

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-96SR18500 with the U.S. Department of Energy.

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Rapid Method for Plutonium, Americium and Curium in Very Large Soil Samples

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Abstract

The analysis of actinides in environmental soil and sediment samples is very important for environmental monitoring. There is a need to measure actinide isotopes with very low detection limits. A new, rapid actinide separation method has been developed and implemented that allows the measurement of plutonium, americium and curium isotopes in very large soil samples (100-200 g) with high chemical recoveries and effective removal of matrix interferences. This method uses stacked TEVA Resin[®], TRU Resin[®] and DGA-Resin[®] cartridges from Eichrom Technologies (Darien, IL, USA) that allows the rapid separation of plutonium (Pu), americium (Am), and curium (Cm) using a single multi-stage column combined with alpha spectrometry. The method combines an acid leach step and innovative matrix removal using cerium fluoride precipitation to remove the difficult soil matrix. This method is unique in that it provides high tracer recoveries and effective removal of interferences with small extraction chromatography columns instead of large ion exchange resin columns that generate large amounts of acid waste. By using vacuum box cartridge technology with rapid flow rates, sample preparation time is minimized.

Key Words

Large soil samples

Plutonium

Americium

Curium

Extraction chromatography

Actinide separations

Resin

Introduction

The analysis of plutonium, americium and curium at extremely low levels requires the analysis of very large soil samples. The analysis of these very large samples (100-200 g), however, is very difficult and requires efficient removal of a large amount of matrix interferences. A preconcentration method using cerium fluoride matrix removal to allow actinide analysis in 5 to 10 gram soil samples has been reported by this laboratory previously.¹ This method offers total dissolution, very good tracer recoveries (<90%) and excellent removal of interferences such as polonium-210 and thorium isotopes. There is a need to achieve lower detection limits for plutonium, americium and curium isotopes by analyzing much larger sample sizes. Other methods have been used that rely on Diphonex resin², but they require actinides be removed from Diphonex Resin using 1-

hydroxyethane-1, 1-diphosphonic acid (HEDPA) extractant. The HEDPA extractant can be destroyed via a manual hot plate digestion prior to further analysis, but this method generates a large amount of residual phosphate and often requires much larger extraction columns to separate the actinides. Other labs have used calcium oxalate and iron hydroxide precipitation to remove soil matrix components³, but the calcium oxalate precipitation requires a precise pH adjustment and can be technique-dependent. As a result, tracer recoveries can vary quite a bit and large separation columns are still required. Another method using calcium oxalate precipitation has been reported, but sample size was limited to ~10g and tracer recoveries were 40-60%.⁴ This method uses a large anion resin column with large acid rinse volumes as well as a relatively large TRU Resin column (~4 ml resin). A method using acid leaching followed by lanthanum fluoride precipitation to remove soil matrix components has been reported recently for larger soil samples, but the plutonium yields were 60-70% and the Am yields were 50-65%. Large anion resin columns were still required and oxalates in this method had to be ashed at 450C overnight. In addition, the alpha spectra seemed to show somewhat poor peak resolution, particularly between Am-243 tracer and Am-241 peaks.⁵ Although acid leaching is not appropriate for soil samples containing refractory PuO₂⁶, it has been shown to be acceptable for fallout-derived radionuclides not associated with refractory components in the sample.^{7,8}

A new matrix removal technique was developed in the SRS Environmental Laboratory that is simple, effective and allows the use of small resin cartridges to separate plutonium, americium and curium isotopes from 100-200g soil samples. After an acid leach of the soil matrix and an iron hydroxide precipitation to collect the actinides, a novel cerium fluoride precipitation is used to effectively eliminate the soil matrix. This new method uses stacked

TEVA Resin[®], TRU Resin[®] and DGA-Resin[®] cartridges from Eichrom Technologies (Darien, IL, USA) that allows the rapid separation of plutonium, americium, and curium using a single multi-stage column (using 2 ml resin volumes) to separate actinide isotopes for alpha spectrometry. DGA-Resin[®], which has very strong retention for americium and curium, is used to enhance chemical recoveries of those analytes.⁹ DGA Resin has also been used to separate Ac-225 from americium and curium isotopes to eliminate interference on Cm isotopes in alpha spectrometry.¹⁰

The new SRS soil method effectively separates plutonium, americium and curium for analysis from very large soil samples (100-200g) for environmental monitoring. It provides rapid leaching of actinides in soil samples and uses a stacked cartridge technology that allows for sequential actinides separations with minimal waste generation. By adjusting the valence of uranium to select against uranium precipitation during the cerium fluoride matrix removal step, interference from the potentially large amount of uranium present is minimized.

Experimental

Reagents

The resins employed in this work are TEVA Resin[®] (Aliquat[™]336), TRU-Resin[®] (tri-n-butylphosphate (TBP) and N,N-diisobutylcarbamoymethylphosphine oxide (CMPO)), DGA Resin (N,N,N',N' tetraoctyldiglycolamide), and Prefilter Resin (Amberchrome-CG-71) available from Eichrom Technologies, Inc., (Darien, Illinois). Nitric, hydrochloric and hydrofluoric acids were prepared from reagent-grade acids

(Fisher Scientific, Inc.). All water was obtained from a Milli-Q2™ water purification system. All other materials were ACS reagent grade and were used as received. Radiochemical isotope tracers Pu-242 and Am-243 were obtained from Analytix, Inc. (Atlanta, GA, USA) and diluted to the approximately 2 pCi/ml (0.074 Bq/ml) level were employed to enable yield corrections. Laboratory Control Standards (LCS) were analyzed using Pu-238, Am-241 and Cm-244 standards that were obtained from Analytix, Inc. (Atlanta, GA, USA) and diluted to approximately 2 pCi/ml (0.074 Bq/ml).

Procedures

Column preparation. TEVA, TRU, and DGA Resin columns were obtained as cartridges containing 2 ml of each resin from Eichrom Technologies, Inc..¹¹ Small particle size (50-100 micron) resin was employed, along with a vacuum extraction system (Eichrom Technologies). Flow rates of 1 -2 ml/min were typically used, much faster than the 0.25 ml/min gravity flow rates typically observed. Sample loading and column stripping steps were performed at ~1 drop/second, while column rinse steps were usually performed at 1 to 2 drops per second.

Sample Preparation. Soil samples were dried at 110C and blended prior to taking sample aliquots. 100 and 200 g sample aliquots were analyzed. After samples were aliquoted into 11 glass beakers, tracers were added and the samples were placed in a furnace at 550C for 4 hours or more. After adding 75 to 100 ml of concentrated nitric acid and 25 ml of concentrated hydrochloric acid to each beaker, samples were heated to dryness on a hot plate. Fifty to seventy-five milliliters of concentrated nitric acid were

added to each sample. The beakers were warmed on a hot plate and the leachate and solids were transferred to a 225 ml centrifuge tube. Twenty-five milliliters of concentrated nitric acid were added to each beaker, warmed on a hot plate and the leachate plus additional solids were transferred to the centrifuge tube. This step was repeated once more with twenty-five milliliters of concentrated nitric acid. The centrifuge tubes were centrifuged at 3500 rpm for 15 minutes. The leachate was filtered through a 0.45 micron filter and transferred to a 600 ml beaker. This beaker was placed on a hot plate for evaporation of the filtered leachate to dryness. Twenty-five milliliters of concentrated nitric acid was added to each tube to rinse the solids. This solution was filtered in the same manner as above after changing out the 0.45 micron filter and added to the evaporating filtrate. Twenty-five milliliters of 4M hydrochloric acid were added to each beaker, warmed on a hot plate and this solution plus additional solids were transferred to the centrifuge tubes. Twenty-five milliliters of 4M hydrochloric acid were added to each beaker and this solution plus additional solids were transferred to the centrifuge tubes. This solution was filtered in the same manner as above after changing out the 0.45 micron filter (if needed) and added to the evaporating filtrate. Twenty-five milliliters of 4M hydrochloric acid were added to each tube to rinse the solids. This solution was filtered in the same manner as above after changing out the 0.45 micron filter (if needed) and added to the evaporating filtrate. The filtered leachate solutions were evaporated to dryness on a hot plate on low heat as needed to prevent splattering. To each beaker, 10-20 ml of concentrated nitric acid were added and evaporated to dryness. This step was repeated once more. After ashing to remove hydrochloric acid, the residual solids were transferred to 250 ml zirconium crucibles (Metal Technology, Inc., Albany, OR, USA). The beakers

were rinsed with concentrated nitric acid, transferred to the crucible and the crucible contents were evaporated on a hot plate to dryness.

After removing the crucibles and allowing them to cool, 20 to 25 grams of sodium hydroxide were added to each crucible. The crucibles were covered with a zirconium lid and placed into a furnace at 600C for ~20 minutes.

After removing the crucibles from the furnace, they were cooled for a few minutes and water was added to transfer the solids to 225 ml centrifuge tubes. The residual solids were removed from the crucibles by adding water and heating the crucibles on the hot plate as needed. Seven milligrams of cerium as cerium nitrate were added to each tube. These solutions were diluted to ~180 ml with water and cooled to room temperature in an ice bath as needed. Six milliliters of 20% titanium chloride reductant were added to each tube, followed by 1 ml of 10% barium nitrate to complex any carbonate present. The tubes were centrifuged at 3500 rpm for 5 minutes and the supernate was poured off. The remaining solids were dissolved in a total volume of 60 ml of 1.5 M HCl. This solution was diluted to 170 ml with 0.01M HCL. Two milligrams of cerium as cerium nitrate were added to each sample. To ensure that uranium is oxidized to the hexavalent state and minimize precipitation, ten milliliters of 30 wt% hydrogen peroxide were added to each sample. Twenty-two milliliters of 28M hydrofluoric acid were added to each sample. The samples were placed on ice for ~10 minutes to reduce solubility and centrifuged for 25 minutes at 3500 rpm. The supernate was removed and the residual solids containing the actinides were dissolved in 5 ml of warm 3M HNO₃-0.25M boric acid, 6 ml of 7M HNO₃ and 7.5 ml of 2 M aluminum nitrate. Figure 1 shows the small cerium fluoride precipitate containing Pu, Am and Cm after soil matrix removal just prior to dissolution into the

column load solution. The solids were transferred to 100 ml teflon beakers during this step and warmed to redissolve the solids.

Column separation. TEVA, TRU, and DGA Resin cartridges were stacked on the vacuum box from top to bottom, in that order.¹⁰ Fifty milliliter centrifuge tubes were used to collect the rinse or final purified fractions.

A valence adjustment was performed by adding 0.5 ml of 1.5M sulfamic acid and 1.25 ml of 1.5M ascorbic acid. After a three-minute wait step, one milliliter of 3.5M sodium nitrite (freshly prepared) to adjust plutonium to Pu⁺⁴. After the valence adjustment, the sample solution was loaded onto the stacked column at approximately 1 drop per second. Column reservoirs may be replaced and/or the TEVA cartridge frits (top) removed if any solids form in the load solution and affect column flow. After the sample was loaded, a beaker rinse of 3 ml of 6M HNO₃ was transferred to the stacked column. At this point the TRU and DGA cartridges were removed and the DGA Resin cartridges were placed on a second vacuum box. Five milliliters of 0.25M nitric acid were added to each DGA column to remove any residual uranium that may have been retained on the DGA cartridges. This rinse was discarded to waste. The TEVA Resin was rinsed with 7 ml of 3M HNO₃ to remove residual uranium, which was also discarded to waste. The TEVA cartridge was rinsed with 10 ml of 5M nitric acid and then 10 ml of 3M nitric acid to remove matrix components. To elute thorium from TEVA Resin, 23 ml of 9M hydrochloric acid were added.

A 5 ml volume of 3M HNO₃ was added to TEVA Resin (and discarded) to reduce the amount of any residual extractant before stripping the plutonium from the resin. The plutonium was stripped from TEVA Resin with 20 ml of 0.1M hydrochloric acid-0.05M

hydrofluoric acid –0.03M titanium chloride (freshly prepared). A 0.5 ml volume of 30 wt% hydrogen peroxide was added to each Pu strip solution to oxidize any residual uranium to U^{+6} as a precaution to prevent coprecipitation. Fifty micrograms of cerium as cerium nitrate was added, along with 1 ml of concentrated hydrofluoric acid (49%). After waiting 30 minutes, the solutions were filtered onto 0.1 micron 25 mm polypropylene and counted by alpha spectrometry .

The TRU cartridges were placed above the DGA cartridges and 15 ml of 4M HCL was used to strip Am and Cm from TRU Resin onto the DGA Resin. After removal of the TRU cartridges, the DGA cartridges were stripped with 10 ml of 0.25M HCl. These strip solutions containing americium and curium were transferred to 50 ml glass beakers using ~3 ml of concentrated nitric acid and 0.05ml of 1.8M sulfuric acid was added to enhance destruction of any extractant in this solution. The Am/Cm strip solutions were evaporated to dryness on a hotplate. These fractions were ashed one time on the hot plate using 2 ml of concentrated nitric acid and 2 ml of 30 wt% hydrogen peroxide. This ashing step removes any residual extractant that may have bled off the resin. The samples were redissolved in 5 ml of 4M ammonium thiocyanate-0.1M formic acid, warming gently as needed. These solutions were loaded onto a TEVA cartridge to remove rare earths present, which interfere with alpha spectrometry peak resolution. The TEVA cartridges were rinsed with 10 ml of 1.5M ammonium thiocyanate-0.1M formic acid to remove rare earths, and the americium and curium were stripped using 25 ml of 1M HCl. The original load solution beaker was rinsed with 5 ml of warm 1M HCL to ensure all the americium and curium was removed from this beaker. This solution, followed by 20 ml of 1M HCL also used to rinse the beaker, was passed through the TEVA Resin to remove the

americium and curium. Fifty micrograms of cerium as cerium nitrate was added, along with 2 ml of concentrated hydrofluoric acid (49%). After waiting 30 minutes, the solutions were filtered onto 0.1 micron 25 mm polypropylene and counted by alpha spectrometry.

Figure 2 shows the vacuum box apparatus and the stacked TEVA, TRU and DGA Resin cartridges. A second vacuum box was used after the cartridges were split apart so that the cartridges could be processed on two boxes for enhanced productivity. DGA and TRU Resin cartridges were moved to the second box and stripped as described above. Figure 3 summarizes the new soil method in a flow diagram.

Apparatus

Plutonium, americium, and curium measurements were performed by alpha-particle pulse-height measurements using Passivated Implanted Planar Silicon (PIPS) detectors. Polycarbonate vacuum boxes with 24 positions and a rack to hold 50 ml plastic tubes were used. Two boxes were connected to a single vacuum source by using a T-connector and individual valves on the tubing to each box. Vacuum boxes were obtained from Eichrom Technologies (Darien, IL, USA).

Results and Discussion

Table 1 shows the performance of the method on 100 g soil samples with MAPEP -05-S14 standard (Department of Energy (DOE) – Radiological and Environmental Sciences Laboratory (RESL), Idaho Falls, ID, USA) added. The average Pu-242 tracer recovery was 85.6% and the average tracer recovery for Am-243 was 94.3%. The unspiked sample contained 0.120 Pu-238 Bq/kg and 0.152 Am-241 Bq/kg. 0.0608 Bq Pu-238 and 0.0811 Bq Am-241 were added per gram of S14 standard added. The measured standard was determined by subtracting the unspiked sample measurement from the spiked sample measurement. The MAPEP acceptance limit for the ratio of the measured to reference values is 0.8-1.2 (0.7-1.3 acceptable with warning). The ratio of the measured Pu-238 to the MAPEP reference value was 0.97 and the ratio for Am-241 was 0.90, showing excellent agreement with the reference values. Table 2 shows the performance of the method on 200 g soil samples with MAPEP -05-S14 standard added. Even though the soil aliquot was doubled, the tracer recoveries were still very high. The average Pu-242 tracer recovery was 81.8% and the average tracer recovery for Am-243 was 93.3%. The ratio of the measured Pu-238 to the MAPEP reference value was 1.02 and the ratio for Am-241 was 0.81, within acceptance limits. Table 3 shows the performance of the method on 100 g soil samples with MAPEP -04-S12 standard added. The average Pu-242 tracer recovery was 81.2% and the average tracer recovery for Am-243 was 80.2%. The ratio of the measured Pu-238 to the MAPEP reference value was 0.94 and the ratio for Am-241 was 0.87, within acceptance limits. Table 4 shows the performance of the method on 200 g soil samples with MAPEP-04-S12 standard added. The average Pu-242 tracer recovery was 80.1% and the average tracer recovery for Am-243 was 80.9%. The ratio of the measured Pu-238 to the MAPEP reference value was 0.95 and the ratio for Am-241 was

0.82, within acceptance limits. Figure 4 shows a typical spectra for the plutonium isotopes for a 200 g sample. The Pu-242 tracer recovery was 82.1% and the Full Width Half Maximum (FWHM) was 53 keV, showing good alpha peak resolution. Figure 5 shows an example of spectra for the Am/Cm isotopes for a 100 g sample. The Am-243 tracer recovery was 97.5% and the FWHM was 66 keV. Based on other studies in this laboratory, if Pu-236 tracer is used instead of Pu-242, neptunium can also be measured.¹²
¹³ To facilitate Np reduction to Np^{+4} in the column load solution, a small amount of iron (~2 mg) may be added along with the ascorbic acid in the column load solution.

After initial drying, blending and heating at 550C for 4 hours, the time required to prepare a batch of soil samples is about 8-10 hours. The column work usually takes about 4 to 5 hours for the respective actinides to be separated and purified. Samples were typically counted 16 hours. The minimum detectable concentration for Pu, Am and Cm is approximately $3\text{E}-5$ pCi/g (0.001 Bq/kg) for a 200g sample counted for 16 hours.

Conclusions

The new soil method developed in the SRS Environmental Laboratory is a rapid method for plutonium, americium and curium isotopes that provides very low detection limits. The minimum detectable concentration for Pu, Am and Cm is approximately $3\text{E}-5$ pCi/g (0.001 Bq/kg). This method has high tracer recoveries, effectively removes interferences and combines the sample preparation for plutonium, americium and curium into a single multi-stage column extraction method. Other methods have typically required very large anion resin columns which generate large volumes of acid waste. The soil matrix removal effectively removes uranium and other interferences and allows the use of

small, stacked resin cartridges (2 ml) instead of large anion resin columns. In addition, it utilizes the new resin, DGA Resin, which has very strong retention for americium and curium.

Acknowledgment

This work was performed under the auspices of the Department of Energy, DOE Contract No. DE-AC09-96SR18500. The authors wish to acknowledge Gene Cooke, Becky Chavous and Jack Herrington for their assistance in testing this method.

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Table Captions

Table 1 Performance on 100 g Samples with MAPEP-05-S14 Soil Standard Added

Table 2. Performance on 200 g Samples with MAPEP-05-S14 Soil Standard Added

Table 3. Performance on 100 g Samples with MAPEP-05-S12 Soil Standard Added

Table 4. Performance on 200 g Samples with MAPEP-05-S12 Soil Standard Added

Figure Captions

Figure 1. Vacuum Box System with Stacked Cartridges

Figure 2. Pu, Am, Cm in Cerium Fluoride Precipitate After Soil Matrix Removal

Figure 3. Soil Method Flow Diagram

Figure 4 Alpha Spectrometry Spectra showing Pu Isotopes

Figure 5. Alpha Spectrometry Spectra showing Am Isotopes

Table 1. Performance on 100 g Samples with MAPEP-05-S14 Soil Standard Added

Soil Sample	Pu-242 Tracer Recovery	Pu-238 Measured/ Reference	Am-243 Tracer Recovery	Am-241 Measured/ Reference
100g + 1 g S14	77.2 %	0.99	90.0%	0.95
100g + 3 g S14	91.5%	0.97	97.5%	0.87
100g + 3 g S14	88.2%	0.95	95.3%	0.86
Avg.	85.6%	0.97	94.3%	0.90

Unspiked sample=0.120 Pu-238 Bq/kg and 0.152 Am-241 Bq/kg

0.0608 Bq Pu-238 and 0.0811 Bq Am-241 added per 1 gram of S14

Table 2. Performance on 200 g Samples with MAPEP-05-S14 Soil Standard Added

Soil Sample	Pu-242 Tracer Recovery	Pu-238 Measured/ Reference	Am-243 Tracer Recovery	Am-241 Measured/ Reference
200g + 3 g S14	82.1 %	1.00	96.6%	0.80
200g + 3 g S14	81.4%	1.04	90.0%	0.82
Avg.	81.8%	1.02	93.3%	0.81

Unspiked sample=0.120 Pu-238 Bq/kg and 0.152 Am-241 Bq/kg

0.0608 Bq Pu-238 and 0.0811 Bq Am-241 added per 1 gram of S14

Table 3. Performance on 100 g Samples with MAPEP-05-S12 Soil Standard Added

Soil Sample	Pu-242 Tracer Recovery	Pu-238 Measured/ Reference	Am-243 Tracer Recovery	Am-241 Measured/ Reference
100g (no S12)	89.3 %	N/A	80.4%	N/A
100g + 3 g S12	60.6%	0.93	76.2%	0.84
100g + 3 g S12	93.8%	0.94	83.9%	0.90
Avg.	81.2%	0.94	80.2%	0.87

Unspiked sample=0.012 Pu-238 Bq/kg and 0.0365 Am-241 Bq/kg

0.0354 Bq Pu-238 and 0.067Bq Am-241 added per 1 gram of S12

Table 4. Performance on 200 g Samples with MAPEP-05-S12 Soil Standard Added

Soil Sample	Pu-242 Tracer Recovery	Pu-238 Measured/ Reference	Am-243 Tracer Recovery	Am-241 Measured/ Reference
200g (no S12)	87.4 %	N/A	78.7%	N/A
200g + 3 g S12	77.1%	1.03	85.1%	0.75
200g + 3 g S12	75.9%	0.87	79.3%	0.88
Avg.	80.1%	0.95	80.9%	0.82

Unspiked sample=0.012 Pu-238 Bq/kg and 0.0365 Am-241 Bq/kg

0.0354 Bq Pu-238 and 0.067Bq Am-241 added per 1 gram of S12

Figure 1 Pu, Am, Cm in Cerium Fluoride Precipitate After Soil Matrix Removal



Figure 2 Vacuum Box with Stacked Cartridges (TEVA+TRU+DGA Resin)

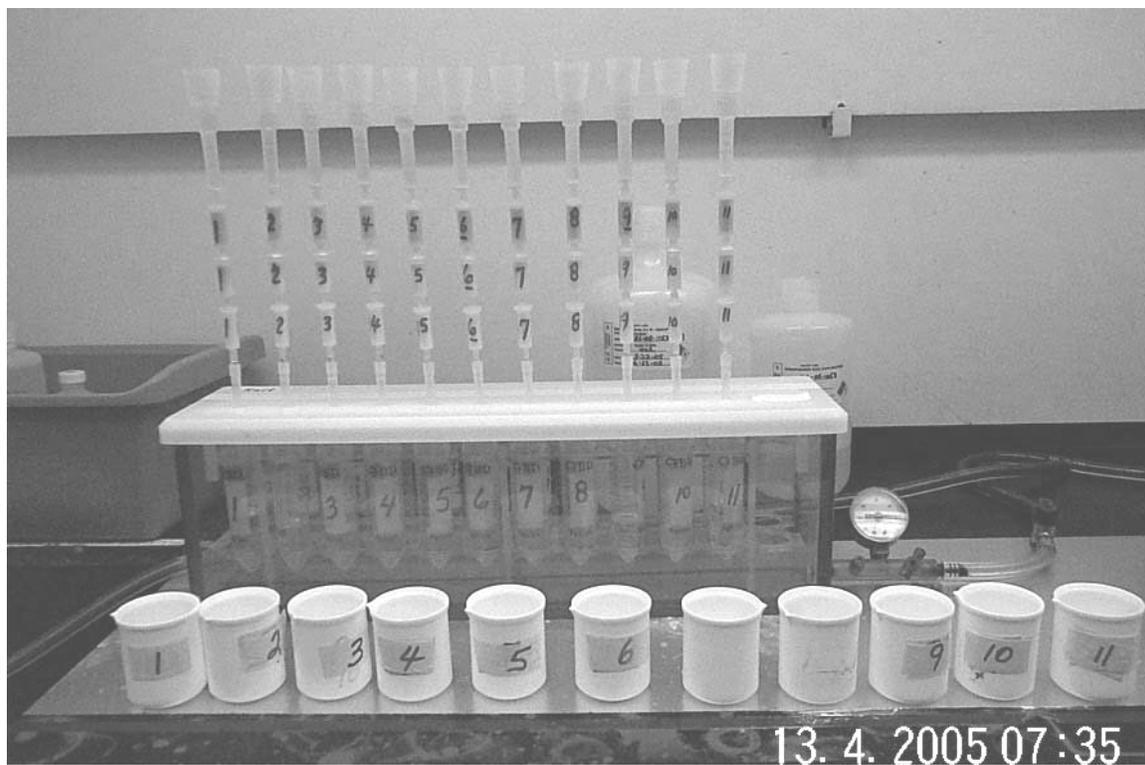


Figure 3. Soil Method Flow Diagram

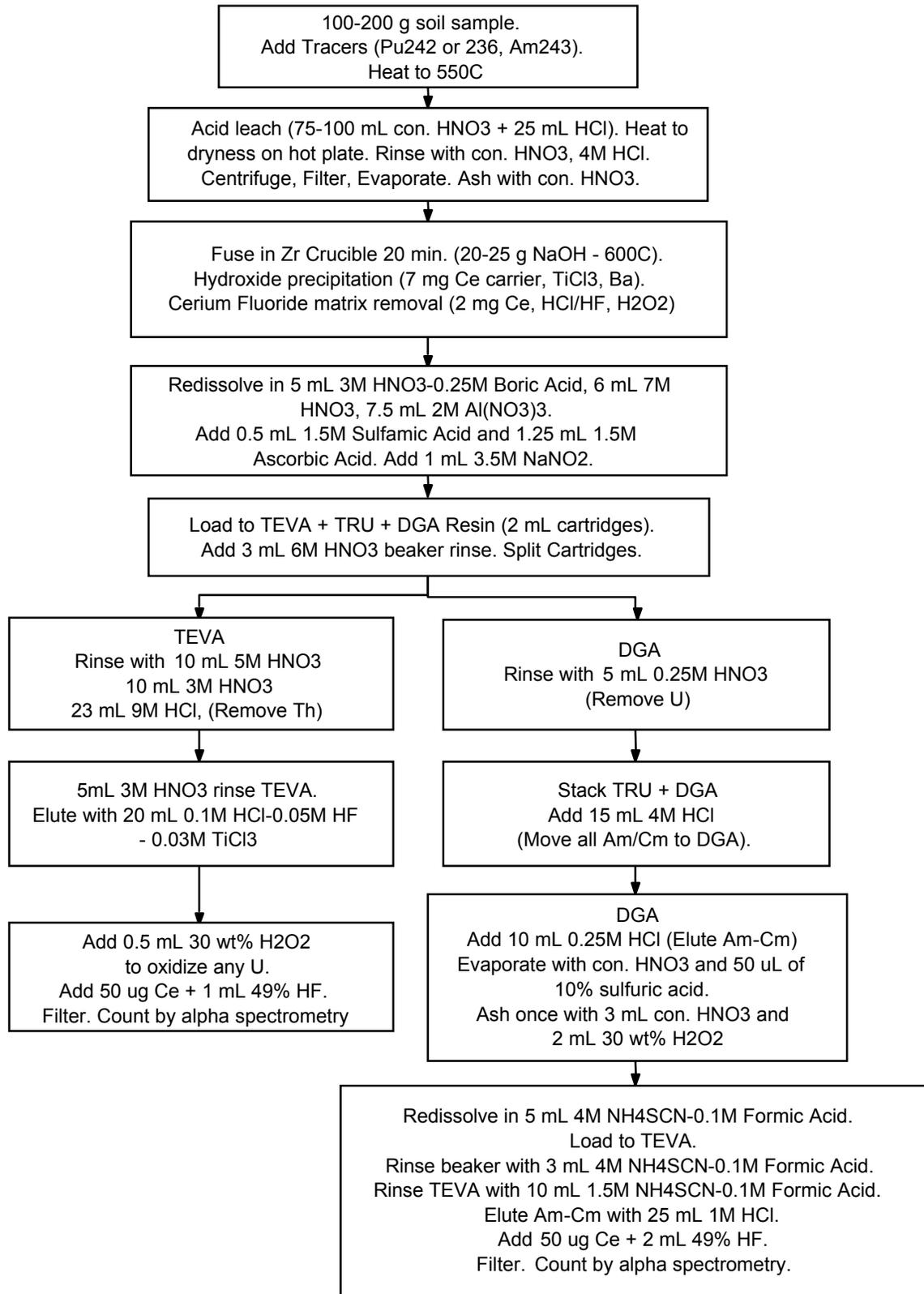


Figure 4 Alpha Spectrometry Spectra showing Pu Isotopes

Figure 5 Alpha Spectrometry Spectra showing Am Isotopes

