

Rapid Determination of ²²⁶Ra in 1g Soil Samples

Summary of Method ²²⁶Ra is separated from 1 gram samples of soil and measured by alpha spectrometry. Samples are fused with sodium hydroxide at 600°C. The fusion cake is dissolved in water, and radium is precipitated from samples with calcium carbonate. The calcium carbonate precipitate is dissolved in hydrochloric acid, and cation exchange chromatography is used to purify radium and barium from matrix ions. Barium is removed from samples using Eichrom Sr Resin. Eichrom DGA Resin is used to remove other alpha emitting nuclides from radium. Samples are prepared for alpha spectrometry by barium sulfate micro-precipitation onto Eichrom[®] Resolve Filters. Sample preparation, including alpha spectrometry source preparation, for batches of 12-24 samples can be completed by a single operator in as little as 6 hours. Yields are traced with ²²⁵Ra(²²⁹Th) by alpha spectrometry. At least 8 hours of ingrowth time for the alpha emitting ²¹⁷At daughter of ²²⁵Ra is required prior to measurement by alpha spectrometry.

Reagents

Cation Exchange Resin (Eichrom C8-B500-F-H)	
Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)	
DGA Resin, Normal 2mL Cartridges (Eichrom DN-R50-S)	
Nitric Acid (70%)	Hydrochloric Acid (37%)
Deionized Water	²²⁵ Ra(²²⁹ Th) Tracer
1.25M Ca(NO ₃) ₂	2M Na ₂ CO ₃
Barium Carrier (1mg/mL)	Isopropyl Alcohol
Ammonium Sulfate	Denatured Ethanol
Ascorbic Acid	Sodium Hydroxide
H ₂ O ₂ (30%)	

Equipment

Plastic Chromatography Column (Eichrom AC-50E-5M)
 Column Extension Funnel (Eichrom AC-20X-20M)
 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 Filter in Disposable Funnel (Eichrom RF-DF-25-25PP01)
 50mL and 250mL Centrifuge Tubes
 Centrifuge
 Stainless Steel Planchets with adhesive tape
 Hotplate
 Alpha Spectrometry System
 150mL Glass beakers
 Vacuum Pump
 250mL Zirconium Crucible w/ lid
 Muffle Furnace

Figure 1. Sample Preparation

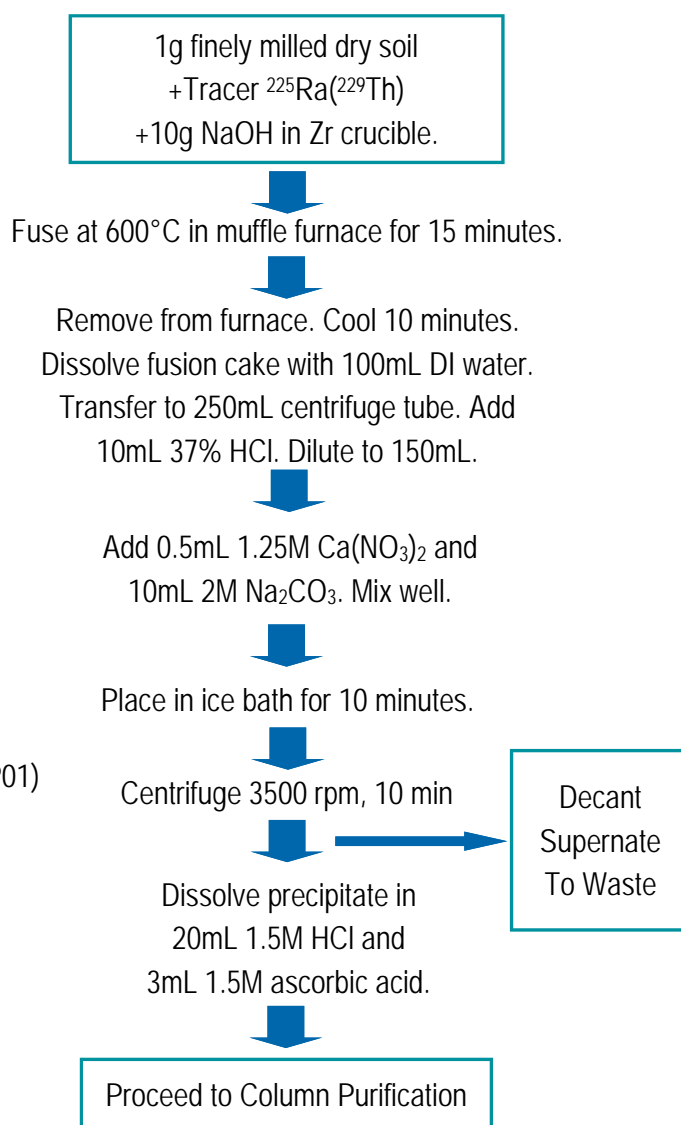



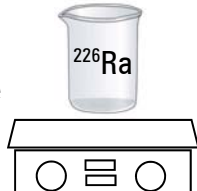
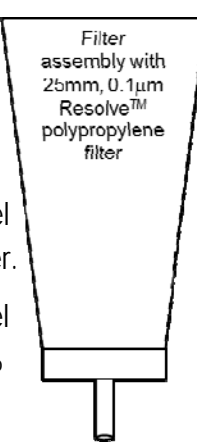
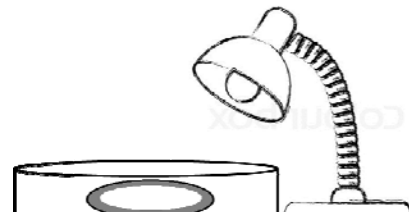


Figure 2. Column Purification and Alpha Source Preparation

<p>(1) Prewash 5.0g 50Wx8 200-400 mesh, cation exchange resin column: -10mL deionized water -20mL 6M HCl -10mL 0.5M HCl</p> <p>(2) Load Sample</p> <p>(3) Rinse 30mL 3M HCl</p> <p>(4) Strip Ra/Ba with 25mL 5M HNO₃.</p>	<p>(11) Add 50ug Ba carrier. Mix well.</p> <p>(12) Add 3g (NH₄)₂SO₄ and 5mL iso-propanol. Mix well.</p> <p>(13) Place in ice bath for 30 minutes.</p> <p>(14) Set up Resolve® Filter Funnel on vacuum box.</p> <p>(15) Wet filter with 3mL 80% ethanol followed by 3mL DI water.</p> <p>(16) Filter sample.</p> <p>(17) Rinse sample tube with 5mL DI water and add to filter.</p> <p>(18) Rinse filter funnel with 3mL DI water.</p> <p>(19) Rinse filter funnel with 1-2mL 100% ethanol.</p> <p>(20) Draw vacuum until filter is dry.</p>	<p>(21) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.</p> <p>(22) Dry filter under heat lamp for 3-5 minutes.</p> <p>(23) Measure ²²⁶Ra and ²²⁵Ra(²¹⁷At) by alpha spectrometry after >8 hours ²¹⁷At ingrowth.</p>
		
<p>(5) Add 2mL 30% H₂O₂. Evaporate to dryness.</p> <p>(6) Dissolve residue in 5mL 3M HNO₃.</p> <p>(7) Pass through 2mL Sr + DGA Resin Cartridges.</p> <p>(8) Rinse Sr + DGA with 6mL 3M HNO₃.</p> <p>(9) Evaporate (7) + (8) to dryness.</p> <p>(10) Dissolve residue in 10mL 1.5M HCl.</p>		
		

¹If using ¹³³Ba tracer, 3.0g of cation exchange resin and proportionally smaller rinse volumes may be used.

²If tracing with ²²⁹Th, a 20mL 1M HCl-1M H₃PO₄ rinse following the sample load can improve purity of final ²²⁶Ra fraction.

Method Performance ²²⁶ Ra in 1g Soil Samples				
Sample	²²⁵ Ra(²¹⁷ At)	²²⁶ Ra(mBq/g)	²²⁶ Ra(mBq/g)	% Bias
	% Yield*	Reference	Measured	
1	75.2	184.5	185.9	0.8
2	77.9	184.5	192.0	4.1
3	74.8	184.5	176.9	-4.1
4	73.3	184.5	184.7	0.1
AVG	75 ± 2	184.5	185 ± 6	0.3

*²²⁵Ra tracer is added as ²²⁹Th in equilibrium with its daughters and measured by its alpha emitting ²¹⁷At daughter (7.066MeV) after >8 hr ingrowth.

References

1) Sherrod L. Maxwell, Brian K. Culligan, "Rapid Determination of ²²⁶Ra in Environmental Samples," *J. Radioanal. Nucl. Chem.*, 293(1), 149-155 (2012).