

AP10

ANALYSIS OF PLUTONIUM-241 IN ENVIRONMENTAL SAMPLES BY LIQUID SCINTILLATION ANALYSIS

PART A

PRINCIPLE

Solid, unfiltered aqueous and filtered aqueous samples are processed and all plutonium isotopes are separated from the other actinides using procedure AP11. The Pu isotopes are coprecipitated on cerium fluoride and counted with an alpha spectrometry system for chemical yield determination. The sample is then placed in a scintillation cocktail and counted using a liquid scintillation analyzer (LSA).

Other matrices, such as air filters and biotic material, are ashed and the residue is treated as soil.

REFERENCES

1. T. K. Ikaheimone, J. of Radioanalytical and Nuclear Chemistry 243, No.2, 535 (2000).
2. Claude W. Sill, Kenneth Puphal, and Forest D. Hindman, Anal. Chem. 46, 1725 (1974).
3. Claude W. Sill and Roger L. Williams, Anal. Chem. 53, 412 (1981).
4. Claude W. Sill, Anal. Chem. 46, 1426 (1974).
5. Claude W. Sill, Anal. Chem. 36, 675 (1964).
6. Roger P. Bernabee, Donald R. Percival, and Forest D. Hindman, Anal. Chem. 52, 2351 (1980).

CERTIFICATION RECORD FOR

AP10

**ANALYSIS FOR PLUTONIUM-241 IN SOIL, SEDIMENT, WATER, AND OTHER
MATRICES BY LIQUID SCINTILLATION ANALYSIS**

CHECKPOINTS

- 1. **JOB HAZARD ANALYSIS (JHA)** _____
- 2. **MSDS/HAZARDS DISCUSSED** _____
- 3. **FLUORIDE FUSION** _____
- 4. **PYROSULFATE FUSION** _____
- 5. **BARIUM SULFATE PPT** _____
- 6. **EXTRACTION CHROMATOGRAPHY** _____
- 7. **SAMPLE DEPOSITION** _____
- 8. **LIQUID SCINTILLATION COUNTING** _____

ANALYST'S SIGNATURE: _____

CERTIFIED BY: _____

DATE: _____

ANALYSIS VALUE: _____

KNOWN VALUE: _____

MEASURED/KNOWN: _____

See Task _____, Batch _____ for the original data.

COMMENTS: _____

PART B

1.0 PURPOSE AND SCOPE

This procedure provides a method for determination of Pu-241 in environmental samples.

2.0 REAGENTS

All chemicals are hazardous. See MSDS for specific precautions. **See step 2.0 of AP11 JHA.** Unless otherwise indicated, all references to water should be understood to mean reagent grade water.

Acetone, ACS reagent

Scintillation cocktail, Packard Instrument Ultima Gold XR, or equivalent

See reagent list in AP11

3.0 APPARATUS

Liquid Scintillation Analyzer (LSA)

See equipment list in AP11

4.0 PROCEDURE

4.1 General Requirements

Before proceeding, you must be certified as indicated in QCP1 of this manual and Section 3 of the Quality Program (QP) Manual. See preceding page for a copy of the certification record.

All procedures require gloves, lab coats and eye protection at all times.

NOTE: If Pu-241 is being analyzed along with Am-241, one LCS may be used if the activity of Am-241 is known from the Pu-241 parent; otherwise two separate batch Laboratory Control Standards (LCS) must be used; one for the Pu-241 and the other for the Am-241.

4.2 Sample Preparation (Yield Determination)

Follow procedure AP11 for the separation and coprecipitation of Pu. For water samples, begin at **section 4.2** of AP11. For soils and other matrices that are treated as soils, begin at **section 4.3** of AP11. The JHA steps in AP11 are followed for this procedure.

4.3 Sample Preparation (Pu-241 Determination)

4.3.1 After the samples have completed counting on alpha spectrometers for the yield determination, remove the Pu filter, using a small amount of acetone, from the stainless steel disk and place in a glass scintillation vial.

4.3.2 Add 15 mL of scintillation cocktail, shake well, and submit for counting by LSA.

5.0 CALIBRATIONS

Using a NIST traceable Pu-241 standard, prepare a counting efficiency standard by adding an appropriate amount of activity of the standard to a glass scintillation vial containing a 0.1 µm filter. Add 15 mL of scintillation cocktail, shake well, and submit for LSA counting. The region of interest for Pu-241 is 0.5 keV to 7.0 keV. The Laboratory Manager must review and approve the counting efficiency.

6.0 CALCULATIONS

Critical data values will be documented on standard forms maintained as critical records. The following equations define the critical data values. All data will be recorded and reduced according to these calculations.

$$\text{Concentration} = \frac{G - B}{E \cdot Y \cdot Q} = pCi / unit$$

$$2\sigma \text{ Error} = \frac{1.96 \sqrt{(G + B) \cdot T}}{T \cdot E \cdot Y \cdot Q} = pCi / unit$$

$$2\sigma \text{ TPU} = C \cdot 1.96 \sqrt{\frac{(G + B) \cdot T}{((G - B) \cdot T)^2} + RE^2 + RY^2 + RQ^2} = pCi / unit$$

$$\text{MDC} = \frac{3 + 4.65 \sqrt{B \cdot T}}{T \cdot E \cdot Y \cdot Q} = pCi / unit$$

To calculate efficiency:

$$E = \frac{G_E - B}{E_{ACT}} = cpm / pCi$$

where:

B	=	background cpm
C	=	concentration in pCi/unit
E	=	counting efficiency based on Pu-241 (cpm/pCi)
E_{ACT}	=	activity of Pu-241 added for efficiency standard (pCi)
G	=	sample gross counts per minute (cpm)
G_E	=	efficiency standard gross counts per minute (cpm)
MDC	=	minimum detectable concentration
Q	=	sample quantity
RE	=	1σ relative error of the counting efficiency
RQ	=	1σ relative error of the sample quantity
RY	=	1σ relative error of the chemical recovery
T	=	sample count time (minutes)
TPU	=	total propagated uncertainty
Y	=	chemical recovery determined from alpha spectroscopy

7.0 RECORDS

- 7.1 Reference QP Manual for general record requirements.
- 7.2 The raw count data is saved during the weekly backup of the Liquid Scintillation Analyzer to the ORISE network disks.
- 7.3 Hard copies of the assignment and calculation sheets are maintained in the archived site file. Electronic copies of assignment and calculation sheets are saved during the daily incremental backup of the network system. The following data sheets should be completed and retained:
- Pu-241 Analysis Assignment form
 - Pu-241 Lab Data Sheet
 - Pu-241 Concentration and Uncertainty Report (This report may be generated using approved Excel spreadsheets or from the database, if available.)

(AP 10, Rev 3) Pu-241 ANALYSIS ASSIGNMENT FORM

Assigned To: _____ Date: _____ Batch: _____

Task #: _____ LWR #: _____ Activity Lev*: _____

Sample #'s: _____

Analysis Required:

Eff. Spike Pu-241 STD # _____ Volume: _____

(see Special Instructions)

Pipet # _____ Volume: _____ Weight: _____

QC REQUIRED:

BLANK

REPLICATE

LCS

MATRIX SPK

Sample # _____ # Replicates: _____

Pu-241 STD # _____ QUANTITY: _____
UNITS: _____

INITIALS

Sample # _____ QUANTITY: _____
Pu-241 STD # _____ UNITS: _____

SPECIAL INSTRUCTIONS: _____

* If Activity Level is indicated as Moderate or High, perform area survey.

(AP 10, Rev 3) Pu-241 DATA SHEET

Sample #							
Quantity							
Units							

Sample #							
Quantity							
Units							

Sample #							
Quantity							
Units							

Sample #			
Quantity			
Units			

(AP10, Rev 3) Pu-241 CONCENTRATION AND UNCERTAINTY REPORT

INPUT BY:

DATE:

Background CPM
Eff. Spike CPM
pCi Added to Eff. Spike
pCi Added Error

TASK#

Count Time (min)
Efficiency (CPM/pCi)
Eff. Error (CPM/pCi)
Eff. Relative Error

BATCH#

Position #	SID	Sample CPM	SQ	Units (F, g, L, or T)	SQ RE	Yield	Y RE	CT, min	CD
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									

