Rapid Determination of Actinides in Animal Tissue Samples

AN-1408-10

Summary of Method Uranium, Plutonium, and Americium-Curium are separated and concentrated from up to 200g tissue samples. Samples are digested with aqua regia, wet ashed with HNO_3-H_2O_2 and muffled over night at 550°C to destroy organic content. Actinides are separated from matrix impurities and potentially interfering radionuclides in the sample using stacked 2mL cartridges of Eichrom TEVA, TRU and DGA Resin. Actinides are measured via alpha spectrometry following cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Average chemical recoveries of Pu for 100-200g samples are 93-101%. Typical americium recoveries are 93-105%. Typical uranium recoveries are 82-96%. A single operator can complete the sample preparation for 12-24 samples, including 16 hours for muffling, in less than 24 hours.

Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Hydrofluoric Acid (49%) or Sodium Fluoride
Iron Carrier (50mg/mL Fe, as ferric nitrate)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrogen Peroxide (30%)
Deionized Water
Cerium Carrier (1mg/mL)
2M $Al(NO_3)_3$
Sodium nitrite
Sulfamic acid
Ascorbic acid
10% (w:w) $TiCl_3$
Denatured Ethanol

Equipment

Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Cartridge Reservoir, 20mL (Eichrom AR-200-RV20)
Inner Support Tubes-PE (Eichrom AR-1000-TUBE-PE)
Yellow Outer Tips (Eichrom AR-1000-OT)
Resolve Filters in Funnel (Eichrom RF-DF25-25PP01)
Muffle Furnace
Hot Plate
Analytical Balance
600mL Glass Beakers
Stainless Steel planchets with adhesive
Vacuum Pump
Alpha Spectrometry System
Heat Lamp

Figure 1. Sample Preparation

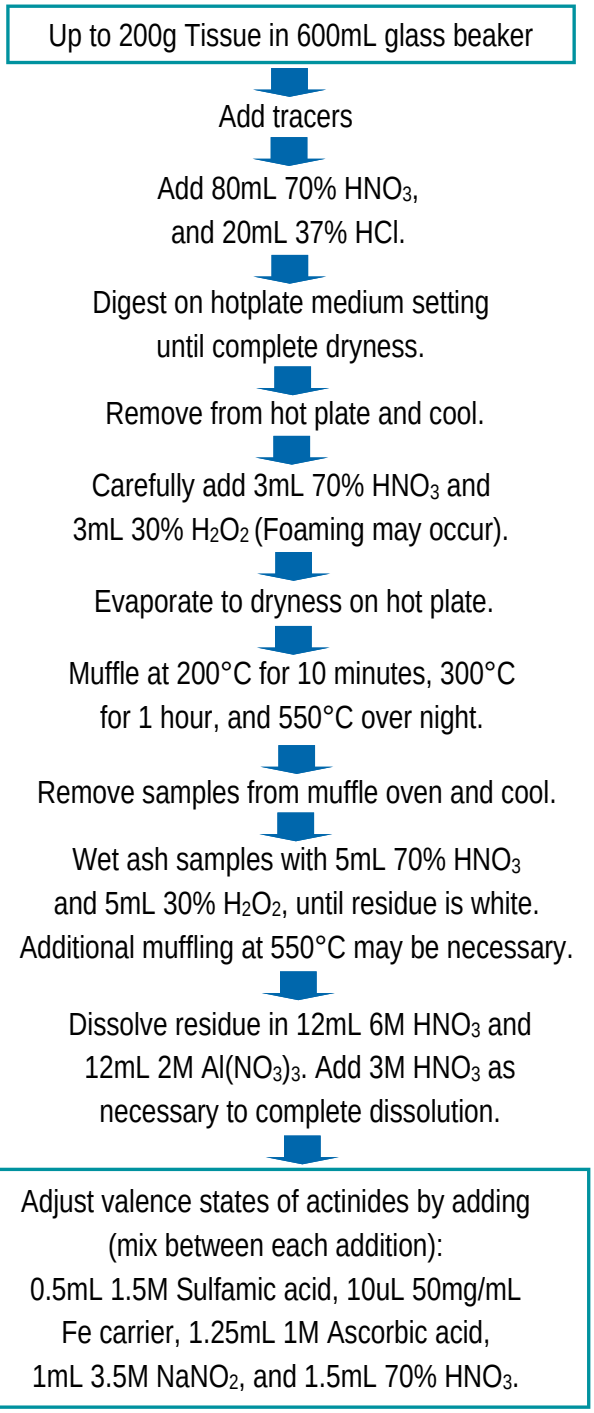
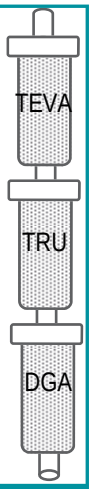


Figure 2. Actinide Separation on TEVA - TRU - DGA*

(1) Precondition stacked 2mL TEVA, TRU, DGA with 10mL 3M HNO_3.
(2) Load sample solution.
(3) Rinse sample tube with 5mL 6M HNO_3.** Add tube rinse to cartridges.
(4) Rinse cartridges with 10mL 3M HNO_3.
(5) Separate TEVA, TRU, and DGA cartridges.



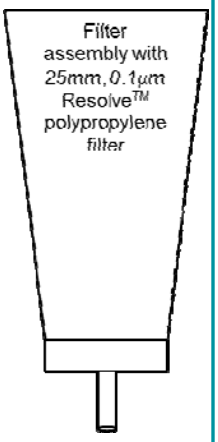
(6) Rinse TEVA cartridge with:
-10mL 3M HNO_3
-20mL 9M HCl(remove Th)
-5mL 3M HNO_3
(7) Strip Pu(Np) from TEVA with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

(8) Rinse DGA with 8mL 0.1M HNO_3.
(9) Place TRU cartridge above DGA .
(10) Strip Am/Cm from TRU onto DGA with 15mL 3M HCl.
(11) Separate TRU and DGA. Set TRU aside for U recovery.
(12) Rinse DGA with:
-5mL 3M HCl
-3mL 1M HNO_3
-15mL 0.05M HNO_3

(13) Strip Am and Cm from DGA with 10mL 0.25M HCl.

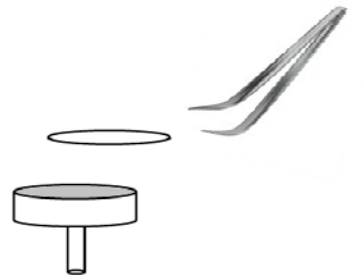
(14) Rinse TRU cartridge with 15mL 4M HCl-0.2M HF-0.002M $TiCl_3$.
(15) Strip U from TRU with 15mL of 0.1M ammonium bioxalate.
(16) Add 0.5mL 10% $TiCl_3$ to U samples, 0.5mL 30% H_2O_2 to Pu and 0.2mL 30% H_2O_2 to Am/Cm samples.

(17) Add 50-100ug Ce carrier to all samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.
(18) Set up Resolve® Filter Funnel on vacuum box.
(19) Wet filter with 3mL 80% ethanol followed by 3mL DI water.
(20) Filter sample.
(21) Rinse sample tube with 5mL DI water and add to

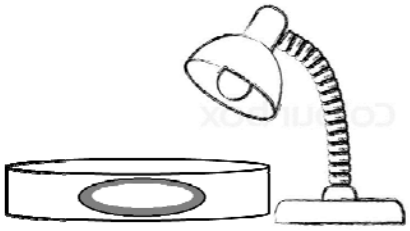


(22) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

(23) Draw vacuum until filter is dry.
(24) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(25) Dry filter under heat lamp for 3-5 minutes.
(26) Measure actinides by alpha spectrometry.



*Radiostrontium may also be measured by adding a 2mL + 1mL Sr Resin cartridge below DGA and following separation scheme in Eichrom application note AN-1407, "Rapid Determination of Sr in Animal Tissue Samples."
**Adding 50uL of 30% H_2O_2 to tube rinse can improve U recoveries and decontamination in Pu/Np samples.

Method Performance for 100-200g Tissue Samples

			% Tracer Recovery		
Sample	**mass, g**	**replicates**	**Pu-236**	**Am-243**	**U-232**
Beef	100	6	98.7 ± 5.7	97.1 ± 8.4	93.4 ± 4.7
Deer	100	59	99.3 ± 12	93.4 ± 10	90.4 ± 8.0
Fish-Bass	200	72	96.2 ± 14	102 ± 13	95.1 ± 8.1
Fish-Bream	100	57	96.6 ± 12	98.4 ± 7.7	91.1 ± 6.3
Fish-Catfish	200	69	98.3 ± 12	103.7 ± 7.6	89 ± 12
Hog	100	17	93 ± 20	96.4 ± 9.7	86 ± 15
Shelfish	100	5	101.3 ± 2.2	97.4 ± 7.1	81.7 ± 3.2

Reference Sherrod L. Maxwell, Donald M. Faison, "Rapid column extraction method for actinides and strontium in fish and other animal tissue samples," *J. Radioanal. Nucl. Chem., 275(3), 605-612* (2007).