AN-1418-10

Rapid Determination of ^{226}Ra in Water Samples

Summary of Method ^{226}Ra is separated from up to 1 liter water samples and measured by alpha spectrometry. 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. Eichrom DGA Resin is used to remove other alpha emitting nuclides from radium. Samples are prepared for radium measurement by alpha spectrometry using barium sulfate micro-precipitation method 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 3-4 hours. Yields can be traced with ^{133}Ba by gamma spectrometry or $^{225}Ra(^{229}Th)$ by alpha spectrometry. If tracing with ^{225}Ra, >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)
DGA Resin, Normal 2mL Cartridges (Eichrom DN-R50-S)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
^{133}Ba or $^{225}Ra(^{229}Th)$ Tracer
Nitric Acid (70%)
1.25M $Ca(NO_3)_2$
Barium Carrier (1mg/mL)
Ammonium Sulfate
Denatured Ethanol
H_2O_2 (30%)
Hydrochloric Acid (37%)
2M Na_2CO_3
Isopropyl Alcohol
Ascorbic Acid
Deionized Water

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
Gamma Spectrometry System (if ^{133}Ba tracer used)
150mL Glass beakers
Vacuum Pump
Heat Lamp

Figure 1. Sample Preparation

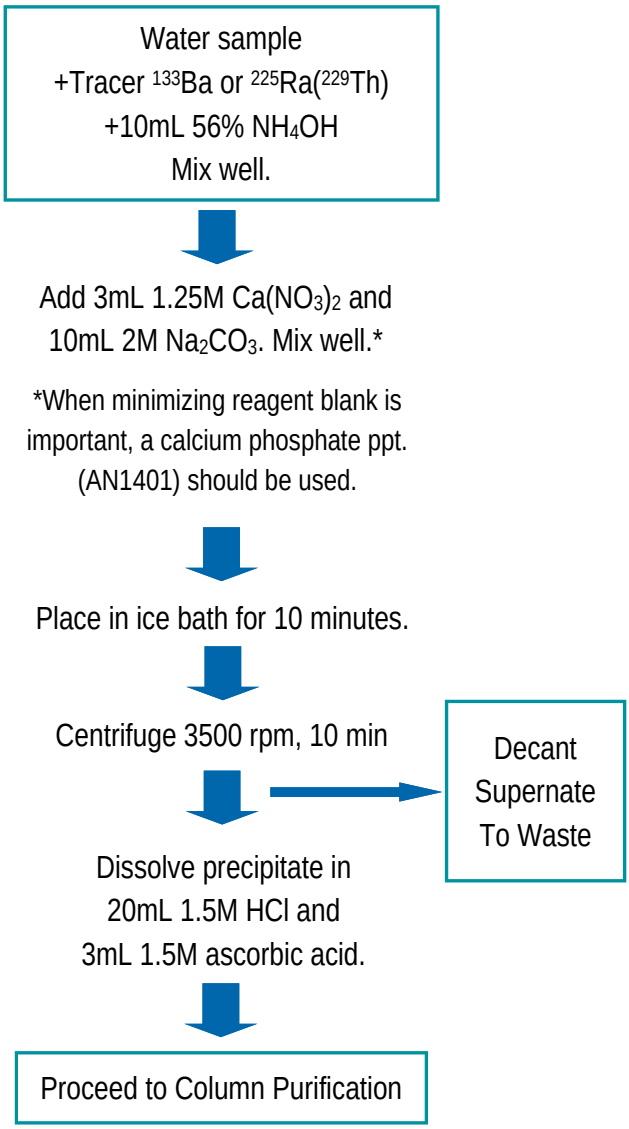


Figure 2. Column Purification and Alpha Source Preparation

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

(2) Load Sample[2]

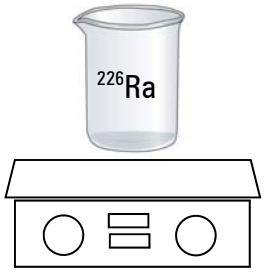
(3) Rinse 30mL 3M HCl

(4) Add 2mL DGA cartridge below cation exchange column.

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

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

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



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

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

(10) Place in ice bath for 30 minutes.

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

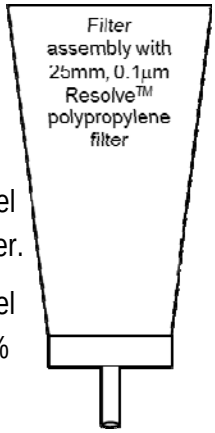
(12) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(13) Filter sample.

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

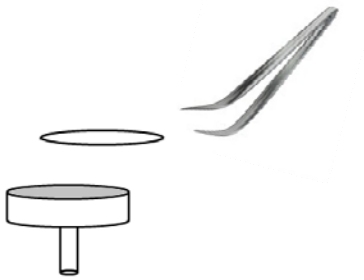
(15) Rinse filter funnel with 3mL DI water.

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



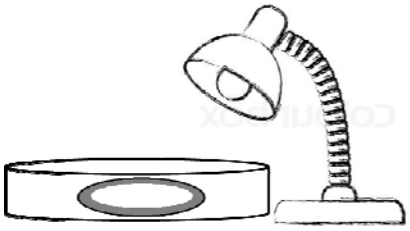
(17) Draw vacuum until filter is dry.

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



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

(20) Measure ^{226}Ra and $^{225}Ra(^{217}At)$ by alpha spectrometry after >8 hours ^{217}At ingrowth. (^{133}Ba by gamma, if necessary.)



[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 Water

Sample	$^{225}Ra(^{217}At)$ %Yield*	^{226}Ra(mBq/L) Reference	^{226}Ra(mBq/L) Measured	%Bias
1	84.8	73.8	69.6	-5.7
2	87.3	73.8	75.7	2.6
3	86.2	73.8	71.3	-3.4
4	98.7	73.8	66.9	-9.3
AVG	89 ± 6	73.8	70.9 ± 3.7	-3.9

*^{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 hr ingrowth.

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).