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THE ISOLATION AND PURIFICATION OF AMERICIUM

D. O. Campbell

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THE ISOLATION AND PURIFICATION OF AMERICIUM

D. O. Campbell

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1.0 ABSTRACT

Gram amounts of americium were separated quantitatively from kilogram quantities of lanthanum to yield an americium product approaching 90% purity. The remaining impurity was chiefly yttrium.

Elution of americium from a 25% loaded Dowex 50 resin column with 0.15 M citric acid—0.10 M diammonium citrate—0.3 M ammonium nitrate, pH 3.3, gave a product containing 99% of the americium with a La/Am ratio of 1/100 or less in one fourth of a column volume, in this case about a 100-fold volume reduction. Approximately 9 g of americium was purified by this method.

Elution with 12.8 M hydrochloric acid from a 20 to 30% loaded column gave 90% of the americium in two column volumes of product with a La/Am ratio of about 1/4. About 1 g of americium was purified by this method.

2.0 INTRODUCTION

The americium recovered from Hanford slag and crucible waste by the Amex process was concentrated and purified by an ion-exchange method. The 215-liter Amex product contained 10 g of americium, 3000 g of lanthanum, smaller amounts of corrosion products and other impurities, and large amounts of ammonium acetate, acetic acid, and ammonium nitrate. Because of a commitment to send 1 g of americium with a La/Am ratio of less than 10/1 to another laboratory in a short time, the experiments were concerned with finding a satisfactory procedure for separating americium from lanthanum rather than with studying the separation under varying conditions. It is not known whether the conditions used in the two procedures developed are optimum.

The Amex program[1] was carried out in conjunction with a program

[redacted]

for recovering plutonium from the Hanford waste. The Amex process[2] consisted of tributyl phosphate extraction of the americium from the neutralized aqueous waste from the plutonium recovery process, sorption on Dowex 50W resin, and elution with 3 M ammonium acetate—1.5 M acetic acid and nitric acid. The presence of large amounts of lanthanum, whose chemical properties are very similar to those of americium, was not indicated in preliminary considerations of the Hanford waste, and the resin column used in the Amex process was expected to hold all the americium from the entire program. Actually, lanthanum is sorbed on Dowex 50 resin somewhat better than americium under Amex isolation conditions and after a few days' operation the column became loaded. It was necessary to elute the Amex column 22 times during the program. Each elution used 6 to 10 liters of acetate eluant, about 2 liters of nitric acid, and large volumes of wash water. Economic considerations made it desirable to carry out the americium purification and concentration from this large-volume Amex product on a laboratory scale.

Because of its low energy, the americium gamma radiation is not readily detected by standard health physics survey instruments. Therefore, special precautions, such as surveys using film, were taken to prevent personnel overexposures in handling gram quantities of americium.

Many persons contributed to the work reported here, particularly W. W. Crane of the University of California Radiation Laboratory at Livermore, who interrupted his vacation to work on the problem; J. T. Roberts of the Chemical Technology Division, Oak Ridge National Laboratory, who investigated the citrate elution method; P. C. Stevenson of the University of California Radiation Laboratory at Livermore and G. E. Boyd and G. D. O'Kelley of the Chemistry Division at Oak Ridge National Laboratory, with whom consultations were held; J. H. Cooper and his group, G. W. Leddicotte and his group, and M. M. Murray of the Analytical Chemistry Division at Oak Ridge National Laboratory, who made the numerous americium determinations, lanthanum determinations, and spectrographic analyses. C. W. Greene was the technician on the program and R. L. Hickey the operator.

3.0 INVESTIGATION OF LANTHANUM-AMERICIUM SEPARATION METHODS

Satisfactory separation of americium and lanthanum on a laboratory scale was obtained by selective elution of americium from a cation-exchange resin with either citrate eluant, similar to the method used for rare earth separation,[3] or concentrated hydrochloric acid. Selective sorption of americium on an anion-exchange resin column from a solution of americium and lanthanum in a highly concentrated lithium chloride solution was unsatisfactory.

Separation of americium and lanthanum by tributyl phosphate extraction was briefly considered, since some separation had been obtained in the Amex solvent-extraction system. Measurement of the distribution of the two elements between 8 M ammonium nitrate—0.1 M nitric acid solution and 30% tributyl phosphate in Amsco showed the americium distribution coefficient to be about twice that of lanthanum. Separation by this method would therefore be possible but would require a large number of stages. Extraction with thenoyl trifluoroacetone was also rejected because of the large-scale equipment required.

Americium can be oxidized to the V or VI state[4] and then easily separated from lanthanum. An oxidation method had been used at Los Alamos[5] for final purification of an americium product contaminated with lanthanum but containing no acetate or other organic material. However, direct oxidation of the Amex product was impossible because of the large amount of acetate, and removal of such reducing material prior to the oxidation would probably be about as difficult as the separation of americium from lanthanum.

A brief investigation indicated that precipitation methods would not give the necessary separation.

3.1 Citrate Elution of Cation-Exchange Resin

Ninety-nine percent of the americium from a feed with a La/Am ratio of 1600/1 was obtained in 6 to 8 volume changes of product with a La/Am ratio* considerably less than 1/1 (Table 1). The method consisted of elution of a Dowex 50 resin column partially loaded with americium and lanthanum by 0.25 M citrate solution of pH 3.3 (0.15 M citric acid-0.10 M diammonium citrate).

Initial eluate cuts were acid, since the eluant was converting the hydrogen-form resin to an ammonium form. The americium began to come off the column when the pH of the product increased to 3. In the first run, with a single column, this took place after about 20 column volume changes (2200 to 2300 ml).

In the second experiment two columns were used. The first, containing hydrogen-form resin, was loaded essentially to breakthrough. It was connected to the top of another column filled with ammonium-form resin. The americium started coming off sooner than in the first experiment, i.e., after 600 to 700 ml. The americium peak was at 750 ml, and lanthanum was not detected until 2100 ml had passed through. There were two plutonium peaks, Pu(IV) at 350 ml and Pu(VI) at 1200 ml. This product was much purer than that in the first experiment.

The column used in the first experiment was 220 ml in volume, 3/4 in. diam. and 31 in. high. The columns in the second experiment were of the same diameter; the one containing hydrogen-form resin was 10.5 in. high and the one containing ammonium-form resin was 34.5 in. high. The eluant flow rate was 2 $ml/m/cm^2$ and the resin loading was about 20% in both experiments.

---

*Until after the isolation of the first gram of americium (Sec. 4.2), separated La/Am ratios may have been too high because of the presence at the time of analysis of lanthanum formed by fissioning of plutonium during the neutron activation. Although the amount of plutonium in the feed to the Amex process was much less than 1% of that present in the Hanford waste, it was significant in comparison with the americium, and some of this plutonium was carried over into the americium products.

Table 1. Separation of Americium and Lanthanum by Citrate Elution

Resin: Dowex 50, 12% cross-linked, 50-100 mesh
Feed: Amex product solution, La/Am = 1600/1

Yield of Am (%)	Vol. of Product (ml)	La/Am Ratio[a]	
		Maximum	Estimated Actual
Column: 3/4 in. diam., 31 in. high; 220 ml Eluant: 0.15 M citric acid, 0.10 M diammonium citrate, pH 3.27; 2 ml/m/cm^2 flow rate Loading: 3.3 g of La, 2.1 mg of Am; about 20% of column length			
90	500	1/1	1/10
99	900	2/1	3/10
99.6	1200	3/1	1/1
99.8	1400	10/1	8/1
99.9	---	30/1	27/1
Column: 3/4 in. diam., 10.5 in. high; loaded to breakthrough and eluted through an ammonium-form resin column 3/4 in. in diam. and 34.5 in. high Eluant: same as above except pH of 3.31 Loading: 4.7 g of La, 3.0 mg of Am; about 20% of column length			
99	900	---	3/100,000
99.9	1300	---	1/100

[a]Figures in column headed "maximum" are based on radiolanthanum found in sample after neutron activation. Figures in column headed "estimated actual" are corrected for lanthanum formed by fission of plutonium during neutron activation analysis (see footnote, p. 4).

3.2 Hydrochloric Acid Elution of Cation-Exchange Resin

From an Amex product feed with a La/Am ratio of 315/1, 80% of the americium was obtained with a La/Am ratio of 9.2/1 by concentrated hydrochloric acid elution of a Dowex 50 resin column 5% loaded with lanthanum and americium. With 30% loading the La/Am ratio was 17/1 and with 55% loading it was 30/1. With 50% recovery, for all column loadings the La/Am ratio was lower, viz., 3.7/1, 7.7/1, and 20/1 (Figs. 1-3). The product La/Am ratio was decreased when the resin particle size was decreased from 50-100 to 100-200, but at the flow rates used the percentage of cross linking in the resin (4, 8, and 12%) had little effect.

The distribution coefficient (resin/aqueous) of lanthanum on Dowex 50 is larger than that of americium at all aqueous phase HCl concentrations.[6] The difference increases as the HCl concentration increases above 7 M. As was expected, with Amex product solution the separation of americium from lanthanum increased as the eluant HCl concentration was increased from 6 to 12 M.

In all runs the americium product concentration reached a peak near the end of the first volume change following one displacement volume.* In the run with 100-200 mesh resin, the americium peak was sharper than in that with the 50-100 mesh resin, probably because the smaller resin size permitted equilibrium conditions to be more nearly approached. There was evidence of a small lanthanum peak, but this may have resulted from the formation of lanthanum from plutonium during the neutron activation analysis (footnote, p. 4) and not from lanthanum in the column effluent.

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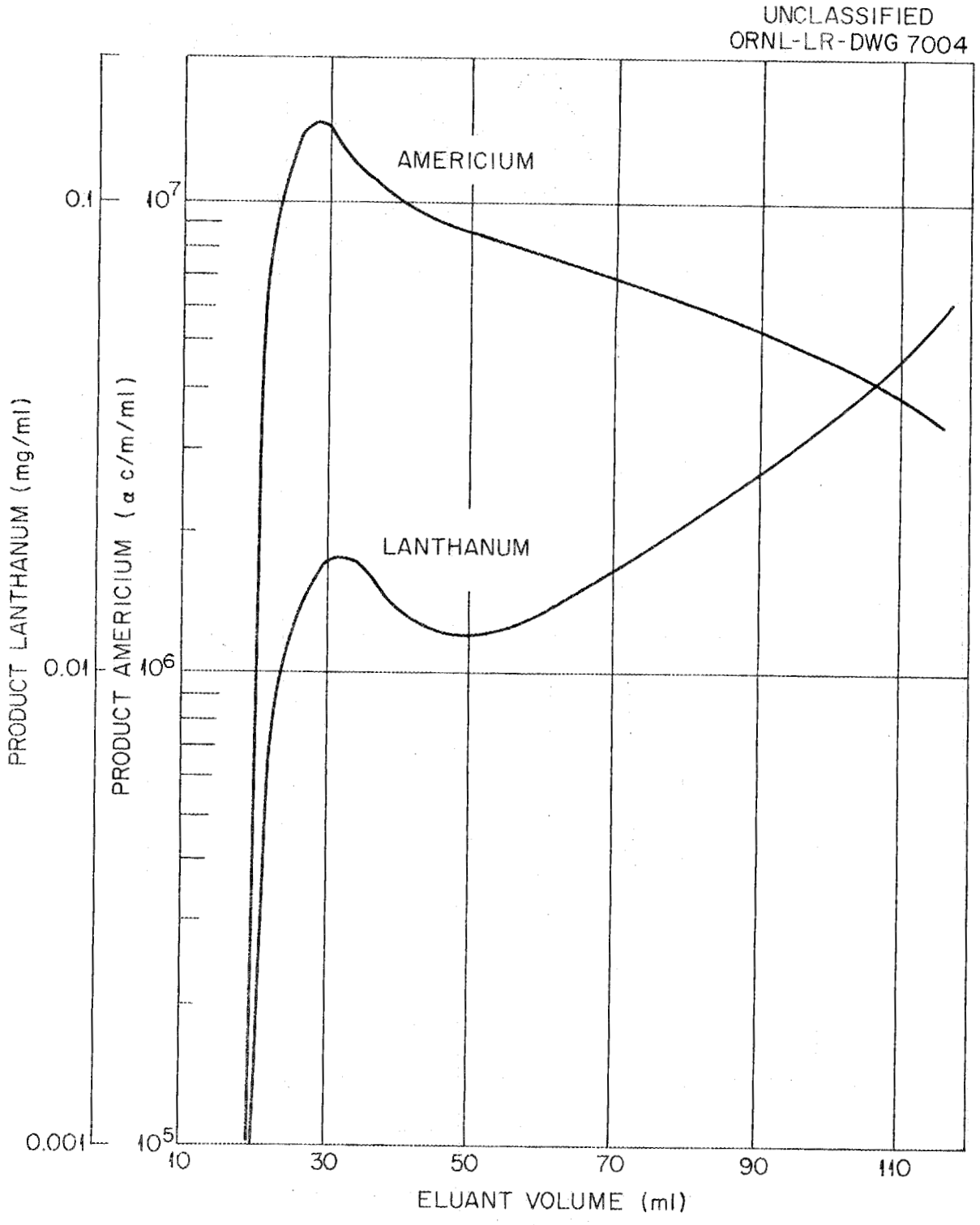
*The displacement volume could be observed visually by the first appearance of mixing in the product caused by the higher refractive index of the hydrochloric acid solution. A volume change is defined as one half the apparent volume of resin.

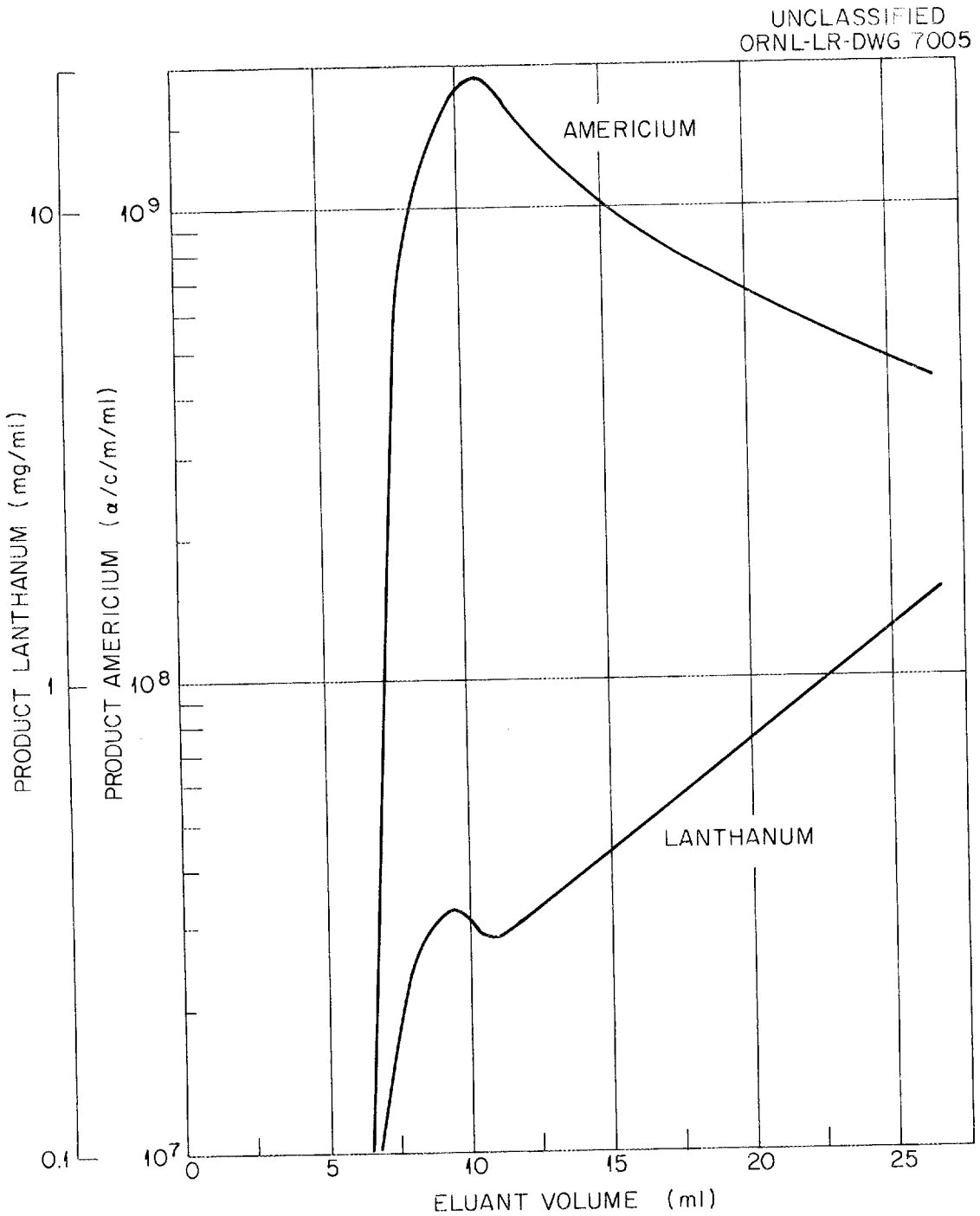


Dowex 50W resin, 12% cross-linked, 50-100 mesh; 26 ml, 0.57 cm^2 cross section

Feed: 74 mg of La, 8.65×10^8 α c/m/ml of Am (La/Am ratio = 315)

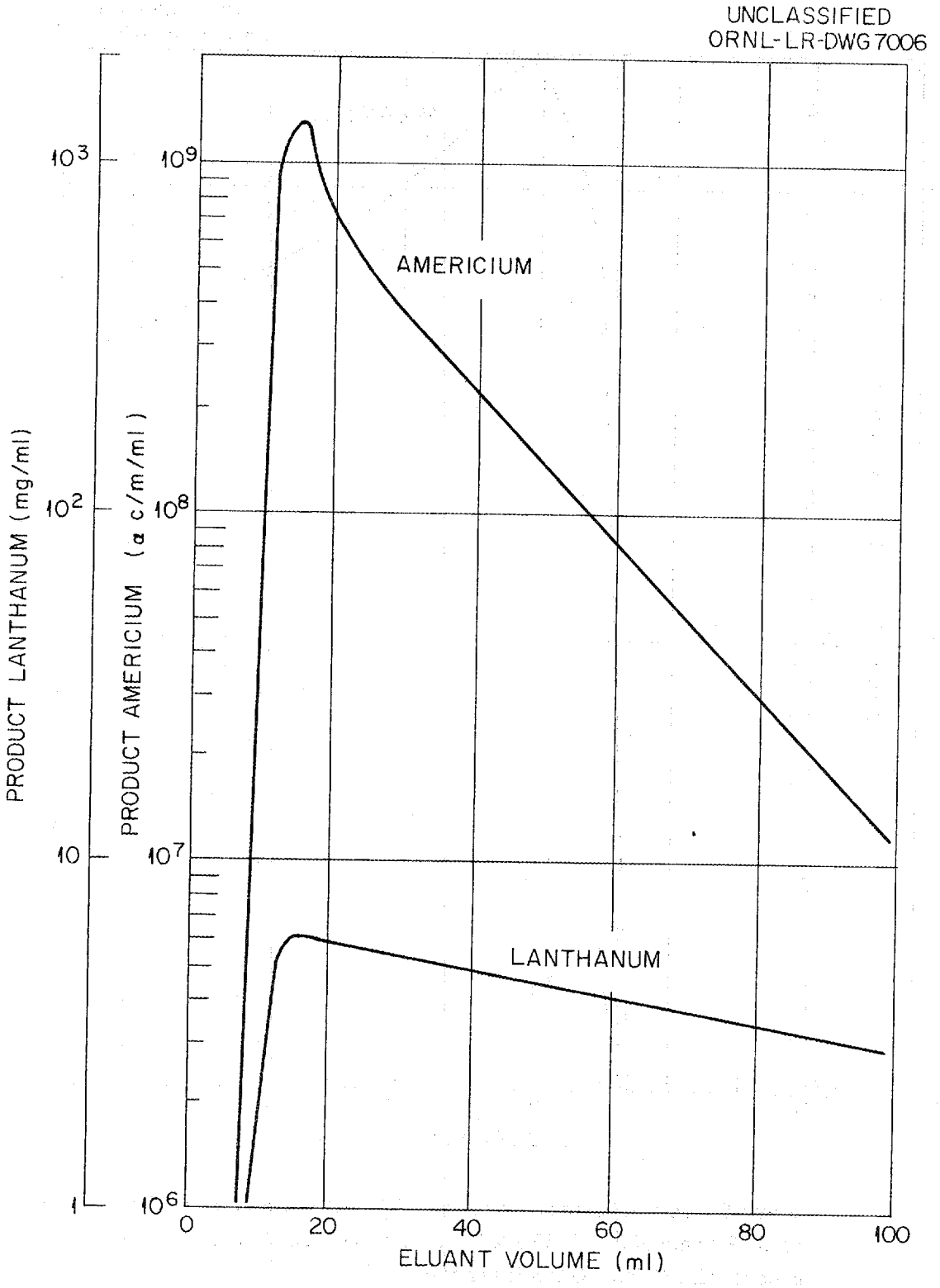
Eluant: 12*M* HCl, 0.5 ml/m/cm^2 flow rate

Fig. 1 . Separation of Americium from Lanthanum by Elution with Hydrochloric Acid from 5% Loaded Resin.



Dowex 50 resin, 8 % cross linked, 50-100 mesh; 10 ml, 0.57 cm^2 cross section
Feed: 225 mg of La, 2.39 x 10^9 α c/m of Am (La/Am ratio = 315)
Eluant: 12.1 *M* HCl, 0.5 ml/m/cm^2

Fig. 2. Separation of Americium from Lanthanum by Elution with Hydrochloric Acid from 30% Loaded Resin.



Dowex 50 resin, 12 % cross-linked, 100-200 mesh; 15.3 ml, 0.57 cm^2 cross section

Feed: 820 mg of La, 2.17×10^{10} α c/m/ml of Am (La/Am ratio = 137)

Eluant: 12.1 *M* HCl, 0.5 ml/m/cm^2 flow rate

Fig. 3 . Separation of Americium from Lanthanum by Elution with Hydrochloric

The columns used were 6 cm high by 0.5 cm^2 in cross-sectional area. The Amex product was acidified to 0.1-0.2 M H^+ and diluted until the ammonium ion concentration was less than 8.5 M to improve the sorption. The resin capacity was less than the theoretical value of 106 mg/ml. Americium broke through at 1% of feed concentration after an average lanthanum loading of 54 mg/ml, and the amount of americium in the effluent reached the feed concentration (lanthanum was about half the feed concentration) at a lanthanum loading of about 75 mg/ml. The entire Amex product, containing about 3000 g of lanthanum, would therefore load over 40 liters of resin, but much more than 40 liters would be required because only partial loading would be used.

In experiments with lanthanum nitrate solution and column loadings of 5 to 55%, elution with 12.4 M hydrochloric acid in water gave no lanthanum elution peak. Instead, the lanthanum came off the column at a nearly constant rate of 2 to 5% of the column loading per volume change, the higher percentages being obtained with the higher loadings. The eluate lanthanum concentration generally did not vary by more than a factor of 2 during the first 10 volume changes. The explanation of this is probably that equilibrium was not attained in the column and the concentration of lanthanum in the product was controlled by the rate at which it left the resin. Elution with a 12.8 M hydrogen chloride solution in 20% ethanol gave an eluate lanthanum concentration about half that obtained in an elution with 12.8 M hydrochloric acid solution in water. These experiments were carried out with Dowex 50 resin, 12% cross linked, 100-200 mesh, in a 10-ml column, 0.51 cm^2 in cross section. The eluant flow rate was 0.5 $ml/m/cm^2$.

3.3 Lithium Chloride--Anion Exchange Resin Separation

It has been reported that in the presence of high concentrations of lithium chloride, americium sorbs on anion-exchange resin.[5] Lanthanum

could be washed from the americium with several column volumes of lithium chloride reagent, and the americium then eluted with two column volumes of 6 M hydrochloric acid. The method was investigated only briefly since during the sorption studies very little solution was passed through the column before americium broke through and no volume reduction was obtained.

For the first experiment solid lithium chloride was added to some of the Amex process product, but a precipitate, probably ammonium chloride, formed at low lithium chloride concentrations. At 80°C lithium chloride was soluble to a concentration of about 12 M before any precipitation occurred. In a column heated to 90°C, the americium band front, detected by the gamma radiation, with a Victoreen survey meter, moved rapidly down the column and americium came off the bottom at feed concentration by the end of the second column volume.

For the second experiment the feed was made from a hydrochloric acid solution of lanthanum and americium, obtained from a hydrochloric acid elution experiment (Sec. 3.2), in order to avoid heating the system or preliminary removal of substances that caused precipitation. The hydrochloric acid was neutralized with lithium hydroxide to give a feed 0.5 M in hydrochloric acid and 12.9 M in lithium chloride. In this experiment the americium began to break through at the end of three column volumes.

Since americium has anionic properties in high concentrations of lithium chloride, it should be eluted from cation-exchange resin by this reagent. However, 10 M lithium chloride, both neutral and with 0.5 M hydrochloric acid added, did not effectively elute americium. With either solution only about 5% of the americium on the column was eluted with each column volume of eluant.

4.0 PROCEDURE USED

As a result of the laboratory studies, citrate elution from Dowex 50 resin was chosen for purification of most of the americium required for the program. Considerable concentration was achieved in this step. Further purification was achieved by passing this product, in concentrated hydrochloric acid, through a Dowex 1 anion-exchange column. The first gram of americium prepared for shipment was purified by hydrochloric acid elution from Dowex 50 followed by fluoride precipitation. Purification was satisfactory, but the ion-exchange product was dilute. Since citrate elution gave a better separation and a more concentrated product, it was used to process the remainder of the americium.

4.1 Citrate Elution

In laboratory studies the two-column citrate elution method gave better separation and used less eluant than the one-column method, but was cumbersome. For the actual separation work, elution from a single partially loaded column was used, with the eluant ammonium ion concentration increased from 0.2 M to 0.5 M. With this solution the hydrogen-form resin was converted to the ammonium form by only 40% as much eluant. The column was 18 liters in volume, a 6-in.-diam. glass pipe with 176 cm^2 cross-sectional area. The resin was Dowex 50, 12% cross linked, 100-200 mesh, with the fines removed. The work was carried out in one bench hood and one floor hood of a laboratory. Polyethylene bottles up to 50 liters in volume were used for containers, Tygon tubing for connections, and a finger pump for pumping the solutions.

Practically all the americium was obtained in less than one volume change (Table 2), in one case 99% in less than 4 liters. Collection of this middle cut, while the column effluent line (1/4-in.-diam. Tygon tubing) read greater than 10 mr/hr with a

Table 2. Separation of Americium from Lanthanum by Citrate Elution from 18-liter Resin Column

Column: 6-in.-diam. glass pipe, 176 cm^2 cross-sectional area
Resin: Dowex 50, 12% cross-linked, 100-200 mesh; 18 liters
Eluant: 0.10 M diammonium citrate--0.15 M citric acid--0.30 M ammonium nitrate, pH 3.30-3.32; 80-100 ml/min flow rate

Cut No.	Vol. of Cut (liters)	Total Vol. (liters)	Americium[a] (% of total)	La/Am Ratio[b]
Feed, 955 mg of Am, 243 g of La; about 20% column loading				
Displacement	8.0	---	---	
1	21.5	21.5	0.001	
2	4.0	25.5	0.007	
3	3.1	28.6	0.04	
4	10.0	38.6	99.9	<0.2/1
5	24.0	62.6	100.0	
6	22.0	84.6	100.0	
Feed, 1800 mg of Am, 389 g of La; about 30% column loading				
Displacement	8.0	---	---	
1	21.0	21.0	---	
2	4.0	25.0	---	
3	4.0	29.0	<0.1	
4	0.5	29.5	0.02	
5	8.5	38.0	97.3	1.5/1[c]
6	4.2	42.2	98.6	
7	25.0	67.2	99.9	
8	20	87.2	100.0	
Feed,[d] 4700 mg of Am, 140 g of La; about 10% column loading				
Displacement	8.0	---	---	
1	24.0	24.0	---	
2	10.0	34.0	---	
3	4.1	38.1	0.1	
4	2.7	40.8	98.9	<0.005/1[e]
5	4.0	44.8	99.9	
6	4.0	48.8	100.0	
7	50.0	98.8	100.0	

[a]The amount of americium loaded on a column was known only approximately; these numbers are based on the americium eluted from the column.

[b]Lanthanum analyses for only the product cut; all other cuts containing americium were recycled to a later elution.

[c]The high lanthanum was probably the result of incomplete removal from the column after the preceding run.

[d]Products of previous two runs plus enough additional Amex product to give the 10% loading.

[e]No lanthanum detectable by neutron-activation analysis.

Victoreen survey meter, gave most of the americium at a concentration of about 1 g/liter. In a subsequent run (Fig. 4), 98% of the americium was obtained in 3.3 liters of product solution with a La/Am ratio of 1/5.* Still greater economy could be achieved by higher loadings, the combined products from several elutions being recycled with only a small fraction of the resin loaded in this final elution. The cuts containing low concentrations of americium could be recycled after acidification to 0.1-0.2 M H^+.

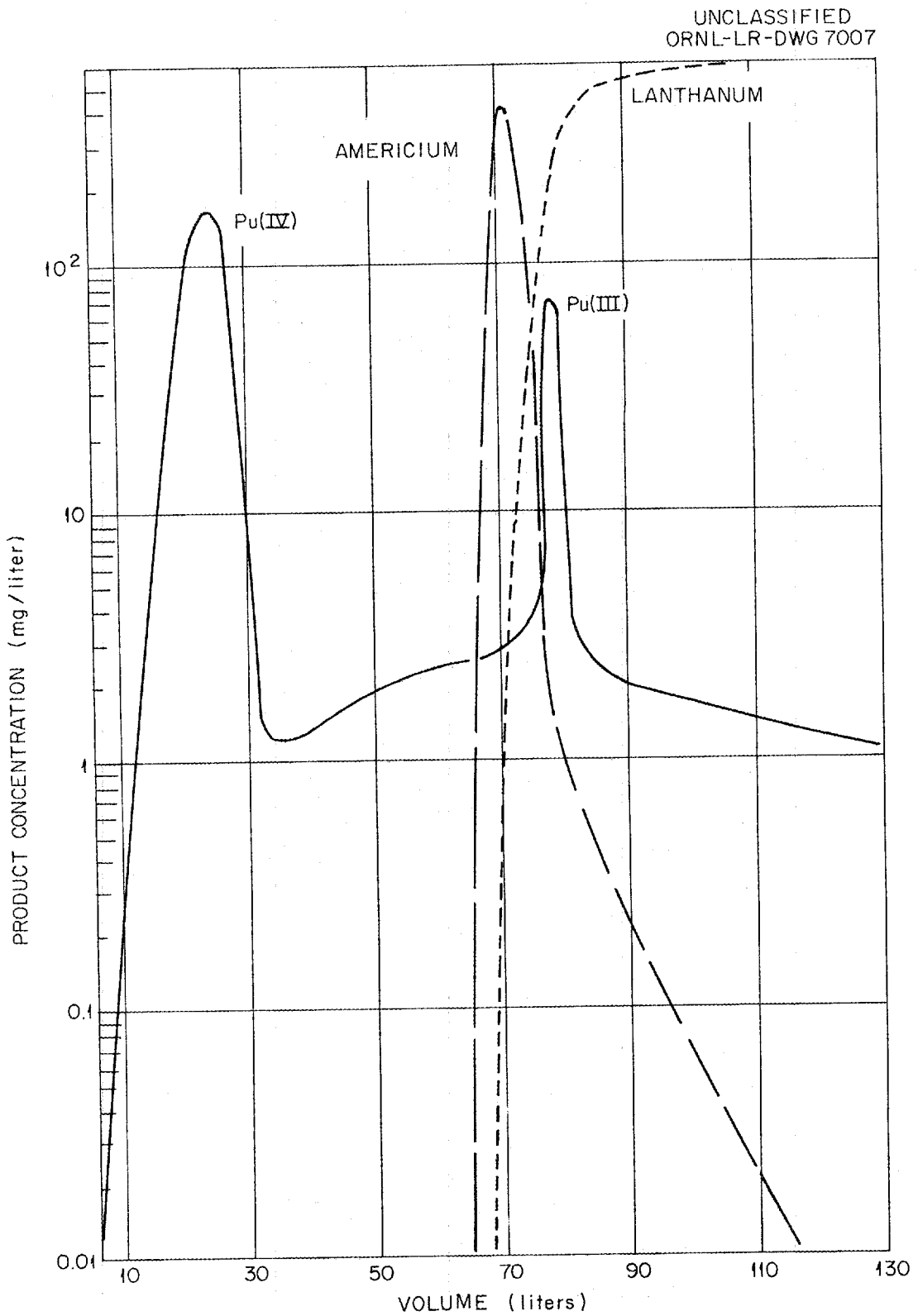
The product was purified by acidifying with hydrochloric acid to a pH of 1, reloading on a Dowex 50 column, and eluting with concentrated hydrochloric acid. The americium chloride solution was then run through a 300-ml Dowex 1 anion-exchange resin column. Iron (about 0.5 g) and plutonium were successfully removed. From this chloride solution approximately 5 g of americium was precipitated as the hydroxide. Analysis of the material gave the following data:

Volume	760 ml
Gross α activity	2.17×10^{10} c/m/ml
Americium (by TTA extraction)	2.24×10^{10} α c/m/ml = 4677 mg total
Plutonium (by TTA extraction)	1.92×10^{5} α c/m/ml
Pulse analysis of Pu fraction:	
55% Pu^{239}	~1.0 mg total
25% Pu^{240} or Am^{241}	---
20% Np^{237} (decay product of Am^{241})	~ 50 mg total
Lanthanum (by neutron activation)	< 0.027 mg/ml = < 20 mg total

A qualitative spectrographic analysis of the product gave the following estimates for impurities (accurate to about a factor of 2):

	mg/g of Am
Y	~ 100
Eu, Gd, Nd, Sm	~ 10
Al, Ca, Dy, Er	~ 3
Mg, Si, Sr	< 3
Others (including Ce, Cr, Ho, La, Lu, Mn, Mo, Ni, Pr, Si, Tb, Tm, Yb)	No lines found

*These La/Am ratios are probably accurate. Plutonium was removed from the samples before the neutron-activation analysis (see footnote p. 4).



Dowex 50 resin, 12% cross-linked, 100 to 200 mesh; 18 liters, 6-in.-diam. glass pipe
Feed: 1.45 g of Am, ~700 g of La
Eluant: 0.10*M* diammonium citrate – 0.15*M* citric acid – 0.30*M* ammonium nitrate, ~100 ml/min flow rate

Fig. 4 . Separation of Americium from Lanthanum by Citrate Elution from 50% Loaded Resin.

The chief impurity was yttrium; the elution procedure may not have given any separation of americium from yttrium.

After all the americium had been removed from the column, lanthanum was eluted with 1.5 M acetic acid--3.0 M ammonium acetate. A precipitate often formed in the column or in the product bottle during this part of the elution, but it quickly dissolved as more eluant was run through. The precipitate was identified spectroscopically as a relatively pure lanthanum compound (about 95%). At least five volume changes of this eluant were required to remove all the lanthanum so that the next americium elution would give a satisfactory product. It would be advisable, in order that lanthanum be minimized in the final product, to use more than five volume changes of this eluant before the final column elution if earlier products are recycled to the column.

The concentration obtained in the larger scale work was considerably greater than that in laboratory-scale work. This may have been due to the lower flow rate used in the actual separation work. As the elution proceeded, the americium band became narrower and more intense. Apparently the americium moved down the column faster behind the ammonium band front (where the pH was 3.3) than the ammonium band front itself moved. The americium could not move ahead of the ammonium band front, since the pH was lower, so it concentrated immediately behind it.

4.2 Hydrochloric Acid Elution

As a result of the experimental work described in Sec. 3.2, the following conditions were chosen for the hydrochloric acid elution: elution with 12.8 M HCl at a flow rate of 0.25 $ml/m/cm^2$; Dowex 50 resin, 12% cross linked, 200-400 mesh with the fines removed, loaded to 30% of capacity. This procedure gave a satisfactory yield of an acceptable product in 10-ml columns, and the product improved as the process was scaled up. The chief disadvantages were the dilute product obtained and the inconvenience of handling large volumes of

concentrated hydrochloric acid.

In a 2-in.-diam. column holding 600 ml of resin (Table 3), 80% of the americium was obtained with a La/Am ratio of 5/1 with a 15% loading, and 55% with the same ratio with a 25% loading. In both experiments 80% of the americium was obtained in four volume changes. In the first experiment the apparent lanthanum concentration increased uniformly throughout the elution, from 0.15 mg/ml in the first two volume changes to 0.7 mg/ml in the tenth. In the second experiment, with the higher loading, the lanthanum concentration reached a peak in the fourth volume change as 1.5 mg/ml and decreased slightly to 1.2 mg/ml in the tenth.

In a 4-in.-diam. column holding 3.75 liters of resin (Table 4) a 60% yield was obtained in about two volume changes with a La/Am ratio of 1/1,* and 90% yield in four volume changes with a La/Am ratio between 1/1 and 2/1. A further increase in column size, especially length, would almost certainly have further improved the results with the hydrochloric acid elution. The product of three elutions of this column added to the products from the smaller column gave the first gram of purified americium product.

Although the lanthanum concentration in the products of these runs varied, it was generally less than 0.1 mg/ml for the first four volume changes. The cuts with low La/Am ratios (A-1, 2; B-1; C-1, 2; D-1, 2; E-1, 2, 3) were combined and concentrated, by boiling, from 20.1 to 1.8 liters. Americium was precipitated from the concentrate with excess hydrofluoric acid, filtered off, and dissolved in saturated boric acid and concentrated nitric acid. The americium was precipitated from this solution with excess 10 M sodium hydroxide, washed with 1 M sodium hydroxide, filtered off, and dissolved in nitric acid. Analysis gave the following data:

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*Probably nearer 1/5; see footnote p. 4.

Table 3. Separation of Americium from Lanthanum by Hydrochloric Acid Elution from 600-ml Resin Column

Column: 2-in.-diam. glass pipe, 20 cm^2 cross-sectional area
Resin: Dowex 50, 12% cross-linked, 200 mesh, 600 ml in water
Eluant: 12.8 M hydrochloric acid, 5 ml/min flow rate

Cut No.	Cut Vol. (ml)	Total Vol. (ml)	Am (% of Total)	La/Am Ratio	
				In Cut	In Total Vol.
Run A: feed, 6.93 g of La, 54.4 mg of Am; about 15% column loading					
Displacement	515	---	0.5	---	---
1	500	500	55.8	3.0	3.0
2	500	1000	78.3	9.4	4.9
3	300	1300	84.8	33	7.0
4	250	1550	88.2	66	9.4
5	250	1800	90.4	125	12.0
6	250	2050	92.3	140	14.5
7	250	2300	93.9	167	17.3
8	250	2550	95.1	290	20.6
Run B: feed, 10.6 g of La, 87.6 mg of Am; about 25% column loading					
Displacement	421	---	0.091	---	---
1	500	500	55.8	4.7	4.7
2	515	1015	79.4	36.6	14.2
3	250	1265	83.4	108	18.7
4	250	1515	86.7	123	22.7
5	250	1765	89.1	158	26.3
6	250	2015	90.7	257	30.4
7	500	2515	94.6	185	36.5

Table 4. Separation of Americium from Lanthanum by Hydrochloric Acid Elution from 3.75-liter Resin Column

Column: 4-in.-diam. glass tube, 73 cm^2 cross-sectional area
Resin: Dowex 50, 12% cross-linked, 200 mesh; 3.75 liters in water
Eluant: 12.8 M hydrochloric acid, 18 ml/min flow rate

Cut No.	Cut Vol. (ml)	Total Vol. (ml)	Am (% of Total)	La/Am Ratio	
				In Cut	In Total Vol.
Run C: feed, 441 mg of Am, 57 g of La; about 25% column loading					
Displacement	2960	---	0.01	---	---
1	3100	3100	66.4	0.80	0.80
2	3100	6200	91.4	1.2	0.9
3	4000	10200	98.1	61	5.0
4	4100	14300	99.3	748	14.2
5	2000	16300	99.3	7000	14.5
Run D: feed, 481.5 mg of Am, 67.7 g of La; about 30% column loading					
Displacement	2740	---	< 0.01	---	---
1	3100	3100	59.5	0.77	0.77
2	3100	6200	81.9	5.1	1.95
3	4000	10200	94.5	21.7	4.58
4	4000	14200	98.1	103	10.5
5	4100	18300	99.1	380	14.3
6	4000	22300	99.2	---	---
Run E: feed, 250.6 mg of Am, 50.4 g of La; about 25% column loading					
Displacement	2690	---	0.02	---	---
1	1000	1000	12.9	1.5	1.5
2	2100	3100	62.6	1.2	1.3
3	3100	6200	91.0	0.5	1.0
4	4000	10200	98.1	20.1	2.4
5	4000	14200	99.3	510	9.3
6	4000	18200	99.7	3100	20

Volume	410 ml
Gross α activity	1.04×10^{10} c/m/ml
Americium* (by TTA extraction)	9.86×10^{9} α c/m/ml = 1.11 g total
Plutonium* (by TTA extraction)	3.70×10^{8} α c/m/ml = 2.14 g total
Lanthanum (by neutron activation)	2.77 mg/ml = 1.13 g total

*The amounts of plutonium and americium were calculated on the basis of a specific activity of 7.07×10^{7} and 3.64×10^{9} c/m/ml, respectively (52% geometry). The specific activity of plutonium was higher because of the presence of some Pu^{240}.

The americium was then precipitated again with 10 M sodium hydroxide, washed, and filtered off. The precipitate was dissolved with a minimum amount of concentrated hydrochloric acid and transferred to a 250-ml polythene bottle which served as the shipping bottle. The americium was precipitated in this bottle with sodium hydroxide, and the product was shipped as an americium hydroxide slurry.

5.0 REFERENCES

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Appendix

6.0 AMERICIUM GAMMA RADIATION

Americium is generally considered to be an alpha emitter, but it also emits low-energy gamma radiation, which introduced problems in the americium separation work. This low energy is recorded on survey instruments to a much smaller extent than on film badges, and, unless precautions are taken, personnel exposures may be higher by a factor of about 10 than would be expected from cutie-pie survey readings.

Without shielding, 25 mg of americium in a small volume in the tip of a glass centrifuge cone may read 1-2 r/hr with a cutie-pie at contact. However, 1/8 in. of lead will decrease the radiation from any reasonable amount of americium to a tolerable level, and 1/4 in. will absorb it essentially completely. Self-shielding in large volumes is considerable—1 g of americium in a 400-ml volume in a graduated cylinder reads about 4 r/hr with a cutie-pie at contact. A lead sheet wrapped around equipment such as columns and bottles was an effective shield. Shadow shielding was not effective because of the strong back-scattering of the low-energy radiation. As an example of the type of problem encountered, survey instruments showed essentially no radiation from large bottles containing only a few milligrams of americium, but a film badge showed a significant amount, even several feet away. It is important that personnel working with large amounts of americium shield and keep away from equipment containing even a few milligrams of americium, even though a survey meter indicates a radiation level well below tolerance. In the americium separation work reported here, with experience and care, personnel exposures were held to entirely reasonable amounts, even without elaborate shielding or remote operation.