PAPER

Separation and Analysis of Am and Pu from Large Soil and Sediment Samples

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Americium and plutonium occur in soil and sediment samples as a result of various anthropogenic sources. In most cases their concentration is very low, requiring analytical techniques with low detection limits. To this end, we have developed a method for the determination of Am and Pu in 50-g samples. Diphonix Resin, an ion-exchange resin with geminally substituted diphosphonic acid groups, is used for group separation of actinides and lanthanides from the accompanying matrix. These groups are then eluted from the resin with 0.5 M 1-hydroxyethane-1, 1-diphosphonic acid (HEDPA). After decomposing the HEDPA using a very effective and simple "Fenton's Reagent" technique, Am and Pu are separated from the lanthanide elements and from each other via standard extraction chromatographic techniques. Because the matrix has been effectively eliminated by the Diphonix treatment, each sample behaves uniformly and predictably throughout the chemical separations. We also describe a problem concerning an intensive reaction of a load solution with TRU Resin that was encountered during the development stages of the new method. The problem became very apparent when larger amounts of ascorbic acid were added to reduce Fe^{3+} to Fe^{2+}. A revised load solution, using $NaNO_2$, has eliminated this problem. We also present findings on how the CeF_3 micro-coprecipitation source-preparation method can be improved by adding sufficient amounts of HF to obtain quantitative recoveries.

Introduction

Measurement of radionuclides in environmental samples is an important part of environmental monitoring and radiation protection. Major components of the environment include soil and sediments and measurement of actinide elements in such samples is frequently required. Besides natural uranium and thorium and decay products, the transuranium species americium (Am) and plutonium (Pu) are the most often monitored alpha-particle-emitting radioactive elements. Under most conditions, the concentration of Pu and Am

in environmental samples is negligible compared to primordial radionuclides, requiring very sensitive analytical techniques with low minimum-detectable activities.

This work describes a method of Am and Pu determination of large, 50-g soil and sediment samples. The method is an extension of the procedure developed by Kim et al. (2000) in which Am, Pu, U, and Th were analyzed for 10-g samples. In the analysis of any complex environmental sample, one often encounters difficulties caused by so-called "matrix effects," i.e., chemical interferences resulting from various ions present in the sample. The main strategy of the present procedure is to separate the actinide elements from the soil matrix early in the procedure to avoid these effects. We use Diphonix ion-exchange resin as the matrix elimination approach. The actinides are than separated from each other as well as lanthanides and other minor impurities using TRU and TEVA Resin columns. The final sources are prepared by cerium fluoride micro-precipitation/filtration and measured by alpha-particle spectrometry.

Experimental
Reagents and materials
The 1-hydroxyethane-1, 1-diphosphonic acid (HEDPA) solution used in our procedure is industrial grade obtained from Albright & Wilson Americas Inc. (4851 Lake Brook Drive, Glen Allen, Virginia 23060). High-purity HEDPA may be obtained from Fluka (P.O. Box 2060, Milwaukee, WI 53201). All other reagents are commonly available analytical grade acids and chemicals. A complete listing of all reagents as well as a step-by-step protocol of the method presented in this paper is available by contacting the authors. The resins used in this procedure (Diphonix Resin, TRU Resin and TEVA Resin) are available from Eichrom Technologies (8205 S. Cass Avenue, Suite 111, Darien, IL).

Diphonix resin
An ion-exchange resin that contains geminally substituted diphosphonic acid groups chemically bounded to a styrene-based polymer matrix. The resin also contains strongly hydrophilic sulfonic acid groups. It has the capability to selectively remove actinide and lanthanide elements from solution, even in the presence of macro concentrations of many elements that occur commonly in environmental samples (Horwitz et al., 1993; Chiarzia et al., 1997).

TRU resin
A resin containing the extractant octylphenyl-N, N-di-isobutyl carbamoylphosphine oxide (CMPO) dissolved in tri-n-butyl-phosphate (TBP) and coated onto inert plastic beads. TRU Resin extracts tri-, tetra-, and hexa-valent actinides. It has high-extraction capacity and excellent selectivity for transuranium elements over common ions.

TEVA resin
A resin containing an aliphatic quaternary amine extractant coated onto an inert support. It has a very high selectivity for tetravalent actinides. In our case it is used to separate Am from lanthanides in a thiocyanate medium.

Samples
For testing this procedure samples were obtained from DOE's Environmental Measurements Laboratory (EML) and the International Atomic Energy Agency (IAEA). In addition, during the development stages of this research we used some soil and sediment samples collected for another project in Scarboro (abbreviated SCS), a small town near Oak Ridge, Tennessee.

Apparatus
Plutonium and Am measurements were performed using an Ortec (Perkin-Elmer, Oak Ridge, Tennessee) Octete alpha-particle-spectrometry system containing eight ion-implanted silicon detectors. The average counting efficiency of the detectors is around 20%. The efficiencies and the backgrounds of all detectors are monitored on regular basis. Samples used for developmental work with high (>16 Bq) activity Pu and Am tracers were measured by liquid scintillation analysis

(LSC) using a low-background Wallac 1414 "Guardian" LSC with alpha-/beta-particle separation.

Overview of method, sample preparation

Flow diagrams showing the overall strategy for matrix removal by Diphonix and separation via TRU and TEVA Resins are shown in Figures 1 and 2, respectively. For 50-g samples, leaching with mineral acids is the most convenient way to recover anthropogenically-derived radionuclides such as Am and Pu. We used this approach despite the possibility that, in some cases, some insoluble actinides might remain unleached (e.g.,

highly fired Pu oxides). In such cases, total dissolution techniques for soil and sediments are preferred but are best suited for smaller sample amounts (Croudace et al., 1998).

To remove possible interference from organic matter, each sample is combusted for 4 hours at 550 °C in a muffle furnace prior to the leaching procedure. After cooling, calibrated radiotracers (125 mBq ^{243}Am and 150 mBq ^{242}Pu), 150 mL 6 M HCl and 150 mL 8 M HNO$_3$ are added to each sample in a 600-mL Pyrex beaker. The sample is refluxed for 4 hours at ~90 °C. The leachable fraction is than separated from the residual soil by
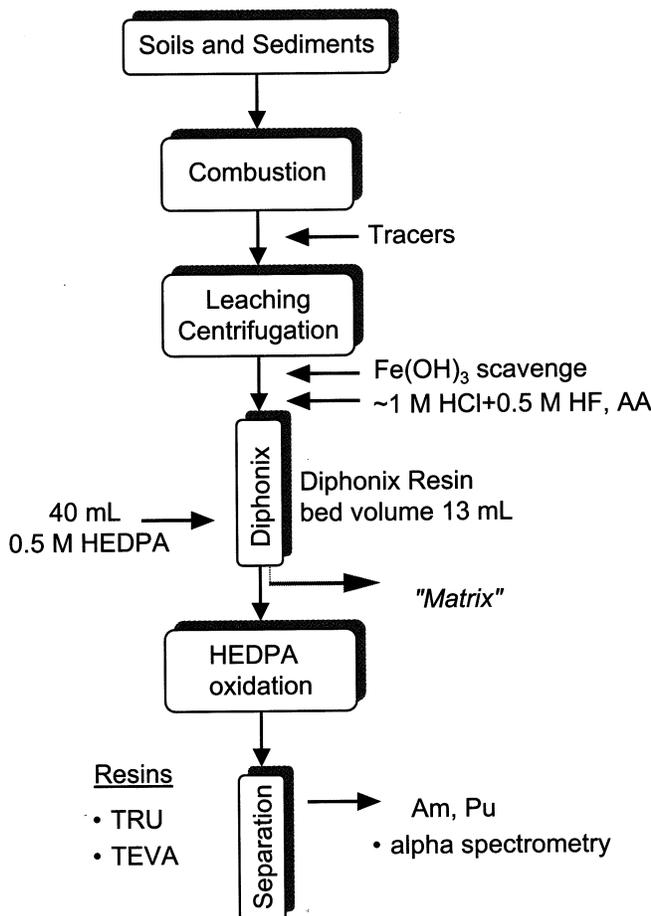

Figure 1   Schematic diagram of the overall analysis scheme for Am and Pu in 50-g soil/sediment samples.
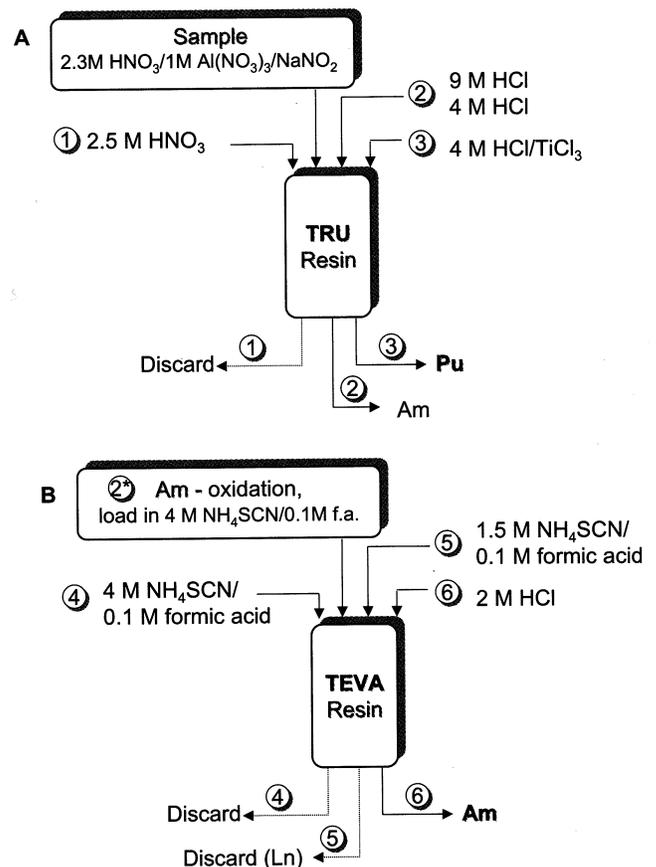

Figure 2   (A) Separation scheme of Am and Pu using TRU Resin column with 2.5 M HNO$_3$-1 M Al(NO$_3$)$_3$-0.06 M NaNO$_2$.
(B) Separation of Am from lanthanides on TEVA Resin column from 4 M NH$_4$SCN / 0.1 M HCOOH.

centrifugation. Actinides are than co-precipitated from this solution with $Fe(OH)_3$ at pH ~8 by careful addition of NH_4OH.

Matrix removal

According to the data of Horwitz et al. (1993) and Chiarzia et al. (1997) the adsorption efficiency of Am on Diphonix resin increases with decreasing acidity. In this procedure we prepare a load solution of ~1 M HCl + 0.5 M HF by dissolving the $Fe(OH)_3$ precipitate with sufficient amounts of concentrated HCl. The HF is employed to fully complex Al^{3+} with F^- and solubilizes any Si that leached off the soil. According to our experience, 35-mL concentrated HCl and 10-mL concentrated HF diluted to 500 mL with deionized water results in a solution which will dissolve the $Fe(OH)_3$ precipitate even from samples containing relatively high amounts of iron (e.g., EML 0009 Quality Assurance Program soil sample), and gives essentially 100% Am and Pu recovery on Diphonix Resin. Ascorbic acid is added to the load solution to reduce Fe^{3+} to Fe^{2+} that eliminates the Fe^{3+} sorption onto the Diphonix resin. To ensure that all Fe is reduced before loading onto the Diphonix column, a small drop of the sample is added to a grain of ammonium thiocyanate that turns red in the presence of Fe^{3+} and remains clear if all iron has been reduced to Fe^{2+}.

In some samples, insoluble precipitates were observed after the addition of HF. Since such insoluble fluorides could scavenge actinides as well, we took steps to ensure that the precipitates were dissolved. We first centrifuged the load solution and loaded the supernatant onto a Diphonix resin column (1.5-cm-diameter Kontes borosilicate glass column; 13-mL wet resin vol., 100-200 mesh, preconditioned with 10-mL 2 M HCl + 0.5 M HF). The residue was then treated with a small volume of 2 M HCl and addition of solid boric acid until it redissolved. This solution was loaded onto the Diphonix column separately after the supernatant solution already passed the column.

We have found that the soluble matrix from 50-g samples can be handled with a wet volume of 13 mL of Diphonix resin. After loading of the sample solution, 60 mL of 2 M HCl - 0.5 M HF is added to the column to rinse the resin (2 M HCl is used for efficient removal of Ca^{2+} and Fe^{2+}) to ensure complete remove of matrix elements. We maintain the flow rate of the solution at less than ~1 mL/min (0.57 mL/cm²/min). Actinides (and lanthanides which are also retained) are then eluted from the column using 40 mL of 0.5 M HEDPA solution.

Using a high ^{241}Am spike in a soil matrix solution, Kim et al. (2000) found that the minimum volume of 0.5 M HEDPA solution required to elute Am from 10 mL (wet vol.) Diphonix resin was about 20 mL. Using a similar approach, we found that 40 mL of 0.5 M HEDPA is sufficient for complete elution of both Am and Pu from 13-mL (wet vol.) Diphonix.

Oxidation of HEDPA

Two techniques were described by Kim et al. (2000) for the oxidation of HEDPA solution in the "10-g-sample procedure": (1) "Fenton's Reagent" technique (Fenton, 1894); and (2) ozonation. It was concluded that the "Fenton's Reagent" approach is more convenient for its speed and simplicity. To summarize this procedure briefly, 1 mL of concentrated HNO_3 and 40 mL of 30% H_2O_2 is added to a 600-mL Pyrex beaker containing the eluate. The sample is warmed to ~90 °C, 0.17 g of ammonium ferrous sulfate is added, and the heating is continued for 40 min. A loose-fitting glass cover is placed on top of the beaker allowing the solution to evaporate slowly. We add 10-mL increments of 30% H_2O_2 every 10 min during the 40 min heating period. After 40 min, only 5-10 mL of solution remains (total volume of H_2O_2 added to this point is 70 mL). A t this stage the cover lid is removed and a final 5 mL of 30% H_2O_2 is added to ensure complete oxidation of HEDPA. A spontaneous thorough bubbling of the solution when the H_2O_2 is added indicates that the oxidation is complete (Figure 3). At this point, a sample is evaporated without the lid until only about 3-mL solution remains.

drop remains. The purpose of the sulfuric acid addition is to keep the material in solution and not let the fraction dry into the beaker. The residue is dissolved in ~1 drop of concentrated formic acid, evaporated again until a very small drop remains, another drop of concentrated formic acid is added and heated carefully until the walls of the 100-mL beaker are completely dry. The residue is then dissolved in 10 mL 4 M ammonium thiocyanate - 0.1 M formic acid and loaded onto a standard-size TEVA Resin column. We rinse the beaker with 5 mL of 4 M NH$_4$SCN/0.1 M formic acid and pour this solution onto the column. The lanthanides are completely rinsed out of the column with an additional 10 mL of 1.5 M NH$_4$SCN/0.1 M formic acid. Finally, the Am is eluted with 20 mL of 2 M HCl.

Source preparation and alpha-particle counting

Alpha-particle sources for Am and Pu are prepared by CeF$_3$ micro co-precipitation and filtration (Sill, 1987). Americium is recovered from TEVA Resin in 20 mL of 2 M HCl. We add 100 µg of cerium carrier and 2 mL of concentrated HF. Plutonium is recovered from TRU Resin in 30 mL of 4 M HCl/1 mL 20% TiCl$_3$. Following past procedures, we add an additional 0.5 mL of 20% TiCl$_3$ to insure that the valency of Pu is Pu^{3+} or Pu^{4+}, followed by 100 µg of cerium carrier and 2 mL of concentrated HF.

After 20 min the resulting precipitates are filtered onto 0.1-µm, 25-mm Metricel membrane filters on a filter apparatus with screen, funnel, and filtering flask. The filters are dried for about 10 min in a <60 °C oven and then attached to a stainless steel disc using glue stick. All samples are counted in an alpha-particle spectrometer using low-background ion-implanted silicon detectors.

During experiments performed while developing this procedure, the average Am recovery was over 90%. However, the Pu recoveries were much lower, around 30-40%. On the other hand, experiments using a high Pu spike and LSC measurements of the fractions, gave consistently high (>80%) Pu recoveries through the elution step from the TRU Resin (Figure 4). We were apparently losing a significant fraction of the Pu during the very last step in the entire procedure, i.e. the source preparation. Apparently, the problem was that the Pu was not completely co-precipitating with the CeF$_3$ under the conditions used. We discovered that almost all the missing Pu was in the filtrate. Collecting the filtrate and co-precipitating CeF$_3$ a second time confirmed this hypothesis. In every case we tested the missing 60-70% of the Pu was there. A similar, but much smaller (~3%), amount of Am was recovered by a second co-precipitation of the Am filtrate in sample EML 0009.

In light of these observations, we performed a few simple tracer tests and observed 60-70% recovery of Pu when a tracer was co-precipitated from 20 mL 4 M HCl while the recoveries were close to 100% when done in the same volume of 2 M HCl. In our procedure Pu is eluted with 30 mL of 4 M HCl/1 mL 20% TiCl$_3$ (added to reduce Pu^{4+} to Pu^{3+}). Because of the presence of Ti, we cannot easily evaporate the sample and adjust the acidity. After presenting this problem to other scientists on the "Radiochemist's Mail List" (RADCH-L@in2p3.fr), we received several valuable replies including the following: "As HCl concentration increases, so will the solubility of rare earth fluorides. To compensate, one needs to add more HF...In addition, Ti forms a very stable complex with fluoride. If the amount of HF you are adding is too small, some fluoride is unavailable because it is tied up with titanium..." (B. Stewart, pers. comm.).

Following this lead, we investigated to what extent the CeF$_3$ co-precipitation of Pu is affected by the concentration and volume of HCl with Ti added. To find the optimal amount of HF needed for quantitative precipitation, we prepared 6 solutions of 20 mL of 4 M HCl - 0.5 mL 20% TiCl$_3$, 0.33 Bq ^{239}Pu spike, and an additional 0.5 mL 20% TiCl$_3$. One hundred micrograms of cerium carrier was added to each vessel and gradually increased the amount of concentrated HF from 1 to 3.5 mL. The samples were filtered and measured in the same way as the real samples. Our results show that for this system the amount of concentrated HF needed for quantitative co-precipitation of Pu is 2.5 mL (Figure 5A). The resolution of the spectral peaks was not significantly effected (total change in FWMH of ~3 keV) by the slightly increased mass loading.
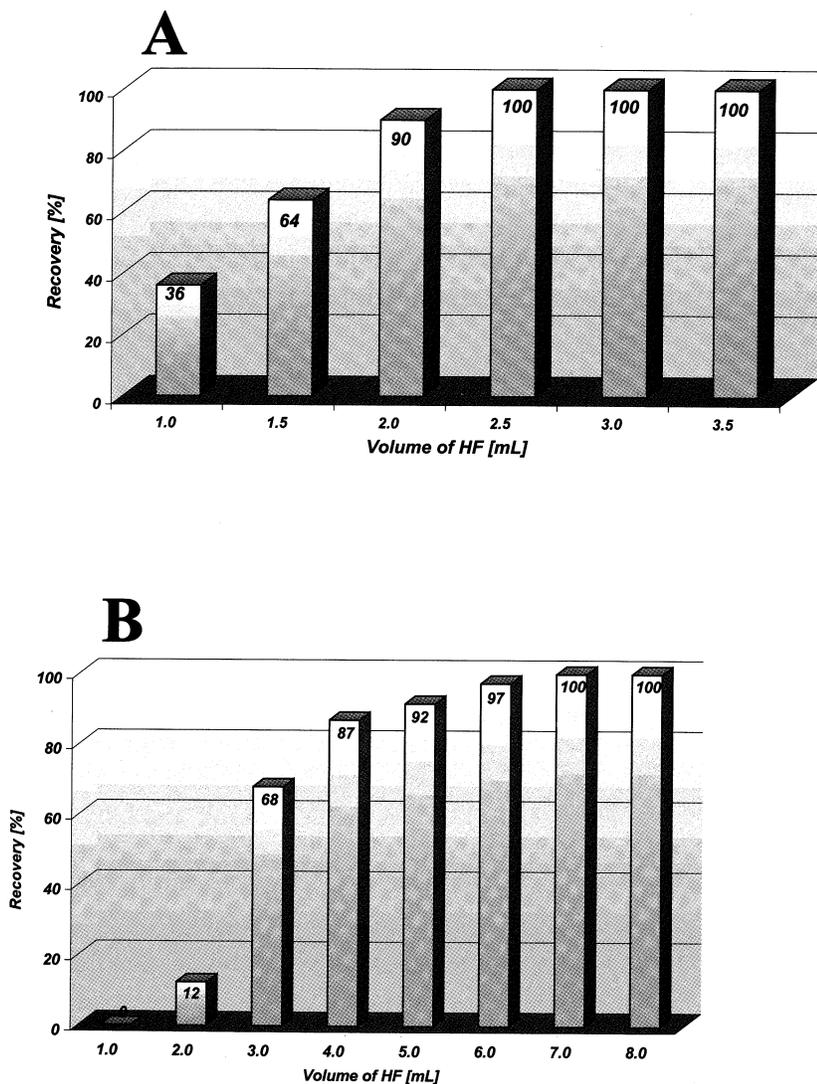
A

B

Figure 5   Dependence of [239]Pu recovery on the volume of HF added during CeF$_3$ co-precipitation. We added 0.33 Bq [239]Pu spike and 100 μg Ce carrier to
(A) 20 mL 4 M HCl/1 mL 20% TiCl$_3$
(B) 30 mL 4 M HCl / 1.5 mL 20% TiCl$_3$.

each vessel and the amount of HF was varied from 1 to 8 mL. The samples were processed the same way as mentioned above. The results (Figure 5B) show that near quantitative results are not obtained until one adds 6 mL of HF, which gave a recovery of 97%.

We also performed one experiment with a smaller amount of Ti^{3+} in the solution to observe whether the HF complexing of Ti^{3+} does influence the amount of F$^-$ available or just the 4 M HCl itself affects the solubility of CeF$_3$. As before, we prepared a solution of 30 mL 4 M HCl, 1 mL 20% TiCl$_3$, and 0.33 Bq [239]Pu spike but without the additional 0.5 mL 20% TiCl$_3$. We added 100 μg of cerium carrier and 3 mL HF. The chemical recovery increased to 92% compared to 68% observed when using a total of 1.5 mL 20% TiCl$_3$.

We thus feel that both the amount of Ti^{3+} added to the sample and the volume and concentration of hydrochloric acid have a significant influence on the extent of Pu co-precipitation by CeF$_3$. By simply adjusting the amount of HF added to compensate for these effects, one can achieve quantitative recoveries.

We also investigated the possibility of using 0.1 M hydro-quinone, which would be relatively easy to destroy by wet oxidation, instead of Ti^{3+} in 4 M HCl to elute Pu from the TRU Resin column. Although this procedure does work, and is an option for those who cannot have Ti in the final solution (e.g., for sources prepared by electrodeposition), we found the elution efficiency to be generally lower.

We repeated this experiment under conditions that would more exactly match our procedure by preparing 8 solutions of 30 mL of 4 M HCl, 1 mL 20% TiCl$_3$, 0.33 Bq [239]Pu spike and additional 0.5 mL 20% TiCl$_3$. One hundred micrograms of cerium carrier was added to

Therefore, the procedure for the alpha-particle-source preparation of Pu was modified accordingly. We add to the 30 mL 4 M HCl, 1 mL 20% $TiCl_3$ solution eluted from TRU Resin an additional 0.5 mL of 20% $TiCl_3$ (to insure that Pu is in a reduced valency state), 100 μg of cerium carrier, and 7 mL of concentrated HF. After 20 min the precipitate is filtered onto a 0.1-μm Metricel filter. The drying and counting procedures remain the same as described earlier.

Time requirements

The average time needed for the radiochemical part of the procedure is spread over a period of about four days. Our typical schedule for a 50-g soil or sediment sample analysis is as follows: (Day 1) - combustion of the soil sample, 4 h; (Day 2) - leaching of the soil, co-precipitating of the actinides by ferric hydroxide, and finally loading of the solution onto the Diphonix column (we allow the solution to pass through the column overnight); (Day 3) - elution of the actinides, oxidation of the HEDPA, and separation of the actinides on the TRU column. The Pu source is prepared on the same day; and (Day 4) - the Am is separated from the lanthanides on TEVA Resin and the Am source is prepared the same day. The sources are counted on our alpha-particle-spectrometric system for up to a few days, depending on their activity and the required counting statistics. If we wanted to compress this schedule, the steps performed on Days 1 and 2 could easily be carried out on the same day. In addition, one could process several samples simultaneously in order to best utilize analyst time.

Results and Discussion

During the development stages of the procedure, we ran soil samples with addition of high (>16 Bq) ^{241}Am and ^{239}Pu spikes so we could evaluate each step of the process. A small aliquot of sample was removed after every step to check the chemical recovery for every significant part of the procedure. These aliquots were analyzed by liquid scintillation counting. The estimated chemical recoveries of these test samples are shown on

Figure 4. Sample "*Am SCS*" contained high ^{241}Am spike and "*Pu EML*" contained high ^{239}Pu spike. Both samples were carried through the whole procedure, which at that time included separation of actinides on TRU Resin using the earlier Fe(II) sulfamate/AA process. Note that the results indicate a significant loss of Pu in the EML sample during separation on the TRU Resin. This was probably a result of the reaction of the resin and the load solution as discussed earlier. In general, we had to add more ascorbic acid to the EML samples than to SCS samples, which may explain why the recovery of Am was higher than that of Pu. We suspect that these EML samples contained more Fe than the SCS samples. Our spike experiments also show that there are no significant losses of Am and Pu in any step when we use 2.5 M HNO_3-1M $Al(NO_3)_3$-0.06 M $NaNO_2$ as a load solution for separation of actinides on TRU Resin. Samples "*Am SCS (NaNO₂)*" and "*Pu EML (NaNO₂)*" show high overall recoveries (~90%). The apparent low recovery of Pu after source preparation was solved later by the addition of more F⁻ (see previous discussion).

Realistic tests of the method were performed by the analysis of reference materials. We analyzed 50 g each of *EML 0009* QAP soil, *IAEA 326* soil, and *IAEA 368* Pacific-ocean sediment using the finalized procedure. The results of ^{241}Am analysis (Table 1) show that the ^{243}Am tracer recoveries were above 90%, except sample IAEA 368, which was just 30%. We feel that this one rather low Am result is an exception as we experienced 90+% Am recoveries throughout the procedure for many test samples. Our ^{241}Am results (FSU) compare well to the recommended standard values (Figure 6). The average FSU ratio to standard value is 0.97.

Results for Pu isotopes are shown in Table 2. Samples *EML 0009* and *IAEA 368* were analyzed before the source-preparation problem was resolved (although it was recognized that some Pu was not co-precipitating). The chemical recoveries of ^{242}Pu tracer after the source preparation by CeF_3 co-precipitation were 61 and 46%, respectively. The second co-precipitation of the filtrates showed that the whole procedure works effectively, with resulting Pu recoveries of at least 90 and 72%. Sample *IAEA 326* was analyzed after solving the co-precipitation problem. Its recovery, at 85%, was still

Sample ID	Description	Weight [g]	Recovery [%]	^{241}Am Activity [Bq/kg]		
				FSU	Certified (confidence int.)	FSU/ certified
EML 0009*	Soil	49.99	92	7.0 ± 0.1	8.3 ± 0.7	0.85
IAEA 368	Pacific ocean sediment	49.97	30	1.34 ± 0.03	1.30 (1.2-1.5)	1.03
IAEA 326	Reference soil	50.00	94	0.20 ± 0.01	0.19 (0.16-0.22)	1.04
* EML 0009 contains ~ 130 Bq/kg ^{244}Cm					Average	0.97

Table 1 Measured ^{241}Am activities in the laboratory at Florida State University (FSU) and their comparison to the recommended values.
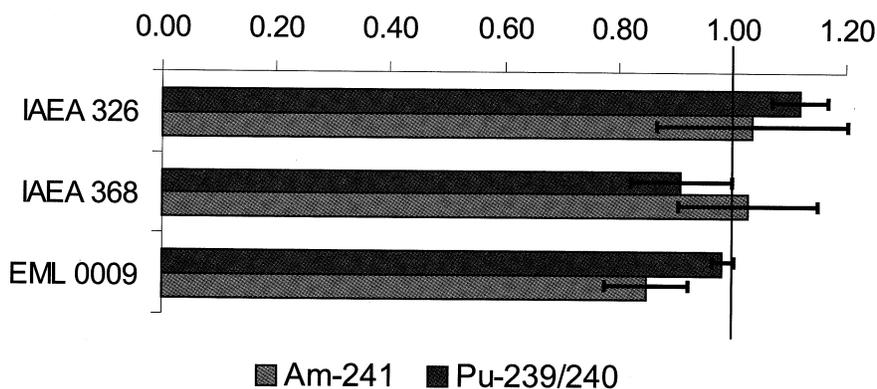
Activity ratio: FSU/Standard

Figure 6 Results for ^{241}Am and $^{239+240}$Pu results to certified values. The error bars are based on error propagation from the counting statistics at the 1σ level.

not representative of the system recovery as we still found 10% of the Pu in a second precipitate. We had mistakenly used only 6-mL HF in the Pu-source preparation for that sample. For further analyses we recommend using at least 7-mL HF in the source preparation. The average ratio to standard for values ^{238}Pu and $^{239+240}$Pu is 1.10 and 1.01 respectively (Figure 6).

Conclusions

The Diphonix Resin approach is very effective in preconcentration of actinides and elimination of sample matrix. Elution of actinides with HEDPA and its subsequent oxidation on a hotplate has an advantage over the microwave destruction of the resin itself (Maxwell et al., 2000). Although the microwave approach works very well, it is limited by the volume of the resin (<3 mL) that is practical to destroy. There are no limits on the volume of neither the Diphonix Resin nor the HEDPA solution in our method. Our experiments also showed that the phosphoric acid, produced by the oxidation of HEDPA solution, does not cause any problem on the uptake of actinides on the TRU Resin. The results obtained by the modified methodology were close to the certified values and the ratios to standard values are very good for both Am and Pu isotopes. The high recoveries show that the method is effective in the near quantitative extraction of Pu and Am from a 50-g soil sample. Furthermore, the overall recoveries are comparable to those observed in

Sample ID	Description	Weight [g]	Recovery [%]	^{238}Pu activity [Bq/kg]			$^{239+240}$Pu activity [Bq/kg]		
				FSU	Certified (confidence int.)	FSU/ certified	FSU	Certified (confidence int.)	FSU/ certified
EML 0009	Soil	49.99	61 (90)^y	18.7 ± 0.2	19.1 ± 0.2	0.98	16.5 ± 0.2	16.8 ± 0.3	0.98
IAEA 368	Pacific ocean sediment	49.97	46 (72)	7.3 ± 0.1	8.5 (7.6-8.9)	0.86	28.2 ± 0.3	31.0 (29.0-34.0)	0.91
IAEA 326	Rederence Soil	50.00	85 (95)	0.028 ± 0.003	0.02 (0.017-0.021)	1.47	0.56 ± 0.01	0.5 (0.48-0.52)	1.12
^{y} estimated total recovery					**Average**	**1.10**		**Average**	**1.01**

Table 2 Measured ^{238}Pu and $^{239+240}$Pu activities in the laboratory at Florida State University (FSU) and their comparison to the recommended values.

methods for 5- to 10-g samples. We therefore conclude that Diphonix Resin has the outstanding ability to preconcentrate actinides from large soil samples to the exclusion of common elements. This seems to solve the "matrix effect" burden so commonly present in environmental radiochemistry. An additional advantage of the method is that the procedure doesn't require any specialized equipment and the time necessary for the analysis is approximately the same as established procedures.

Acknowledgments

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Biographies

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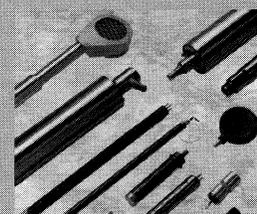8205 S. Cass Avenue, Suite 111
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