USTUR 510: ELECTRODEPOSITION OF AMERICIUM, PLUTONIUM, THORIUM, AND URANIUM

Purpose	Electrodeposition of americium, plutonium, uranium, and thorium	**Method Number**	USTUR 510
Original Date	12/17/96	**Author**	USTUR Radiochemistry
Revision Number	3	**Approved By**	Jim Elliston
Revision Date	3/1/00	**Approval Date**	1/31/01

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Section 3 of this procedure.

1. Principle of Method

1.1. Radionuclides isolated using the various radiochemical separation methods for americium, plutonium, thorium, and uranium are electrodeposited from a sulfate electrolyte onto stainless-steel disks.

2. Apparatus

2.1. Stainless-steel disks: 5/8-in. or 11/16-in. diameter disk, polished on one side with protective tape (A.F. Murphy Die and Machine Co.).

2.2. Coin holders.

2.3. Electrolytic cell: Nalgene 30 mL narrow mouth polypropylene bottle cell body (Fig. 1).

2.4. Plastic covers for cells (analytical funnel with stem removed).

2.5. Electrodeposition apparatus: dc power supply to provide up to 1 A of regulated current to each electrolytic cell.

2.6. Hot plate.

2.7. Plastic transfer pipettes.

3. Reagents

3.1. Thymol blue indicator. Mix 0.2 g of thymol blue powder with 21.5 mL of 0.02 M NaOH and dilute to 500 mL with nanopure water.

3.2. Sodium Bisulfate (fused) (0.36 M): 42.23 g of $NaHSO_4$ (MW 120.07 g/mole) in 1000 mL of nanopure water.

3.3. Sulfuric acid (0.75 M). 42 mL of concentrated (18 M) H_2SO_4 with 958 mL of nanopure water.

3.4. Ammonium hydroxide (concentrated, reagent-grade).

3.5. Ammonium hydroxide (1.5 M). Mix 100 mL of concentrated NH_4OH with 900 mL of nanopure water.

4. Preparation of Sample Disks and Electrodeposition Cell

4.1. Engrave sample name, date, and operator initials onto a new 5/8 inch planchet.

4.2. Remove tape with tweezers and place in indentation of cell cap (see figure 1) with polished side up.

4.3. Rinse a Quad ring with acetone and allow to dry. Place the Quad ring over the planchet.

4.4. Tightly screw a new 30 mL bottle into the cell cap.

4.5. Remove the bottom of the bottle with a razor or other sharp instrument.

5. Electrodeposition

5.1. Add 2 mL of 0.36 M $NaHSO_4$ to final separated fraction and take to dryness. If an Eichrom column was used or if concerned about organic interferences, also add 1 mL of concentrated H_2SO_4 prior to drying the sample.

5.2. Wet ash the sample: Add 5 mL of concentrated HNO_3 to the sample and take to dryness at 120-150°C. Repeat if necessary (especially for samples with H_2SO_4).

5.3. Allow the sample to cool.

5.4. Add 5-6 mL of 0.75 M H_2SO_4 to the sample and add 3-4 drops of thymol blue indicator.

5.5. Transfer sample to cell with plastic transfer pipette.

5.6. Rinse beaker twice with 3 mL of 0.75 M H_2SO_4 and add each wash to the cell using the same transfer pipette.

5.7. Adjust the pH of the solution in the electrolytic cell to a salmon pink end point (pH 2-2.3) using concentrated NH_4OH. The pH of samples being deposited for thorium should be slightly more acidic (pH 1.5 to 2.0).

5.8. Place the cell in the electrodeposition rack and cover with a plastic cover. Attach cathode lead to the cell cap.

5.9. Insert platinum wire anode (mounted on glass rod) to a distance of ~1 cm of the cathode.

5.10. Electrodeposit plutonium, americium, thorium, and uranium for 1 hour at a current of 0.75 A.

5.11. At the end of 1 hour fill the cell with 1.5 M NH_4OH and continue deposition for 1 min.

5.12. Turn off the main power switch of the electrodeposition unit.

5.13. Disconnect the cathode lead.

5.14. Remove the plating cell from the rack and discard contents into the electro-deposition radioactive waste.

5.15. Rinse the cell twice with 1% NH_4OH.

5.16. Rinse the cell three times with nanopure water.

5.17. Disassemble the cell, rinse the disk with nanopure H_2O, and touch the disk edge to a paper towel to remove excess water.

5.18. Place the disk on a flat Pyrex dish and heat at a minimum temperature of 300°C for 10 minutes to drive off Po-210.

5.19. Place the disk in a coin holder, label the holder with sample number, and submit to count room for alpha spectrometric measurement.

Electrodeposition Parameters

Element	Deposition Time	Current	Initial pH
U, Pu, Am	1 hour	0.75A	2-2.3
Th	1 hour	0.75A	1.5-2

6. Source Materials

6.1. H.A. Boyd, B.C. Eutsler, J.F. McInroy, "Determination of Americium and Plutonium in Autopsy Tissue: Methods and Problems," in *Actinides in Man and Animals*, Proceedings of the Snowbird Actinide Workshop, Oct. 14-17, 1979, M.E. Wrenn, scientific editor (R.D. Press, Salt Lake City, Utah, 1981), pp. 43-52.

6.2. N.A. Talvitie, "Electrodeposition of Actinides for Alpha Spectrometric Determination," *Anal. Chem.* **44**, 280-283 (1972).

6.3. C.W. Sill, D.G. Olson, "Sources and prevention of recoil contamination of solid state and alpha detectors," *Anal. Chem*. **42**, 1596-1607 (1970).

6.4. S.E. Glover, R.H. Filby, S.B. Clark, "Optimization and Characterization of a Sulfate Based Electrodeposition Method for Alpha Spectroscopy of Actinide Elements Using Chemometric Analysis." Radioanalytical and Nuclear Chemistry, Vol. 1-2, 1988.

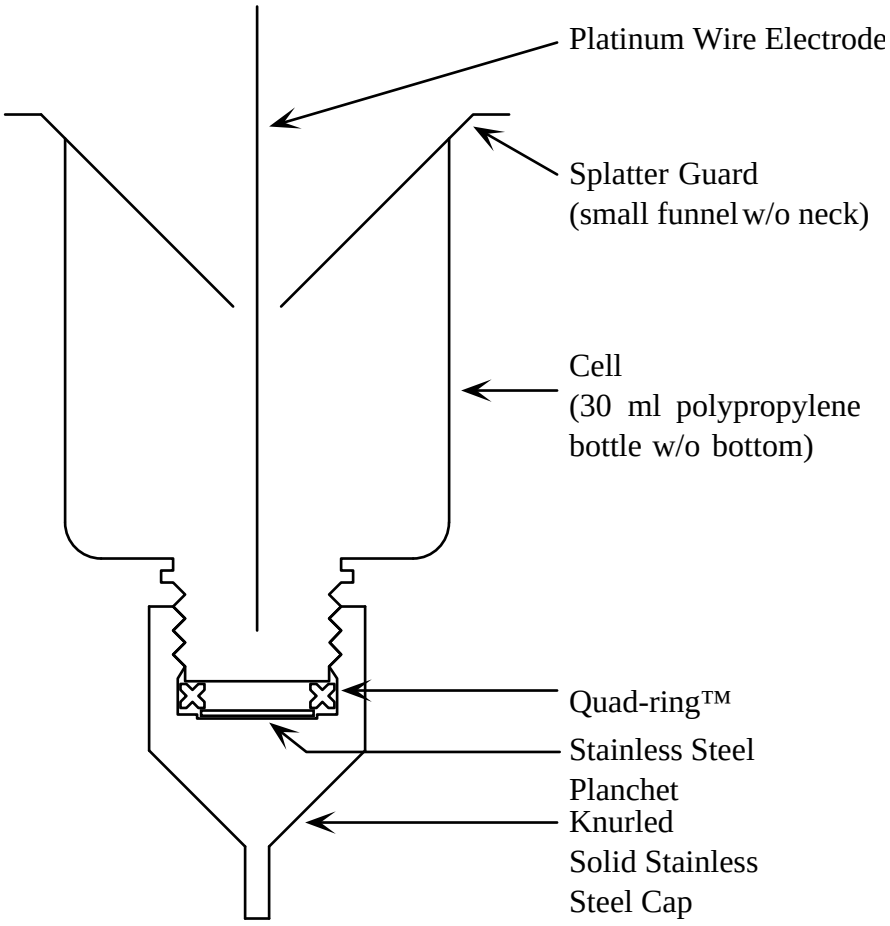

Fig. 1. Electrolytic cell.