Rapid Determination of $^{89/90}$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
- HF (49%)
- Hydrochloric Acid (37%)
- 1.25M Ca(NO$_3$)$_2$
- 2M Al(NO$_3$)$_3$
- Oxalic acid
- Boric acid
- Sodium Hydroxide
- 3.2M (NH$_4$)$_2$HPO$_4$
- Deionized Water
- 1.25M Ca(NO$_3$)$_2$
- 2M Al(NO$_3$)$_3$
- Nitric Acid (70%)
- Hydrochloric Acid (37%)
- 3.2M (NH$_4$)$_2$HPO$_4$
- Oxalic acid
- 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
- Gas Flow Proportional Counter
- Muffle Furnace
- Hot Plate/Heat Lamp
- Analytical Balance
- Vacuum Pump

**Figure 1. Sample Preparation**

1. 1.5g finely ground sample in zirconium crucible
2. Add 4mg Sr carrier.
3. Heat samples to dryness on hot plate.
4. Add 15g of NaOH.
5. Cover crucibles with zirconium lid and place in muffle furnace at 600°C for 15-20 minutes.
6. Carefully remove samples from furnace and cool in fume hood.
7. Add 25-50mL of water and heat on hot plate to dissolve fusion cake.
8. Transfer to a 250mL centrifuge tube. Rinse crucible with water. Dilute to 160mL with water.
9. Add 50mg Fe Mix.
10. Add 8.5mL 3.2M (NH$_4$)$_2$HPO$_4$ . Mix.
12. Dissolve precipitate in 80mL 1.5M HCl. Dilute to 170mL. Add 15mL 49% HF. Mix. Cool in ice bath 10min.
14. Dissolve precipitate in 7mL 3M HNO$_3$-0.25M Boric acid, 7.5mL 7M HNO$_3$, and 7mL 2M Al(NO$_3$)$_3$. Warm as needed.
**Figure 2. Strontium Resin Separation (Optional \(^{90}\)Y Ingrowth)**

(1) Precondition Sr Resin with 10mL 8M HNO\(_3\).
(2) Load sample at 1-2mL/min.
(3) Rinse sample tube with 5mL 8M HNO\(_3\).
(4) Add tube rinse to Sr Resin. Elute at 1-2mL/min.
(5) Rinse Sr Resin sequentially with:
   - 10 mL 8M HNO\(_3\)
   - 10mL 3M HNO\(_3\) - 0.05 oxalic acid
   - 10mL 8M HNO\(_3\)
(6) Dispose of (1) to (5) as waste.
(7) Strip Sr with 20mL 0.05M HNO\(_3\) at 1mL/min.
(8) Evaporate samples to dryness on tared cupped stainless steel planchets.
(9) Rinse Sr sample vials with 2mL 0.05M HNO\(_3\). Transfer vial rinse to planchets. Evaporate to dryness.
(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable Sr(NO\(_3\))\(_2\).
(11) Measure radiostrontium in samples on low background gas flow proportional counter.

**Method Performance**

<table>
<thead>
<tr>
<th>Sample</th>
<th>% Sr tracer recovery</th>
<th>(^{90})Sr Bq/g reference</th>
<th>(^{89})Sr Bq/g measured</th>
<th>% bias</th>
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**References**