USTUR 300: ANION EXCHANGE ISOLATION OF AMERICIUM FROM PREPARED TISSUE SOLUTIONS

Purpose	Anion exchange for ^{241}Am	**Method Number**	USTUR 300
Original Date	10/10/95	**Author**	Radiochemistry Staff
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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. Radionuclides of americium are isolated from ashed and acid-dissolved tissue samples.

1.2. An aliquot of the tissue sample solution is selected for analysis based on wet sample mass and estimated sample alpha activity.

1.3. Americium-243 tracer is added to the aliquot. Americium is separated from prepared tissue solutions by solvent extraction and anion exchange and is electrodeposited onto stainless-steel disks.

1.4. Concentrations of ^{241}Am are determined by alpha spectrometry.

1.5. Chemical losses are identified and corrected on the basis of the recovery of the added ^{243}Am tracer.

NOTE: If only americium is being analyzed, begin with procedure USTUR 210, then follow with this procedure (USTUR 300).

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 dis/min) of ^{241}Am.

3. Accuracy and Precision

3.1. Average blank recovery of ^{243}Am tracer is equal to 76% ± 16% at the 0.16-Bq (10 dis/min) activity level.

4. Apparatus

4.1. Fume Hood.

4.2. Beakers: 100- and 150-mL.

4.3. Magnetic stirring hot plates: Corning PC-351 or equivalent.

4.4. Stir bars: Teflon-coated.

4.5. Wash bottle: 250-mL.

4.6. Hot plates: Thermolyne Model 2200 or equivalent.

4.7. Separatory funnels: 125-, 250-, or 500-mL.

4.8. Funnel support: adjustable height.

4.9. Watch glasses: assorted sizes.

4.10. Bio-Rad Ion-exchange columns (Fig. 1): Econopak borosilicate glass column with polypropylene reservoir, stop-cock, and bed support, 20 cm long by 1.0 cm i.d.

4.11. Rack: to support ion exchange columns.

4.12. Funnel: powder, 50-mL.

4.13. Graduated cylinders: 10-, 100-, 500-, and 1000-mL.

4.14. Thermometer: dial-type, metal, 0-550°C (VWR; Catalog #61157-254).

4.15. Glass beads: 2 or 3 mm diameter.

5. Reagents

5.1. Nitric acid (concentrated 69-71%, reagent-grade).

5.2. Silver nitrate (1%). Add 1 g of $AgNO_3$ to 100 mL of nanopure water.

5.3. Bio-Rad anion-exchange resin (AG MP-1, 100-200 mesh) chloride form. Make a slurry of half resin, half nanopure water in a wash bottle.

5.4. Sodium nitrite (reagent grade).

5.5. DDCP (Dibutyl-N,N-diethylcarbamoyl phosphonate, technical-grade).

5.6. Dodecane (A.C.S. reagent-grade).

5.7. Nitric acid (2 M). Add 125 mL of concentrated HNO_3 to 875 mL of nanopure water.

5.8. Nitric acid (2.5 M). Add 78 mL of concentrated HNO_3 to 422 mL of nanopure water.

5.9. Nitric acid (6 M). Add 375 mL of concentrated HNO_3 to 625 mL of nanopure water.

5.10. Nitric acid (12 M). Add 750 mL of concentrated HNO_3 to 250 mL of nanopure water.

5.11. Ethanol (absolute).

NOTE: For the alcohol/nitric acid mixtures, limit the amount made (to minimize volume of waste collected). For each sample run you need: 31 mL Reagent A, 30 mL Reagent B, 30 mL Reagent C, and 40 mL Reagent D. Reagents A-D should be kept well-covered, to prevent evaporation, until use.

5.12. Ethanol-nitric acid, Reagent A (60:40 mixture of EtOH and 6 M HNO_3). Add 140 mL of 6 M nitric acid to 210 mL of ethanol. Prepare fresh.

5.13. EtOH-$NaNO_2$ (saturated). Add solid $NaNO_2$ to ethanol and shake well.

5.14. Methanol (absolute, ACS reagent-grade).

5.15. Methanol-nitric acid, Reagent B (75:25 mixture of MeOH and 6 M HNO_3). Add 100 mL of 6 M nitric acid to 300 mL of methanol. Prepare fresh.

5.16. Methanol-nitric acid, Reagent C (60:40 mixture of MeOH and 6 M HNO_3). Add 140 mL of 6 M nitric acid to 210 mL of methanol. Prepare fresh.

5.17. Methanol-nitric acid, Reagent D (60:40 mixture of MeOH and 2.5 M HNO_3). Add 140 mL of 2.5 M nitric acid to 210 mL of methanol. Prepare fresh.

CAUTION: Any remaining reagent from 5.13, 5.16 and/or 5.18 after column completed must be diluted with a minimum of twice the volume of H_2O and discarded into waste container labeled alcohol/nitric acid mixture. The container should be kept tightly capped.

6. Procedure

6.1. Sample preparation.

6.1.1. Sample aliquot is obtained from USTUR 200, Step 6.3.9.

6.2. Sample dissolution.

6.2.1. Add 5 mL of concentrated HCl to the sample and heat to 80-100°C.

6.2.2. Add 20 mL of 12 M HNO_3 and heat, using a magnetic stirrer if necessary, to dissolve the sample. Add 15 mL of concentrated HNO_3. This brings solution concentration back up to 12 M HNO_3. If the sample does not dissolve, add an additional 5 mL of concentrated HCl, 20 mL of 12 M HNO_3, and 15 mL of concentrated HNO_3. Repeat these additions until the sample is dissolved. Bring the final volume to 40, 90, 140, 190 mL, etc., by adding 12 M HNO_3.

NOTE: An alternate method may be more successful for certain samples, especially those containing cartilage. To the 5 mL of concentrated HCl added in Step 6.2.2, slowly add 5 mL of H_2O. Heat until the sample is dissolved or nearly dissolved and then add 30 mL of concentrated HNO_3. It may be necessary to use multiples of these amounts depending on the size and difficulty of the sample.

6.3. Extraction of americium.

6.3.1. Transfer the solution to an appropriate size (125-, 250-, 500-mL) separatory funnel. Rinse the beaker with 5 mL of 12 M HNO_3 then add the rinse to the separatory funnel. Repeat rinse.

6.3.2. Add 1 mL of undiluted DDCP per 50 mL of solution and shake for 20 s. Allow to stand until the phases separate (approximately 1 h).

6.3.3. Drain the lower aqueous phase and discard into waste container labeled "12 M HNO_3 only from Am Extraction".

6.3.4. Add 10 mL of 12 M HNO_3 to the DDCP solution. Shake for 10 s. Allow the phases to separate.

6.3.5. Drain and discard the aqueous phase into waste container labeled "12M HNO_3 only from Am Extraction".

6.3.6. Add 20 mL of 2 M HNO_3 for each 1 mL of DDCP used in Step 6.3.2, and shake for 10 s. Allow the phases to separate.

6.3.7. Drain the aqueous solution into a 100- or 150-mL labeled beaker.

6.3.8. Repeat Steps 6.3.6 through 6.3.7.

NOTE: Evaporation of the collected stripping solution from Step 6.3.7 may be started while phases are separating.

6.3.9. Discard the organic solution into a plastic waste container labeled "DDCP/Dodecane." Rinse separatory funnel with 5-10 mL dodecane and add to the waste container.

6.3.10. Place the beaker on a hot plate with a surface temperature of 120-140°C and take to dryness.

6.3.11. Add 10 mL of concentrated HNO_3 to the beaker and take to dryness.

6.3.12. Repeat Step 6.3.11 at least twice to remove organics.

6.4. Methanol-nitric acid anion exchange.

6.4.1. Add 5 mL of 6 M HNO_3 to the sample. Cover with a watch glass and heat between 80-90° C for 10-15 min to dissolve the sample.

NOTE: It may be necessary to increase the volume of 6 M HNO_3 in order to get the sample into solution. Increase the volume of ethanol used in Step 6.4.3 proportionately. If the sample appears to be insoluble in 6 M HNO_3, evaporate it to dryness, dissolve the sample in concentrated HNO_3, add enough deionized water to make the solution 6 M HNO_3, and multiply the total volume by 1.5 to find the amount of ethanol to use in Step 6.4.3.

6.4.2. Cool to room temperature.

6.4.3. Add 7.5 mL of EtOH saturated with $NaNO_2$, to the dissolved sample. Cover and allow to stand while the column is prepared.

6.4.4. Measure 7 mL of water into the column and mark the column at the water level. Discard the water. Add a slurry of AG MP-1 (100-200 mesh) resin, allowing it to settle to about 1 cm above the mark. (The resin should settle to the mark after being washed with HNO_3.)

6.4.5. Rinse all resin down from the sides of the column using distilled water then 6.0 M HNO_3. Once all the acid has drained, add approximately 1 cm depth of glass beads on top of the resin to prevent disturbance as reagents are added.

6.4.6. Wash the column with a 15 mL aliquot of 6 M HNO_3.

6.4.7. Test the last 5 mL of effluent for Cl^- by adding one drop of 1% $AgNO_3$.

6.4.8. If a white precipitate forms, continue washing the column with 6 M HNO_3 until no precipitate is formed when 1% $AgNO_3$ is added.

6.4.9. Add 21 mL of 60:40 ethanol nitric acid, Reagent A, to the column, drain, and discard, with 50 mL H_2O added to effluent, into the waste container marked alcohol/nitric acid mixture.

NOTE: From this step to the stripping of americium, do not let the column dry at any time. Once liquid levels are down to the glass beads, add the next reagent.

6.4.10. Transfer the sample to the column funnel. Rinse the beaker with 5 mL of 60:40 ethanol nitric acid, Reagent A, and add to the column. Repeat this rinse and add it to the column.

6.4.11. Wash the column with 30 mL of 75:25 methanol nitric acid, Reagent B.

6.4.12. Wash the column with 30 mL of 60:40 methanol nitric acid, Reagent C.

6.4.12.1. Add 200 mL H_2O to the column washes and discard into waste container for alcohol/nitric acid mixture, which is kept tightly capped.

6.4.13. Elute the americium from the column with 40 mL of 60:40 MeOH and 2.5 M HNO_3, Reagent D, and collect in a 100-mL beaker for electrodeposition.

6.4.14. Evaporate the eluate to dryness on a low temperature (<80° C) hot plate.

6.4.15. Add 10 mL concentrated HNO_3 and heat to dryness. Repeat several times to remove any organics.

6.5. Proceed to procedure USTUR 500, "Electrodeposition of Americium, Plutonium, and Uranium."

7. Source Materials

7.1. J.F. McInroy, H.A. Boyd, B.C. Eutsler, and D. Romero, *Health Phys.* **49**, 587-621 (1985).

7.2. D. Knab, *Anal. Chem.* **51**, 1095 (1979).

7.3. LANL method RT300.

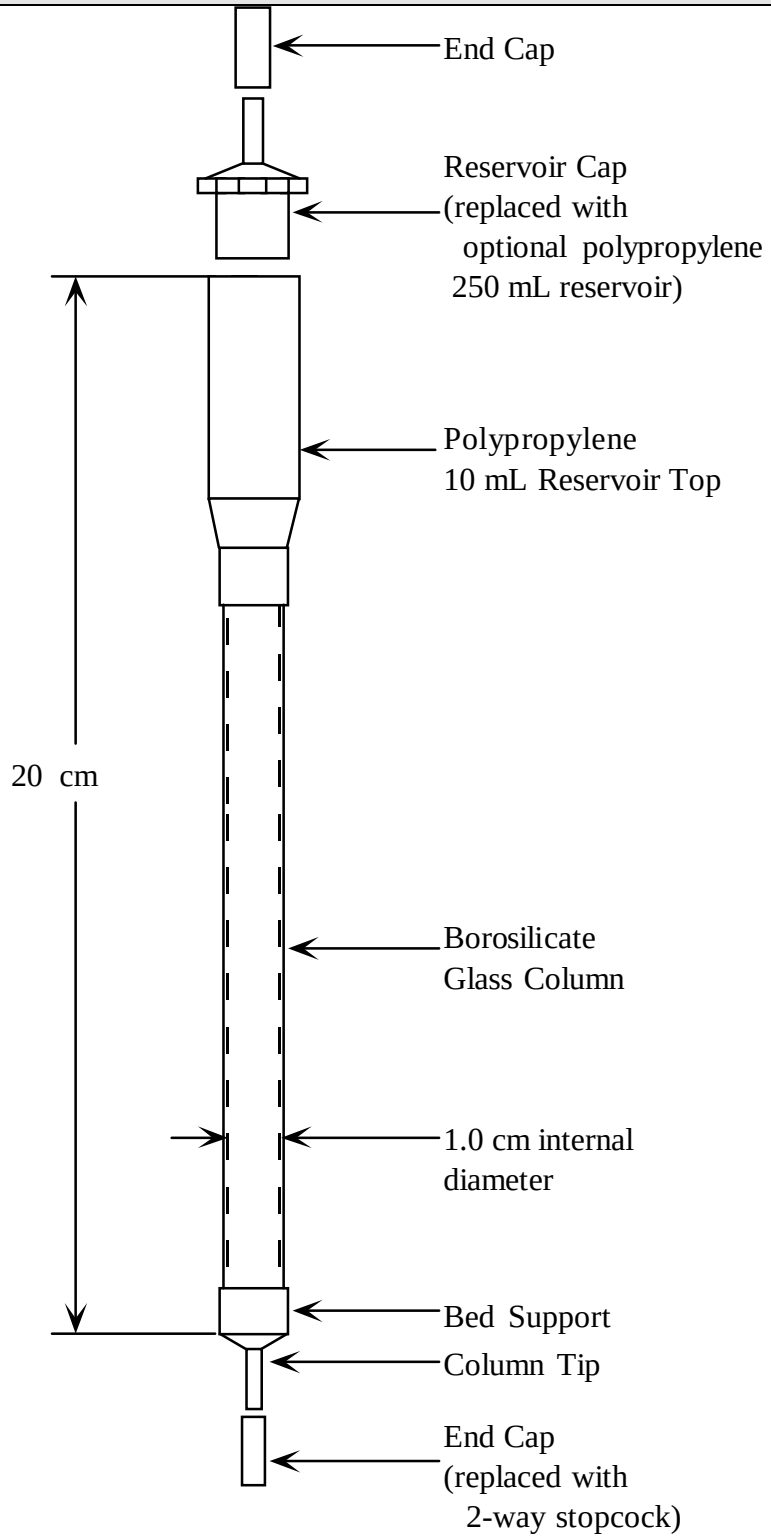

Fig. 1. Ion exchange column for americium.