

UNITED STATES TRANSURANIUM AND URANIUM REGISTRIES
ANALYTICAL PROCEDURE MANUAL

USTUR 310: SEPARATION AND PURIFICATION OF AMERICIUM FROM PRE-CONCENTRATED TISSUE SOLUTIONS

Purpose	Separation and purification of Americium from pre-concentrated tissue solutions	Method Number	USTUR 310
Original Date	11/18/99	Author	Dorothy Stuit
Revision Number	2	Approved By	Jim Elliston
Revision Date	04/28/05	Approval Date	04/28/05

SAFETY NOTE: Before beginning this procedure read all of the Material Safety Data Sheets for the chemicals listed in Section 5 of this procedure.

1. Principle of Method

- 1.1. The americium portion of the sample, previously separated from plutonium in USTUR 220, is separated and purified by extraction chromatography from any remaining radionuclides or interfering elements.
- 1.2. The final americium aliquot is wet-ashed with sulfuric acid to remove traces of organic material from the extraction resin and then prepared for electrodeposition following procedure USTUR 510.

2. Minimum Detectable Activity (MDA)

- 2.1. MDA is limited by the counter background.
- 2.2. For routine measurements with a 75,000-s counting period, a tracer recovery of 60%, and a counting efficiency of 25%, L_d is $8E-4$ Bq per sample (0.05 dpm) of ^{241}Am .

3. Accuracy and Precision

- 3.1. Average blank recovery of ^{243}Am tracer is equal to $76\% \pm 16\%$ at the 0.16-Bq (10 dpm) activity level.

4. Apparatus

- 4.1. Hotplate.
- 4.2. Hotplate thermometer with range to 200°C.
- 4.3. Eichrom TRU resin column.

UNITED STATES TRANSURANIUM AND URANIUM REGISTRIES
ANALYTICAL PROCEDURE MANUAL

- 4.4. Bio-Rad plastic column reservoir.
- 4.5. Rack: to support columns.
- 4.6. Beakers: various sizes.
- 4.7. Watchglasses: assorted sizes to fit beakers.
- 4.8. Graduated cylinders: various sizes.
- 4.9. Disposable plastic transfer pipet.

5. Reagents

- 5.1. 18 M Ω Type 1 deionized water (D.I. water).
- 5.2. Nitric acid (concentrated 69-71%, reagent-grade).
- 5.3. Nitric acid (3 M). Add 190 mL of concentrated HNO_3 to 810 mL of Type 1 deionized water.
- 5.4. Nitric acid (0.025 M). Add 1 mL of concentrated HNO_3 to Type 1 deionized water and bring to a volume of 640 mL with Type 1 deionized water.
- 5.5. Sulfuric acid (concentrated 96-98%, reagent grade).

6. Procedure

- 6.1. Sample preparation.
 - 6.1.1. Americium aliquot from step 6.3.8, USTUR 220, is run in this procedure.
- 6.2. Sample dissolution.
 - 6.2.1. To each dried americium aliquot, add 25 mL of 3 M HNO_3 .
 - 6.2.2. Cover each sample with a watchglass and warm on hotplate set at 120°C to completely dissolve the sample. Remove from hotplate and cool completely before adding solution to column.
- 6.3. Extraction Separation.
 - 6.3.1. Remove the cap from a TRU column and fit a Bio-Rad reservoir onto its top.

Note: Column resin should be covered with several milliliters of liquid, if the column is dry do not use it.

UNITED STATES TRANSURANIUM AND URANIUM REGISTRIES
ANALYTICAL PROCEDURE MANUAL

- 6.3.2. Snap bottom tip from column and allow storage solution to drain.
- 6.3.3. When the column has drained, add 8 mL of 3 M HNO₃ and allow it to drain completely.
- 6.3.4. Add the prepared sample solution to the column; rinse the beaker twice with approximately 5 mL of 3 M HNO₃ and adding each rinse to the column. Allow the column to drain completely.
- 6.3.5. Add 12 mL of 3 M HNO₃ to the column and allow it to drain completely.
- 6.3.6. Using a disposable pipet, collect any remaining droplets left in the column top and reservoir and add to column. Allow the column to drain completely.
- 6.3.7. Place a clean 100 or 150 mL beaker, labeled with sample number and "Am", under the column.
- 6.3.8. Add 15 mL of 0.025 M HNO₃ to the column to remove americium. Allow it to drain completely.
- 6.3.9. Add 2 mL of concentrated sulfuric acid to the sample in the collection beaker.
- 6.3.10. Bring the sample to dryness by starting on a hotplate set at 120°C and gradually raising the temperature up to 250°C, until the sulfuric acid is fumed off.
- 6.3.11. Continue preparing the sample for electrodeposition by following USTUR 510.

Note: Add 4 mL instead of 2 mL of 0.36 M NaHSO₄ to sample for optimum recovery and proceed to USTUR 510.

7. Source Materials

- 7.1. Hongguo Qu, et al, J. Radioanalytical and Nuclear Chem. 234 (1-2), 175 (1998).