Rapid Determination of Sr in 50g Soil Samples

**Summary of Method**  
Strontium is separated and concentrated from 50 gram soil samples. Soils are leached with concentrated nitric and hydrochloric acid. The leachate is evaporated to dryness, and the residue is dissolved in 1M HCl. A ferric hydroxide-calcium phosphate precipitate concentrates strontium and removes matrix components leached from the soil. A calcium fluoride precipitate further concentrates and purifies the strontium fraction. Strontium is separated from matrix impurities and potentially interfering radionuclides in the sample using two stacked 2mL cartridges of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter. Average chemical recovery of strontium, determined by gravimetric yield of stable strontium carrier, is 91 ± 4%. Measured values of 90Sr agreed to within 2% of reference values for 90 minute count times. The minimum detectable activity for 90Sr in 50g samples with 90 minute count times is 0.41Bq/g. A single operator can prepare batches of 12 samples for the measurement of 90Sr in less than 16 hours.

**Reagents**
- Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
- Nitric Acid (70%)
- Hydrochloric Acid (37%)
- Hydrofluoric Acid (49%) or Sodium Fluoride
- Ammonium Hydroxide (listed as 28% NH₃ or 56% NH₄OH)
- Deionized Water
- 1.25M Ca(NO₃)₂
- 3.2M (NH₄)₂HPO₄
- Strontium Carrier (10mg/mL)
- 2M Al(NO₃)₃
- Sr-90 standard
- 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
- Centrifuge
- Cupped Stainless Steel Planchets (~5mL volume)
- Gas Flow Proportional Counter
- Muffle Furnace
- Hot Plate
- Analytical Balance
- 600mL Glass Beakers
- Vacuum Pump

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**Figure 1. Sample Preparation**

Dry soil at 110°C. Blend and Size.
Remove 50g aliquot into 600mL glass beaker.
Muffle at 550°C for 1-2 hours.
Add 6mg Sr Carrier*, 50mL 70% HNO₃, and 25mL 37% HCl.
*may need to adjust Sr carrier amount to account for native Sr content in soil.
Heat to dryness on hot plate, medium setting.
Add 50mL 70% HNO₃. Warm sample. Transfer solids and liquid to 250mL centrifuge tube.
Centrifuge 3500 rpm, 10 min.
Transfer supernate to 600mL beaker.
Add 25mL 70% HNO₃ to Solids. Mix and Centrifuge. Transfer supernate to same 600mL beaker. Repeat once.
Discard solids to waste.
Evaporate supernate in 600mL beaker to dryness.
Dilute to 160mL. Add 1mL 1.25M Ca(NO₃)₃, 2mL 3.2M (NH₄)₂HPO₄, and 25mL 57% NH₄OH. Mix.
Centrifuge. Decant supernate to waste.
Continue to load solution preparation.
Figure 2. Load Solution Preparation and Strontium Separation

Dissolve residue in 40mL 1.5M HCl. Dilute to 170mL with H2O. Add 25mL 49% HF. Mix well. Centrifuge 10 min. Discard Supernate. Dissolve residue in 7mL 70% HNO3, 7mL 3M HNO3-0.25M Boric Acid, 7mL 2M Al(NO3)3.

(1) Precondition Sr Resin with 10mL 8M HNO3.
(2) Load sample.
(3) Rinse sample tube with 5mL 8M HNO3.
(4) Add tube rinse to Sr Resin.
(5) Rinse Sr Resin sequentially with:
   - 15 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 18mL 0.05M HNO3.

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 89/90Sr 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). 89/90Sr can then be measured by adding liquid scintillation cocktail.
(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO3 after >7 days of 90Y ingrowth. 89/90Sr 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.

Method Performance for 50g Soils Spiked with 90Sr

<table>
<thead>
<tr>
<th>Sample replicates</th>
<th>90Sr Reference Value (mBq/g)</th>
<th>90Sr Measured Value (mBq/g)</th>
<th>% Bias</th>
<th>Sr Carrier % Yield</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>5.92</td>
<td>5.95 ± 0.22</td>
<td>5.0</td>
<td>94.0 ± 2.6</td>
</tr>
<tr>
<td>7</td>
<td>11.8</td>
<td>11.5 ± 0.7</td>
<td>-2.5</td>
<td>89.6 ± 2.7</td>
</tr>
<tr>
<td>7</td>
<td>59.2</td>
<td>57.8 ± 1.7</td>
<td>-2.4</td>
<td>89.3 ± 4.7</td>
</tr>
</tbody>
</table>

MDA 90Sr, 90 minute count, 50g Soil = 0.41 mBq/g

References