AN-1803-10

Rapid Determination of ^{226}Ra in Steel Samples

Summary of Method ^{226}Ra is isolated from 1 gram steel samples. Samples are digested with concentrated nitric, hydrochloric, and hydrofluoric acids. The digestate is evaporated to dryness, the residue dissolved in HNO_3/H_3BO_3 and calcium fluoride precipitate is used to concentrate the radium and remove matrix. Radium is separated from matrix impurities and potentially interfering radionuclides in the sample using cation exchange and DGA Resin. Radium is measured by alpha spectrometry following barium sulfate microprecipitation onto Eichrom Resolve Filters. The chemical recovery, determined by ^{133}Ba tracer, was 89–95%. Measured values of ^{226}Ra agreed to within 5% of reference values for 16 hour count times. The minimum detectable activity for ^{226}Ra in 1 g samples with 16 hour count times was 0.5 mBq/g. A single operator can prepare batches of 12 samples for the measurement of ^{226}Ra in less than 8 hours.

Reagents

Cation Exchange Resin (Eichrom C8-B500-F-H)
DGA Resin, Normal 2 mL Cartridges (Eichrom DN-R50-S)
Ammonium Hydroxide (Listed as 28% NH_3 or 56% NH_4OH)
Nitric Acid (70%)
Deionized Water
^{133}Ba Tracer
1.25M $Ca(NO_3)_2$
Barium Carrier (1 mg/mL)
Isopropyl Alcohol
Ammonium Sulfate
Denatured Ethanol
Hydrochloric Acid (37%)
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, 20 mL (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)
50 mL Centrifuge Tubes
Centrifuge
Hotplate
150 mL Glass beakers
Vacuum Pump
Heat Lamp
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Gamma Spectrometry System (^{133}Ba tracer)

Figure 1. Sample Preparation

Add 1 g steel sample to Teflon beaker.

↓

Add ^{133}Ba tracer, 5 mL 70% HNO_3,
20 mL 37% HCl, and 5 mL 49% HF.
Digest on Hotplate to dryness.

↓

Add 5 mL 70% HNO_3,
10 mL 37% HCl, and 1 mL 49% HF.
Digest on Hotplate to dryness.

↓

Add 5 mL 3M HNO_3-0.25M H_3BO_3
and 5 mL 37% HCl.
Digest on Hotplate to dryness.

↓

Add 10 mL 0.25M HCl. Warm to dissolve.
Transfer to 50 mL centrifuge tube.
Repeat 2 additional times.

↓

Add 2 mL 1.25M $Ca(NO_3)_2$ and 6 mL 49% HF.

↓

Mix Well. Allow to sit 20 min.
Centrifuge. Discard supernate.

↓

Dissolve precipitate in 10 mL 1M HCl
-0.25M H_3BO_3 and 10 mL 1M HCl.
Mix. Warm if necessary. Centrifuge.
Check for solids.

↓

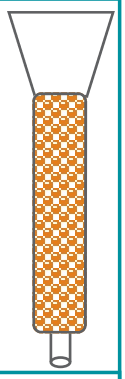
Continue to Ra Separation.

Figure 2. Column Purification and Alpha Source Preparation

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

(2) Load Sample.

(3) Rinse 30 mL 3M HCl.

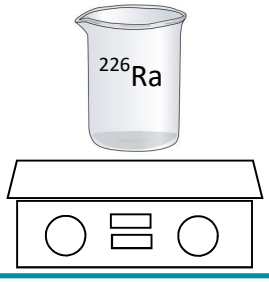


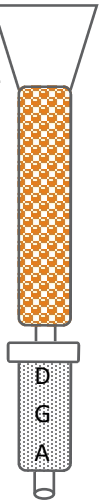
(4) Add 2 mL DGA cartridge below cation exchange column.

(5) Strip Ra/Ba with 35 mL 5M HNO_3.

(6) Add 3 mL 30% H_2O_2. Evaporate to dryness.

(7) Dissolve residue in 10 mL 1.5M HCl. Transfer to 50 mL c-tube with 2 x 7 mL 1.5M HCl





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

(9) Add 3 g $(NH_4)_2SO_4$ and 5mL isopropanol. Mix well.

(10) Place in ice bath for 30 minutes.

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

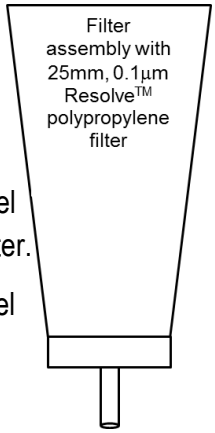
(12) Wet filter with 3 mL 80% ethanol followed by 3 mL DI water.

(13) Filter sample.

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

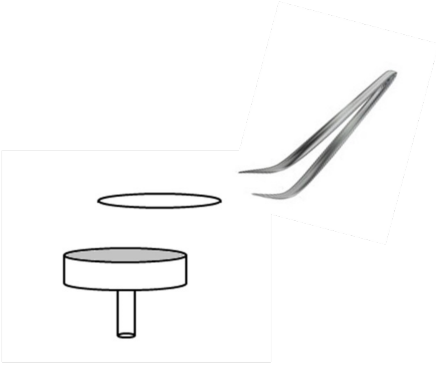
(15) Rinse filter funnel with 3 mL DI water.

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



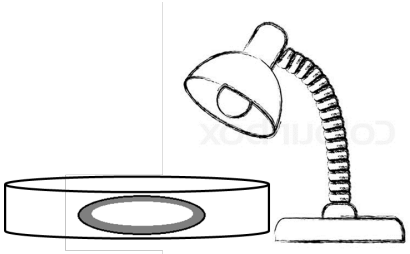
(17) Draw vacuum until filter is dry.

(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 by alpha spectrometry and ^{133}Ba by gamma spectrometry.



Method Performance for 1 gram Steel Samples

Sample replicates	Reference (mBq/sample)	Measured (mBq/sample)	Average % Diff.	133Ba tracer % Yield
5	36.8	36.5 ± 0.8	1.9	95.4 ± 5.9
5	73.7	74.9 ± 3.1	3.7	88.8 ± 1.8
5	184	183 ± 5	1.9	90 ± 13

References

1) Sherrod L. Maxwell, Brian K. Culligan, Robin C. Utsey and Daniel R. McAlister, "Rapid Method to Determine ^{226}Ra in Steel Samples," *J. Radioanal. Nucl. Chem., 314*(2), 1417-1423 (2017).