USTUR 200: ANION EXCHANGE ISOLATION OF PLUTONIUM FROM PREPARED TISSUE SOLUTIONS

Purpose	Anion exchange for isotopes of plutonium	**Method Number**	USTUR 200
Original Date	10/10/95	**Author**	Radiochemistry Staff
Revision Number	3	**Approved By**	James T. Elliston
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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 plutonium are isolated from ashed and acid-dissolved tissue samples (USTUR 100).

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

NOTE: A concentration of 25 mg ash per mL of 9 M HCl-Cl_2 should not be exceeded. Ash weights for bones are recorded, while ash weights of soft tissues are assumed to be 1% of wet weights.

1.3. Plutonium-242 tracer is added to the aliquot. Plutonium is isolated by anion exchange and is electrodeposited onto stainless-steel disks.

1.4. Concentrations of ^{238}Pu and ^{239}Pu are determined by alpha spectrometry.

1.5. Chemical losses are identified and corrected on the basis of the recovery of the added ^{242}Pu tracer.

NOTE: If only americium is being analyzed, use procedure USTUR 210.

2. Minimum Detectable Activity (MDA)

2.1. MDA is limited by the counter background.

2.2 For routine measurements with a 75,000-s sample counting period; 600,000-s background count (20 counts), 60% tracer recovery, and a counting efficiency of 25%, the L_D is 0.001 Bq per sample (0.05 dis/min) of ^{238,239}Pu. See USTUR 600 for alpha spectrometry procedures.

3. Accuracy and Precision

3.1. Average blank recovery of ^{242}Pu tracer is equal to 87% ± 8% at the 0.15-Bq (9 dpm) concentration level as of 6/10/96.

4. Apparatus

4.1. Hot plates.

4.2. Magnetic stirring hot plates.

4.3. Stirring bars: Teflon-coated.

4.4. Hot plate thermometer with a range to 200°.

4.5. Watch glasses: assorted sizes to fit beakers used.

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

4.7. Rack: to support ion-exchange columns.

4.8. Glass beads: 2 or 3 mm diameter.

4.9. Graduated cylinders: various sizes.

4.10. Beakers: various sizes.

4.11. Wash bottle, 500 mL.

5. Reagents

5.1. 18 M Ω (nanopure) deionized water

5.2. Hydrochloric acid (concentrated, 36.5 – 38%, reagent grade).

5.3. Hydrogen peroxide (30%, reagent grade).

5.4. Chlorinated hydrochloric acid solution (9 M HCl-Cl_2). Add 5 mL H_2O_2 (30%) and 750 mL concentrated HCl to 250 mL nanopure H_2O. Heat for 1 hour at 120°C, noting formation of yellow color. Make fresh just before use.

5.5. Hydrochloric acid (0.6 M). Add 50 mL of concentrated HCl to 950 mL of nanopure H_2O.

5.6. Bio-Rad anion-exchange resin (AG 1-X4, 100-200 mesh) chloride form. Make a slurry of half resin, half nanopure water in a wash bottle.

5.7. Nitric acid (concentrated, 69 – 71%, reagent grade).

5.8. Nitric acid (8M). Add concentrated HNO_3 to an equal volume of nanopure H_2O.

5.9. Hydrochloric acid (1.2 M). Add 100 mL of concentrated HCl to 900 mL nanopure H_2O.

6. Procedure

6.1. Sample preparation.

6.1.1. Refer to procedure USTUR 100, Tissue Ashing, Sample Dissolution, Sample Aliquot Selection, and Tracer Addition.

6.1.2. Do not add U-232 tracer to an aliquot to be analyzed for Pu. The order of stripping will allow Th-228 (U-232 daughter) to interfere with Pu-238. An alternate elution step must be used!

6.2. Sample dissolution.

6.2.1. To the previously prepared sample, add enough 9 M HCl-Cl_2 (a minimum of 100 mL is needed, keeping a concentration of 25 mg ash per mL or less) to completely dissolve the sample when heated to 120°C on a hot plate. Use stirring hot plates with magnetic stir bars to hasten the dissolution of large samples. Cover the beaker with a plain watch glass while heating.

6.2.2. When the sample is dissolved, heat it for an additional 5-10 min on a hot plate with a surface temperature of 120°C. Remove the sample from the hot plate and allow it to cool to room temperature.

6.3. Anion exchange separation.

6.3.1. Add 12-mL of nanopure water to the column and mark the column at the water level. Drain off the H_2O.

6.3.2. Fill the column with a slurry of AG 1-X4 resin to a settled volume of slightly more than 12 mL (approx. 0.5 cm above the mark).

6.3.3. Rinse all of the resin down from the sides of the column using nanopure water, then 0.6 M HCl. Once all of the acid has drained, add approximately 1 cm depth of glass beads on top of the resin to prevent

disturbance as reagents are added. Place a reservoir on the top of the column.

6.3.4. Wash the column with 60 mL (5 column volumes) of 9 M HCl-Cl_2, allowing the solution to drip into a waste beaker. Discard this solution into a hazardous waste container (Non-rad).

6.3.5. If americium is also to be determined in the sample, place a clean labelled collection beaker under the column. This must be large enough to contain the sample volume plus the 85 mL (7 column volumes) of 9 M HCl-Cl_2 wash solution that will follow.

6.3.6. Transfer the sample to the ion exchange column. Rinse the beaker twice with 9 M HCl-Cl_2, adding the washings to the column.

6.3.7. Adjust the flow rate to between 1 and 3 mL/min.

6.3.8. When the solution has drained to the top of the resin bed, add 85 mL (7 column volumes) of 9 M HCl-Cl_2 to the column.

6.3.9. When the solution has reached the top of the resin bed, remove the americium collection beaker, if used, and replace it with a waste beaker for the next wash. Evaporate the americium fraction to dryness on a hot plate with a surface temperature not greater than 120°C.

NOTE: Proceed to procedure USTUR 300, "Anion Exchange Isolation of Americium from Prepared Tissue Solutions," for the determination of americium.

6.3.10. Wash the column with 120 mL (10 column volumes) of 8 M HNO_3. If the sample is also to be analyzed for uranium, discard the first 30 mL (2.5 column volumes) of this wash into a radioactive waste container. Replace the waste beaker with a clean labeled 150-mL beaker and collect the remaining 90 mL (7.5 column volumes) for the uranium procedure, USTUR 400. Evaporate this uranium fraction to dryness on a hot plate set to 150°C, but do not bake.

NOTE: Washing with 8 M HNO_3 removes the iron from the resin bed. At this point the deep yellow color is removed from the column.

6.3.11. When the wash solution has reached the top of the resin bed, replace the waste beaker with a labeled 150-mL beaker for the collection of the plutonium labeled fraction.

6.3.12. Elute the plutonium with 85 mL (7 column volumes) of 1.2 M HCl containing 2 mL 30% H_2O_2 per 100 mL 1.2 M HCl.

6.3.13. Evaporate the eluate to dryness on a hot plate set to 120°C.

6.3.14. Add 5-10 mL of concentrated HNO_3 to the beaker and evaporate to dryness at 150°C. Repeat this step as necessary until no (or little) residue is present.

6.4. Proceed to procedure USTUR 500, "Electrodeposition of Americium, Plutonium, and Uranium" or USTUR 510.

7. Source Materials

7.1. I.K. Kressin, *Anal. Chem.* **49**, 842 (1977).

7.2. I.K. Kressin, W.D. Moss, E.E. Campbell, and H.F. Schulte, *Health Phys.* **28**, 41 (1975).

7.3. LANL method RT 400.

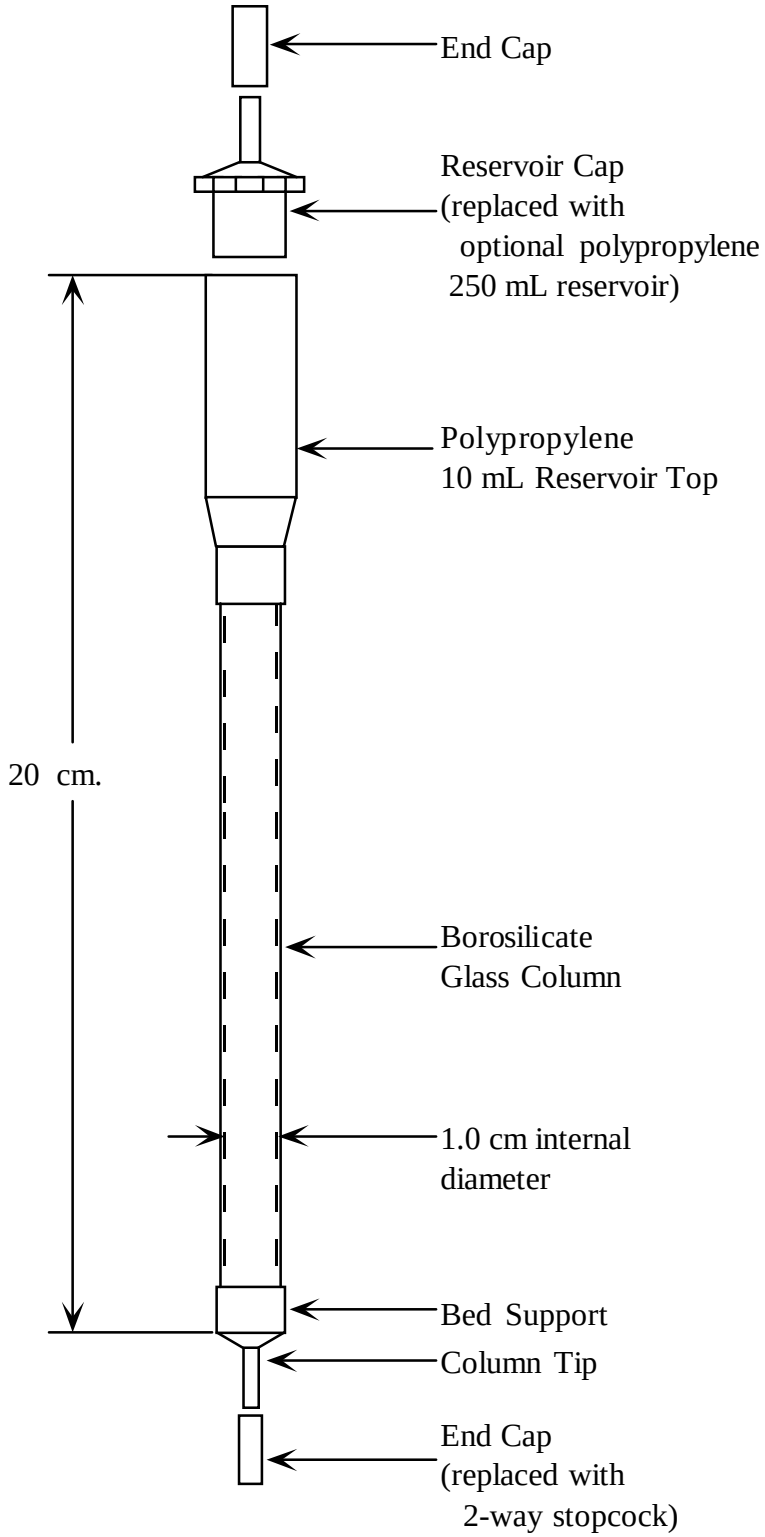

Figure 1. Ion Exchange Column