USTUR 220: SEPARATION AND PURIFICATION OF PLUTONIUM FROM AMERICIUM IN PRE-CONCENTRATED SAMPLES

Purpose	Separation and purification of plutonium from Americium in pre-concentrated samples	**Method Number**	USTUR 220
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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. Plutonium and americium (pre-concentrated from tissue samples in USTUR 150) are quantitatively separated by anion exchange.

1.2. The plutonium portion is prepared for electrodeposition (USTUR 510).

1.3. The americium portion is purified through further separation in procedure USTUR 310.

2. Minimum Detectable Activity (MDA)

Not applicable.

3. Accuracy and Precision

Not applicable.

4. Apparatus

4.1. Hot plates.

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

4.3. Hot plate thermometer with range to 200ºC.

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

4.5. Rack: to support ion-exchange columns.

4.6. Glass beads: 2 or 3 mm diameter.

4.7. Graduated cylinders.

4.8. Beakers: various sizes.

4.9. Metric ruler.

4.10. Wash bottles: 500 mL.

5. Reagents

5.1. 18 M Ω deionized water (D.I. water).

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

5.3. Nitric acid (8M). Dilute concentrated HNO_3 with an equal volume of D.I. water by adding the acid to the water.

5.4. Sodium nitrite (reagent-grade).

5.5. Bio-Rad anion exchange resin (AG1-X4, 100-200 mesh) chloride form. Make a slurry of half resin, half D.I. water in a wash bottle.

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

5.7. Hydrochloric acid (9M). Add 750 mL of concentrated HCl to 250 mL of D.I. water.

5.8. Hydrochloric acid (0.6 M). Add 50 mL of concentrated hydrochloric acid to 950 mL of D.I. water.

5.9. Hydroxylamine hydrochloride (reagent grade crystals).

5.10. Ammonium iodide (reagent grade crystals).

5.11. 0.6 M hydrochloric acid/0.05 M ammonium iodide. Dissolve 0.72 g of NH_4I in 0.6 M HCl. Dilute to 100 mL with 0.6 M HCl. Make fresh just prior to use.

6. Procedure

6.1. Sample preparation.

6.1.1 Sample aliquot is obtained from USTUR 150, step 6.4.15.

6.2 Sample dissolution.

6.2.1. Add 50 mL of 8 M HNO_3 to sample aliquot and cover with watchglass. Warm on 150ºC hotplate, if necessary, to dissolve sample. Remove samples from the hotplates.

6.2.2. Turn hotplate up to 300-350ºC (with samples off hotplate).

6.2.3. Add approximately 0.1 g sodium nitrite (tip of large spatula full) to each sample. Swirl sample, replace watchglass, and place on hotplate. Heat until just starting to boil.

NOTE: Solution should be maintained at a slight yellow color. Do not heat until clear! Once sodium nitrite has been added to the samples, the separation must be completed in a timely manner. Columns should not be left dry for longer than 20-30 minutes.

6.2.4. Remove sample from hotplate and bring to room temperature before proceeding with anion exchange.

6.3. Anion exchange separation.

6.3.1. Using a ruler, mark the ion exchange column 8 cm above the frit.

6.3.2. Fill the column with the slurry of AG1-X4 resin to a settled depth at the 8g cm mark.

6.3.3. Rinse all the resin down from the sides of the column using deionized water, then 8 M HNO_3. Once all of the acid has drained, add approximately 1 cm depth of glass beads on top of the resin.

6.3.4. Wash the column with 50 mL of 8 M HNO_3. Discard the wash in a hazardous waste acid container.

6.3.5. Place a clean 250 mL beaker labeled with sample number and “Am” to catch the americium portion of the sample.

6.3.6. Add cooled sample to column reservoir. Rinse beaker twice with approximately 5 mL 8 M HNO_3, adding the rinse to the column. Allow column to drain completely.

6.3.7. Rinse the column with 60 mL of 8 M HNO_3, collecting the column effluent in the beaker for americium analysis. Allow column to drain completely.

6.3.8. Remove americium beaker and bring to dryness on a hotplate set at 120°C. Cover with a watch glass or parafilm and store for purification by USTUR 310.

6.3.9. With a waste beaker under the column, rinse column with 30 mL of 9 M HCl. Allow column to drain completely. Discard effluent in a Radioactive waste container.

6.3.10. Place a clean 150 mL beaker labeled with sample number and "Pu" under column to catch plutonium portion of sample.

6.3.11. Cover tops of glass beads (in column) with hydroxylamine hydrochloride crystals.

6.3.12. Add 30 mL of 0.6 M HCl to the column and allow it to drain completely.

6.3.13. Add 20 mL of 0.6 M HCl/0.05 M NH_4I solution to the column and allow it to drain completely.

6.3.14. Add 20 mL of concentrated HNO_3 to plutonium beaker and bring solution to dryness on a hotplate set at 120ºC.

6.3.15. Continue with step 5.1 in USTUR 510 to prepare the plutonium portion for electrodeposition.

6.3.16. Rinse the column with 50 mL of D.I. water. Discard the rinse into a radioactive waste container. Place the used resin into a resin collection beaker.

7. Source Materials

7.1. "Isolation of Plutonium, Americium, and Uranium from Urine, by Precipitation, Ion Exchange, and Extraction," 4-16200-RHL-0037, Rocky Flats Plant.

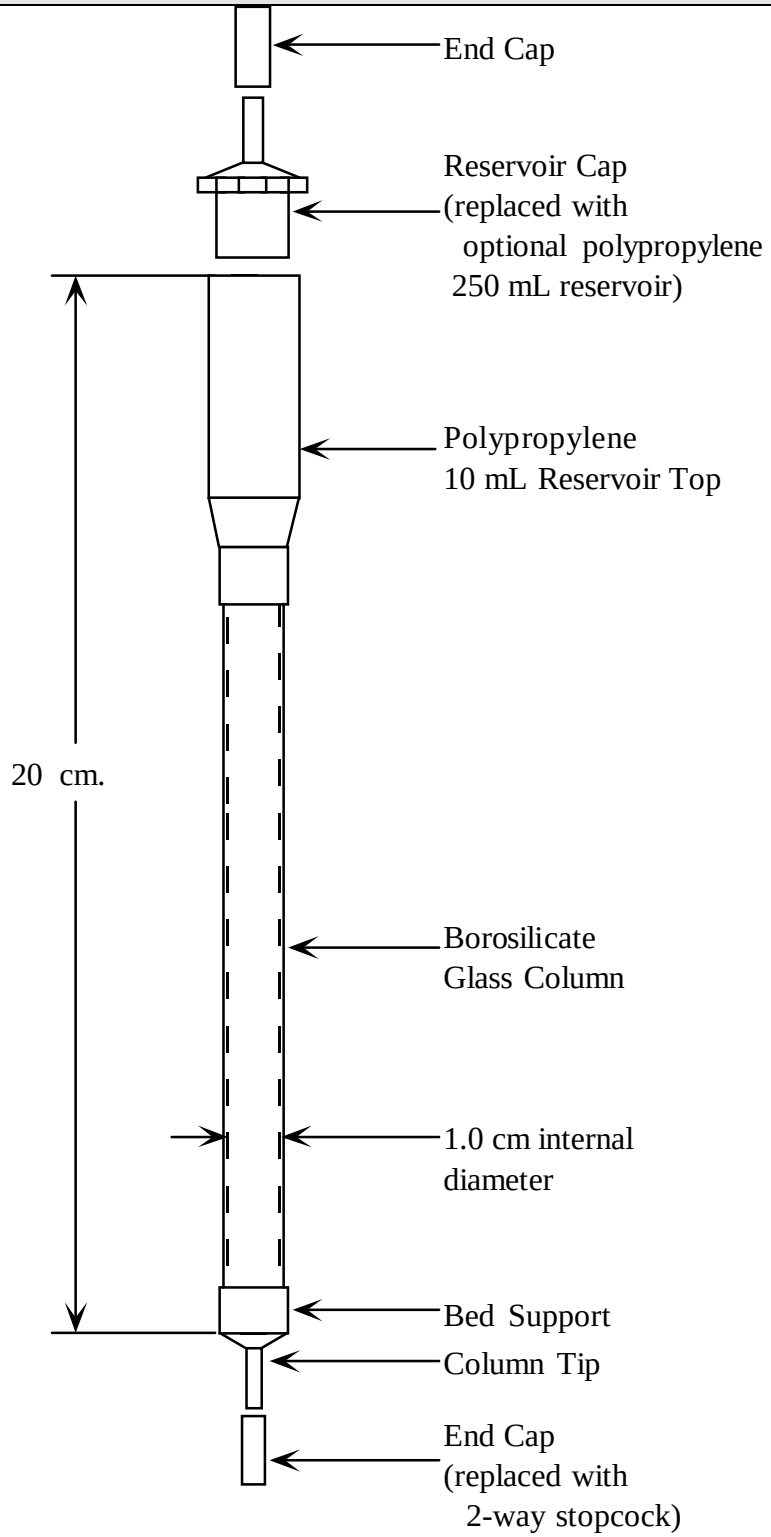

Figure 1. Ion Exchange Column