SEPERATION OF AMERICIUM FROM RARE EARTHS

(WITH VACUUM BOX SYSTEM)

1. SCOPE

1.1. This is a method for separation of americium (and curium) from rare earths prior to alpha spectrometry source preparation.

1.2. This method does not address all aspects of safety, quality control, calibration or instrument set-up. However, enough detail is given for a trained radiochemist to achieve accurate and precise results for the analysis of the analyte(s) from the appropriate matrix, when incorporating the appropriate agency or laboratory safety, quality and laboratory control standards.

2. SUMMARY OF METHOD

2.1. Americium is separated from rare earths using Eichrom TEVA resin prior to measurement by alpha spectrometry. Am and Cm are retained on TEVA from a thiocyanate solution, while rare earths pass through the resin. Am and Cm are then recovered in a matrix that will allow alpha spectrometry source preparation by rare earth fluoride micro precipitation (Eichrom SPA01) or electrodeposition (Eichrom SPA02).

3. SIGNIFICANCE OF USE

3.1. This method is used to improve the alpha spectrometry peak resolution for americium (and curium) samples containing significant amounts (>100μg) of rare earths.

4. INTERFERENCES

4.1. Rare earths present in samples may not be separated from americium or curium using standard separation chemistries on TRU or DGA Resins. Light rare earths, such as La and Ce, can be separated from Am/Cm using DGA resin and rinsing with 10-15 bed volumes of 3M HCl. However, heavier rare earths and yttrium and scandium will co-elute with Am/Cm. The presence of rare earths (>100μg) can lead to poor alpha spectrometry peak resolution through self-absorption in alpha spectrometry sources. This method will effectively separate Am and Cm from rare earths.

5. APPARATUS

- Analytical balance, 0.0001 g sensitivity
- Beakers, glass
- Centrifuge tubes, 50mL
- Fume hood
- Hotplate with stirrer
- Vacuum box (Eichrom Part: AR-24-BOX or AR-12-BOX)
- Vacuum box inner support tubes (Eichrom Part: AR-1000-TUBE-PE)
- Vacuum box yellow outer tips (Eichrom Part: AR-1000-OT)
- Vacuum pump - 115 V, 60 Hz Fisher Part: 01-092-25 (or equivalent) or house vacuum
- Vortex mixer
- Optional: vacuum box inner liner - For collection of load and rinse fractions, Eichrom Part: AR-12-LINER or AR-24-LINER

6. REAGENTS

Note: Analytical grade or ACS grade reagents are recommended.

Ammonium thiocyanate, NH_4SCN
Deionized water, all reagents are prepared with deionized water
Formic acid, HCOOH
Hydrochloric acid (37%), concentrated HCl
TEVA® resin, 2mL prepacked cartridge, 50-100μm, Eichrom Part TE-R50-S

6.1. *Ammonium thiocyanate (4M)* - formic acid (0.1M) - Dissolve 304g NH_4SCN in water, add 4.25mL formic acid and dilute to 1L with water. Prepare fresh prior to use.

6.2. *Ammonium thiocyanate (1.5M)* - formic acid (0.1M) - Dissolve 114g NH_4SCN in water, add 4.25mL formic acid and dilute to 1L with water. Prepare fresh prior to use.

6.3. *Hydrochloric acid (1M)* - Add 83mL concentrated HCl to 750mL of water and dilute to 1L with water.

7. PROCEDURE

7.1. Evaporate each americium/curium sample to dryness in a glass beaker.

7.2. Dissolve the residue in 5mL 4M NH_4SCN/0.1M formic acid

7.3. Place the inner tube rack (supplied with vacuum box system) into the vacuum box with the centrifuge tubes in the rack. Fit the lid to the vacuum system box. Alternatively, a vacuum box inner liner may be used.

7.4. Place yellow outer tips into all 12 or 24 openings in the lid of the vacuum box. Fit a white inner support tube into each yellow tip.

7.5. For each sample solution, fit a TEVA cartridge on to the inner support tube.

7.6. Add syringe barrels (funnels/reservoirs) to the top end of each TEVA cartridge.

7.7. Connect the vacuum pump to the box. Turn the vacuum pump on and ensure proper fitting of the lid.

Note: The unused openings on the vacuum box should be sealed. Vacuum manifold plugs can be used to plug unused white tips to achieve good seal during the separation. Alternatively, unused vacuum box holes can be sealed with scotch tape.

7.8. Add 5mL of 4M NH_4SCN/0.1M formic acid into each TEVA cartridge reservoir to condition the resin. Adjust the vacuum to achieve 1mL/min flow rate. Allow the solution to drain.

7.9. Transfer each solution from step 7.2. to the appropriate TEVA cartridge reservoir. Allow the load solution to completely pass through each cartridge at 1mL/min.

7.10. Add 3mL of 4M NH_4SCN/0.1M formic acid to rinse each sample beaker. Add the rinse solution to the appropriate TEVA cartridge reservoir. Allow the rinse to completely pass through each cartridge at 1mL/min.

7.11. Add 10mL of 1.5M NH_4SCN/0.1M formic acid to rinse each TEVA cartridge. Allow the rinse solution to completely pass through each cartridge. Disengage vacuum. Discard the solution collected to this point as waste.

Note: Ammonium thiocyanate is compatible with $KClO_3$ and $Pb(NO_3)_2$. Do not mix the waste solution with either of these chemicals.

7.12. Ensure that clean, labeled centrifuge tubes are below each TEVA cartridge.

7.13. Add 20mL 1M HCl to each TEVA cartridge reservoir to elute americium and curium. Engage vacuum. Allow the solution to completely pass through each cartridge at 1mL/min.

7.14. Prepare samples for measurement of americium and curium by electrodeposition (Eichrom SPA02) or rare earth fluoride micro precipitation (Eichrom SPA01).

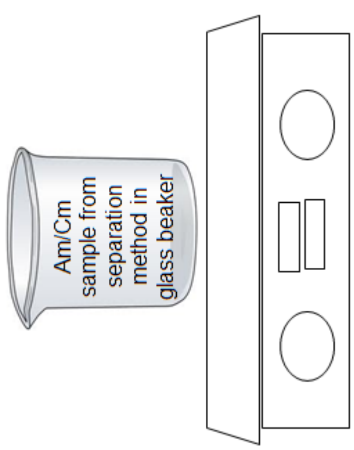
8. REFERENCES

1) Horwitz, E.P., et al. "Separation and Preconcentration of Actinides from Acidic Media by Extraction Chromatography," Analytica Chimica Acta, 281, 361-372 (1993).

2) Chiarizia, R., et al. "Am(III) and Eu(III) Extraction by Aliquat·336 and Benzyl Substituted Quaternary Ammonium Salts from Nitrate and Thiocyanate Solutions," Solvent Extraction and Ion Exchange, 13(4), 614-645 (1995).

1) Evaporate Am/Cm samples to dryness in glass beaker.

2) Dissolve residue in 5mL of 4M $(NH_4)SCN$-0.1M Formic acid.



3) Precondition 2mL TEVA with 5mL 4M $(NH_4)SCN$-0.1M Formic acid.

4) Load Sample on 2mL TEVA.

5) Rinse sample beaker with 3mL 5mL 4M (NH4)SCN-0.1M Formic acid. Add rinse to the appropriate 2mL TEVA.

6) Rinse 2mL TEVA with 5mL 1.5M (NH4)SCN-0.1M Formic acid.

7) Place a clean beaker or centrifuge tube below each 2mL TEVA.

8) Strip Am/Cm from 2mL TEVA with 20mL 1M HCl.