AN-1409-10

Rapid Determination of Sr in Building Materials

Summary of Method Strontium is separated and concentrated from 1.5 gram samples of concrete or brick. 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 and decreases the volume of precipitate. The calcium fluoride precipitate is dissolved with nitric acid-boric acid-aluminum nitrate to form the Sr Resin load solution. Strontium is separated from remaining matrix and potentially interfering radionuclides using stacked 2mL and 1mL Sr Resin cartridges. Batches of 12-24 samples can be prepared for analysis in less than 8 hours. Radiostrontium is measured by gas flow proportional counting or liquid scintillation counting. Chemical yield of strontium is determined by gravimetric yield of stable strontium or ICP-AES measurement.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Sr Resin, 1mL Cartridges (Eichrom SR1ML-R50-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrofluoric Acid (49%) or Sodium Fluoride
Deionized Water
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
2M $Al(NO_3)_3$
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Strontium Carrier (10mg/mL)
^{90}Sr standard
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 250mL Centrifuge Tubes
Centrifuge
Cupped Stainless Steel Planchets (~5mL volume)
Gas Flow Proportional Counter
Muffle Furnace
Hot Plate
Analytical Balance
250mL Zirconium crucibles with zirconium lids
Vacuum Pump

Figure 1. Sample Preparation

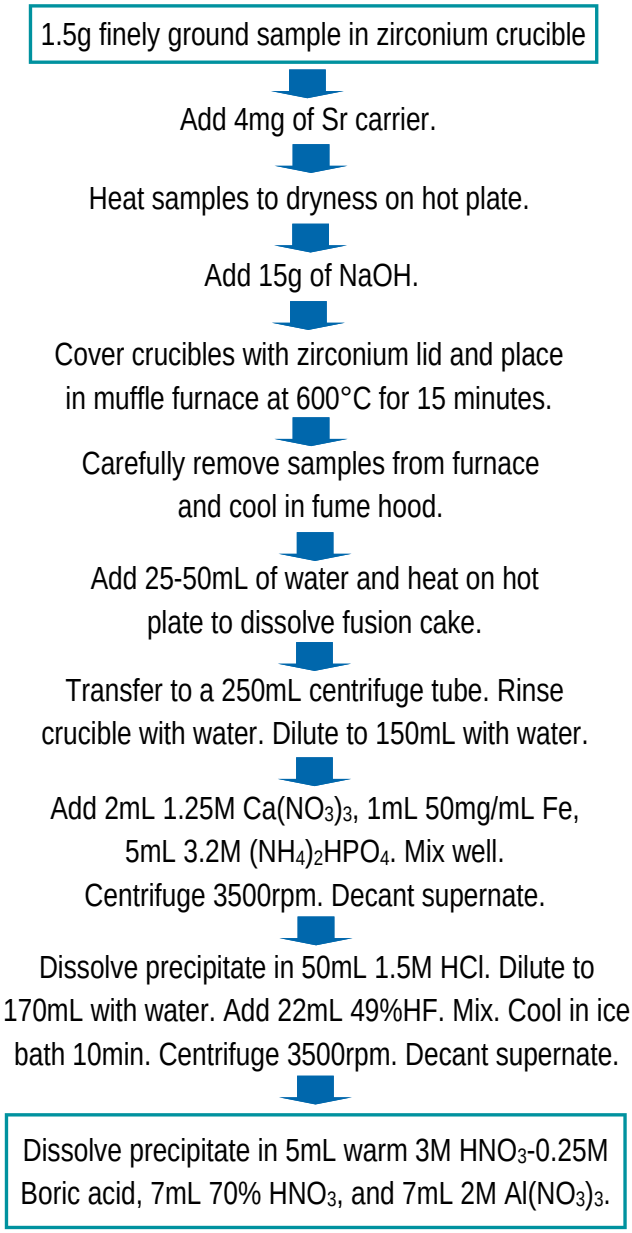


Figure 2. Strontium Resin Separation (Optional ^{90}Y Ingrowth)

(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.

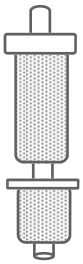
(4) Add tube rinse to Sr Resin. Elute at 1-2mL/min.

(5) Rinse Sr Resin sequentially with:

- 15 mL 8M HNO_3
- 10mL 3M HNO_3 - 0.05 oxalic acid
- 10mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

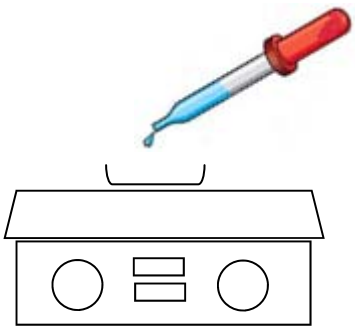
(7) Strip Sr with 20mL 0.05M HNO_3 at 1mL/min.



Gas Flow Proportional Counting:*

(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.

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

When necessary to obtain ^{89}Sr and ^{90}Sr data:

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (without LSC cocktail).

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

References

1)"Rapid radiochemical method for total radiostrontium (Sr-90) in building materials for environmental remediation following radiological incidents," U.S. Environmental Protection Agency, National Analytical Radiation Environmental Laboratory, EPA 402-R14-001.

2)"Rapid method for sodium hydroxide fusion of concrete and brick matrices prior to americium, plutonium, strontium, radium, and uranium analyses for environmental remediation following radiological incidents,"U.S. Environmental Protection Agency, National Analytical Radiation Environmental Laboratory, EPA 402-R-14-004.