145
8-15-78
LA-7057

14. 1842
UC-4

Issued: February 1978

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Daryl Knab

los alamos
scientific laboratory
of the University of California
LOS ALAMOS, NEW MEXICO 87545

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**UNITED STATES
DEPARTMENT OF ENERGY
CONTRACT W-7405-ENG. 36**

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A PROCEDURE FOR THE ANALYSIS OF AMERICIUM IN COMPLEX MATRICES

by

Daryl Knab

ABSTRACT

A radioanalytical procedure for the analysis of americium in complex matrices has been developed. Clean separations of americium can be obtained from up to 100 g of sample ash, regardless of the starting material. The ability to analyze large masses of material provides the increased sensitivity necessary to detect americium in many environmental samples. The procedure adequately decontaminates from rare earth elements and natural radioactive nuclides that interfere with the alpha spectrometric measurements.

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I. INTRODUCTION

The Environmental Studies Group at the Los Alamos Scientific Laboratory (LASL) is investigating the biogeochemical behavior of ^{241}Am in the natural environment.^{1,2} Analysis of americium in environmental samples is difficult because of the very low concentrations available and inherent problems of chemically isolating americium from complex matrices.^{3,4} A radiochemical procedure suitable for the simultaneous analysis of several samples for americium from large quantities of several matrices was necessary for this study. A procedure has been developed that meets these requirements and is also applicable to other analytical problems relative to americium. The procedure yields clean separations of americium from up to 100 g of sample ash, regardless of the starting material. It adequately decontaminates from rare earth elements (REE), and natural and man-made actinides and their decay products (ACT) that interfere with alpha spectrometric measurements. The procedure is designed for large samples, but can be shortened for smaller or less complex samples by omitting portions of the procedure.

The procedure is lengthy, but not esoteric or complex and is therefore suitable for simultaneous analysis of many samples. The procedure is outlined here and a detailed procedure is given in the Appendix. The extraction is insensitive to chemical conditions, therefore samples can be dissolved by any method suitable for the matrix being analyzed. Even the final acid dissolution before extraction is not critical, but 6 M HCl is convenient and is recommended. The initial step

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is a liquid-liquid extraction of americium into DEHPP that makes the procedure extremely versatile and selective. The extraction procedure is based on work reported by Myasoedov et al.^{5,6} and has been adapted for environmental samples.⁷ No matrix encountered has interfered with the extraction, although bones do require a more concentrated extracting solution. The plutonium anion exchange column is the classic method for plutonium separation.⁸ It can be run before or after the extraction or eliminated if plutonium is not being analyzed. It is necessary that plutonium be in the correct oxidation state for each step. The methanol-nitric acid column reported by Majors et al.⁹ is an excellent cleanup step for separating residual macromaterials including alkaline earths, other actinides, and most rare earths. Many americium samples can be plated directly after the methanol-nitric acid column. For samples with a high REE content, an additional Am-REE separation step is required. The thiocyanate anion column reported by Livingston et al. is used for this purpose.¹⁰ The resultant americium from either column is then electrodeposited before measurement by alpha spectroscopy.

II. RESULTS

Some results obtained from the procedure are tabulated in Table I. A Health and Safety Laboratory (HASL) soil standard is the only external standard analyzed so far. The recoveries and internal agreement were good and accuracy relative to the reported HASL value was acceptable. The analyses results averaged about 10% high, which probably reflects calibration differences. Recoveries from routine analysis of soils and vegetation samples are adequate. The average for the 56 vegetation samples includes four samples with <20% recovery, which is about the expected level of anomalous low recoveries for routine analysis. Bone sample recoveries reported here are unacceptable, but they reflect changes in the extraction procedure not applicable to bone sample analysis, which can be circumvented.

TABLE I
AMERICIUM RESULTS

Sample Type	Sample Weight (g)	Number of Samples	Recovery (av %)	Result (PCI/g)
HASL soil standard	50	2	90	0.20 ± 0.01 (1.0) ^a
HASL soil standard	10	6	85	0.218 ± 0.003 (1.1) ^a
Blank soil	100	4	83	0.0001 ± 0.0003
Lichen	100	18	83	0.020 ± 0.001
Vegetation	10-300	56	65	---
Bone	50-300	13	41	0.0003 ± 0.0002

^aLASL value/HASL standard value.

III. EXTRACTION

Figure 1 shows some detail on the extraction procedure. It uses a synergistic extractant composed of $\sim 0.045M$ HDEHP $\cdot$ $0.015M$ P_2O_5 in cyclohexane ($0.015M$ DEHPP). The DEHPP extracts actinides, lanthanides (elements containing f shell electrons), and radium from very strong acid solutions, even up to concentrated solutions of HCl and $HClO_4$ acids. Other elements, including common matrix materials, do not extract nor do they interfere, making the method applicable to a wide variety of samples.

The DEHPP is prepared by adding P_2O_5 to bis (2-ethylhexyl) hydrogen phosphate (HDEHP) and allowing the mixture to react overnight before diluting with cyclohexane. If the P_2O_5 is added to HDEHP prediluted with cyclohexane, a weaker and less consistent product is obtained. The stronger reagent yields quantitative extraction at lower concentrations, $0.015M$ opposed to $0.1M$, and gives better phase separation and less phosphate residues on decomposition of the back extract.

The extraction is sufficiently strong that back extraction is difficult, but carbonate solutions effectively back extract americium and anything else that extracts, plus P_2O_5 products and part of the HDEHP. A saturated solution of ammonium carbonate is recommended, although it is necessary to dilute the organic phase with ethyl acetate to effect complete back extraction. Acid decomposition of the back extract with $HClO_4$ is necessary to destroy residual HDEHP, which interferes with subsequent plutonium analysis. The decomposition results in insoluble phosphates that must be dissolved in HF, and americium is then conveniently coprecipitated with YF_3 .

Extraction of americium from large bone samples is not complete with $0.015M$ DEHPP, although earlier studies using $0.1M$ DEHPP gave good americium recoveries through the extraction steps from up to 100 g of bone ash.⁷ Consequently, it appears that bone samples must be treated as special cases in the extraction steps. The remainder of the procedure is unchanged for bone samples. The $0.1M$ DEHPP for extracting americium from bone samples is prepared by adding 14 g P_2O_5 to 97 g HDEHP dissolved in 300 ml of cyclohexane; after stirring for 1 h, the mixture is further diluted to 1 l with cyclohexane and allowed to settle overnight.

IV. PLUTONIUM ANION EXCHANGE COLUMN

The extraction step is highly selective, but still leaves REE, radium, ACT, and a small percentage of matrix material with the americium. The anion column for plutonium shown in Fig. 2 effectively removes plutonium and thorium, but is only necessary for samples requiring plutonium analysis or for samples with high actinide concentrations. In our work, very high levels of plutonium are not uncommon and plutonium analysis is almost always required. Therefore, this step is used routinely. Although the extraction is designed basically for collecting americium, it is also applicable to plutonium samples whether americium analysis is required or not. The plutonium anion column is relatively insensitive to conditions, but large soil and bone samples can adversely affect the plutonium recoveries, necessitating an initial collection step.

Fig. 1.
DEHPP extraction.

Fig. 2.
Plutonium ion exchange column.

V. METHANOL-HNO₃ ANION EXCHANGE COLUMN

Additional decontamination of americium from residual matrix material, ACT, radium, and most REE is accomplished with the methanol-nitric acid anion column shown in Fig. 3. Radium decontamination has been a chronic problem for environmental americium samples because some of its isotopes and their daughters interfere directly with alpha spectroscopy of americium, radium is frequently present in much higher concentrations than americium and radium thinks it is an actinide. Being an alkaline earth, radium should not extract, but apparently does and remains with americium through the entire procedure, except for the methanol-HNO₃ column where it is eliminated effectively.

The column originally was designed as an Am-REE separation step,⁹ but was only partially successful. Heavy rare earths, samarium to lutetium, scandium and yttrium, elute with 60% methanol + 40% 6M HNO₃, but light rare earths, promethium to lanthanum, which account for 60% of the REE by weight, remain on the column and elute with americium in 6M HNO₃. We found that americium can be eluted with 60% methanol + 40% 2.5M HNO₃, leaving praeosodymium, cerium, and lanthanum on the column. Neodymium tends to split under the conditions used and promethium, which has no natural abundance, has not been studied. For fission product separation, promethium undoubtedly will be a problem.

The AG MP-1 resin, an anion exchange resin available from Bio Rad, Richmond, California, is recommended for the column. The macroporous resin alleviates the problem of slow chemical equilibration of alcohol-mineral acid solutions in ion exchange systems. Although this column will work using standard resins, the separation is improved significantly by using the MP-1 resin. Based on available data obtained from this column, it appears possible to design a system applicable for sequential elution of all the lanthanides and actinides.

The methanol-nitric acid column also separates americium from Pu⁴⁺ and thorium. Plutonium can be eluted and plated selectively, but the column takes over 12 h to run through the complete procedure. The column also separates curium from americium. We assume that curium elutes ahead of americium, but this has not been verified. If ²⁴⁴Am is being used as a tracer for curium, this column will not be useful in its present form.

Americium samples low in REE content can be plated directly after the methanol-HNO₃ column. The decision to plate directly or run an additional REE separation step is determined subjectively by experience. Samples containing 1 g of local soil yield alpha spectra that are slightly degraded, but resolvable. Thus, samples containing less than 1 g of soil such as tissue, bone, and small clean vegetation samples can be electrodeposited directly.

VI. THIOCYANATE ANION COLUMN

For soil samples and samples that contain high soil content such as large vegetation samples, hides, feathers, and animal stomachs, an additional REE separation is required. The thiocyanate anion column shown in Fig. 4 yields a true group separation of REE and actinides. The REE pass through with 2M SCN⁻ and the actinides can be eluted as a group with dilute mineral acid; radium also elutes with the actinides. Iron and other strongly complexed metals remain on the column. It is necessary to control the pH of the 2M NH₄SCN solution. Above pH 5 the REE elution is retarded and below pH 2 actinides are eluted with SCN⁻. Optimum separation is obtained at about pH 3.

The main disadvantage to the column is the residual SCN⁻ that elutes with the americium. It must be decomposed before electrodeposition, which results in H₂SO₄ and elemental sulfur and

Fig. 3.
Methanol-nitric acid anion column.

Fig. 4.
Ammonium thiocyanate anion column.

leaves a mixture difficult to work with, although the electrodeposition is not affected. Attempts to run the thiocyanate column before the methanol-nitric acid column have been unsuccessful. If the thiocyanate column is run directly after a fluoride precipitation step, the column does not run properly. Something in the fluoride precipitation or dissolution interferes with the REE elution causing them to partly load on the column and elute with the actinides. No convenient method of eliminating the interference has been found.

VII. AMERICIUM ELECTRODEPOSITION

Electrodeposition of americium shows some interesting characteristics. A pure sample plated by a given procedure plates at a given rate. A sample passed through some chemistry plates at a slower rate with plating taking two to five times longer for complete electrodeposition. This poses some problems because it is difficult to observe. When optimizing a plating procedure, pure standards usually are used and contaminants are added directly to check their effect, but the slower plating rate may not be observed. The result is development data that indicate the plating procedure works very well, but may actually give low recoveries. The electroplating rate of pure standard samples passed through either anion column of this procedure is slower than for pure samples, and slower than for pure standard samples contaminated with small amounts ($<50 \mu\text{g}$) of material that might be present as interferences in the americium plating solution.

Plating from NH_4Cl solution titrated to the methyl red end point¹¹ is recommended over the oxalate method¹² or the $\text{Na}_2\text{SO}_4 \cdot \text{NaHSO}_4$ method¹³ because it is affected least and can be forced to completion. The electrodeposition is allowed to proceed at 0.6 A until the plating solution evaporates to the top of the anode. At some point in the evaporation, the conditions are right for quantitative deposition. It is necessary to quench and remove the sample from the plater before the solution evaporates too far. If electrical contact is broken, americium and steel are stripped from the plate.

VIII. CONCLUSIONS

The procedure is long, but not overly complex, and is quite reliable. In early stages of production, tracer recoveries were greater than 50% on more than 90% routine samples using the complete procedure. The procedure is applicable to large amounts of all matrices and adequately decontaminates americium from REE and ACT at levels encountered in environmental samples. Currently, 24 samples are being processed simultaneously so that about 48 samples can be completed in 3 wk, requiring about 3 h per sample to process americium and plutonium through the complete procedure. For samples not requiring the complete procedure, cost effectiveness is improved.

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APPENDIX

AMERICIUM AND PLUTONIUM FROM LARGE ENVIRONMENTAL SAMPLES

The procedure is suitable for all sample types containing up to 100 g of sample ash. The final thiocyanate column is unnecessary for samples low in rare earth elements.

The sample dissolution method has no effect on the extraction, thus the best method for dissolving the sample matrix being analyzed is recommended.

Bone sample analysis requires a different organic extractant.

DEHPP EXTRACTION PROCEDURE

1. Add 20 ml HCl to the dissolved sample residue. Evaporate just dry. Add 200 ml HCl. Boil 1 h to convert the solution to 6M HCl. (If the sample is not dissolved, add additional 6M HCl until dissolution is complete.) Let cool to room temperature. Add 10 ml H₂O, and let set overnight.

2. Add 50 ml 0.015M DEHPP (0.1M DEHPP for bone samples) to the solution. (Maintain an organic:aqueous ratio of >1:5.) Stir 10 min on a magnetic stirrer. Pour the mixture into a separatory funnel. Let the phases separate for 10 min or until separation is complete. Return the aqueous phase and interface to the beaker.

3. Add 50 ml DEHPP to the aqueous phase. Stir 5 min. Gently pour the mixture into the separatory funnel. Let the phases separate. Drain the aqueous phase into the beaker. Retain the interface.

4. Add 100 ml 6M HCl to the organic. (Maintain an organic:aqueous ratio of 1:1.) Mix 1 min and let the phases separate. Drain the acid wash into the beaker.

5. Dilute the organic 1:1 with ethyl acetate, and let set 30 min. Add 50 ml saturated (NH₄)₂CO₃ · 10% NH₄OH to the organic. (Maintain an organic:aqueous ratio of 4:1.) Carefully release the gas pressure. Shake for 3 min. Let the phases separate. Drain the aqueous phase into a 200-ml tall form beaker.

6. Add 50 ml (NH₄)₂CO₃ · 10% NH₄OH to the organic. Shake for 3 min. Let the phases separate. If separation is slow, let set overnight. Drain the aqueous phase into the beaker. Include the interface.

7. Evaporate the solution dry on low heat. Add 20 ml HNO₃, 20 ml HCl and evaporate dry. Add 10 ml HClO₄ and 10 ml HNO₃. Cover and evaporate to 1 ml. Do not let the beaker dry spot.

8. Transfer the residue to a 50-ml plastic tube with 3M HCl · 3M HF to about 30 ml. Heat in a hot bath 10 min. If a large precipitate remains, add HF 1 ml at a time and heat 10 min between additions until most of it dissolves. Add 2 ml 5M NH₄OH · HCl. Let set 10 min.

9. Add 3 ml HF. Add 5 mg Y carrier; stir and heat 20 min. Add 5 mg Y carrier; stir and heat 20 min. Cool 20 min. Centrifuge for 5 min. Decant off the supernate. Wash the precipitate with 25 ml 1M HCl · 1M HF. Centrifuge and discard the wash.

10. Add 3 ml saturated H₃BO₃. Slurry the ppt. Add 3 ml HNO₃ and heat in a hot-water bath until the ppt dissolves. Dilute to 30 ml with 7.2M HNO₃. Add 1 ml 1M NaNO₂. Save for the plutonium anion column. Centrifuge off any insoluble residue before loading on the plutonium column.

PLUTONIUM ION EXCHANGE SEPARATION

1. Add 1 ml of 1M NaNO₂ to the sample solution and mix thoroughly. Allow the sample to set for 1 h.

2. Prepare anion exchange columns as follows.

(a) Pack a small glass wool plug tightly into the tip of the ion exchange tube, filling only the tapered tip.

(b) Transfer the AG 1x2, 50 - 100 mesh resin slurry to the ion exchange tube until the settled resin bed is just up to the neck of the column (19 cm high by 0.7 cm i.d.).

(c) Wash the resin bed with three 20-ml portions of 7.2M HNO₃. The final wash should be done with a wash bottle making sure all resin is washed from the reservoir into the column proper. The final preconditioned column should be 13 cm high.

(d) Pack a glass wool plug on top of the resin to contain it.

3. Pass the sample solution through the resin column in 10-ml portions, allowing each portion to drain to the top glass plug. Wash the tube with 5 ml 7.2M HNO₃ and add to the column.

4. Wash the column with 20 ml of 7.2M HNO₃. Save the load solution and the first wash for the americium procedure.

5. Wash the column twice more with 50 ml 7.2M HNO₃.

6. Wash the column with two 20-ml portions of 8M HCl. Discard these washes.

7. Elute the plutonium from the column into a 100-ml beaker with four 20-ml portions of a freshly prepared solution containing 1 g/l NH₄I in 1M HCl. Save for the plutonium electrodeposition procedure.

PLUTONIUM ELECTRODEPOSITION PROCEDURE

1. Add 1 ml HNO₃ to the eluate and evaporate to dryness. (The solution may be evaporated on an asbestos-covered hotplate at low heat overnight.)

2. Add 1 ml HNO₃ and 5 ml HCl to the residue and evaporate *just* to dryness. (*Avoid baking the sample.*)

3. Add 5 ml HCl and evaporate to the first dry spots on low heat. (*Avoid baking the sample.*)

4. Wash down the sides of the beaker with about 5 ml HCl, cover and store for at least 24 h.

5. Evaporate the solution at medium heat just to the point at which a dry spot cannot be covered by swirling the beaker and allow the beaker to cool.

6. Add 3 ml of 4% (NH₄)₂C₂O₄ and transfer the solution to an electrodeposition cell with 0.1M HCl washes until the cell is about half full.

7. Electrodeposit for 30 min at 1 A.

8. With the current on, quench the reaction by filling the cell with 1% NH₄OH solution and remove the cell from the electrodeposition apparatus.

9. Empty the cell and rinse the planchet with water and then with ethanol.

10. Ignite the planchet to a dull red in the flame of a bunsen burner, allow it to cool, and store it in a labeled envelope.

METHANOL-NITRIC ACID ANION COLUMN

1. Evaporate the americium fraction from the plutonium column just dry. Add 5 ml 6M HNO₃ and heat gently to dissolve the residue. Cover and let set.

2. Prepare the anion exchange column as follows.

(a) Pack a small glass wool plug loosely into the tip of the ion exchange tube.

(b) Transfer MP-1, 100-200 mesh resin slurry to the tube to a settled height of 11 cm by 0.7 cm i.d.

(c) Wash the resin with two 20-ml portions of 60% ethanol · 40% 6M HNO₃. The final wash should be done with a wash bottle making sure all resin is washed into the column tube.

(d) Cover the column with a watch glass to minimize alcohol evaporation.

3. After adding the first wash solution, add 7.5 ml ethanol saturated with NaNO₂ to the sample. Mix and transfer the sample to the column with a disposable pipet. Wash the beaker and column with 5 ml 60% ethanol · 40% 6M HNO₃ solution.

4. Wash the column twice with 20 ml 75% methanol · 25% 6M HNO₃.
5. Wash the column twice with 20 ml 60% methanol · 40% 6M HNO₃. Discard the load and wash solutions.
6. Elute americium with 30 ml 60% methanol · 40% 2.5M HNO₃ into a 100-ml beaker. Evaporate to 2 ml on low heat.

AMMONIUM THIOCYANATE ANION COLUMN

1. Evaporate the americium fraction from the methanol column just dry. Do not bake. Add 5 ml HCl and evaporate to ~2 ml.
2. Prepare an ion exchange column as follows.
 - (a) Pack a small glass wool plug tightly into the tip of the ion exchange tube filling only the tapered tip.
 - (b) Transfer the AG 1x8 resin slurry to the ion exchange tube until the settled resin is 17 cm by 0.7 cm i.d.
 - (c) Wash the resin bed with two 25-ml portions of 2M NH₄SCN, pH 3. The final wash should be done with a wash bottle making sure all the resin is washed into the column proper.
3. Add 2 ml 6M NH₄SCN to the sample. Titrate the pink Fe(SCN)₃ color to clear with dropwise addition of NH₄OH. Back titrate just to the pink color with 1M HCl. Add 1 drop excess 1M HCl.
4. Load the sample on the column with a transfer pipet. Wash the tube with 10 ml 2M NH₄SCN. Load on the column. Allow each fraction to drain down before adding the next.
5. Elute rare earths with four 25-ml portions of 2M NH₄SCN. Let each addition drain to the plug before adding the next.
6. Elute americium into a 100-ml beaker with two 25-ml portions of 1M HCl. Save for the americium electrodeposition procedure.

AMERICIUM ELECTRODEPOSITION PROCEDURE

1. Evaporate the americium fraction to dryness on low heat. Cautiously add HNO₃ a little at a time until the violent reaction ceases. Add 10 ml HNO₃ and 10 ml HCl. Evaporate to 1 ml. Add 10 ml HNO₃ and 10 ml HCl. Evaporate until most of the H₂SO₄ has fumed off.
2. Add 2 ml HClO₄ to the sample. Evaporate just dry. Add 5 ml HCl. Evaporate to 0.5 ml. Add 5 ml HCl. (Sample may be covered and stored at this point.) Evaporate to 0.5 ml. Let cool.
3. Add 2 ml 4M NH₄Cl and 1 drop methyl red. Titrate from red to yellow with dropwise addition of NH₄OH. Back titrate just to the red endpoint with 1M HCl. Transfer to a plating cell with H₂O to a total volume of 8.0 ml.
4. Connect the cell to the power supply and adjust the current to 600 mA. Plate onto a 12-mm-diam stainless steel surface until the solution in the cell evaporates to the top of the anode. *Do not* let the solution evaporate until contact is broken.
5. Fill the cell with 5% NH₄OH and continue plating for 30 s. Remove the cell from the plater and rinse with 5% NH₄OH into the original beaker.
6. Dismantle the cell. Rinse the disk with H₂O and then with alcohol.
7. Submit the plate for alpha spectroscopy.

SPECIAL REAGENTS AND MATERIALS

1. 0.015M DEHPP. Add 2.13 g P_2O_5 to 14.5 g bis (2 ethylhexyl) hydrogen phosphate. Allow the mixture to react overnight. Dilute to 1ℓ with cyclohexane and let settle overnight.

2. 0.1M DEHPP. Dissolve 97 g bis (2 ethylhexyl) hydrogen phosphate in 300 ml cyclohexane. Add 14 g P_2O_5 and stir 1 h. Dilute to 1ℓ with cyclohexane and let settle overnight.

3. Plutonium Anion Exchange Resin. AG 1x2, 50-100 mesh, Cl form resin is available from Bio-Rad Laboratories, Richmond, California.

4. Methanol-Nitric Acid Anion Exchange Resin. AG MP-1, 100-200 mesh resin is available from Bio-Rad Laboratories, Richmond, California.

5. Ammonium Thiocyanate Anion Exchange Resin. AG 1x8, 100-200 mesh resin is available from Bio-Rad Laboratories, Richmond, California.

6. Ammonium Thiocyanate 2M. Dissolve 1 lb NH_4SCN in 3ℓ H_2O . Adjust the pH to 3.0 with HCl.