# USTUR 210: REMOVAL OF PLUTONIUM FROM SAMPLES TO BE ANALYZED FOR AMERICIUM ONLY

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| **Purpose** | Sample preparation for Am determination | **Method Number** | USTUR 210 |
| **Original Date** | 10/1/96 | **Author** | Dorothy Stuit |
| **Revision Number** | 0 | **Approved By** | Jim Elliston |
| **Revision Date** | 10/1/96 | **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. This procedure is for removal of plutonium from samples being analyzed only for americium. This ensures that $^{238}$Pu does not interfere with the $^{241}$Am peak in alpha spectrometry measurements.

1.2. An aliquot of the tissue sample solution is selected for analysis based on wet sample mass (or bone ash weight) and estimated sample alpha activity.

1.3. Both Americium-243 and Plutonium-242 tracers are added to the aliquot. Americium is separated from plutonium by anion exchange.

1.4. The americium portion is purified by continuing with procedure USTUR 300: Anion Exchange Isolation of Americium from Prepared Tissue Solutions.

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

1.6. Separation of Pu from Am is identified by absence of $^{242}$Pu during alpha spectrometry.

## 2. Minimum Detectable Activity (MDA)

Not applicable.

## 3. Accuracy and Precision

Not applicable.

## 4. Apparatus

4.1. Hot plates.

4.2. Magnetic stirring hot plates.

4.3. Hot plate thermometer (100 - 150°C).

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

4.5. 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.

4.6. Rack: to support ion-exchange columns.

4.7. Glass beads: 2 or 3 mm diameter.

4.8. Graduated cylinders.

4.9. Beakers: various sizes.

4.10. Metric ruler.

4.11. Stir bars: Teflon-coated.

4.12. Wash bottles: 50 mL.

## 5. Reagents

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

5.2. Nitric acid (8M). Add 500 mL concentrated $HNO_3$ to 400 mL of nanopure . Dilute to 1000 mL with nanopure water.

5.3. Sodium nitrite (reagent-grade).

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

5.5. Plutonium-242 tracer solution.

5.6. Americium-243 tracer solution.

**6. Procedure**

5.7. Sample preparation.

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

NOTE: Add both $^{243}$Am and $^{242}$Pu tracers (~10 dpm each).

6.2 Sample dissolution.

6.2.1. To the previously prepared sample, add 80 mL of 8 M $HNO_3$. Use a stirring hot plate with a stir bar to ensure complete dissolution of the sample. Cover the beaker with a watchglass while heating (100-130ºC).

6.3. Anion exchange separation.

6.3.1. Using a ruler, mark the ion exchange column 9 cm above the frit.

6.3.2. Fill the column with the slurry of AG1-X4 resin to a settled depth of 9 cm.

6.3.3. Rinse all of the resin down from the sides of the column using distilled 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 to prevent disturbance as reagents are added.

6.3.4. Wash the column with 45 mL of 8 M $HNO_3$, collecting the solution in a waste beaker. Discard this solution into a hazardous waste car boy.

6.3.5. While conditioning the column, add approximately 0.5 g of sodium nitrite to the sample solution.

6.3.6. Swirl the beaker, cover with a watchglass, and heat the sample quickly, just to a boil, with a minimum amount of sample volume reduction.

6.3.7. Cool the sample to room temperature.

6.3.8. Place a 250 mL collection beaker under the column to catch the americium portion of the sample.

6.3.9. Pour the sample onto the column.

6.3.10. When the solution has drained to the top of the resin bed, rinse the empty sample beaker with 25 mL of 8 M $HNO_3$, adding the rinse to the column.

6.3.11. Repeat step 6.3.10.

6.3.12. When the solution has reached the top of the resin bed, remove the americium collection beaker and replace it with a waste beaker. Evaporate the americium fraction to dryness on a hot plate (~120-150ºC).

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

6.3.13. Rinse the column with 50 mL nanopoue water. Discard the rinse into a radioactive acid waste container. The resin is placed into a resin collection beaker for disposal.

## 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.

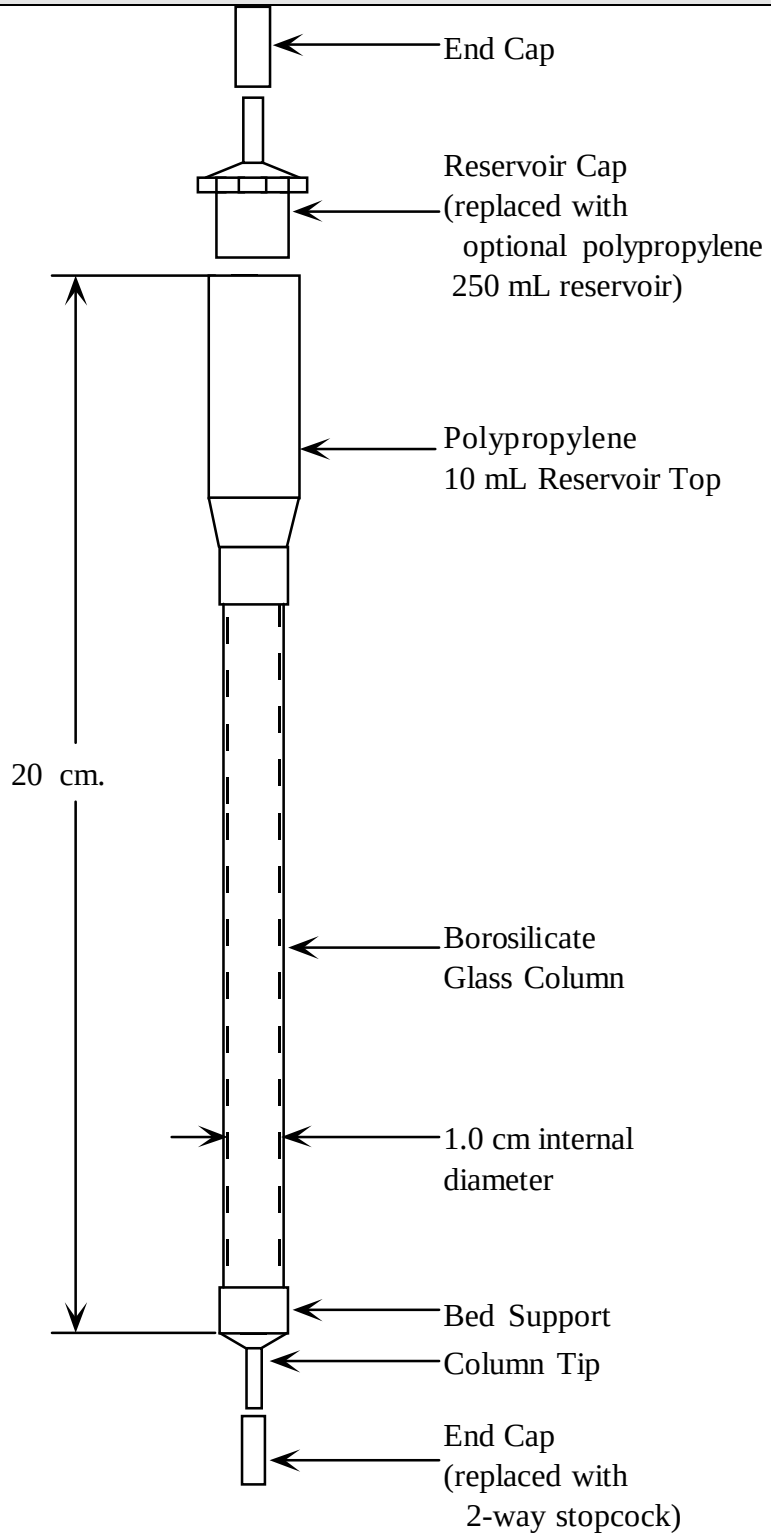


Figure 1. Ion Exchange Column