Rapid Determination of Actinides in Emergency Air Filter Samples

Summary of Method

U, Pu, Np, Am and Cm are separated and concentrated from air filters. Samples are digested in Teflon beakers once with HNO₃-H₂O₂-HF and then several times with HNO₃-H₂O₂. After evaporating to dryness from HNO₃-H₃BO₃ to complex any residual fluoride, actinides are valence adjusted and separated on stacked 2mL cartridges of Eichrom TEVA and TRU resins. Actinides are measured by alpha spectrometry following CeF₃ microprecipitation onto Eichrom Resolve® Filters. Chemical yields of tracers averaged from 94±12% for ²⁴²Pu, 87±6% for ²⁴³Am, and 67±32% for ²³²U. Poor ²³²U recoveries in some samples were traced to insufficient mass of Ce carrier in the source preparation step. Recovery of ²³²U improved upon increasing to 100µg of Ce carrier. Measured values typically agreed to within 10% of reference values. Sample preparation for batches of 12 samples can be completed by a single operator in <8 hours. Alpha spectrometry count times will depend on detection limit and data quality objectives.

Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
²⁴²Pu (or ²³⁶Pu if meas. Np), ²⁴³Am and ²³²U tracers
Oxalic acid/Ammonium oxalate
Ce carrier (1mg/mL)
Deionized water 2M Al(NO₃)₃
10% (w:w) TiCl₃ HNO₃ (70%)
HCl (37%) HF (49%) or NaF
Boric acid H₂O₂ (30%)
NaNO₂ Denatured ethanol
Sulfamic Acid Ascorbic Acid

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)
50mL Centrifuge Tubes
Centrifuge
Heat Lamp
Hot Plate
Analytical Balance
250mL Teflon beakers
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Vacuum Pump

Figure 1. Sample Preparation

Air Filter + tracers in Teflon beaker.

Wet ash to dryness with 3mL 70% HNO₃, 3mL 30% H₂O₂ and 5mL 49% HF.

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Add 3mL 3M HNO₃ –0.25M H₃BO₃.
Evaporate to dryness.
Dissolve residue in 6mL 6M HNO₃ and 6mL 2M Al(NO₃)₃.
Cool samples to room temperature.

Fix valence states. Mix between each addition of:
0.5mL 1.5M sulfamic acid, 10uL 50mg/mL Fe, 1.5mL 1M ascorbic acid, 1mL 3.5M NaNO₂.
References


**89/90Sr can also be measured by placing a 2mL Sr Resin cartridge below DGA and following the separation scheme in application note AN-1434

**Adding 50uL of 30% H2O2 to the 6M HNO3 tube rinse can help improve U recoveries and decontamination in the Pu/Np fraction.