

Rapid Determination of $^{89/90}\text{Sr}$ in 5g Concrete Samples

Summary of Method Strontium is separated and concentrated from 5 gram concrete samples. Samples are finely ground and fused in a zirconium crucible for 30 minutes at 600°C with 30 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. Analytes are separated from remaining matrix and potentially interfering radionuclides using two stacked 2mL 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. For aged samples, where the shorter lived ^{89}Sr ($t_{1/2} = 50.55$ days) is unlikely to be present, ^{90}Sr can be determined from the direct separation of its ^{90}Y daughter from up to 10g concrete samples, using Eichrom Application Note AN-1606.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)

Strontium Carrier (10mg/mL)

Iron Carrier (50mg/mL Fe, as ferric nitrate)

^{90}Sr standard

HF(49%)

30% H_2O_2

Nitric Acid (70%)

Hydrochloric Acid (37%)

Deionized Water

1.25M $\text{Ca}(\text{NO}_3)_2$

3.2M $(\text{NH}_4)_2\text{HPO}_4$

2M $\text{Al}(\text{NO}_3)_3$

Oxalic acid

Boric acid

Sodium Hydroxide

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 500mL Centrifuge Tubes

Cupped Stainless Steel Planchets (~5mL volume)

250mL Zirconium crucibles with zirconium lids

Centrifuge

Gas Flow Proportional Counter

Muffle Furnace

Hot Plate/Heat Lamp

Analytical Balance

Vacuum Pump

Figure 1. Sample Preparation

5g finely ground sample in zirconium crucible

Add 6mg Sr carrier.

Heat samples to dryness on hot plate.

Add 30g of NaOH.

Cover crucibles with zirconium lid and place in muffle furnace at 600°C for 30 minutes.

Carefully remove samples from furnace and cool in fume hood.

Add 50-100mL of water and heat on hot plate to dissolve fusion cake.

Transfer to a 500mL centrifuge tube. Rinse crucible with water. Dilute to 450mL with water.

Cool to room temp. Add 50mg Fe. Mix.

Add 8.5mL 3.2M $(\text{NH}_4)_2\text{HPO}_4$. Mix.

Centrifuge 10min. Decant supernate.

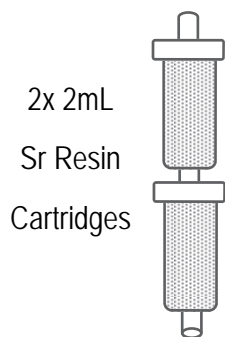
Dissolve precipitate in 200mL 1.5M HCl. Add 50mL 0.01M HCl and 15mL 49%HF. Mix. Cool in ice bath 10min.

Centrifuge 10min. Decant supernate.

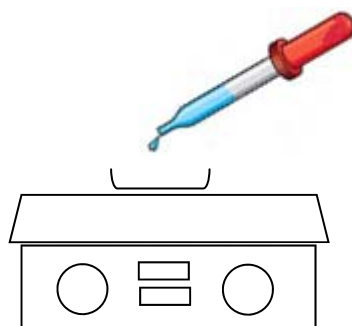
Dissolve precipitate in 7mL 3M HNO_3 -0.25M Boric acid, 7mL conc. HNO_3 , 7mL 8M HNO_3 and 7mL 2M $\text{Al}(\text{NO}_3)_3$. Warm as needed.

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

- (1) Precondition 2x2mL 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:
 - 20 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).

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

Method Performance (5g gram Concrete, Sr Resin Method)

Sample	% Sr tracer recovery	⁹⁰ Sr Bq/g reference	⁹⁰ Sr Bq/g measured	% bias
1	78.5	1.416	1.51	6.6
2	77.8	1.416	1.35	-4.6
3	80.5	1.416	1.42	0.2
4	62.2	1.416	1.49	5.2
AVG	75 ± 8		1.44 ± 0.07	

References

- 1) Maxwell, Culligan, Hutchinson, Utsey, Sudowe, McAlister, "Rapid Method to Determine 89/90Sr in Large Concrete Samples," *J. Radioanal. Nucl. Chem.* accepted (2016). DOI 10.1007/s10967-016-4787-4