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ISOLATING ^{241}Am FROM WASTE SOLUTIONS CONTAINING Al, Ca, Fe, AND Cr

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ABSTRACT

About 2.4 kg of ^{241}Am contaminated with calcium and aluminum had been recovered from low-activity waste during recycle of 11% ^{240}Pu. A process was developed and demonstrated to purify the americium before shipment as $^{241}AmO_2$. The americium and some of the calcium were batch extracted into 50% TBP-n-paraffin from 2.2M $Al(NO_3)_3$ - 0.3M HNO_3 solution in a canyon tank. Pregnant solvent was scrubbed first with 2.1M Al^{3+}-0.3M Li^+-6.7M NO_3^- and then with 7M $LiNO_3$ to reduce the calcium content and to displace the aluminum. Americium was then stripped from the solvent with water and concentrated by evaporation. Before precipitating the americium with oxalic acid, the nitric acid was adjusted with NH_4OH to yield a 1M NH_4NO_3 solution. Recovery across the batch extraction step was 97.8%, while 93% of the calcium and >99% of the aluminum was rejected. Recovery across precipitation averaged >96% while producing a product which was >99.3% pure $^{241}AmO_2$. The major impurities were water, carbon, calcium, iron, and zinc.

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INTRODUCTION

Extensive research[1-1/] has been devoted to methods of separation of americium isotopes from various contaminants. The method used is determined by (1) the composition of the starting material, (2) the amount of material to be handled, (3) the availability of appropriate equipment, (4) the time constraints of product delivery to a customer, (5) the purity required by the customer, and (6) the availability of chemicals of sufficient purity to carry out the scheme selected.

Previous campaigns[12-1/] at the Savannah River Plant (SRP) to produce kilogram amounts of $^{241}AmO_2$ have used a combination of solvent extraction, cation exchange, and oxalate precipitation to produce acceptably pure product. In general, the contaminants removed in the earlier campaigns have been plutonium, sodium, iron, chromium, nickel, aluminum, sulfate ion, and nitric acid. For the americium purification campaign reported herein, the major contaminants removed were aluminum, chromium, iron, calcium, formic acid, and nitric acid.

As a result of rapid formation of Al(III) and Fe(III) anionic complexes with oxalate ion and the very slow kinetics of the reaction between oxalate ion and Cr(III), separation of americium from those three elements can be accomplished fairly easily by oxalate precipitation. Calcium oxalate, however, is a good carrier for americium oxalate.[8] In fact, calcium oxalate and uranium-(IV) oxalate are perhaps the best carriers for the recovery of americium from very dilute solutions.[8] Therefore, sufficient separation of americium and calcium to produce high purity product by oxalate precipitation is difficult.

Two techniques are, however, useful for americium purification: prior separation of calcium-americium (by either solvent extraction or ion exchange) or selectively masking the calcium before precipitation with oxalic acid. The sequence of methods chosen at the SRP for the separation was batch solvent extraction followed by selective masking of residual calcium with ammonia prior to oxalate precipitation of americium oxalate.

CONCEPTUAL PROCESS

Because it was necessary to use a process that would work in existing equipment, a process was designed involving the following operations:

- **Acid Adjustment.** The excess formic acid was oxidized by heating with nitric acid to yield a solution which contained low concentrations of both formic acid and nitric acids.

- **Aluminum Nitrate Adjustment.** Technical grade 60% aluminum nitrate nonhydrate (60% ANN) was added, and the solution evaporated to yield a 2.2M $Al(NO_3)_3$ solution.

- **Solvent Preparation.** A solution of 50% tri-n-butyl phosphate (TBP) was prepared in a normal paraffin hydrocarbon (NPH). This solution (4000 liters) was washed with 4% $NaHCO_3$ and with 0.75% HNO_3.

- **Batch Extraction.** The adjusted feed was transferred to the extraction tank followed by two small aluminum nitrate flushes to remove the americium in the liquid heel and combined with the solvent in the extraction tank. The aqueous to organic ratio at this point was about 1. The solvent and aqueous was agitated to extract the americium into the solvent, the phases were allowed to separate, and the aqueous raffinate steam-jet decanted to a waste-hold tank. Americium concentration in the solvent was about 0.6 gram/liter.

- **Decanting.** To minimize the aqueous heel left behind, an instrument jumper was fabricated and installed, which decreased the instrument liquid level heel to 1.5 inches. A 12-gpm jet was fabricated and installed to decant the aqueous waste from beneath the settled solvent. The aqueous volumes remaining in the tank at instrument heel and at jet heel was determined to be 42 liters and 20 liters, respectively.

  During decanting operations, solution was removed until the specific gravity monitor fell to the specific gravity of solvent (<42 liters of aqueous), and the steam-jet cut off before sufficient additional solution had been removed to lower the solvent to the 20-liter jet heel.

- **Scrubbing with $Al(NO_3)_3$ Solution.** Because there was an aqueous heel left behind in the tank used for batch extraction and because calcium does extract (D_a^o= 0.614) into the organic phase, four scrub steps using 2.1M Al^{3+}- 0.3M Li^+ - 6.6M NO_3^- solutions were performed. These steps removed the bulk of iron, chromium, and calcium.

- **Scrubbing with $LiNO_3$ Solution.** To minimize the volume of solution to be handled in the finishing process, Al^{3+} was displaced from the aqueous heel in the tank by two small-volume 7M $LiNO_3$ scrubs.

- **Stripping.** Three low acid (<0.05M HNO_3) strips were used to remove the americium from the organic phase.

- **First Evaporation, Acid, and Ammonium Nitrate Adjustment.** The strip solution was combined and evaporated to a volume of about 2500 liters. At this point, the acid was adjusted by ammonium hydroxide addition to yield the desirable concentrations at the finishing volume.

- **Organic Wash.** To reduce the possibility of solids produced via the chemical and radio-degradation of the TBP dissolved in the aqueous solution, a one contact batch extraction with NPH was made.

- **Second Evaporation.** At this point, the solution was transferred from the large canyon evaporator to the smaller finishing line evaporator, and the large evaporator was flushed with dilute acid to move all products to the smaller evaporator. The volume was reduced to the precipitator feed concentrations.

- **Finishing.** The adjusted americium concentrate was precipitated in small batches by the addition of 0.9M oxalic acid, digested at ambient temperature, filtered, washed, air dried, calcined at 700°C, and packaged for shipment.

LABORATORY DEMONSTRATIONS

Each step of the process was demonstrated on a laboratory scale by using dysprosium as a stand-in for americium. Once the process had been optimized, actual plant solutions and technical grade chemicals were used to demonstrate the process on a laboratory scale. After plant completion of the batch extraction phase, actual plant solutions were obtained, and the finishing process was again demonstrated on a laboratory scale.

RESULTS

Batch extraction had been used before on a plant scale to isolate ^{243}Am and ^{244}Cm from $Al(NO_3)_3$ - $NaNO_3$ - HNO_3 solutions.[10] It was, therefore, already known for good recovery of the americium, the aqueous solution would have to be adjusted to 6.5-6.8M inextractable nitrate and 0.10-0.35M HNO_3 before extracting the americium with an equal volume of 50 vol % TBP in a normal paraffin hydrocarbon diluent. In this previous campaign, the americium-calcium was back extracted into 0.2M HNO_3 with an overall americium-calcium recovery of 95%. The distribution of calcium across the batch extraction was not known. It was, therefore, necessary to optimize the extraction-scrub sequence to minimize calcium carry-over into the product.

Batch Extraction

Literature sources indicate that it is possible to prepare a 0.99M solution of $Ca(NO_3)_2$ in pure TBP.[18] Rainey[19, 9] developed a continuous countercurrent 30% TBP extraction process for the recovery of grams of americium from Handford slag and crucible waste. Following dissolution of the material in HNO_3-HF media, Pu(IV) was first recovered by extraction with 30% TBP. The excess 5M HNO_3 in the aqueous americium containing raffinate from the plutonium extraction step was neutralized to 0.01M with gaseous ammonia. The resulting feed solution, which contained about 4.9M NH_4NO_3, 0.28M $Al(NO_3)_3$, 0.16M $Ca(NO_3)_2$, and 1.0M $Mg(NO_3)_2$, was contacted with a 30% TBP solution to extract americium. Because both calcium and magnesium are slightly extractable with 30% TBP (distribution coefficients are about 0.068 for calcium and 0.0045 for magnesium), scrubbing of the organic extract was necessary. Although the distribution coefficients are small, this is sufficient extractability to give a calcium plus magnesium concentration of about 10 grams/liter in the unscrubbed product, while the americium concentration is only 5×10^{-4} grams/liter. Scrubbing with 3M ammonium nitrate reduced the calcium plus magnesium content of the final product to less than 0.01 gram/liter. The distribution coefficient of ammonia from the 3M NH_4NO_3 solution was 0.003, thereby, giving about 1.2 grams/liter NH_4NO_3 in the product when the americium was stripped into water. This process was satisfactorily tested in pulse column runs with actual slag and crucible waste solution, but no production-scale use of this separation has been made to date.

Data for $Ca(NO_3)_2$ distribution into 50% TBP solution planned for use in the production scale purification, however, were not available from the literature. Under the conditions of the proposed extraction (2.1M $Al(NO_3)_3$ - 0.3M HNO_3), the distribution coefficient was determined to be 0.614.

Scrubbing Sequences

With the determined distribution coefficient, the theoretical scrub steps necessary to reduce the calcium-americium ratio from about 1 to <0.005 was calculated to be 6 (Table 1). However, several practical problems invalidate the theoretical number:

TABLE 1

Theoretical Calcium Distribution

	Calcium Content, kg			
	Phases at Equilibrium		Phases at Heel Volumes**	
Step	Organic	Aqueous	Organic	Aqueous
Before extraction	-	2.750	-	-
Extraction	1.046	1.704	1.046	0.021
Scrub 1***	0.406	0.661	0.406	0.008
Scrub 2	0.157	0.257	0.157	0.003
Scrub 3	0.061	0.099	0.061	0.001
Scrub 4	0.024	0.038	0.024	0.0005
Scrub 5	0.009	0.015	0.009	0.0002
Scrub 6	0.003	0.006	0.003	0.0001
Scrub 7	0.001	0.002	0.001	<0.0001

---

* To assure a product in solution where Ca/Am <0.005, no more than 5 g Ca could remain after scrubbing.

** A 50-L heel aqueous volume is assumed; at equilibrium, the aqueous and organic volumes are assumed to be 4000 L.

*** All aqueous solutions are 2.1M $Al(NO_3)_3$.

1. The batch extraction was to be done in a tank with an aqueous heel of 50-100 liters left behind after aqueous phase decanting.

2. Chemicals obtainable in bulk quantities to carry out the plant scale process contained calcium as an impurity.

With these physical limitations on the scrub steps, the optimum number of scrubs was both calculated (Table 2) and demonstrated to be 4 (Table 3). Even reagent grade chemicals contained sufficient calcium to render more than 5 scrubs useless.

In an attempt to increase the scrub efficiency, $LiNO_3$, which is known to extract into 50% TBP,[18] was added to the scrub solution. Lithium did improve phase separation; it did not, however, appreciably affect the distribution of calcium. As a result of the enhancement of phase separation, the optimized scrub operation used 2.1M $Al(NO_3)_3$ - 0.3M $LiNO_3$ solution (Table 4).

Aluminum Removal

Previous studies have shown that to obtain high yields of $Am_2(C_2O_4)_3(S)$, the aluminum concentration of the precipitating solution must be less than 0.25M.[13,14,6] These studies had also shown that a 40°C digestion cycle was necessary to obtain crystals of sufficient size to settle well and, therefore, filter rapidly. These data indicated that removal of the 100 to 225 moles of $Al(NO_3)_3$ remaining in the aqueous heel at the end of the scrub steps was necessary for optimum processing rate in the finishing phase of the campaign. Simple calculations show that two one-quarter scrub steps using 7M $LiNO_3$ would remove about 99% of the Al(III) from the tank (Table 5).

The optimized scrub sequence then contained four equal volume 2.1M $Al(NO_3)_3$ - 0.3M $LiNO_3$ scrubs followed by two quarter-volume 7M $LiNO_3$ scrubs. Both calculations from the determined distribution coefficient for calcium and laboratory demonstrations indicated that this sequence would remove ~90% of the calcium and all but about 2 to 4 moles of aluminum from the system (see Table 6).

Finishing Operations

Specifications for the final americium oxide requires a calcium-americium ratio of ≤0.005. Preliminary laboratory demonstrations runs, however, indicated the calcium-americium ratio of the solution transferred to the finishing operation would be about 0.10 to 0.12. Use of the finishing flowsheet used in the previous

TABLE 2

Expected Calcium Distribution with Obtainable Chemicals*

Step	Calcium content, kg			
	Phases at Equilibrium		Phases at Heel Volumes**	
	Organic	Aqueous	Organic	Aqueous
Before extraction	-	2.750	-	-
Extraction	1.046	1.704	1.046	0.021
Scrub 1	0.742	1.209	0.742	0.015
Scrub 2	0.480	0.871	0.480	0.010
Scrub 3	0.378	0.616	0.378	0.008
Scrub 4	0.339	0.551	0.339	0.007
Scrub 5	0.323	0.527	0.323	0.007
Scrub 6	0.317	0.517	0.317	0.006
Scrub 7	0.315	0.512	0.315	0.006
Scrub 8	0.314	0.511	0.314	0.006
Scrub 9	0.313	0.511	0.313	0.006
Scrub 10	0.313	0.510	0.313	0.006

---

* Al scrub solution contains 126 ppm Ca.

** Aqueous heel volume assumed to be 50 L.

TABLE 3

Laboratory Demonstration of Calcium Removal by Scrubbing the Organic Phase

	Calcium Concentration in Aqueous Solution, g/L	
	2.2M $Al(NO_3)_3$ Scrub	2.1M Al-03M Li Scrub
Feed Stock Before Extraction	1.049*	1.049
Extraction Raffinate	0.706	0.694
Scrub 1	0.536	0.310
2	0.203	0.140
3	0.167	0.064
4	0.143	0.029
5	0.128	0.016
6	0.135	0.009
7	0.117	0.013
8	0.120	–
Strip 1	0.097	0.004
2	0.004	0.002

---

* Analysis of solution were by Atomic Absorption.

TABLE 4

Calcium Scrubbing by Various Salting Agents

	Calcium Concentration in Scrub Solutions, g/L			
	2.2M Al	7M Li	2.0M Al - 0.6M Li	2.1M Al - 0.3M Li
Feed Stock Before Extraction	1.00	1.00	1.00	1.00
Raffinate	0.694	0.695	0.693	0.698
Scrub 1	0.108	0.464	0.324	0.282
2	0.106	0.184	0.168	0.162
3	0.067	0.137	0.088	0.059
4	0.061	0.071	0.061	0.029
5	0.074	0.048	0.050	0.037
6	0.047	0.047	0.040	0.016
7	0.047	0.048	0.080	0.013

TABLE 5

Aluminum Removal from System

	Moles Aluminum Remaining Assuming	
	100-L heel	50-L heel
Number of Li Scrubs	210	105
Full-volume Li Scrubs		
1st	5.25	1.72
2nd	0.13	0.02
Half-volume Li Scrubs		
1st	10.50	2.63
2nd	0.53	0.07
Quarter-volume Li Scrubs		
1st	21.0	5.25
2nd	2.1	0.26

TABLE 6

Laboratory Demonstration of Optimized Batch Extraction Flowsheet (Reagent Grade $Al(NO_3)_3$)

	Calcium Concentration in Aqueous Solution, g/L	Ca/Am Ratio
Prepare ^{241}Am Feed Solution	1.078	112.3
Extraction Raffinate	1.089	
Al scrub*		
1	0.052	
2	0.017	
3	0.056	
4	0.014	
Al-Li Scrub**		
1	0.0	
2	0.0	
3	0.003	
Combined Strips	0.036	
Evaporated Strips	0.046	4.8***

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* $Al(NO_3)_3$ scrub solution contained 2 ppm Ca.

** Al-Li scrub solution contained 6 ppm Ca.

*** Ca/Am ratio in the final product must be $\leq$0.005.

campaign reduced this ratio to the range 0.035 to 0.075. Clearly, masking of calcium by some method was needed for the finishing operation.

A survey of the literature indicated CaC_2O_4 solubility[20] was enhanced by the addition of NH_4NO_3. A survey of stability constants indicated NH_3 does form complexes with calcium in 2M NH_4NO_3 solutions. A grid of precipitation experiments was set up by using $Dy(NO_3)_3$ as a stand-in for americium with calcium; NH_4NO_3 and HNO_3 concentrations varied. The simplified grid is given in Table 7. The grid was designed not only to find conditions under which a shipable product could be precipitated but also where product losses would be low and the product filterable at a reasonable rate. The data for the portion of the grid which was used in the actual process are shown in Tables 8 and 9. The conditions which produce both a rapidly filtering and shipable product and had acceptable solubility losses were ~0.75M HNO_3, ~1M NH_4NO_3, and ~3 grams/liter americium.

SUMMARY OF PLANT PROCESSING

Batch Extraction

The calcium and aluminum contaminated ^{241}Am solution was adjusted to 2.2M $Al(NO_3)_3$ - 0.2M HNO_3 before batch extraction into 50% TBP. The aqueous raffinate was removed by transferring it from beneath the solvent by using a steam jet. To remove the contaminants, the ^{241}Am containing solvent was scrubbed four times with 2.1M $Al(NO_3)_3$ - 0.3M $LiNO_3$ solution and two times with 7M $LiNO_3$. Losses of ^{241}Am to the raffinate and scrubs were small (Table 10). After scrubbing, the ^{241}Am was removed from the solvent by two process water strips and one 0.05M HNO_3 strip. The strips were transferred from beneath the solvent, by using the steam jet, into an evaporator for concentration. Total losses for the batch extraction process were 2.2%. Impurity levels before and after the batch extraction are given in Table 11.

Acid and Ammonium Nitrate Adjustment

To obtain the required acid and NH_4NO_3 concentrations for the feed to precipitation, NH_4OH was added. Concentrations before adjustment, after adjustment in the canyon tank, and calculated concentration after evaporation in the MPPF evaporator are given in Table 12.

TABLE 7

Canyon Precipitation Grid

Component	Concentrations, M			
HNO_3	0.10	0.25	0.50	0.75
NH_4NO_3	0.00	0.50	1.00	2.00
$Ca(NO_3)_2$	0.0041	0.012		
$Pb(NO_3)_2$	0.0041	0.012		
$Pb(NO_3)_2$	0.00075			
$LiNO_3$	0.6			
$Dy(NO_3)_3$	0.0124			

TABLE 8

Dysprosium Loss and Calcium Rejection Data for Small Volume, Ammonium Nitrate Masked Precipitation Grid

Solution Number	Molar Conc. of Feed Solution			Concentration, ppm of Supernate Solution	
	HNO_3	NH_4NO_3	$Ca(NO_3)_2$	Dy	Ca
M-1	0.10	0.00	0.01236	4.3 ±0.2	-
M-2	0.10	0.50	0.01236	0.2 ±0.1	114
M-3	0.10	1.00	0.01236	<0.1	96
M-4	0.10	2.00	0.01236	0.8 ±0.1	-
M-5	0.10	0.00	0.00412	4.8 ±0.2	34
M-6	0.10	0.50	0.00412	1.2 ±0.1	72
M-7	0.10	1.00	0.00412	0.7 ±0.1	77
M-8	0.10	2.00	0.00412	3.1 ±0.2	88
M-9	0.25	0.00	0.01236	4.3 ±0.2	-
M-10	0.25	0.50	0.01236	0.9 ±0.1	258
M-11	0.25	1.00	0.01236	3.0 ±0.2	276
M-12	0.25	2.00	0.01236	7.0 ±0.3	219
M-13	0.25	0.00	0.00412	8.2 ±0.3	50
M-14	0.25	0.50	0.00412	16.6 ±0.4	88
M-15	0.25	1.00	0.00412	4.1 ±0.2	93
M-16	0.25	2.00	0.00412	1.3 ±0.1	182
M-17	0.50	0.00	0.01236	11.0 ±0.3	145
M-18	0.50	0.50	0.01236	3.1 ±0.2	279
M-19	0.50	1.00	0.01236	9.3 ±0.3	266
M-20	0.50	2.00	0.01236	4.4 ±0.2	282
M-21	0.50	0.00	0.00412	7.3 ±0.3	93
M-22	0.50	0.50	0.00412	3.2 ±0.2	83
M-23	0.50	1.00	0.00412	1.8 ±0.1	90
M-24	0.50	2.00	0.00412	2.1 ±0.3	-
M-25	0.75	0.00	0.01236	12.4 ±0.3	145
M-26	0.75	0.50	0.01236	9.5 ±0.3	258
M-27	0.75	1.00	0.01236	4.2 ±0.2	269
M-28	0.75	2.00	0.01236	3.4 ±0.2	269
M-29	0.75	0.00	0.00412	10.5 ±0.3	82
M-30	0.75	0.50	0.00412	6.7 ±0.3	88
M-31	0.75	1.00	0.00412	3.7 ±0.2	86

$Dy(NO_3)_3$ = 0.01245M; $Pb(NO_3)_2$ = 0.00075M; $LiNO_3$ = 0.6M;
NA = no analysis.

TABLE 9

Final Product Purity Using the Small Volume, Ammonium Nitrate Masked Precipitation Grid

Solution No.	Molar Concentration of Feed			Impurities in final oxide product, ppm		
	HNO_3	NH_4NO_3	$Ca(NO_3)_2$	Ca	Li	Pb
M-1	0.10	0.00	0.01236	NA	NA	NA
M-2		0.50		1180	10	26390
M-3		1.00		2345	9	45930
M-4		2.00		NA	NA	NA
M-5		0.00	0.00412	2250	4	58620
M-6		0.50		390	80	7640
M-7		1.00		850	25	4090
M-8		2.00		615	40	9500
M-9	0.25	0.00	0.01236	NA	NA	NA
M-10		0.50		660	11	8135
M-11		1.00		1250	13	24325
M-12		2.00		460	5	7405
M-13		0.00	0.00412	9330	1	46640
M-14		0.50		779	10	14705
M-15		1.00		300	5	8270
M-16		2.00		650	35	13010
M-17	0.50	0.00	0.01236	13790	9	65135
M-18		0.50		380	12	6655
M-19		1.00		445	20	5010
M-20		2.00		525	25	4655
M-21		0.00	0.00412	1485	15	27200
M-22		0.50		425	20	11290
M-23		1.00		850	15	8730
M-24		2.00		NA	NA	NA
M-25	0.75	0.00	0.01236	15640	8	42265
M-26		0.50		NA	NA	NA
M-27		1.00		585	7	4880
M-28		2.00		460	9	4590
M-29		0.00	0.00412	360	13	2305
M-30		0.50		570	10	5170
M-31		1.00		310	8	4610
M-32		2.00		325	7	5875

$Dy(NO_3)_3$ = 0.01245M; $Pb(NO_3)_2$ = 0.00075M; $LiNO_3$ = 0.6M;
NA = no analysis.

TABLE 10

^{241}Am Loss to Waste

	Americium Losses, g	
	Tank 16.3	Tank 16.2
Raffinate	12.0	12.0
Al-Li Scrubs		
1	5.4	7.7
2	5.0	5.5
3	11.3	10.2
4	13.0	8.4
Li Scrubs		
1	2.6	4.5
2	1.6	
Total	50.9 (2.2%)	48.3 (2.1%)

TABLE 11

Canyon Batch Extraction Purification

Component	Component Weight, g Before Extraction	 After Extraction
Am^{3+}	~2484	2361
Al^{3+}	~3000	0.2
Ca^{2+}	~2800	190
Fe^{3+}	~2000	<0.1
Cr^{3+}	~2000	<0.1
Ni^{2+}	~800	<0.1
Pb^{2+}	~20	<1
SO_4^{2-}	~1000	90
Li^+	Trace	~8000

TABLE 12

MPPF Feed Adjustment

	Before Acid adjustment	After Acid adjustment	After Concentration
Volume, L	2,480	2,720	600
HNO_3, M	0.55	0.18	0.8
NH_4NO_3, M	---	0.38	1.7
^{241}Am, g/L	0.9	0.8	3.7

Hydrocarbon Wash

A demonstration run, by using the feed after the canyon adjustment but before the MPPF evaporation, yielded white waxy solids during evaporation. A single batch wash with normal paraffin hydrocarbon (NPH) removed the TBP precursor of these solids. A one-fourth volume NPH wash was used for the canyon product.

TRANSFER AND EVAPORATION

After the final product wash, the product solution was transferred in batches to the smaller MPPF evaporator for the final evaporation. Then, to remove the aqueous heel from the storage tank, approximately 0.05M nitric acid was used as a flush. However, due to the misvalving, a portion of 10M HNO_3 was added to the product solution. Whereas the final nitric acid concentration had been expected to be 0.8M, the actual final nitric acid concentration obtained was 2.2M. It was, therefore, necessary to dilute this solution with water before the precipitation with oxalic acid. Composition of the actual MPPF feed is given in Table 13.

FINAL PRODUCT LABORATORY DEMONSTRATION

As a final check on the scaleup of the developed flowsheet, a laboratory demonstration of the MPPF portion of the flowsheet was run with feed prepared by the canyon batch extraction. Using a single precipitation and a small volume HNO_3 - $H_2C_2O_4$ wash of the precipitate, a product of marginal purity with respect to calcium was produced. Addition of sequential NH_4NO_3 - HNO_3 washes, however, produced a product well within the product guidelines.

PRODUCT FINISHING IN MPPF

Batches of solution containing 60 to 70 grams ^{241}Am were transferred to the precipitators; sufficient water was added to dilute the HNO_3 concentration to 1.2 to 1.7M, and the product was precipitated with 0.9M $H_2C_2O_4$. After washing several times with NH_4NO_3 - HNO_3 solution, several batches of filtered-washed precipitates were combined for calcination. The calcined oxide product was then packaged for shipment. A summary of the product feed conditions is given in Table 13; a summary of the product quality is given in Table 14. The mean value for calcium is skewed high by 250 ppm due to an incomplete wash of one batch of material.

TABLE 13

Actual MPPF Feed

	Concentrate	Diluted Solution
Am, g	2361	
Batches, g/L	4.59	3.7 - 3.6
HNO_3, M	2.2	1.2 - 1.7
NH_4NO_3, M	1.8	1.0 - 1.3

TABLE 14

Product Summary

	Mean	Median	Mode
$^{241}AmO_2$, %	99.46	99.41	99.51
Wt loss, %	0.41	0.43	0.41
Ca, ppm	1500	1250	1500
C, ppm	235	202	100
Fe, ppm	800	800	-

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