AN-1405-10

Rapid Determination of Sr in Vegetation Samples

Summary of Method Strontium is separated and concentrated from 5-10 gram vegetation samples. Samples are muffled in zirconium crucibles 2-4 hours to destroy organic content. The residue is wet ashed with HNO_3-H_2O_2 and then fused with 15g NaOH at 600°C for ten minutes. The fusion cakes are dissolved in water, transferred to 250mL centrifuge tubes and precipitated with calcium phosphate to facilitate matrix removal. Strontium is separated from matrix impurities and potentially interfering radionuclides in the sample using stacked 2mL and 1mL cartridges of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter or liquid scintillation counter. Chemical yield of strontium is determined by gravimetric recovery of stable strontium or ICP-AES measurement. Average chemical recovery of strontium is 64 ± 4% for 5g samples and 70 ± 8% for 10g samples. Measured values of ^{90}Sr agreed to within 12% of reference values for 90 minute count times. The average time to complete the sample preparation is <8 hours.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Sr Resin, 1mL Cartridges (Eichrom SR1ML-R50-S)
Nitric Acid (70%)
Hydrogen Peroxide (30%)
Deionized Water
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Strontium Carrier (10mg/mL)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
^{90}Sr standard
3.2M $(NH_4)_2HPO_4$
Sodium Hydroxide
Oxalic 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
250mL Zirconium crucibles with zirconium lids
Vacuum Pump

Figure 1. Sample Preparation

5-10g Vegetation sample in zirconium crucible

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Muffle at 600°C.
2 hours for 5g sample.
4 hours for 10g sample.

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Wet ash on hotplate with 5mL 70% HNO_3 and 5mL 30% H_2O_2.

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Fuse samples with 15g NaOH at 600°C for 10 minutes.

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Dissolve fusion cake with H_2O.
Transfer to 250mL centrifuge tube.
Add 125mg Fe and 4mg Sr. Dilute to 180mL.

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Add 4mL 1.25M $Ca(NO_3)_2$, 5mL 3.2M $(NH_4)_2HPO_4$.
Mix. Cool in ice bath for 10min.

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Centrifuge at 3500rpm. Decant Supernate.

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Dissolve precipitate in 5mL warm 3M HNO_3, 7mL 70% HNO_3, and 7mL 2M $Al(NO_3)_3$.

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.

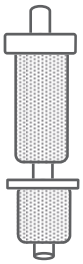
(5) Rinse Sr Resin sequentially with:

- 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.

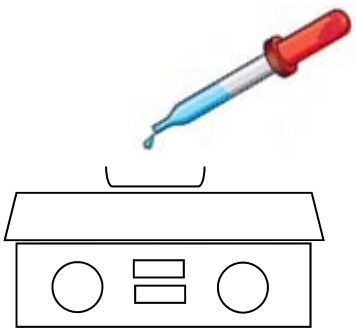
2mL + 1mL Sr Resin Cartridges



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 Discrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}$Sr can then be measured by adding liquid scintillation 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.

*Actinides may also be measured by adding a 2mL TEVA, TRU and DGA cartridges above Sr Resin and following the separation scheme in Eichrom application note AN-1406, "Rapid Determination of Actinides in Vegetation Samples."

Performance of ^{90}Sr Method 5-10g Vegetation Samples

Sample Replicates	Sample Mass, g	^{90}Sr, Reference Value (Bq/g)	^{90}Sr, Measured Value (Bq/g)	% Bias	Sr carrier % Recovery
6	5.0	0.255	0.285 ± 0.03	12	64 ± 4
2	10.0	0.156	0.156 ± 0.001	0.0	69 ± 2
2	10.0	0.110	0.109 ± 0.003	-0.1	70 ± 7

90 minute count times

References

1) Sherrod L. Maxwell, Brian K. Culligan, Gary W. Noyes, "Rapid separation of actinides and radiostrontium in vegetation samples," *J. Radioanal. Nucl. Chem., 286(1), 273-282* (2010).