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EICHROM TECHNOLOGIES



Eichrom Method ACS07-VBS U in Soil

Outline

- Steps
 - Dry soil, pulverize, homogenize. Sample aliquot.
 - Add U-232 tracer
 - Ash sample in oven.
 - Digest HNO_3 , HCl , HF (HF needed for silicates).
 - Dissolve residue in $\text{HNO}_3\text{-Al}(\text{NO}_3)_3$.
 - UTEVA separation.
 - Prepare alpha spectrometry source.

Treatments for solid samples

1) Ash/burn to remove organic material.

- a) Important to remove organics prior to further treatment.
- b) Can slow reactions to dissolve inorganic material.
- c) Could catch fire if using $\text{HNO}_3/\text{H}_2\text{O}_2$ or other oxidizers.

2) Treat inorganic material

a) Leach

- Selectively dissolve select elements, leave bulk solids behind
- May not dissolve difficult samples or equilibrate tracer.

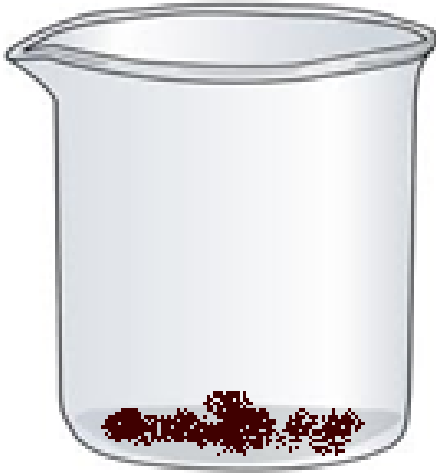
b) Acid/Wet digestion

- **More complete digestion. HF to dissolve silicates.**
- **May leave behind refractory or difficult residues.**

c) Fusion

- Complete sample dissolution including refractory components.

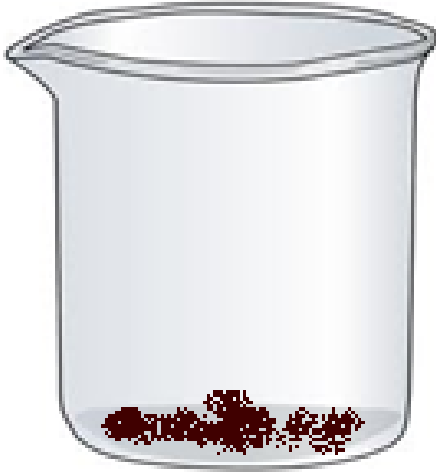
Treatments for solid samples



Soil in 200 mL
Glass Beaker. Dry,
weigh, then ash.

- 1) Weigh up to 2 grams of soil in 200 mL glass beaker.
- 2) Heat sample at 110C until dry.
- 3) Cool sample. Weigh. Record dry weight.
- 4) Ash sample overnight at 510C.
- 5) Transfer ashed soil to 125 mL Teflon beaker. Rinse glass beaker with 10 mL conc. HNO_3 . Add to Teflon beaker.
- 6) Add 10 mL conc. HNO_3 to Teflon beaker. Cover with watchglass. Heat to near boiling for 3 hours.

Treatments for solid samples



Soil in Teflon Beaker.
Glass beaker not
compatible with HF.

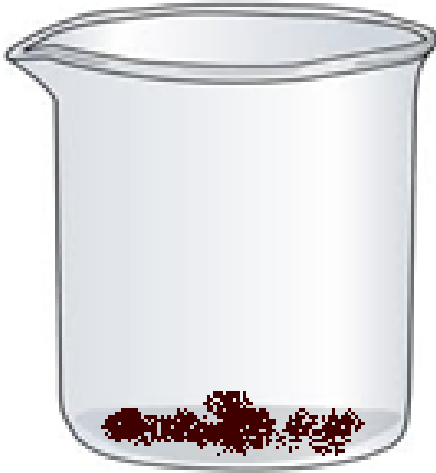
7) Transfer liquid and solids to 50 mL centrifuge tube. Rinse with DI water to complete transfer. Dilute to 50 mL with DI water.

8) Centrifuge. Decant liquid to clean 125 mL Teflon beaker. Set aside.

9) Transfer solids to original 125 mL Teflon beaker. Rinse centrifuge tube with 10 mL conc. HNO_3 to complete transfer. Add 10 mL conc. HNO_3 and 15 mL conc. HF.

Cover with Teflon lid. Digest on hotplate until solids dissolved. Add more HNO_3 -HF if needed.

Treatments for solid samples



Soil in Teflon Beaker.
Glass beaker not
compatible with HF.

- 10) Transfer liquid from dissolved solids to 125 mL Teflon beaker containing the liquid fraction. Evaporate to dryness.
- 11) Add 5 mL conc. HNO_3 . Evaporate to dryness.
- 12) Add 10 mL 3M HNO_3 + 1M $\text{Al}(\text{NO}_3)_3$.
- 13) Transfer to a 50 mL centrifuge tube. Rinse beaker with 5 mL 3M HNO_3 + 1M $\text{Al}(\text{NO}_3)_3$. Add rinse to centrifuge tube.

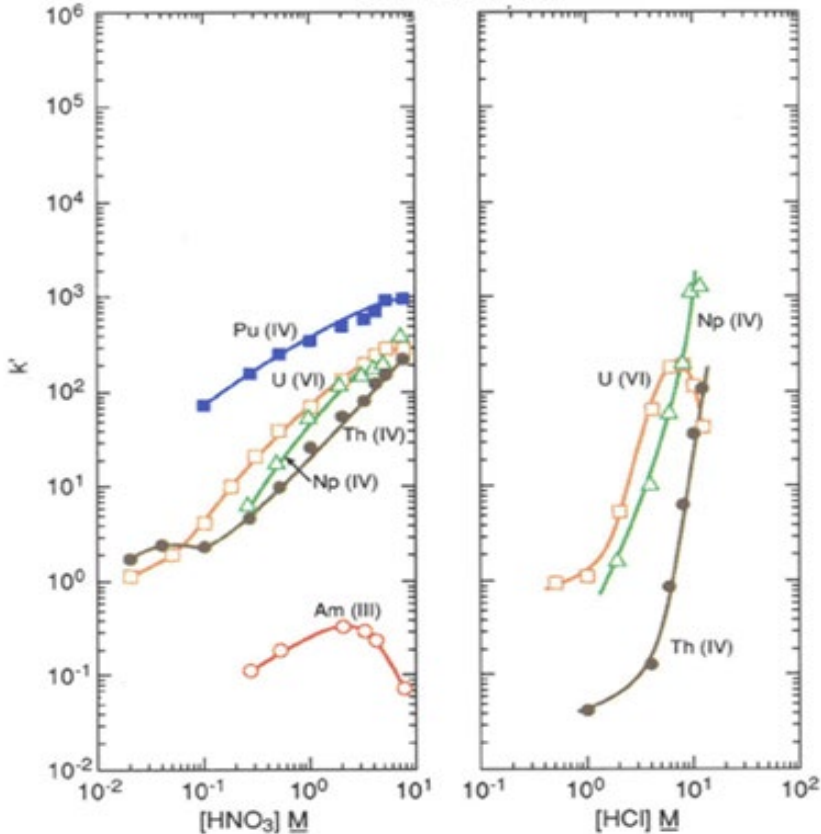
Optional: centrifuge to ensure any remaining solids are removed.

Note: Remaining silicates can cause gelling which will clog separation columns/cartridges.

UTEVA Resin

Acid dependency of k' for various ions at 23-25°C.

UTEVA Resin



14) Precondition UTEVA 5mL 3M HNO_3 .

15) Load sample onto UTEVA at 1-2 mL/min.
UTEVA retains U(VI).

16) Rinse sample tube with 5mL 3M HNO_3 .
Add rinse to UTEVA. Allow liquid to drain.

17) Rinse UTEVA with 5mL 3M HNO_3 .
Allow liquid to drain.

18) Rinse UTEVA with 15mL 8M HNO_3 .
(Additional Po removal. Po-210 overlaps with U-232)

19) Rinse UTEVA 15mL 9M HCl. (convert to HCl).

20) Rinse UTEVA with 20 mL 5M HCl-0.05M oxalic acid.
Removes Th(IV) and any traces of Pu(IV)/Np(IV).

21) Place a clean 50 mL centrifuge tube below each
UTEVA cartridge. Elute U with 15 mL 1M HCl.
(set aside for CeF_3).

Alpha Source Preparation (CeF₃)

Rapid method with adequate resolution for most analyses.

- No evaporations

Add 50-100 ug of Ce

Add TiCl₃ to U samples, U(IV) will carry on CeF₃

Add HF (or NH₄HF) to all samples

Alpha Source Preparation (CeF₃)

Typical Performance of CeF₃ Microprecipitation onto Eichrom Resolve Filters

Nuclide	μg Ce	Matrix	Yield	Resolution (FWHM)
²³⁰ Th	50	30 mL 4.5M HCl	>95%	20-30 keV
^{238/234} U	100	20 mL 1M HCl	>95%	30-40 keV
²³⁹ Pu	50	20 mL 0.1M HCl-0.05MHF-0.01MTiCl ₃	>95%	30-40 keV
²⁴¹ Am	50	15 mL 4M HCl	>95%	22-28 keV

1) Dilute samples as necessary and add Ce Carrier (See Table I).

2) **U Samples**, Add 0.5mL 10% $TiCl_3$

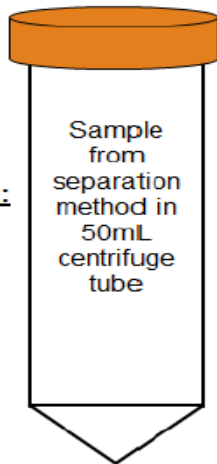
3) **Th, Np, Pu, Am/Cm samples requiring additional U decontamination:**
Add 50mL 30% H_2O_2 .

4) Swirl to mix.

5) Add 1-3.5mL conc. HF (see Table I).

6) Swirl to mix.

7) Wait 20-30 minutes before filtration.



8) Ensure tight fit of filter assembly.

9) Engage vacuum.

10) Wet filter with 3-5mL 80% ethanol.

11) Wet filter with 3-5mL DI water.

12) Add sample.

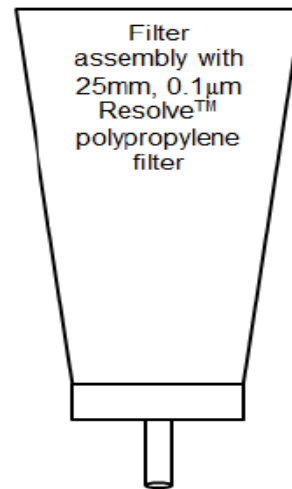
13) Rinse tube with 5mL DI water and add to filter assembly.

14) Allow all fluid to pass through filter.

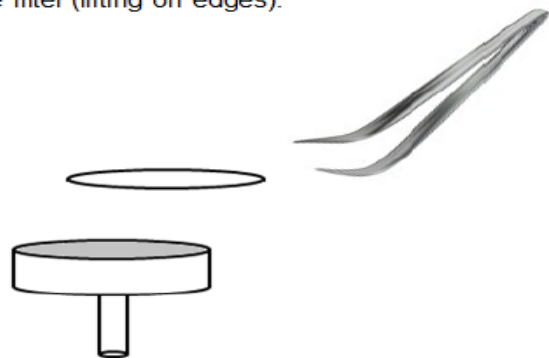
15) Rinse filter funnel with 3-5mL DI Water.

16) Rinse filter funnel with 2-3mL ethanol.

17) Filter until dry.



18) Remove filter (lifting on edges).

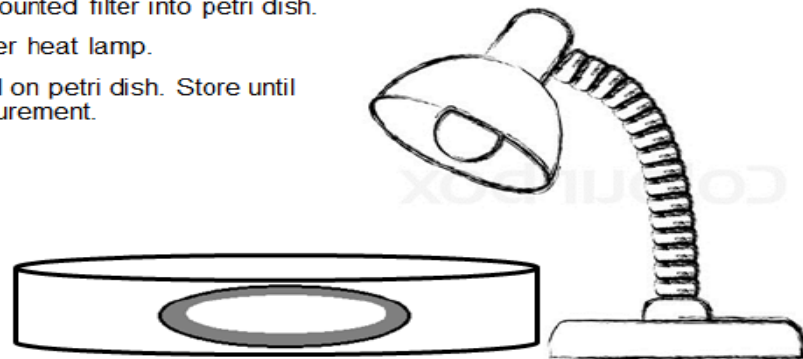


19) Mount filter to stainless steel planchet.

20) Place mounted filter into petri dish.

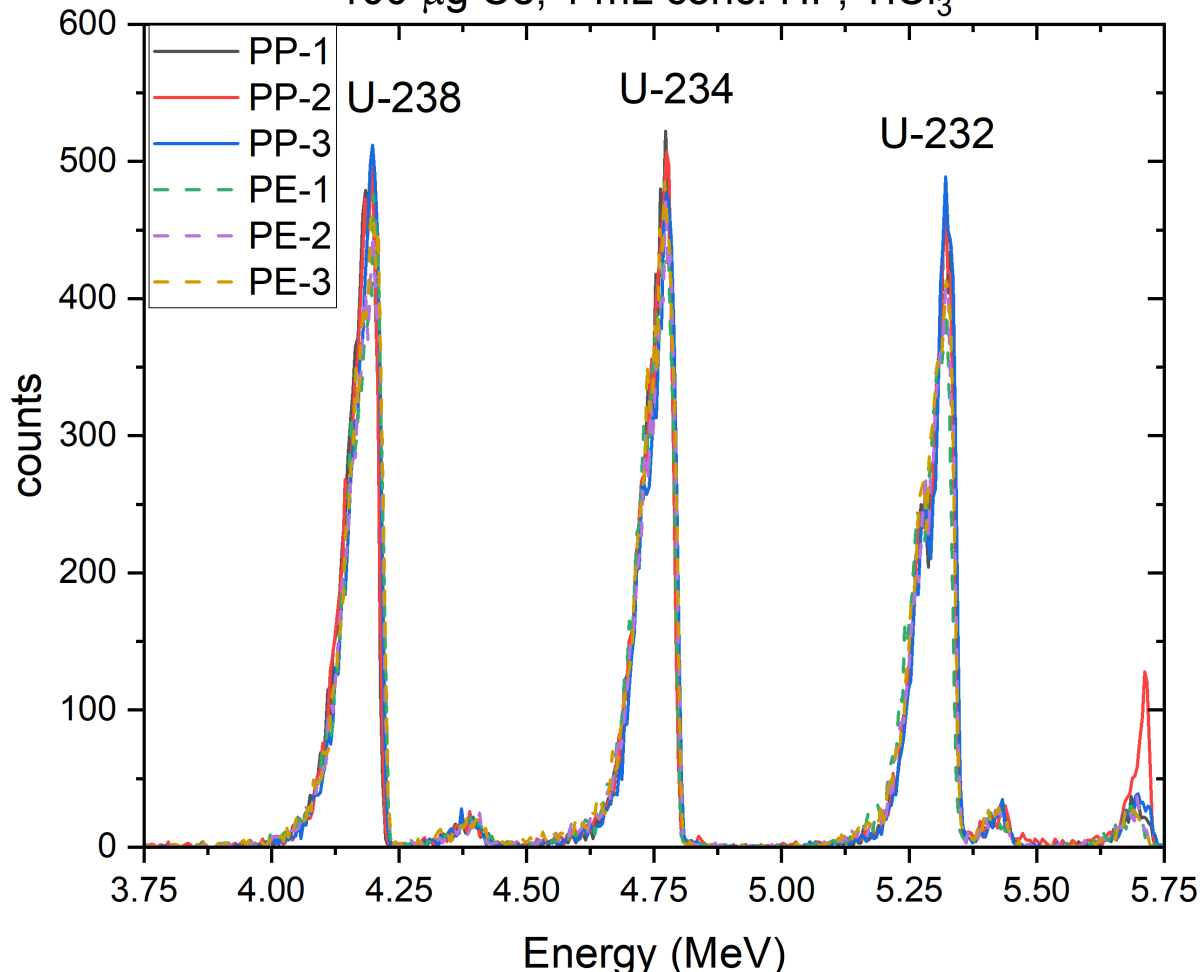
21) Dry under heat lamp.

22) Place lid on petri dish. Store until alpha measurement.



U-238, U-234, and U-232 in 15 mL 1 M HCl

100 μg Ce, 1 mL conc. HF, TiCl_3



Questions????

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