

Rapid Determination of $^{89/90}\text{Sr}$ in Limestone and Marble

Summary of Method Strontium is separated and concentrated from 1.5 gram samples of limestone or marble. Samples are finely ground and fused in a zirconium crucible for 15 minutes at 600°C with 15 grams of sodium hydroxide. The fusion cake is dissolved in water, and strontium is concentrated and separated from the matrix using a calcium phosphate precipitate enhanced with iron. A secondary precipitation with calcium fluoride removes additional matrix (including silicates) and decreases the volume of precipitate. The calcium fluoride precipitate is dissolved with nitric acid-boric acid-aluminum nitrate to form the load solution. Strontium is separated from remaining matrix and potentially interfering radionuclides using stacked 2mL + 1mL Sr Resin cartridges. Radiostrontium is measured by gas flow proportional counting or liquid scintillation counting. Chemical yields of strontium are determined by gravimetric yield or by ICP-AES. Batches of 12-24 samples can be prepared for analysis in less than 8 hours. Simultaneous separation of actinides can be achieved by using the separation method in AN-1603.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
 Sr Resin, 1mL Cartridges (Eichrom SR1ML-R50-S)
 Strontium Carrier (10mg/mL)
 Iron Carrier (50mg/mL Fe, as ferric nitrate)
 ^{90}Sr standard
 Nitric Acid (70%)
 Deionized Water
 3.2M $(\text{NH}_4)_2\text{HPO}_4$
 Oxalic acid
 Sodium Hydroxide

HF(49%)
 Hydrochloric Acid (37%)
 1.25M $\text{Ca}(\text{NO}_3)_2$
 2M $\text{Al}(\text{NO}_3)_3$
 Boric acid

Equipment

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)
 50mL and 250mL Centrifuge Tubes
 Cupped Stainless Steel Planchets (~5mL volume)
 250mL Zirconium crucibles with zirconium lids
 Centrifuge
 Muffle Furnace
 Analytical Balance

Gas Flow Proportional Counter
 Hot Plate/Heat Lamp
 Vacuum Pump

Figure 1. Sample Preparation

1.5g finely ground sample in zirconium crucible

Add 4mg Sr carrier.

Heat samples to dryness on hot plate.

Add 15g of NaOH.

Cover crucibles with zirconium lid and place in muffle furnace at 600°C for 15-20 minutes.

Carefully remove samples from furnace and cool in fume hood.

Add 25-50mL of water and heat on hot plate to dissolve fusion cake.

Transfer to a 250mL centrifuge tube. Rinse crucible with water. Dilute to 160mL with water.

Add 50mg Fe Mix.

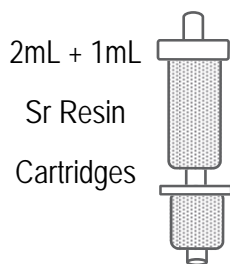
Add 8.5mL 3.2M $(\text{NH}_4)_2\text{HPO}_4$. Mix.
 Centrifuge 10min. Decant supernate.

Dissolve precipitate in 80mL 1.5M HCl. Dilute to 170mL.
 Add 15mL 49%HF. Mix. Cool in ice bath 10min.
 Centrifuge 10min. Decant supernate.

Dissolve precipitate in 7mL 3M HNO_3 -0.25M Boric acid, 7.5mL 7M HNO_3 , and 7mL 2M $\text{Al}(\text{NO}_3)_3$. Warm as needed.

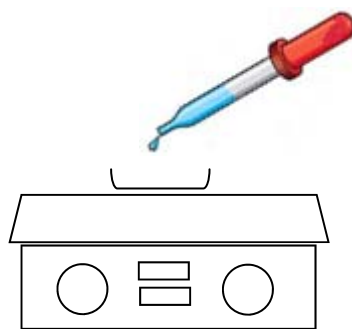
Figure 2. Strontium Resin Separation (Optional ⁹⁰Y Ingrowth)*

- (1) Precondition Sr Resin with 10mL 8M HNO₃.
- (2) Load sample at 1-2mL/min.
- (3) Rinse sample tube with 5mL 8M HNO₃.
- (4) Add tube rinse to Sr Resin. Elute at 1-2mL/min.
- (5) Rinse Sr Resin sequentially with:
 - 10 mL 8M HNO₃
 - 10mL 3M HNO₃ - 0.05 oxalic acid
 - 10mL 8M HNO₃
- (6) Dispose of (1) to (5) as waste.
- (7) Strip Sr with 20mL 0.05M HNO₃ at 1mL/min.



Gas Flow Proportional Counting:**

- (8) Evaporate samples to dryness on tared cupped stainless steel planchets.
- (9) Rinse Sr sample vials with 2mL 0.05M HNO₃. Transfer vial rinse to planchets. Evaporate to dryness.



- (10) Weigh planchets on an analytical balance to determine gravimetric yield of stable Sr(NO₃)₂.

- (11) Measure radiostrontium in samples on low background gas flow proportional counter.

** (Options for ^{89/90}Sr Discrimination)

- (a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ⁸⁹Sr can be measured by Cerenkov counting (no LSC cocktail). ^{89/90}Sr may then be measured after adding liquid scintillation cocktail.
- (b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO₃ after >7 days of ⁹⁰Y ingrowth. ^{89/90}Sr can be removed on Sr Resin. ⁹⁰Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.
- (c) Sr fraction from step (10) can be dissolved in 10mL 8M HNO₃ after >7 days of ⁹⁰Y ingrowth. ⁹⁰Y can be removed on DGA Resin. ^{89/90}Sr will elute in DGA Resin load. Additional rinsing will remove remaining Sr. ⁹⁰Y can be eluted in 0.1M HCl and counted by gas flow proportional counting or liquid scintillation (Cerenkov).

*Actinides may also be measured by adding 2mL TEVA, TRU and DGA Resin cartridges above Sr Resin and following separation scheme in Eichrom Application note AN-1603.

**Additional discussion of ^{89/90}Sr separation and measurement options can be found in Eichrom Application Note AN-1624-10.

Method Performance

Sample	% Sr tracer recovery	⁹⁰ Sr Bq/g reference	⁹⁰ Sr Bq/g measured	% bias
1	84.1	1.415	1.41	-0.1
2	84.8	1.415	1.42	0.4
3	84.8	1.415	1.38	-2.7
AVG	84.6 ± 0.4		1.40 ± 0.02	

References

- 1) Maxwell, Culligan, Hutchinson, Utsey, Sudowe, McAlister, "Rapid Method to Determine Actinides and Sr-89/90 in Limestone and Marble Samples," *J. Radioanal. Nucl. Chem.* accepted (2016). DOI 10.1007/s10967-016-4783-8