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L. W. Gray
G. A. Burney
T. W. Wilson
J. M. McKibben

E. I. du Pont de Nemours & Co.
Savannah River Laboratory
Aiken, SC 29808

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RECOVERY OF Am-Cm FROM HIGH-ACTIVITY WASTE CONCENTRATE BY IN-CANYON-PRECIPITATION AS OXALATES*

L. W. GRAY, G. A. BURNEY, T. W. WILSON and J. M. McKIBBEN

E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, SC 29808

Savannah River Laboratory (SRL) and Savannah River Plant (SRP) have been separating actinides for more than 25 years. Work continues to upgrade processes and to initiate new processes. This report summarizes work on a precipitation process to separate kg amounts of Am and Cm from hundreds of kilograms of $NaNO_3$ and $Al(NO_3)_3$. The developed process includes formic acid denitration of the Am-Cm bearing streams for acid adjustment; oxalate precipitation of the Am-Cm; and Mn^{+2} catalyzed oxidation of oxalate in both the decanted supernate and the precipitated actinides. The new process generates one fourth the radioactive waste as the solvent extraction process which it replaced, and produces a cleaner feed solution for downstream processing to separate the AM and Cm before conversion to their respective oxides.

BACKGROUND OF Am-Cm SOLUTIONS

Large-scale purification of Am, Cm, and Cf with pressurized cation exchange has been planned at SRP for many years (1,2). Initial work involved SRP batch extractions to isolate a crude actinide-lanthanide fraction followed by solvent extraction and ion exchange in the SRL high-level caves. For large-scale purification, a single solvent extraction step was substituted for batch extraction and solvent extraction.

The flowsheet used, as shown in Figure 1, was run a total of seven times in three campaigns to recover a total of ~6.1 kg Am and ~2.3 kg Cm. The overall recovery of Am and Cm was >99%. The Am and Cm were combined in a single tank after evaporation. Unfortunately, the contribution of both entrainment and solubility of $NaNO_3$ from the 1AS stream was sufficient to yield a concentration of 1M $NaNO_3$ in the evaporated, stored Am-Cm solution (1).

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This solution needed to be evaporated by a factor of 55 before being moved to Am-Cm separation by high-pressure ion exchange in the Multi-Purpose Processing Facility (MPPF). A further clean-up of the product was therefore necessary.

Since a campaign to produce ^{242}Pu was to be run, an attempt was made to develop a precipitation process that allowed the recovery of the approximately 8 kg of combined Am-Cm product without using the above solvent extraction flowsheet. The flowsheet was for use with both solutions containing Am-Cm. Whereas previous solutions contained about 1M/L $NaNO_3$, solutions generated by solvent extraction separation of ^{242}Pu from combined Am-Cm would contain about 2.2M/L $Al(NO_3)_3$.

CONCEPTUAL PROCESS

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

Acid Adjustment. The free acid concentration of the Am-Cm solution was reduced to <0.5M by semibatch reaction with formic acid.

Oxalate Precipitation and Digestion. Before precipitation, it was necessary to dilute the interfering cations, Na^+ or Al^{3+}. Individual batches of the acid adjusted material were diluted with process water and heated to >60°C. Oxalate concentration was adjusted by the addition of 0.9M oxalic acid while maintaining the temperature at ~60°C; no precipitation occurs at this temperature. The solution was cooled slowly to 40-45°C to initiate slow precipitation and crystal growth. After a 4-hour digestion cycle, the solution was cooled to <35°C and allowed to settle for 8 hours.

Decanting. A low rate jet jumper (25 gpm) with a short dip tube was used for decanting. The jumper left 2500 L of slurry (precipitate plus supernate) in the precipitator after decanting. This residual volume provided the jet suction of fluid at least 7 inches above the settled precipitate when the total amount of Am-Cm and lanthanide fission products were accumulated in the tank.

Washing and Dissolving. Precipitate which accumulated in the tank was washed several times, at <35°C with a solution of approximately 0.2M oxalic acid and 0.5M nitric acid. In each wash, a volume of solution equal to the residual slurry volume was added and agitated. Solids were allowed to settle before decanting the wash supernate. This procedure was repeated until analysis showed acceptably low concentrations of impurities in the wash solution.

The washed precipitate was agitated and dissolved in 8M nitric acid.

Oxalate Oxidation. Decanted supernate and wash solutions were concentrated and oxalate ion oxidized by reaction with nitric acid using manganous ion catalyst in the evaporator. Off-gas rates (nitrogen oxides, carbon dioxide, and water) were controlled by adjusting the concentrations of nitric acid and manganous ion in the evaporator and controlling the transfer rate during supernate addition.

Am-Cm Finishing. The purified Am-Cm solutions were combined and will be stored until processing can begin in the MPPF. This processing will be a pressurized ion exchange system to separate Am-Cm from fission products and from each other.

LABORATORY DEMONSTRATIONS

Each step of the process was demonstrated on a laboratory scale using dysprosium as a stand-in for the lanthanides and actinides. Initial precipitations of simulated solutions were performed in centrifuge cones to allow fast separation of the precipitate from the pregnant liquor. Simulations of in-canyon-tank precipitation-digestion-settling were performed in a glass tank 5.5 inches in diameter and 32 inches high. The settling rates of various digestion cycles and the volume of slurry produced were then measured. Decanting was demonstrated by vacuum transfer of supernate using various size tubes and adjusting the vacuum to simulate various steam-jet transfer rates. Open beakers were used to simulate product slurry dissolution and oxalate destruction procedures, off-gas rates from oxalate oxidation were measured by water displacement from a water-sealed reaction train.

LABORATORY RESULTS

Formic Acid Denitrations

Simulated solutions were subjected to laboratory formic acid denitrations (Figures 2 and 3). The most usable free acid concentration for the simulated solutions was obtained when a formic acid to free acid ratio of about 1.6 to 1.9 was used. This ratio yields a final free acid of about 0.6 to 0.8M. As a result of Al^{3+} hydrolysis, it was possible to drive the Al-Am-Cm solution to about pH 10. However, acid concentrations less than 0.2M had to be avoided to prevent hydrolysis and precipitation of the actinides.

As a result of the high concentration of nitrate (from sodium and aluminum nitrate) the reaction rate was controlled by the formic acid addition rate until the free acid concentration is reduced to about 0.5M. For semi-batch denitrations it appears that a nitric acid concentration of 1 to 2M at the end of each individual denitration is an excellent stop-point. Using 1 to 2M nitric acid as a projected stop-point assured that there will be no residual formic acid at the end of the reflux and evaporation step. Additional high nitric acid-aluminum nitrate solution can be added to the evaporated-denitrated solution without auto-initiation of a formic acid-nitric acid reaction.

After all the solution has been transferred to the denitration evporator and evaporated, it is possible to drive the denitration reaction to a residual free acid concentration of less than 0.5M.

Precipitation of Simulated Solutions

For the Am-Cm $NaNO_3$ solutions, acceptable losses (<1%) of transplutonium elements could be achieved using 0.3M oxalic acid in the final slurry with a free nitric acid concentration of <0.7M. These conditions were achieved by adjusting the free acid concentration to <1.0M and adding one volume of 0.9M $H_2C_2O_4$ to two volumes of adjusted feed.

As the result of oxalate ion masking of Al^{3+}, precipitation of Am-Cm-$Al(NO_3)_3$ solutions were not straightforward. Using Dy as a stand-in for Am-Cm, simulated solutions were prepared where the ratio of $Al(NO_3)_3$ to $Dy(NO_3)_3$, KF, $NaNO_3$, and $Hg(NO_3)_2$ was held constant as would result in actual process solutions. However, the total ratio of these species to free nitric acid was varied in the stock solutions. Precipitation conditions were simulated by additions of either a half-equal or an equal volume of either an 0.9M oxalic acid or a saturated (~2M) potassium oxalate solution. After precipitation and centrifugation, the residual Dy in solution was determined by flameless atomic absorption. The percent Dy remaining in solution was calculated (Figures 4 and 5).

These tests indicate that to obtain high yields from precipitation, aluminum concentration must be <0.2M (Figure 5). This can best be obtained by evaporating the Am-Cm-Al solution to about 2 to 2.2M $Al(NO_3)_3$ and denitrating this concentrate with formic acid (Figure 3) to about 0.5M nitric acid. Dilution to an aluminum concentration of <0.5M would yield a feed suitable for oxalate precipitation.

Digestion and Settling Rates of the Precipitate

To design the proper short suction leg jet, it was necessary to know the volume of slurry to be expected in the tank, rate of settling, and the minimum distance above the slurry that the jet must be to prevent movement of the slurry.

The volume of precipitate and settling rate was determined by precipitation of 4 L of simulated $NaNO_3$ solution preadjusted to 1M HNO_3 by the addition of 2 L of 0.9M oxalic acid. The settling rate (Figure 6) for the major portion of the precipitate was about 1.25 inches per minute. Fines, however, settled at about 0.8 inch per min. The volume of precipitate-slurry was determined to be ~3.6 L per mole of dysprosium.

Projecting these data to a 12.5 m^3 tank would indicate an approximately 2-hour settling time for the major portion of the precipitate and perhaps 4 to 6 hours for the remainder of the fines to settle. Digestion of the precipitate at ~40°C for about 2 to 4 hours, however, eliminates fines.

The volume of precipitate and settling rate was also determined by precipitation of 2 L of simulated solution, adjusted to ~0.5M Al^{3+} and ~0.25M HNO_3 by the addition of 4 L of 0.9M oxalic acid. When the precipitation was carried out at room temperature, less than 10% of the precipitate had settled after a 60-hour settling period. When the oxalic acid was added to a 60°C solution and then further held at 40-45°C for an additional 2 hours, the settling rate and final volume of precipitate were very similar to the previous Am-Cm material containing $NaNO_3$.

Washing the Precipitated Oxalates

As a result of the 2500-L heel of slurry left in the precipitation tank, the product slurry contained large concentrations of contaminating cations. To yield an acceptable product for downstream processing in MPPF, these ions had to be diluted away from the product. Five equal volume washes of the slurry were calculated to reduce the non-lanthanide impurity concentrations of polyvalent cations to acceptable levels; only four washes are needed to reduce the monovalent cations to acceptable levels.

Jetting of Waste Supernate from Over the Precipitated Slurry

To simulate jetting of waste supernate, solutions were transferred by vacuum from the simulated canyon tank to a second tank through a 1/4-inch-ID tube. At linear velocities through the tube of about 315 linear feet per minute, solution could be transferred at about 1-1/2 inches above the slurry without moving precipitate. Movement of the transfer-tube closer than 1 inch resulted in the movement of precipitate into the tube.

The Purex canyon normally uses two standard transfer jets; one at 75 gal per min, the other at 25 gal per min. The face velocity of the 75 gpm jet is about 460 linear feet per min; the 25 gpm jet is about 155 linear feet per min. Simulations indicate the 25 gpm jet could be used at 6 inches above the slurry without excessive transfer of precipitate.

Dissolution of Washed Precipitate

Downstream processing of the Am-Cm product requires that the precipitate be dissolved, the oxalate ion removed from the solution, and the acid adjusted to $<1M$. Attempts were made to dissolve the precipitate at various nitric acid concentrations. Dissolutions in 1 to 5M nitric acid were successful only if Mn^{2+} was added to catalize the oxidation of oxalate ion. At a concentration of 8M nitric acid, the precipitate could be dissolved at temperatures of 60 to 80°C. Because downstream processing requires that the extraneous polyvalent cation to lanthanide-actinide ratio must be less than 0.14, addition of Mn^{2+} must be kept to a minimum. Because the precipitate can be dissolved in 8M HNO_3 without the addition of Mn^{2+}, 8M HNO_3 should be used to dissolve the precipitate.

Oxalate Destruction in Product Stream

As the Am-Cm will be separated by an ion exchange process, it is necessary to remove all of the oxalate ion. If separated solution is to be stored for an extended period of time before separation, self-radiolysis of the solution will of course destroy a portion of the oxalate. Mousty, Toussaint, and Godfrin (3) have shown that extended boiling of a 10M HNO_3 solution oxidized sufficient oxalate to render a suitable solution for separation. However, extending boiling of $>10M$ HNO_3 solutions in stainless steel leads to excess corrosion of the equipment and, hence, the introduction of polyvalent cations (Fe, Cr, Ni) to the process solution. Koltunov (4) has shown that managanous ion (Mn^{2+}) will catalyze the oxidation of oxalate in nitric acid solutions. Low concentrations of manganous ion were therefore used in an attempt to catalyze the oxidation of oxalate in the precipitated slurries.

Figure 7 shows the destruction of oxalate in the precipitated slurry in 8M nitric acid. A manganous ion concentration of 0.045M Mn^{2+} in 8M HNO_3 will oxidize the oxalate in the slurry in about one-half the time required for a concentration of 0.011M Mn^{2+}.

Because Mn^{2+} is a polyvalent cation, its concentration in the final solution feed to the cation columns should be kept below 0.05M to avoid excess competition for resin sites. For the overall process, it is better to use the longer oxidation times than to use higher Mn^{2+} concentration. Catalyzed oxidation should not be preformed until the volume of solution is reduced to the minimum possible volume.

Oxalate Destruction in Waste Stream

Although the reactions are more rapid at 8M HNO_3, manganous ion catalytizes the oxidation of oxalate at lower acidities. Approximately 1M HNO_3 seems to be required. As the major solids producing reagent in the waste stream was oxalic acid, it was much cheaper to oxidize the oxalic acid to CO_2 gas than to store it as radioactive waste. Waste streams are therefore acidified with nitric acid, $Mn(NO_3)_2$ added and the solutions evaporated. During the evaporation, the oxalate ion will be oxidized to CO_2 gas.

Koltunov (4) has shown that the rate of catalytic oxalic acid oxidation with low concentrations of nitric acid (≤6M) is given by the equation:

$$\frac{-d\ (H_2C_2O_4)}{dt} = \frac{k_1\ (H_2C_2O_4)\ (Mn^{II})}{(H^+)^{0.73}}$$

where $k_1 = 3.29 \pm 0.04$ liter$^{0.27}$ mole$^{-0.27}$ at $u = 3.1$ and 97°C. The overall reaction is given by the equation:

$$3H_2C_2O_4 + 2HNO_3 \rightarrow 6CO_2\ (g) + 2NO\ (g) + 4H_2O$$

Koltunov's equation was applied to two cases. The first considered the maximum off-gas rate under proposed operating conditions. It was assumed that the oxidation started at 97°C with 5000-L volume of 0.02M Mn^{2+}, 5M HNO_3, 0.9M $H_2C_2O_4$, and 0.25M $NaNO_3$. The second case assumed the waste was concentrated by evaporation before adding Mn^{2+} ion.

In the first case, an off-gas rate of 193 cfm was calculated by Koltunov's equation. In the second case, an off-gas rate of 603 cfm was calculated. Laboratory experiments for both conditions were in good agreement with the off-gas rate predicted by Koltunov's equation. Under plant operating conditions, then, a maximum off-gas rate is estimated to be less than 10% of the 6000 cfm venting capabilities of the evaporator proposed as the reaction vessel. No foaming problems were observed during the laboratory experiments.

Flowsheets

Generalized flowsheets for the separation procedures are given in Figures 8 and 9. The flowsheets provide for acid adjustment by formic acid denitration followed by dilution and precipitation in batches with precipitated slurry accumulated in the tank. After washing of the precipitated slurry to remove contaminating cations, the product is dissolved by adjusting the acid concentration to <8M and heating the solution. Product is then stored until the MPPF is available to process the material.

Supernates are transferred to an evaporator containing boiling 5M HNO_3 and 0.02M Mn^{2+}. Solution is transferred at a rate to maintain approximately a constant evaporator volume. Sufficient acid is added at the end of each batch to oxidize oxalate to be transferred in the next batch. All material is accumulated in the evaporator.

When MPPF processing begins, the product solution must be evaporated from the ~8000 L storage volume to about 600 L. If oxalate has survived both the initial heating steps and the high radiation field during storage, it must be oxidized during the evaporator step. Sufficient Mn^{2+} should be added to give approximately 5 moles of Mn^{2+} in the MPPF evaporator. After evaporation the acid must be adjusted to MPPF requirements by formic acid denitration.

SUMMARY OF PLANT PROCESSING

Approximately 8 kg of Am-Cm was recovered from the stored Am-Cm-$NaNO_3$ waste. The process yielded an overall recovery of 94.75% of the Am-Cm while rejecting ~99.5% of the Na, ~95% of the SO_4^{2-} and ~85% of the Fe. Of the losses, 0.25% was dissolved losses and 5% was entrained losses.

Approximately 5 kg of Am-Cm were recovered from the stored Am-Cm-$Al(NO_3)_3$ waste. The process yielded an overall recovery of ~75%. Of the losses, ~2.5% was dissolved losses and the remainder was entrained losses. Of this 25% lost to the waste supernate stream, about 18% is stored for future recovery; the remaining 7% was actually lost to the waste tanks. The major contaminants of the purified Am-Cm product are Fe, Al, and Na.

Characterization of Actual Solutions

The analyses of the concentrate solutions before purification are given in Table I.

TABLE I. Characterization of Actual Solutions

	Am-Cm-Na	Am-Cm-Al
Density, g/cm^3	1.116	1.305
HNO_3, M	2.72	1.67
Sodium, g/L	11.5	-
Aluminum, g/L	0.03	42.0
Iron, g/L	0.62	1.2

Formic Acid Denitrations

Two batches were required to denitrate the $NaNO_3$ solutions, and four batches were required for the $Al(NO_3)_3$ solutions. The final acid concentration of $NaNO_3$ solutions was 0.9M HNO_3; the final acid concentration of the $Al(NO_3)_3$ solutions was 0.2M. All reactions initiated promptly and proceeded smoothly.

Foaming was a problem with the $Al(NO_3)_3$ solutions due to the high salt content; this extended both the denitration runs and the evaporation period before denitration.

Precipitation, Digestion, Washing, Decanting of Am-Cm-$NaNO_3$ Solution

To minimize precipitation of sodium oxalate salts and to minimize the solubility of actinide oxalates, the denitrated solution ws diluted with water and heated to ~60°C before adding oxalic acid. Sufficient 0.9M oxalic acid was added to adjust the final slurry to 0.6M oxalate, 0.18M nitric acid and <0.5M sodium nitrate. The slurry was cooled to 40-45°C and digested for four hours to promote large crystal growth. After cooling to 35°C, the precipitate was settled for eight hours before decanting the supernate.

This procedure was reported with the remainder of the Am-Cm-$NaNO_3$ solution, a third batch which resulted from an ineffective flush of the evaporator and four wash cycles. Other losses to the waste supernate were 5.25%; only 0.25% of the total was dissolved losses, the remainder was entrained losses.

Precipitation Digestion, Washing, Decanting of Am-Cm-Al$(NO_3)_3$ Solution

As a result of Al^{3+} masking of oxalate, it was necessary to divide the Am-Cm-Al$(NO_3)_3$ solution into ten batches. After transfer of about 10% of the solution into the precipitation vessel, dilution water was added to reduce the Al^{3+} concentration and the solution heated to about 60°C. On the first batch, a small amount of La^{3+} carrier was also added before sufficient oxalic acid was added to make the solution 0.6M. The slurry was then cooled to 40-45°C and digested 4 hours to promote crystal growth before decanting the supernate. This procedure was repeated with the remaining nine batches until all the precipitate was accumulated in the tank. The accumulated precipitate was washed four times to dilute out contaminating cations.

Decanting losses were much higher than anticipated. In an attempt to minimize losses, the chemical composition of the precipitation batches were varied to increase the oxalate/aluminum ratio. These variables had little effect on product losses because the solubility losses were very low (<15% of the actual loss). Apparently, Am-Cm oxalate solids are being disturbed prior to and during decanting and a portion of the disturbed precipitate was decanted. A new decant jet, which had a plate welded across the bottom and holes drilled around the pipe above the plate to provide horizonal rather than vertical suction, was fabricated and installed after the fifth batch. Losses, however, remained unchanged.

A total of about 20% of the product (~1600 g Am-Cm) was entrained to the waste evaporator with decanted supernate. Most of the oxidized supernate concentrate containing 1150 g Am-Cm was retained and is available for recovery by another method; the remainder was discarded.

Destruction of Oxalate in Waste Stream

Before supernate or wash solution was added, 5M nitric acid containing 0.02M Mn^{2+} was brought to boiling in the evaporator to ensure prompt and rapid oxidation of the oxalate. Transfer rates of either supernate and wash solution or additional nitric acid, needed to ensure complete destruction of the oxalate, were limited to keep off-gas rates less than 100 cfm. The final oxalate concentration of the waste was <0.02M which indicates >99.8% of the oxalate was oxidized to CO_2 and H_2O.

TABLE II. Composition of Na^+-Am^{3+}-CM^{3+}-NO_3^- Solutions

	Storage Condition Before Purification	MPPF Feed Requirements	Actual Final Solution
Volume, L	17,000	<620	
HNO_3, M/L	2.72	<0.5	
Moles, total	46,240	<310	
$NaNO_3$, M/L	0.50	<1.0*	
Moles, total	8,500	<620*	45*
Ln's + Ac's, total moles	~550	~550	
Moles/L	~0.032	~0.887	
Other polyvalent cations, M/L	~0.015	<0.05**	
Moles, total	~255	<31**	13**

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* This is the feed requirement for the sum of all polyvalent cations (Al^{3+} + Fe^3 + Hg^{2+} + all others).

** This is the feed requirement for the sum of all monovalent cations excluding H^+ (Na^+ + K^+ + any others).

TABLE III. Composition of Al^{3+}-Am^{3+}-Cm^{3+}-NO_3^- Solutions

	Storage Condition Before Purification	MPPF Feed Requirements	Actual Final Solution
Volume, L	13,200	620 L	
HNO_3, M/L	1.0	<0.5	
Moles, total	13,200	<310	
Al($Na\bar{N}O_3$), M/L	2.0	<0.05*	
Moles, total	26,400	<31*	50*
$NaNO_3$, M/L	0.07	<1.0**	
Moles, total	925	<620**	75**
KF, M/L	0.12		
Moles, total	1584		
$Hg(NO_3)_3$, M/L	0.023		
Moles, total	304		
$Fe(NO_3)_3$, M/L	0.023		
Moles, total	304		
H_2SO_4, M/L	0.046		
Moles, total	607		
Ln's + Ac's, M/L	0.006		
Moles, total	80		

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* This is the feed requirement for the sum of all polyvalent cations (Al^{3+} + Fe^{3+} + Hg^{2+} + all others).

** This is the feed requirement for the sum of all monovalent cations excluding H^+ (Na^+ + K^+ + any others).

Dissolution and Characterization of Final Products

The washed product slurry was adjusted to 8M nitric acid and raised to a simmer of about 40°C to dissolve the oxalate precipitate. A comparison of the major impurities before and after the oxalate precipitation purification step for the sodium nitrate solution is given in Table II, for the aluminum nitrate solution in Table III. These two solutions have been combined and will be stored to await processing by high pressure ion exchange in the MPPF.

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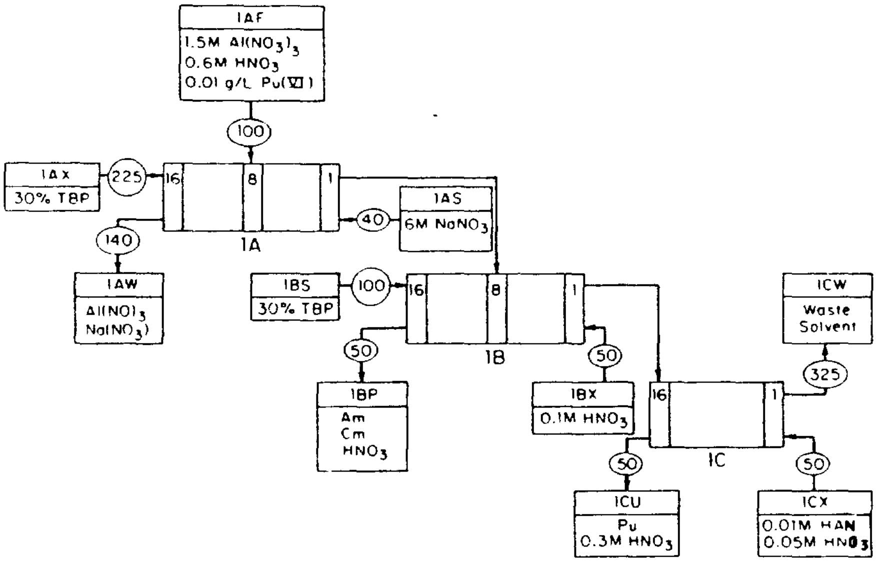

FIGURE 1. Solvent Extraction Flowsheet

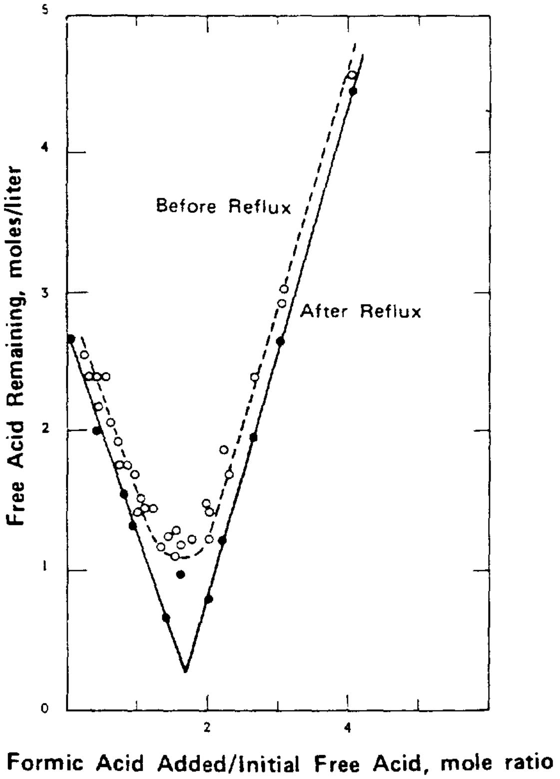

FIGURE 2. Formic Acid Denitration of Simulated Solution

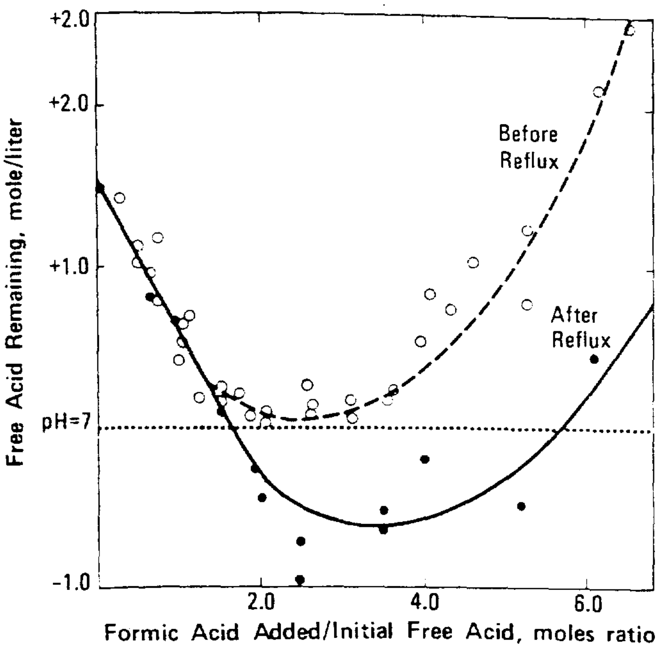

FIGURE 3. Formic Acid Denitration of Simulated Solutions

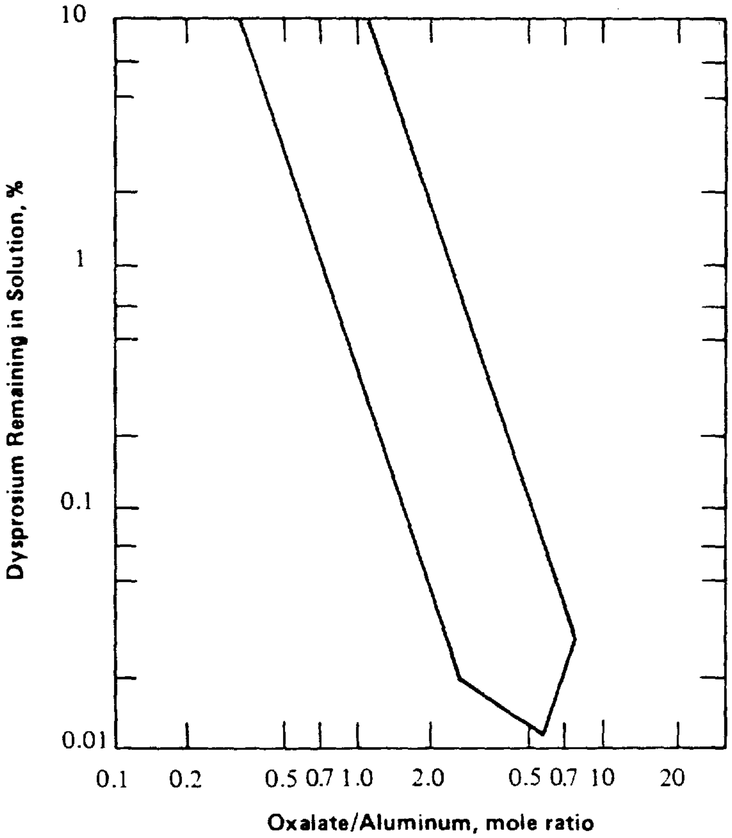

FIGURE 4. Precipitation of Dysprosium from Simulated Waste as a Function of Oxalate Ion

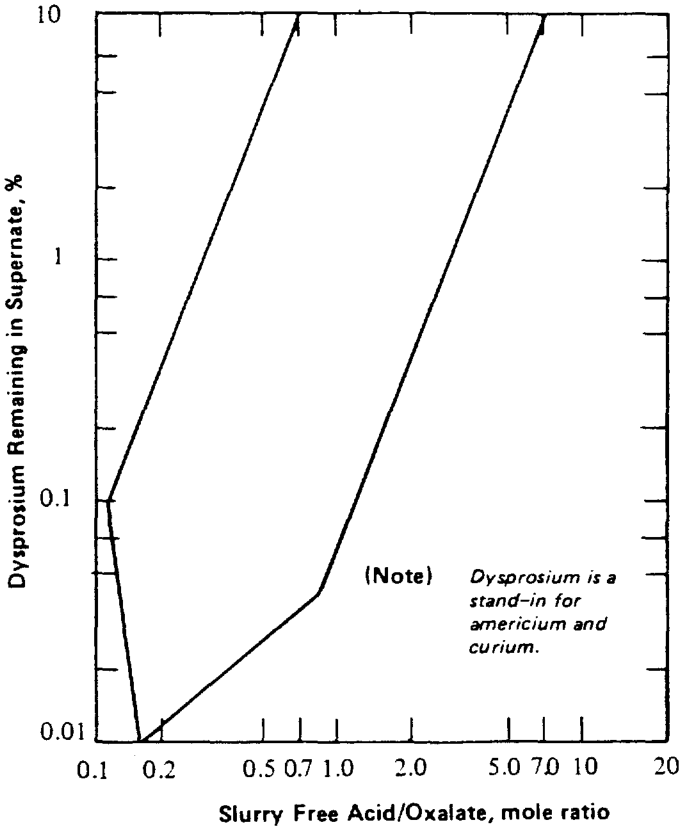

FIGURE 5. Precipitation of Dysprosium from Simulated Waste as a Function of Free Acid

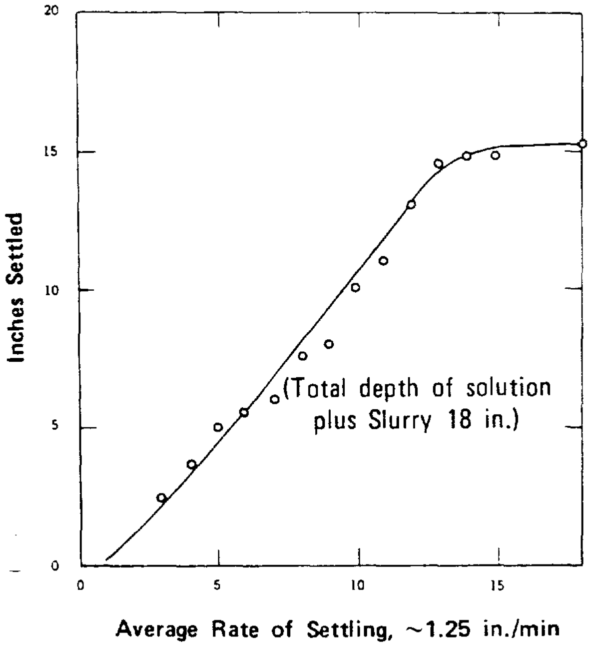

FIGURE 6. Settling Rate of Typical Simulated Oxalate Precipitate

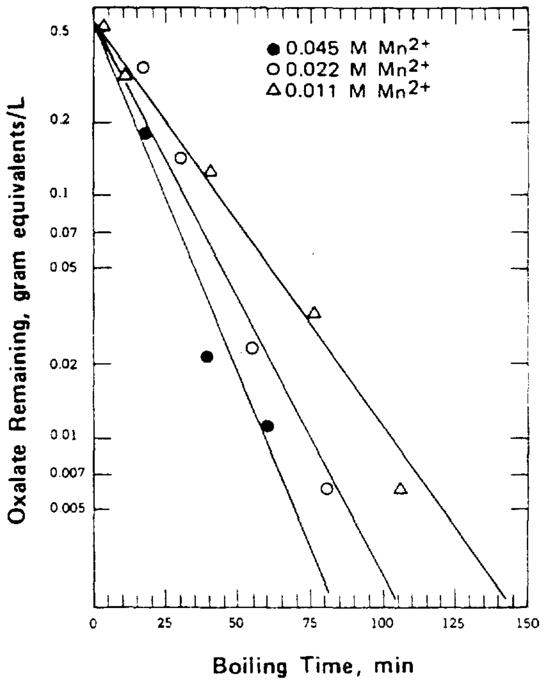

FIGURE 7. Oxidation of Oxalate Ion with 8M Nitric Acid Catalyzed by Manganous Iron

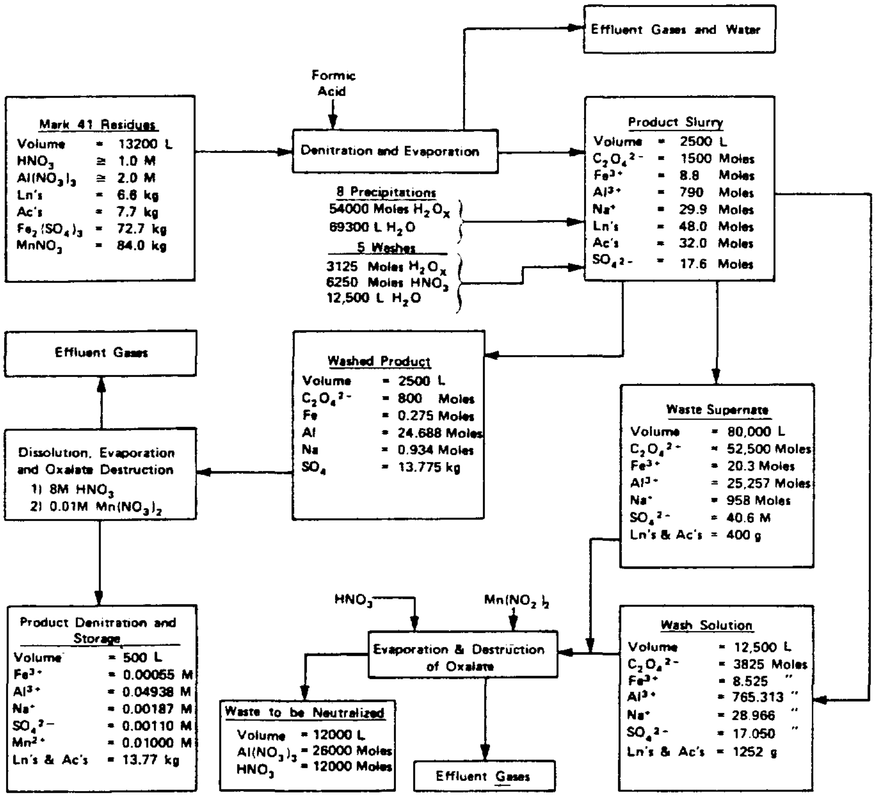

FIGURE 8. Flowsheet for Purification of Mark 41 Residues

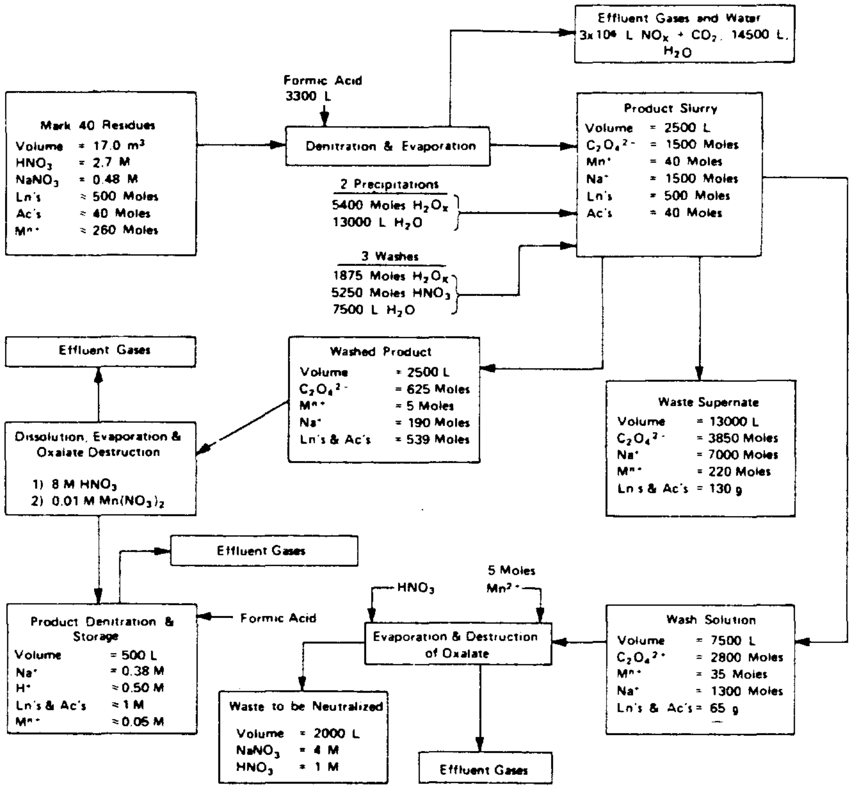

FIGURE 9. Flowsheet for Purification of Mark 40 Residues