*   Dowex 50W-X8 and -X4 are gel-type resins; Bio-Rad AG MP-50 is a macroporous resin

**   Waste chloride solutions containing either Na or Ca and K, Mg.

\+   Mean % + standard deviation on duplicate samples, or single determination

Figure 2. Recovery of Plutonium from MSE Salt Residues by Cation Exchange – Downflow Mode

Y-axis: Plutonium in Eluate, % Plutonium Adsorbed

X-axis: Volume of Eluate, Column Volumes

TEST

Resin: 150ml (column volume), 50 to 100 mesh

Elutrient: 7M HNO_3

Flow Rates: 0.85ml/min-cm² Feed, Elution (6ml/min)
1.84ml/min-cm² Wash (13 ml/min)

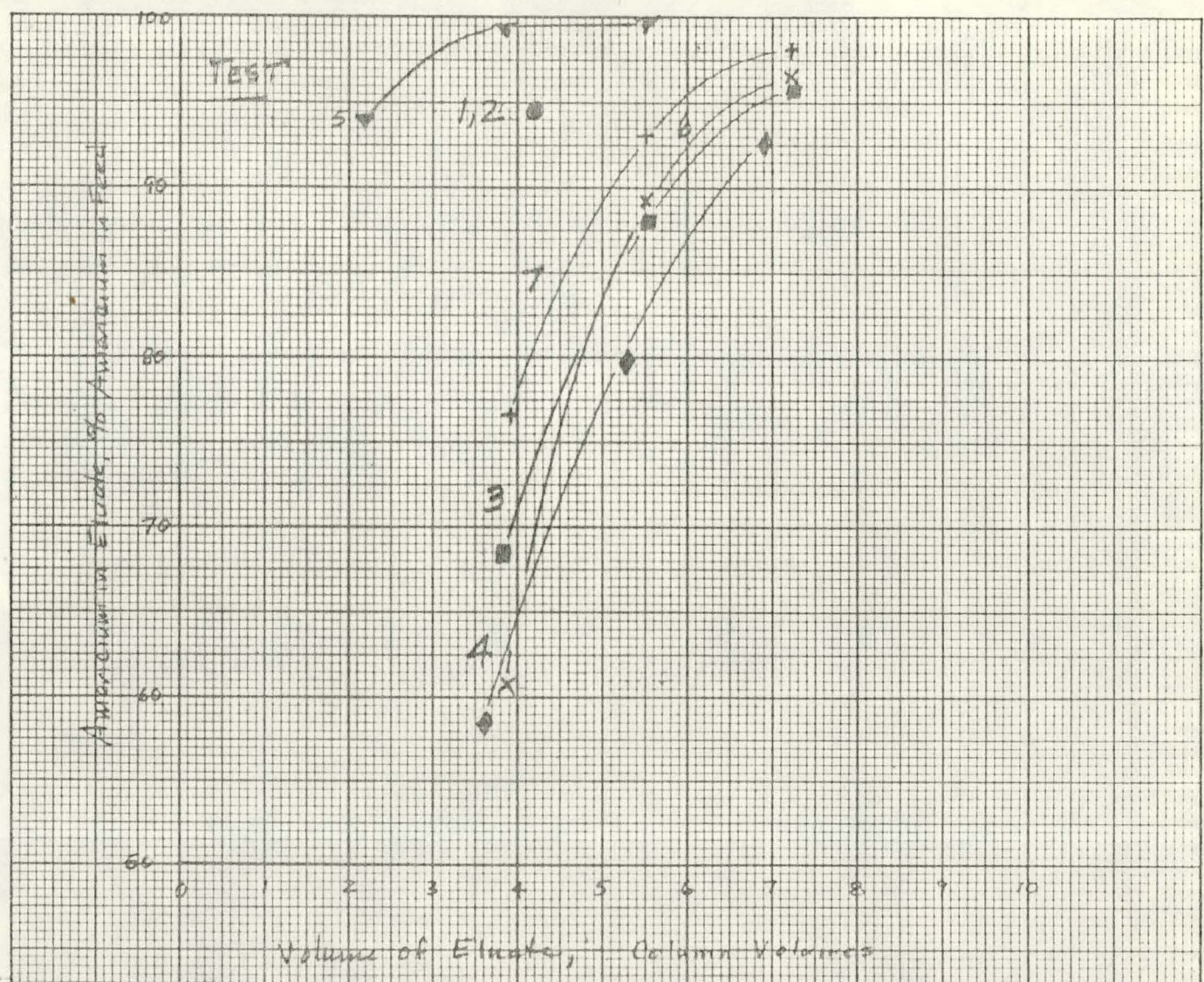Tests 1,2: Na-K-Mg Chloride Solution; 4.6 g Pu; Dowex 50W-X9 Resin

Tests 3,4: Ca-K-Mg Chloride Solution; 3.04 g Pu; " "

Test 5 Na-K-Mg Chloride Solution; 2.19 g Pu Dowex 50W-X8 Resin
Test 6 " " " 5.94 g Pu; Bio Rad AG MP-50 Resin
Test 7 Ca-K-Mg " " 5.21 g Pu; " "

Test

Volume of Eluate, Column Volumes

Resin: 150 ml (Column volume), 50 to 100 mesh

Elutriant: 7M HNO₃

Flow Rates: 0.85 ml/min·cm² Feed, Elution (6 ml/min)
            1.84 ml/min·cm² Wash   (13 ml/min)

Tests 1,2: Na-K-Mg chloride Solution; 0.122 g. Am; Dowex 50W-X8
Test 3,4: Ca-K-Mg chloride Solution; 0.123 g. Am;   "
Test 5: Na-K-Mg chloride Solution; 0.105 g Am; Dowex 50W-X4
Test 6:   "   ; 0.284 g Am; Biorad AG 1P?
Test 7: Ca-K-Mg Chloride Solution; 0.217 g Am;   "

Figure 3. Recovery of Americium from MSE Salt Residues
           By Cation Exchange - Downflow Mode

(Y-axis label: Americium in Eluate, % Americium in Feed)

plutonium. Again, it is suggested that this is because of the oxidation of Pu(III) to Pu(IV) and formation of the hexanitrato plutonium complex; Pu(IV) is more tightly held than Pu(III) and the bulky hexanitrato complex has difficulty diffusing through the polymer matrix. We cannot explain why plutonium recovery is poorer from the Na-K-Mg residue than from the Ca-K-Mg residue whereas americium recovery is better from the Na-K-Mg system and worse from the Ca-K-Mg system. The mean ± standard deviation for the duplicate tests suggest the effect is real.

The macroporous BioRad AG MP-50 resin has approximately 30% more capacity then the Dowex 50W-X8 for the Na-K-Mg solution and 50% more capacity for the Ca-K-Mg solution. This suggests that tervalent plutonium and americium can displace calcium from the macroporous resin more effectively than from the gel-type Dowex 50W-X8 resin. Another way to look at this effect is to consider the two resins individually. For Dowex 50W-X8, only 74% as much actinide can be adsorbed from the Ca-K-Mg system as from the Na-K-Mg system. With BioRad AG MP-50, 87% as much actinide can be adsorbed from the Ca-K-Mg system; calcium is therefore more effectively displaced.

Plutonium elution from BioRad AG MP-50 is faster than from Dowex 50W-X8; as noted previously, this can be explained as the result of a shorter distance for the bulky hexanitrato anion to diffuse through the matrix before reaching a channel. For plutonium, BioRad AG MP-50 macroporous resin gave the best capacity, calcium decontamination, and elution characteristics.

We have no explanation for the slower elution of americium from macroporous resin. Americium recovery from macroporous resin should be no worse than from gel-type resin; one would expect it to be better.

Because of the poor recovery of americium and because RFP operating procedures involve loading and elution in the upflow and downflow modes, respectively, four additional tests were done in this mode with Dowex 50W-X8 and BioRad AG MP-50 resins and the Na-K-Mg and Ca-K-Mg systems. The results are tabulated in Table IV and plutonium and americium elution curves are shown in Figures 4 and 5 respectively.

The analytical results for plutonium in these runs varied so the overall actinide capacities for the resins in Table III are considered more accurate. Less variation was noted in duplicate americium samples. Basing the amount of actinide sorbed on americium, the Dowex 50W-X8 adsorbed 73% as much americium from the Ca-K-Mg system as it did from the Na-K-Mg system; the BioRad AG MP-50 adsorbed 83% as much americium from the Ca-K-Mg system. The results based on americium are similar to those found during the downflow experiments.

The percent recovery of actinide during elution was calculated based on actinide fed to the column and recovered in eluate fractions. Both plutonium and americium recoveries were significantly improved by upflow loading and downflow elution. This shows that americium has an appreciable affinity for the resin even in $\underline{7}M$ HNO_3, and that plutonium may also be retained as cationic

TABLE IV

ACTINIDE ELUTION FROM CATION EXCHANGE RESIN -
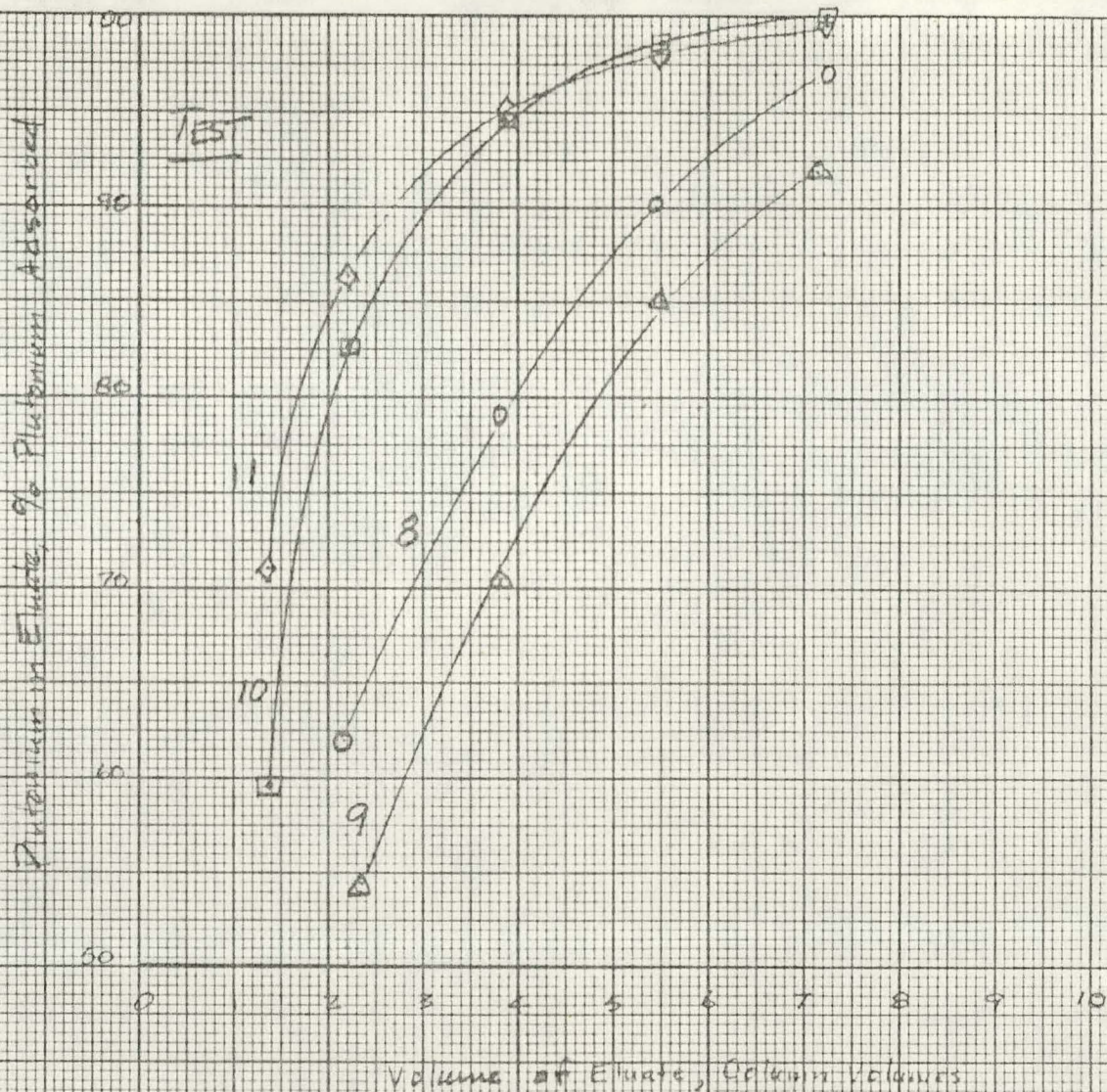### UPFLOW AND DOWNFLOW MODE

Column Volume (CV): 150 ml   Volume of Elutriant: 630 ml (4.2CV)
Elutriant: 7M HNO_3 Temperature: 23°C
Flow Rate: 0.85 ml/min-cm^2 (6 ml/min)   Mode: Load upflow; elute downflow

Resin*	Type of MSE Waste**	Actinide in Feed Pu,g	Am,g	Actinide Recovery[+] Pu, %	Am, %
Dowex 50W-X8	Na-K-Mg	4.05	0.194	82	95
Dowex 50W-X8	Ca-K-Mg	3.43	0.142	75	93
Bio-Rad AG MP-50	Na-K-Mg	5.43	0.260	96	98
Bio-Rad AG MP-50	Ca-K-Mg	5.21	0.215	96	97

*   Dowex 50W-X8 is a gel-type resin; BioRad AG MP-50 is a macroporous resin.

** Waste chloride solutions containing either Na or Ca and K, Mg.

+   Single determination

TEST

Plutonium in Eluate, % Plutonium Adsorbed (y-axis)
Volume of Eluate, Column Volumes (x-axis)

Curve labels: 11, 8, 10, 9

Resin: 150 ml (column volume), 50 to 100 mesh

Elutriant: 7M HNO₃

Flow Rates: Feed; Elution - 0.85 ml/min-cm² (6 ml/min)
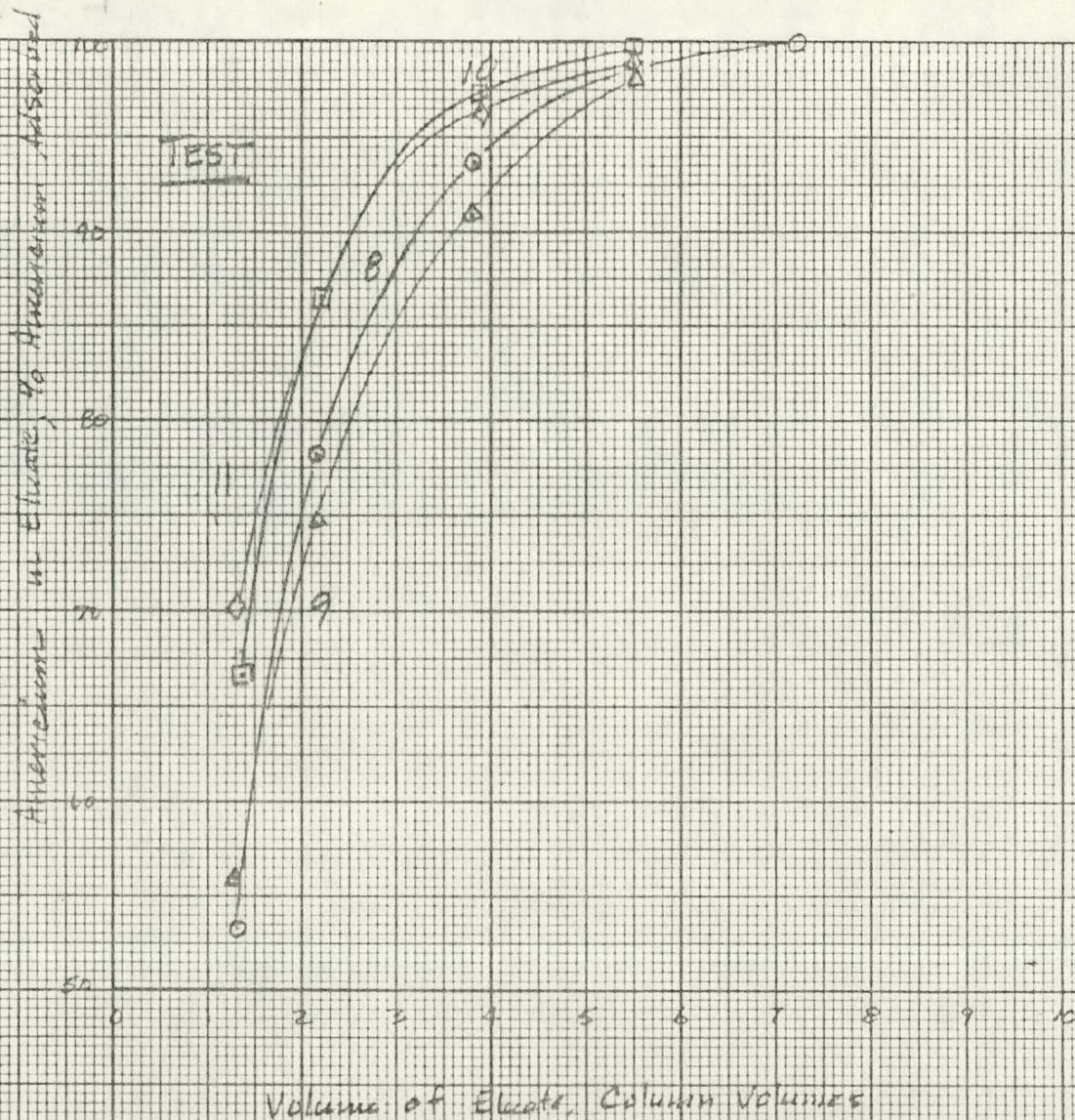   Wash - 1.84 ml/min-cm² (13 ml/min)

Test 8: Na-K-Mg Chloride Solution; 4.05 g Pu; Dowex 50W-X8 Resin

Test 9: Ca-K-Mg Chloride Solution; 3.13 g Pu; Dowex 50W-X8 Resin

Test 10: Na-K-Mg Chloride Solution; 5.135 g Pu; BioRad AG MP-50

Test 11: Ca-K-Mg Chloride Solution; 5.21 g Pu; BioRad AG MP-50

Figure 4. Recovery of Plutonium from MSE Salt Residues
By Cation Exchange - Feed Upflow, Elute Downflow

TEST

Americium in Eluate, % Americium Adsorbed (y-axis)

Volume of Eluate, Column Volumes (x-axis)

Resin: 150 ml (column volume), 50 to 100 mesh

Eluriant: 7 M HNO₃

Flow Rates: Feed, Elution — 0.55 ml/min-cm² (6 ml/min)
            Wash — 1.84 ml/min-cm² (13 ml/min)

Test 8: Na-K-Mg Chloride Solution; 0.194 g Am; Dowex 50W-X8
     9: Ca-K-Mg Chloride Solution; 0.140 g Am; Dowex 50W-X8
    10: Na-K-Mg Chloride Solution; 0.260 g Am; BioRad AG MP-50
    11: Ca-K-Mg Chloride Solution; 0.215 g Am; BioRad AG MP-50

Figure 5 Recovery of Americium from MSE Salt Residues
          By Cation Exchange – Feed Upflow, Elute Downflow

Pu(IV) within the resin matrix even though it forms the anionic hexanitrato complex in $7\underline{M}$ HNO_3. (The mechanism for this could be both the stabilization of Pu(IV) adsorbed on the ion exchange sites and the difficulty of getting six nitrate ions around the Pu(IV) ion in the matrix. Complexation with individual nitrate ions probably occurs simultaneously with diffusion through the matrix and bead channels.) Whatever the elution mechanism, it is evident that back-elution is more effective than attempting to force the actinide on through the column.

These results also suggest that (since plutonium and americium are tightly held) it might be possible to wash impurities loaded with plutonium and americium (e.g. calcium and magnesium) off the column with dilute acid (1 to $3\underline{M}$) before eluting plutonium and americium with $7\underline{M}$ HNO_3.

Conclusions

As a result of this study it was concluded that the increased actinide capacity, faster elution kinetics, and better decontamination properties of macroporous resins justify additional work to further evaluate BioRad AG MP-50 and other macroporous resins for the MSE cation process.

Bidentate Extraction

Recovery of actinides with an organophoshorus bidentate
has been proposed. The conceptual flow sheet is shown in
Figure 6. The bidentate, dihexyl-N,N-diethylcarbamoylmethy-
lenephosphonate (DHDECMP), is especially attractive since
it can recover actinides from MSE residues containing aluminum.[2]
The cation exchange process is unable to effect actinide purifi-
cation when aluminum is present. (DHDECMP extracts actinides
and lanthanides, but does not extract common RFP contaminants,
e.g. aluminum. No lanthanides are used in process streams at
RFP.)

The actinides are extracted from high acid (e.g. $7\underline{M}$ HNO_3)
solutions and can be back-extracted with dilute acid. The method
is therefore easily used with the ion column effluent from the
anion exchange plutonium recovery step.

Two techniques appear to be useful for the bidentate extraction
of actinides. The first is liquid-liquid solvent extraction, a
method which has several advantages. Currently, however, the
type of equipment needed (mixer-settlers, centrifugal contactors,
etc.) is not available at RFP. We are better equipped to use
a column technique. This can be done by sorbing the bidentate
extractant on an inert solid support, loading ion exchange columns
with the sorbent, and proceeding with column runs (extraction chro-
matography).

Both liquid-liquid extraction and extraction chromatography
were tested in this work. Lab and pilot scale recovery tests were
done using liquid-liquid extraction, and laboratory scale tests were

Figure 6

CONCEPTUAL FLOW SHEET FOR PROCESSING ~~PYROCHEMICAL~~ Aluminum RESIDUES
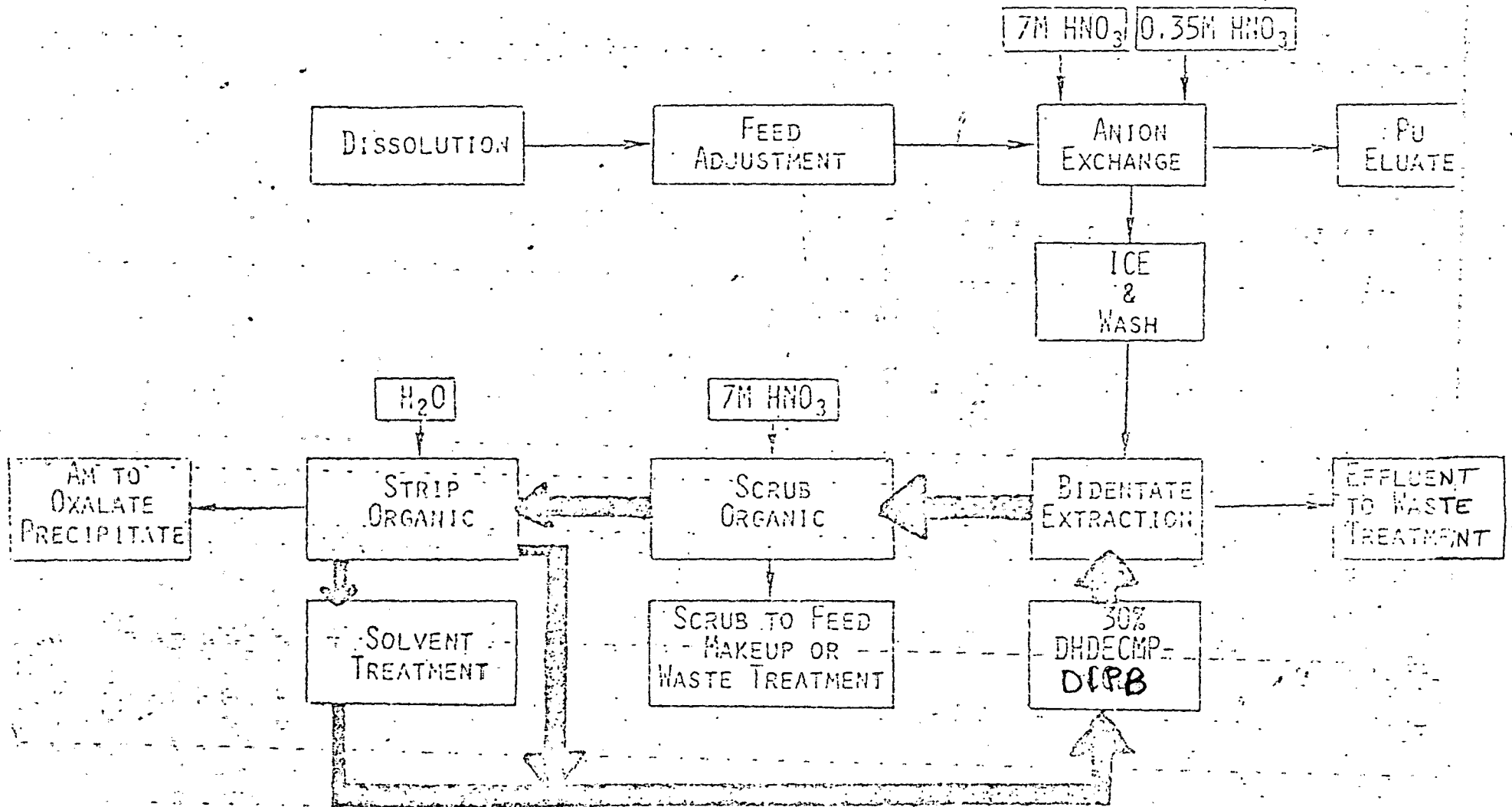
done using extraction chromatography; results of these preliminary tests are described.

Experimental

Materials. The alloy residue contains approximately 15% Al, 44% Mg, 29% Pu, 2% Am, and other impurities. The residues are dissolved by placing the alloy metal in 0.35$\underline{M}$ HNO$_3$ and slowly adding concentrated HNO$_3$. Time is allowed between additions of concentrated HNO$_3$ for the dissolution reaction to subside. The solution is adjusted to 0.1$\underline{M}$ F$^-$ with concentrated HF to remove any polymeric or residual plutonium. After filtration through Whatman®42 filter paper, the solution is adjusted to 7$\underline{M}$ HNO$_3$.

Plutonium in this feed solution is removed by an anion exchange column process. The anion exchange resin is Dowex 1-X4, 50 to 80 mesh nitrate form, obtained from BioRad Laboratories. Ferrous sulfamate (plutonium-to-iron mole ratio of five) is added to the solution to eliminate hexavalent plutonium, and the feed is passed through the column. The ion column effluent (ICE) contains the americium and impurities. Residual americium and impurities are washed from the column with 7$\underline{M}$ HNO$_3$ and the wash is combined with the ICE; this is the feed to the bidentate process. A typical composition is given in Table V. Plutonium is eluted with dilute nitric acid and returned to the production stream.

The DHDECMP is obtained from Wateree Chemical Company, Lugoff, South Carolina, and is purified by an ion exchange method to approximately 67% pure; the method has been described elsewhere.[3] Diisopropylbenzene (DiPB) was obtained from Pfaltz and Bauer, Inc. The extractant is 30% DHDECMP in DiPB.

TABLE V

Average Feed from Dissolution of Al-Mg Alloy

(concentrations in g/l)

Am	Pu	Al	Mg
0.15	8.17×10^{-3}	0.385	1.02

The extraction chromatography method used DHDECMP adsorbed on 20-50 mesh Amberlite[R] XAD-4. The adsorbent was prepared using a column method. The column was loaded with Amberlite XAD-4, washed with acetone, and a mixture of 50 vol% CCl_4 - 50 vol% bidentate was recycled through the column to cause swelling of the beads. The CCl_4-bidentate mixture was then vacuumed from the column and the column was filled with 67% bidentate and permitted to stand over-night. The excess bidentate was vacuumed from the column and the adsorbent was conditioned by washing with $7\underline{M}$ HNO_3.

Procedure. Americium in the combined wash and ICE is removed by liquid-liquid extraction or extraction chromatography using bidentate.

For liquid-liquid extraction, 2.5 ℓ of feed was contacted with 0.5 ℓ of bidentate (A/O = 5) for 15 minutes. This batch process is repeated with fresh batches of extractant until the americium concentration is $<1.9 \times 10^{-2}$ g/l; three contacts were required.

Residual amounts of extracted impurities are removed from the loaded extractant by $7\underline{M}$ HNO_3 washing. Three to five contacts are made using an A/O = 0.2. The wash is either sent to feed

acid adjustment or discarded depending upon americium content. The americium is then stripped from the organic with water (five to seven contacts) using an A/O = 0.2. The americium in the strip solution is precipitated with oxalic acid, and the americium oxalate is calcined to AmO_2. In the lab scale extraction chromatography method, the DHDECMP-impregnated Amberlite XAD-4 was loaded into a column 1.8 cm dia. x 23.5 cm and conditioned with $7\underline{M}$ HNO_3. The americium feed was fed to the column at 1.5 ml/min until a total of 300 ml had been fed. The column was washed with 200 ml of $7\underline{M}$ HNO_3 at 1.7 ml/min, then eluted with deionized water until elution was complete (determined radiometrically). Five to seven 50 ml fractions were required for elution. Samples were submitted for analysis.

Results and Discussion

Table VI shows the results of three laboratory runs of the solvent extraction step (after the plutonium was removed by anion exchange). The major elements are shown before solvent extraction, after solvent extraction, in the $7\underline{M}$ HNO_3 wash, and in the final strip product. The americium remaining in the organic after stripping is also shown. Although there were some analytical discrepancies, the data show that americium was effectively recovered (except in Test 1, for which we have no explanation). Americium was decontaminated from aluminum and magnesium in all three runs.

Results of the pilot plant test of the liquid-liquid solvent extraction test are shown in Table VII. Only 4 ℓ of approximately

TABLE VI

Recovery and Purification of Am from Al-Mg
MSE Residue by Solvent Extraction

Test	Stream	Am, mg	Pu, mg	Al, mg	Mg, mg
1	Feed	21	0.39	149	432
	Raffinate	1	<0.001	143	405
	Wash*	4	<0.001	3	8
	Product**	8	0.06	<0.001	<0.001
	Organic[+]	2	NA[++]	NA	NA
2	Feed	23	0.04	158	459
	Raffinate	1	NA	160	470
	Wash*	1	NA	2	6
	Product**	18	0.004	<0.113	<0.113
	Organic[+]	0.4	NA	NA	NA
3	Feed	44	0.07	211	713
	Raffinate	1	0.01	242	634
	Wash*	1	0.05	<2	8
	Product**	42	0.03	<2	1
	Organic[+]	2	0.36	NA	NA

* $7\underline{M}$ HNO_3 ** Strip   [+] After Strip   [++] Not Available

17 ℓ of americium strip product were used in the precipitation step; the remainder was turned over to Production for final processing. Decontamination from Al and Mg was excellent with decontamination factors (DF) of 72 and 444, respectively. The corresponding DF for plutonium was 10. This is higher than was expected, since bidentate extracts plutonium much more effectively than americium.

Analysis of the final AmO_2 product is shown in Table VIII. Al and Mg were below the detectable limits for these elements. Plutonium was present at a concentration of 3.24 mg/g product. The product met specifications of >95% AmO_2 with less than 0.5% Pu and less than 1% of any other single contaminant. The results of the preliminary lab scale extraction chromatography tests are shown in Table IX. Again, in spite of some analytical problems, it is evident that americium was decontaminated from aluminum and magnesium. A $7\underline{M}$ HNO_3 wash step is assumed to account for the americium loss.

Conclusions

Laboratory results have shown that americium can be recovered and purified by both solvent extraction and extraction chromatography using the bidentate DHDECMP. Solvent extraction recovery of americium was demonstrated on a pilot plant scale. The americium oxide product prepared from this strip solution met specifications.

TABLE VII

Results of Pilot Scale Recovery of Americium
from Dissolved Al-Mg Alloy from MSE Process

Stream	concentration, g/l *			
	Am	Pu	Al	Mg
Feed	0.15	8.17×10^{-3}	0.385	1.02
Strip	0.14	7.5×10^{-4}	0.005	0.002

* Single determination

TABLE VIII

Impurities in Americium Oxide Product*

Impurity Concentrations, ppm**		
Plutonium	Aluminum	Magnesium
3240	<100	<50

* Specifications require >95% AmO_2 with less than 1% of
  any individual impurity and less than 0.5% Pu

** Single determination

TABLE IX

Recovery and Purification of Am from Al-Mg

MSE Residue by Extraction Chromatography

Test	Stream	Am, mg	Pu, mg	Al, mg	Mg, mg
1	Feed	35	0.04	81	204
	Effluent	2	3×10^{-4}	89	200
	Wash*	4	+	+	+
	Product**	22	0.3	0.4	0.18
2	Feed	17	0.02	39	97
	Effluent	0.037	0.0015	25	65
	Wash*	+	+	+	+
	Product**	16	0.2	<0.2	0.2
3	Feed	29	0.04	68	170
	Effluent	0.4	0.003	57	+
	Wash*	3.5	0.0012	+	+
	Product**	25	0.2	<0.45	<0.23

* $7\underline{M}$ HNO$_3$

** Strip

+ Not available

Acknowledgment

References

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3.   J. D. Navratil, et al., "Waste Management Analysis for the Nuclear Fuel Cycle - Parts I and II. Progress Report for Period April 1 to September 30, 1977", USDOE Report RFP-2749, Rockwell International, Energy Systems Group, Rocky Flats Plant, Golden, Colorado, October 23, 1978.