Comparison of Acid Leachate and Fusion Methods to Determine Plutonium and Americium in Environmental Samples

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COMPARISON OF ACID LEACHATE AND FUSION METHODS
TO DETERMINE PLUTONIUM AND AMERICIUM
IN ENVIRONMENTAL SAMPLES

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ABSTRACT

The Analytical Chemistry Laboratory at Argonne National Laboratory performs radiochemical analyses for a wide variety of sites within the Department of Energy complex. Since the chemical history of the samples may vary drastically from site to site, the effectiveness of any analytical technique may also vary. This study compares a potassium fluoride-pyrosulfate fusion technique with an acid leachate method. Both normal and high-fired soils and vegetation samples were analyzed for both americium and plutonium. Results show both methods work well, except for plutonium in high-fired soils. Here the fusion method provides higher accuracy.

INTRODUCTION

Sensitive and reliable analytical techniques to detect and measure actinides in environmental samples are increasingly important due to the expanding public scrutiny of the nuclear industry. Waste burial practices, as well as the impacts of both accidental and routine releases, need to be monitored to allay public concern.

Actinides may enter the environment through several distinct pathways, such as fallout from nuclear testing, leakage from fuel fabrication or reprocessing complexes, and leaching from waste storage facilities. Furthermore, the chemical history of the sample substantially influences its chemical behavior. Plutonium from nuclear testing may exhibit different chemical behavior than plutonium derived from reprocessing or waste disposal. For example, plutonium oxide in the presence of silica or alumina becomes extremely refractory when heated strongly and consequently difficult to dissolve.^{1,2}

Sill et al.³ contend that aqua regia acid leaching techniques are inadequate for samples requiring plutonium analyses. They assert that much of the activity can be left behind in the insoluble residue due to the possible refractory nature of these samples, and that a total dissolution or fusion technique is therefore always indicated. However, this method is very expensive, requiring excessive man-hours and creating large quantities of waste. Bishop et al.⁴ compared an acid leach procedure in which greater than 90% of the plutonium was removed from the soils with the potassium fluoride-pyrosulfate fusion method described by Sill et al. Bishop et al. found that leaching and fusion results essentially agreed for normal soils and that the acid leachate technique requires less analysis time and is therefore more cost-effective.

The Analytical Chemistry Laboratory (ACL) at Argonne National Laboratory (ANL) performs radiochemical analyses for a wide variety of sites within the Department of Energy (DOE) complex. Since the chemical histories of the samples received by the ACL may vary widely from site to site, the ACL must be confident that the chosen dissolution method is appropriate and provides its customers with quality results at reasonable costs. Therefore, ACL's standard repetitive aqua regia leaching technique,⁵ which differs from Bishop's technique, was

compared with Sill's potassium fluoride-pyrosulfate fusion technique³ for both plutonium and americium.

EXPERIMENTAL

Standards

The radionuclides of interest were quantified by isotope dilution employing National Institute of Standards and Technology (NIST) traceable radionuclides. NIST standard reference materials SRM 4350B-Columbia River Sediment and SRM 4353-Rocky Flats Soil #1 were utilized in this study. Environmental Monitoring Laboratory (EML) round-robin soil and vegetation samples, 8803SOAC1 and 8803VEAC1, respectively, were also analyzed.

Sample Preparation

The samples were dried at 80°C for 120 hours. The soils were then milled and sieved to less than 200 mesh for homogeneity. EML samples were analyzed as received. Five to ten grams of sample were employed in each analyses.

Aqua Regia Leachate Method

The samples were slurried with deionized water during the addition of isotopic tracers and then placed in a drying oven to remove the excess water. The soils were then put in a muffle furnace at 510°C overnight to destroy any organic material present in the sample. The aqua regia leaching method requires that the samples boil in 6 N HCl for approximately two hours. The acid is then separated from the soil by centrifuging. The solids are repetitively leached with fresh 6 N HCl, followed by aqua regia, until the color of the leachate clears. The combined leachate solutions are evaporated to incipient dryness, and the residue is redissolved in approximately 40 milliliters of 8 N HNO₃.

The radionuclides of interest are then separated by anion exchange chromatography utilizing Bio-Rad AG 1-X8 resin (chloride form, 100-200 mesh). The plutonium and thorium are initially fixed on the column. Any thorium present is removed by eluting with 40 milliliters of concentrated HCl. The plutonium is then eluted with 25 milliliters of a solution that is 0.1 N HCl and 0.01 N HF. The major soil constituents such as calcium, iron, and phosphates, as well as uranium, americium, and the lanthanides, are not retained and pass directly through the column. This fraction is purified by repetitive oxalic acid precipitations. Uranium is removed through a phosphate coprecipitation of the tervalent actinides and the lanthanides. Finally, americium and the other tervalent actinides are separated from the lanthanide group elements on an ammonium thiocyanate ion exchange column.

Potassium Fluoride-Pyrosulfate Fusion Method

The total dissolution procedure employs a potassium fluoride-pyrosulfate fusion to ensure complete sample dissolution and volatilization of silicon as silica tetrafluoride. The fusion residue is dissolved in dilute hydrochloric acid, and the actinide and lanthanide groups are quantitatively coprecipitated with barium sulfate.

The americium and plutonium are then sequentially separated from an aluminum nitrate-nitric acid system employing 30% Aliquot-336 (Aldrich Chemical Company, Inc.) in xylene as the extractant. In the primary extraction, the acid level is maintained high enough so that the tervalent actinides and the lanthanides do not extract into the organic layer, but remain in the aqueous layer. Thorium is removed from this organic layer with 10 N HCl. Finally, a solution of perchloric and oxalic acids is employed to strip the plutonium from the organic layer.

The original aqueous fraction, which contains the tervalent actinides including americium and the lanthanides, is treated with acid-deficient aluminum nitrate and then extracted with fresh

30% Aliquot-336 in xylene. The trivalent actinides and the lanthanides are back extracted with 8 N HNO₃. Finally, the trivalent actinides are chemically separated from the lanthanides on an ammonium thiocyanate ion exchange column.

Electrodeposition

The analytes of interest were electrodeposited onto 3/4-in. stainless steel disks prior to counting by alpha spectroscopy. A sodium sulfate/sodium bisulfate medium described by Kressin⁶ was utilized for electrodeposition.

RESULTS AND DISCUSSION

Normal soils, high-fired soils, and vegetation samples were analyzed in this study. Table 1 and Table 2 compare analytical results of the leach and fusion methods for ^{239/240}Pu and ²⁴¹Am, respectively. The values reported are either a single analytical result or an averaged value of several determinations, as noted. The differences should reflect both differences in method as well as in sample. Clearly, the leaching method does not remove all of the plutonium from the matrix. However, for those samples that have not been high-fired, the two methods demonstrate relatively good agreement within statistical uncertainty. The refractory nature of the high-fired samples magnify the difference in the methods for plutonium analysis. The americium results exhibit relatively good agreement between both methods.

Table 1. Analysis of $^{239/240}\text{Pu}$ by Leaching and Fusion Methods

Sample Type	$^{234/240}\text{Pu}$, pCi/g ($\pm 2\sigma$) ^a	
	Leach Method	Fusion Method
EML Soil	0.034 (0.004)	0.043 (0.004)
EML Vegetation ^b	0.050 (0.002)	0.058 (0.008)
NIST SRM 4350B	0.011 (0.004)	0.014 (0.006)
NIST SRM 4353 ^b	0.256 (0.026)	0.266 (0.020)
Laboratory High-Fired Soil ^b	0.269 (0.038)	0.396 (0.042)
Environmental High-Fired Soil	0.167 (0.016)	0.256 (0.016)
	3.363 (0.174)	5.716 (0.288)
	22.49 (1.12)	23.72 (1.12)

^aUncertainty based upon propagation of counting statistical error.^bReport values are averages, see Table 5.Table 2. Analysis of ^{241}Am by Leaching and Fusion Methods

Sample Type	^{241}Am , pCi/g ($\pm 2\sigma$) ^a	
	Leach Method	Fusion Method
EML Soil ^b	0.006 (0.002)	0.016 (0.005)
EML Vegetation ^b	0.047 (0.005)	0.046 (0.008)
NIST SRM 4353	0.069 (0.008)	0.042 (0.016)
Laboratory High-Fired Soil	0.530 (0.050)	0.567 (0.110)
Environmental High-Fired Soil	2.560 (0.636)	2.920 (0.422)
	0.015 (0.012)	0.006 (0.004)
	0.562 (0.128)	0.688 (0.052)

^aUncertainty based upon propagation of counting statistical error.^bReported values are averages, see Table 6.

To evaluate the accuracy and the precision of the two methods, standard environmental samples were analyzed. These data are presented in Tables 3-6. At the 99% confidence level, the experimentally determined values generally agree quite well with the accepted values. Our experimental values for NIST SRM 4353 are higher than expected, possibly due to the existence of hot particles in this material. As the data in Tables 5 and 6 demonstrate, the precision of both methods is generally quite good.

Differences between the two methods are most dramatic for the high-fired samples, as expected. However, both techniques are useful under certain circumstances. Clearly, the sample history, as well as the data quality objectives of a study, should play a significant role in the choice of analytical method. For environmental samples that have been high-fired and require high accuracy, the additional cost of a fusion method is warranted. However, for samples that have not been subjected to these extreme conditions, the less-expensive leach method is quite adequate.

Table 3. Experimental Values Versus Accepted Values for $^{239/240}\text{Pu}$

Sample Type	$^{239/240}\text{Pu}$, pCi/g ($\pm 3\sigma$) ^a		
	Leach Method	Fusion Method	Accepted Value
EML Soil ^b	0.034 (0.006)	0.043 (0.006)	0.041 (0.027)
EML Vegetation ^b	0.050 (0.003)	0.058 (0.008)	0.045 (0.003)
NIST SRM 4350B	0.011 (0.006)	0.014 (0.009)	0.014 (0.003)
NIST SRM 4353 ^b	0.256 (0.039)	0.266 (0.030)	0.217 (0.015)

^aUncertainty based upon propagation of counting statistical error.

^bReported values are averages, see Table 5.

Table 4. Experimental Values Versus Accepted Values for ^{241}Am

Sample Type	^{241}Am , pCi/g, ($\pm 3\sigma$) ^a		
	Leach Method	Fusion Method	Accepted Value
NIST SRM 4353	0.069 (0.012)	0.042 (0.024)	0.034 (0.003)
EML Soil ^b	0.006 (0.003)	0.011 (0.008)	0.007 (0.003)
EML Vegetation ^b	0.047 (0.008)	0.058 (0.012)	0.046 (0.006)

^aUncertainty based upon propagation of counting statistical error.^bReported values are averages, see Table 6.Table 5. Reproducibility of the Techniques for $^{239/240}\text{Pu}$

Sample Type	$^{239/240}\text{Pu}$, pCi/g ($\pm 2\sigma$) ^a	
	Acid Leach Method	Fusion Method
EML Vegetation	0.051 (0.002)	0.054 (0.006)
	0.049 (0.002)	0.062 (0.004)
	0.050 (0.002)	
	Avg = 0.050 (0.002)	Avg = 0.058 (0.008)
SRM 4353	0.173 (0.014)	0.238 (0.014)
	0.339 (0.022)	0.294 (0.014)
	Avg = 0.256 (0.026)	Avg = 0.266 (0.020)
Laboratory	0.259 (0.026)	0.390 (0.030)
High-Fired Soil	0.279 (0.028)	0.402 (0.028)
	Avg = 0.269 (0.038)	Avg = 0.396 (0.042)

^aUncertainty based upon propagation of counting statistical error.

Table 6. Reproducibility of the Techniques for ^{241}Am

Sample Type	^{241}Am , pCi/g ($\pm 2\sigma$) ^a	
	Acid Leach Method	Fusion Method
EML Vegetation	0.046 (0.006)	0.039 (0.008)
	0.048 (0.006)	0.062 (0.010)
	0.048 (0.006)	0.054 (0.008)
	Avg = 0.047 (0.005)	Avg = 0.058 (0.008)
EML Soil	0.006 (0.002)	0.006 (0.002)
	0.005 (0.002)	0.009 (0.004)
	0.007 (0.002)	0.017 (0.008)
	Avg = 0.006 (0.002)	Avg = 0.011 (0.005)

^aUncertainty based upon propagation of counting statistical error.

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