USTUR 400: ANION EXCHANGE ISOLATION OF URANIUM FROM PREPARED TISSUE SOLUTIONS

Purpose	Anion exchange for isotopes of uranium	**Method Number**	USTUR 400
Original Date	10/10/95	**Author**	USTUR Radiochemistry Staff
Revision Number	2	**Approved By**	Jim 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 uranium 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. Uranium-232 tracer is added to the aliquot. Uranium is isolated by anion exchange and is electrodeposited on stainless-steel disks.

1.4. Concentrations of ^{234}U, ^{235}U, and ^{238}U are determined by alpha spectrometry.

1.5. Chemical losses are identified and corrected on the basis of the recovery of the added ^{232}U tracer.

2. Minimum Detectable Activity (MDA)

2.1. MDA is limited by the counter background, recovery, efficiency, and count time.

2.2 For routine measurements with a 75,000-s counting period; a 600,000-s background count (20 counts), a tracer recovery of 60%, and a counting efficiency of 15% (shelf 3), L_D is 0.001 Bq per sample (0.08 dis/min) of ^{234}U and ^{238}U. This amount of ^{238}U alpha activity is equivalent to 0.06 μg of normal uranium (0.0715% ^{235}U) if the ratio of $^{238}U/^{234}U$ activity is equal to one and the sample is known to contain only normal uranium.

NOTE: Measurement of samples for their concentrations of ^{235}U will be less exact because of the possible tailing of ^{234}U into the energy spectrum area of ^{235}U. Measurement of samples for ^{235}U concentration may require special counting measures to resolve the ^{234}U and ^{235}U energy peaks (e.g., lower counting efficiency and longer counting periods).

3. Accuracy and Precision

3.1. Average recovery of ^{232}U tracer is equal to 90% ± 8% at the 0.07 Bq (4.2 dis/min) concentration level.

4. Apparatus

4.1. Beakers: 150-, and 250-mL.

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

4.3. 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.

4.4. Rack: to support ion-exchange columns.

4.5. Graduated cylinders: 100-, and 1000-mL.

4.6. Hot plates: Thermolyne Model 2200 or equivalent.

4.7. Glass beads, 2 or 3 mm diameter.

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

4.9. Stirring bars: Teflon-coated.

5. Reagents

5.1. 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.

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

5.3. Hydrochloric acid (8 M). Add 670 mL of concentrated HCl to 330 mL of nanopure water.

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

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

5.6. Nitric acid (8 M). Add 500 mL of concentrated HNO_3 to 500 mL of nanopure water.

6. Procedure

6.1. Sample preparation.

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

NOTE: If the samples contain plutonium, it should be removed using procedure USTUR 200, Anion Exchange Isolation of Plutonium from Prepared Tissue Solutions. Then follow this procedure.

6.2. Sample dissolution.

6.2.1. To the previously prepared sample, add enough 8 M HCl (start with 50 mL) to completely dissolve the sample when heated to 120°C on a hot plate. Use stirring hot plates and magnetic stirring bars to hasten the dissolution of large samples. Cover the beaker with a watch glass while heating.

6.2.2. Remove the sample from the hot plate and allow it to cool to room temperature.

6.3. Anion exchange separation.

6.3.1. Measure 12 mL of water into the column and mark the column at the water level. Discard the water.

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

6.3.3. Rinse all resin down from the sides of the column using first 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 resin to prevent disturbance as reagents are added. Wash column with 60 mL of 0.6M HCl to remove any residual uranium from the resin.

6.3.4. Wash the column with 60 mL (5 column volumes) of 8 M HCl and allow the solution to drain to the top of the resin. Discard wash solutions into Non-Rad acid waste container.

6.3.5. Transfer the sample from Step 6.2.2. to the ion exchange column.

6.3.6. Rinse the beaker twice with 5 mL of 8 M HCl. Add the rinses to the column. Allow the sample and rinses to drain down to the top of the resin.

6.3.7. Wash the column with 85 mL (7 column volumes) of 8 M HCl. Allow the solution to drain to the top of the resin.

6.3.8. Wash the column with 30 mL (2.5 column volumes) of 8 M HNO_3. This will remove iron from the resin bed.

NOTE: This amount of acid will not remove all of the iron, but if larger amounts of 8 M HNO_3 are used, uranium will also be removed from the resin bed.

6.3.9. After all of the wash solution has drained through the column, place a labeled 150 mL beaker under the column to collect eluted uranium.

6.3.10. Elute the uranium with an additional 90 mL (7.5 column volumes) of 8 M HNO_3.

6.3.10.1. For immediate electrodeposition add 2 mL of 0.36 M fused $NaHSO_4$, see USTUR 500 or 510.

6.3.11. Place the sample beaker on a hot plate with a surface temperature of approximately 200ºC and evaporate the sample to dryness.

NOTE: If the eluted sample contains noticeable amounts of iron in the elution residue, as evidenced by yellow color on the bottom of the beaker, a second ion-exchange separation is required. Repeat Steps 6.2.1 through 6.3.10, dissolving the sample in only 10 mL of 8 M HCl and using 3 mL of resin in an ion-exchange column.

NOTE: Column volume is now 3 mL and volumes added in steps 6.3.4, 6.3.7, 6.3.8 and 6.3.10 must reflect this:

Step 6.3.4. 5 column volumes = 15 mL 8 M HCl
Step 6.3.7. 7 column volumes = 21 mL 8 M HCl
Step 6.3.8. 2.5 column volumes = 7.5 mL 8 M HNO_3
Step 6.3.10 7.5 column volumes = 22.5 mL 8 M HNO_3

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

7. Proper Waste Disposal Practices

7.1. Discard the solution from Steps 6.3.6 to 6.3.8 into the radioactive acid waste container.

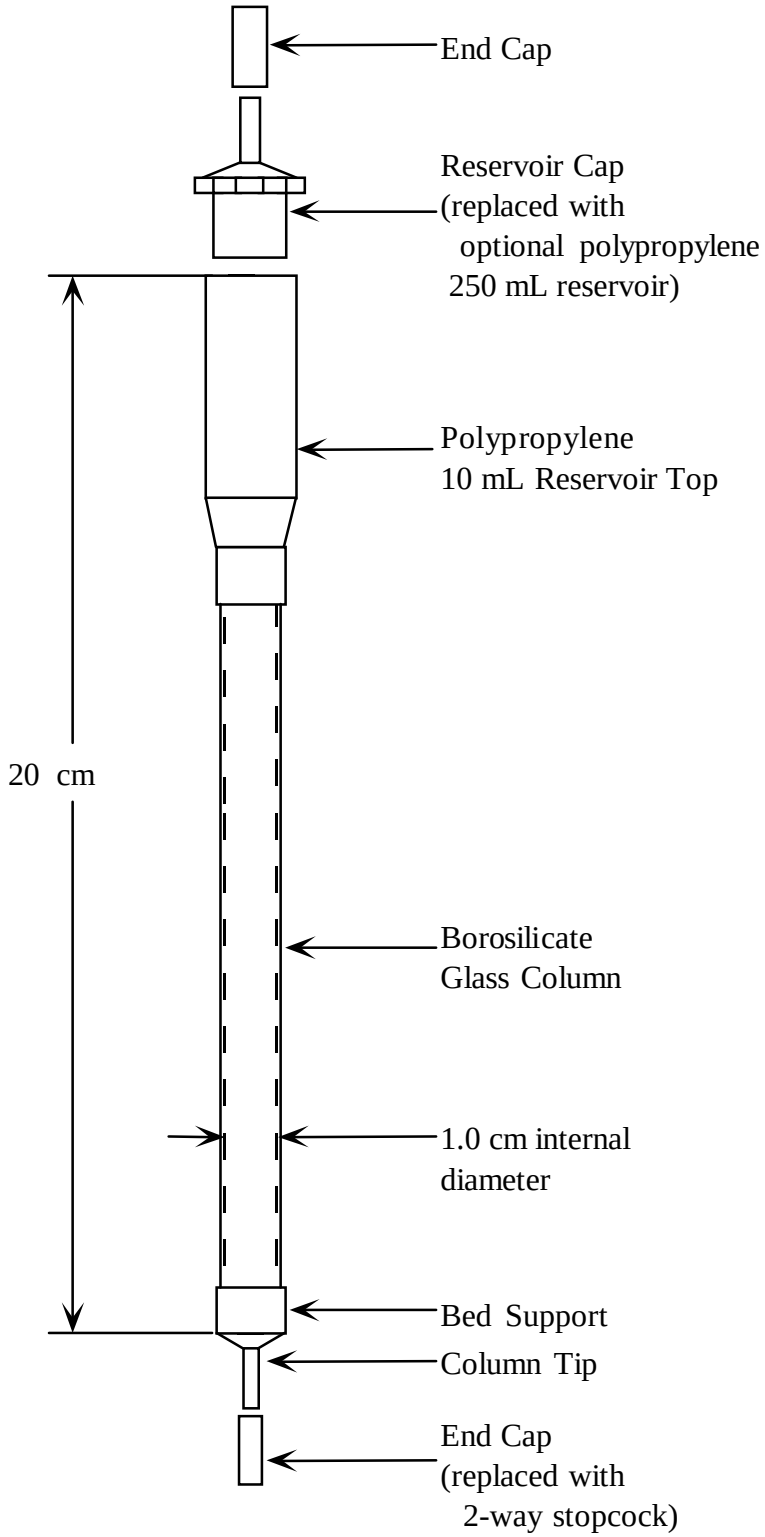

Fig. 1. Ion exchange column for uranium.