eichrom[®]

Rapid Determination of Sr in 50g Soil Samples

AN-1403-10

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 HCI. 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 $91 \pm 4\%$. 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

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% HCI.

*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.

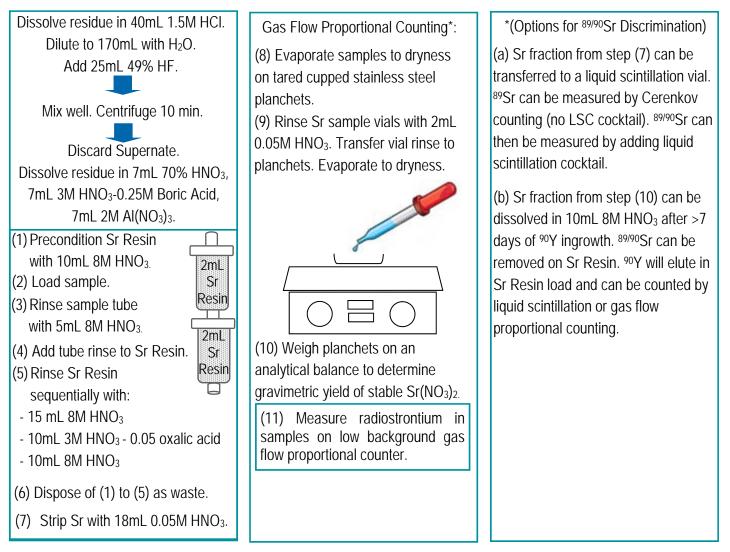
Add 25mL 70% HNO₃ to Solids. Mix and Centrifuge. Transfer supernate to same 600mL beaker. Repeat once. Discard solids to waste.

Evaporate supernate in 600mL beaker to dryness. Dissolve residue in 15-20mL 1M HCI. Transfer to 250mL centrifuge tube.

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



Method Performance for 50g Soils Spiked with ⁹⁰Sr

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

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

References

1) Sherrod L. Maxwell, Brian K. Culligan, Patrick J. Shaw "Rapid determination of radiostrontium in large soil samples," *J. Radioanal. Nucl. Chem., 295(2), 965-971* (2013).