AN-1606-10

Rapid Determination of ^{90}Sr in 10g Concrete Samples

Summary of Method ^{90}Sr is determined by the direct separation of its daughter ^{90}Y from 10 gram concrete samples. Samples are finely ground and fused in a zirconium crucible for 30 minutes at 600°C with 40 grams of sodium hydroxide. The fusion cake is dissolved in water, and strontium is concentrated and separated from the matrix using a ferric hydroxide precipitate. A secondary precipitation with Y/Ca-fluoride removes additional matrix (including silicates) and decreases the volume of precipitate. The Y/Ca-fluoride precipitate is dissolved with nitric acid-boric acid-aluminum nitrate to form the load solution. ^{90}Y is separated from remaining matrix and potentially interfering radionuclides using stacked 2mL TRU and DGA Resin cartridges. ^{90}Y is measured by gas flow proportional counting following microprecipitation onto Resolve® Filters. Chemical yields are determined by ICP-AES analysis. Batches of 12-24 samples can be prepared for analysis in less than 8 hours. This method is only suitable for aged samples, where the shorter lived ^{89}Sr ($t_{1/2}$ = 50.55 days) and fission products such as ^{91}Y are unlikely to be present. For samples not meeting this criterion, $^{89/90}Sr$ can be determined from up to 5g concrete samples, using Eichrom Application Note AN-1605.

Reagents

DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
Yttrium Carrier (10mg/mL)
Iron Carrier (50mg/mL Fe, as ferric nitrate)
^{90}Sr standard
Nitric Acid (70%)
Hydrochloric Acid (37%)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
Boric acid
HF(49%)
Sodium Hydroxide
Deionized Water
3.2M $(NH_4)_2HPO_4$
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)
Resolve Filters with funnel (Eichrom RF-DF25-25PP01)
50mL and 500mL Centrifuge Tubes
Stainless Steel Planchets with two sided tape
250mL Zirconium crucibles with zirconium lids
Centrifuge
Muffle Furnace
Analytical Balance
Gas Flow Proportional Counter
Hot Plate/Heat Lamp
Vacuum Pump

Figure 1. Sample Preparation

10g finely ground sample in zirconium crucible

↓

Add 2mg Y carrier.
Heat samples to dryness on hot plate.

↓

Add 40g of NaOH.
Cover crucibles with zirconium lid and place in muffle furnace at 600°C for 30 minutes.

↓

Carefully remove samples from furnace and cool in fume hood.

↓

Add 50-100mL of water and heat on hot plate to dissolve fusion cake.

↓

Transfer to a 500mL centrifuge tube. Rinse crucible with water. Dilute to 450mL with water. Cool to room temp. Add 125mg Fe Mix. Centrifuge 10min. Decant supernate.

↓

Rinse precipitate with 150mL pH ~9 NaOH. Centrifuge. Decant Supernate. Repeat.

↓

Dissolve precipitate in 200mL 1.5M HCl. Add 50mL 0.01M HCl and 15mL 49%HF. Mix. Cool in ice bath 10min. Centrifuge 10min. Decant supernate.

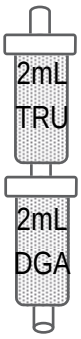
↓

Dissolve precipitate in 7mL 3M HNO_3-0.25M Boric acid, 7mL conc. HNO_3, 7mL 8M HNO_3 and 7mL 2M $Al(NO_3)_3$. Warm as needed.

Figure 2. TRU-DGA Separation and Gas Flow Proportional Counting

(1) Precondition 2mL TRU* + DGA 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 TRU + DGA. Elute 1-2 mL/min.

(5) Rinse sequentially with:
- 5mL 6M HNO_3
- 10mL 3M HNO_3
- 12mL 4M HCl

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

(7) Discard TRU Resin.

(8) Rinse DGA sequentially with:
- 10mL 8M HNO_3 (Ca)
- 15mL 0.1M HNO_3 (U)
- 25mL 3M HNO_3-0.25M HF (Th)

(9) Strip Y with 18mL 0.25M HCl.

(10) Dilute to 20mL with water.

(11) Take 1mL aliquot and dilute to 20mL for ICP-AES yield measurement.

(8) Add 100ug Ce carrier to samples.

(9) Mix well.

(10) Add 2mL 49% HF.

(11) Mix well.

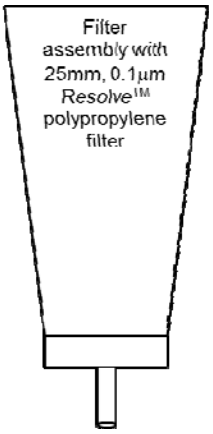
(12) Wait 15-20 minutes.

(13) Set up Resolve® Filter Funnel on vacuum box.

(14) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(15) Filter sample.

(16) Rinse sample tube with 5mL DI water and add to filter.

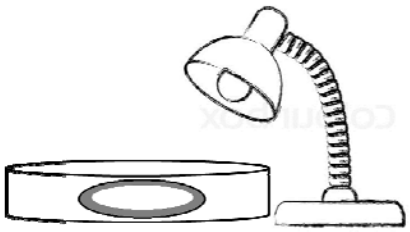


(17) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

(18) Draw vacuum until filter is dry.

(19) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.

(20) Dry filter under heat lamp for 3-5 minutes.



(21) Measure Y-90 by gas flow proportional counting.

*TRU Resin improves decontamination factors for U, Th and Bi isotopes, which could interfere with the measurement of ^{90}Y by gas flow proportional counting.

Method Performance (10g gram Concrete, TRU/DGA Resin Method)

Sample	% Y tracer recovery	^{90}Sr Bq/g reference	^{90}Sr Bq/g measured	% bias
1	81.7	0.0327	0.031	-5.4
2	83.3	0.0327	0.033	1.2
3	83.7	0.0327	0.031	-5.0
4	86.3	0.0327	0.033	-0.6
AVG	84 ± 2		0.032 ± 0.001	

References

1) Maxwell, Culligan, Hutchinson, Utsey, Sudowe, McAlister, "Rapid Method to Determine 89/90Sr in Large Concrete Samples," *J. Radioanal. Nucl. Chem.* accepted (2016). DOI 10.1007/s10967-016-4787-4