AN-1422-10

Rapid Determination of ^{226}Ra in 5g Vegetation Samples

Summary of Method ^{226}Ra is separated from 5 gram samples of vegetation 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 separate 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 samples can be completed by a single operator in as little as 6 hours. Yields are traced with $^{225}Ra(^{229}Th)$ by alpha spectrometry. At least 8 hours of ingrowth time for the alpha emitting ^{217}At daughter of ^{225}Ra 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%)
$^{225}Ra(^{229}Th)$ Tracer	1.25M $Ca(NO_3)_2$
2M Na_2CO_3	Barium Carrier (1mg/mL)
Isopropyl Alcohol	Ammonium Sulfate
Denatured Ethanol	Ascorbic Acid
Sodium Hydroxide	Hydrogen Peroxide (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
Heat Lamp

Figure 1. Sample Preparation

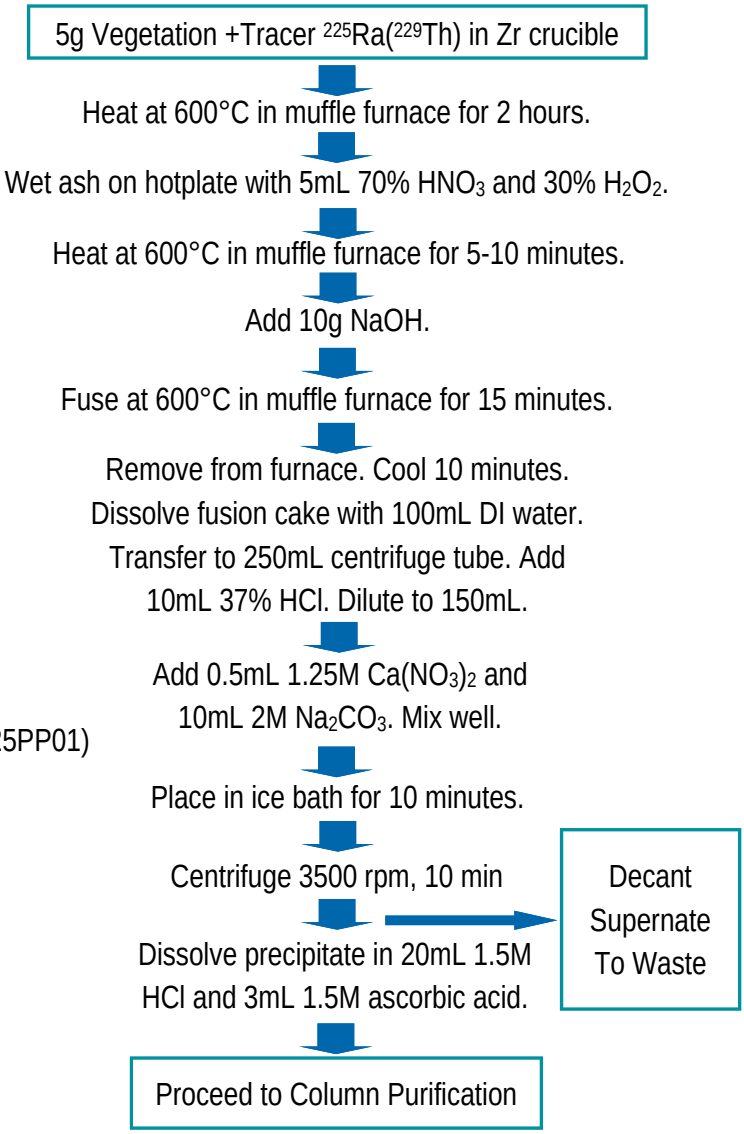


Figure 2. Column Purification and Alpha Source Preparation

(1) Prewash 5.0g 50Wx8 200-400 mesh, cation exchange resin column:
-10mL deionized water
-20mL 6M HCl
-10mL 0.5M HCl

(2) Load Sample

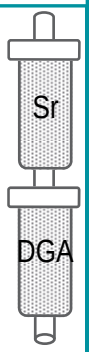
(3) Rinse 30mL 3M HCl

(4) Strip Ra/Ba with 25mL 5M HNO_3.

(5) Add 2mL 30% H_2O_2. Evaporate to dryness.

(6) Dissolve residue in 5mL 3M HNO_3.

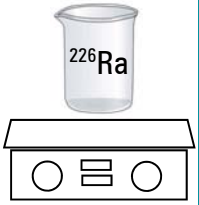
(7) Pass through 2mL Sr + DGA Resin Cartridges.



(8) Rinse Sr + DGA with 6mL 3M HNO_3.

(9) Evaporate (7) + (8) to dryness.

(10) Dissolve residue in 10mL 1.5M HCl.



(11) Add 50ug Ba carrier. Mix well.

(12) Add 3g $(NH_4)_2SO_4$ and 5mL iso-propanol. Mix well.

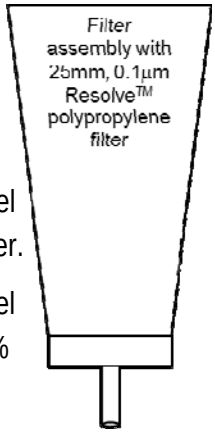
(13) Place in ice bath for 30 minutes.

(14) Set up Resolve® Filter Funnel on vacuum box.

(15) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(16) Filter sample.

(17) Rinse sample tube with 5mL DI water and add to filter.

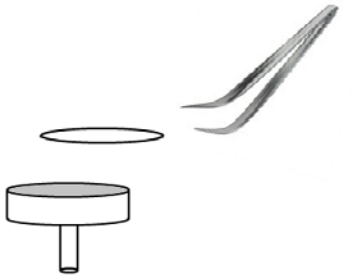


(18) Rinse filter funnel with 3mL DI water.

(19) Rinse filter funnel with 1-2mL 100% ethanol.

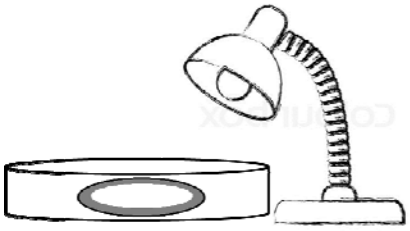
(20) Draw vacuum until filter is dry.

(21) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.



(22) Dry filter under heat lamp for 3-5 minutes.

(23) Measure ^{226}Ra and ^{225}Ra(^{217}At) by alpha spectrometry after >8 hours ^{217}At ingrowth.



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

[2]If tracing with ^{229}Th, a 20mL 1M HCl-1M H_3PO_4 rinse following the sample load can improve purity of final ^{226}Ra fraction.

Method Performance ^{226}Ra in 5g Vegetation Samples

Sample**	^{225}Ra(^{217}At) %Yield*	^{226}Ra(mBq/g) Reference	^{226}Ra(mBq/g) Measured	%Bias
1	91.5	73.8	70.8	-4.1
2	88.3	73.8	73.8	0.0
3	93.1	73.8	69.8	-5.4
4	82.2	73.8	68.5	-7.2
5	80.2	73.8	81.4	10.3
AVG	87 ± 6	73.8	73 ± 5	-1.1

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

**5 grams of blank hay matrix spiked with ^{226}Ra

References

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