AN-1402-10

Rapid Determination of Sr in Emergency Milk Samples

Summary of Method Strontium is separated and concentrated from 100mL milk samples using a calcium phosphate precipitation. The precipitate is dissolved with nitric acid and centrifuged to remove residual protein and fat. The supernate, containing Sr, is wet ashed with HNO_3-H_2O_2 and then heated in a muffle furnace at 550°C for 30-60 minutes to destroy any residual organic matter. The muffled residue is wet ashed again with HNO_3-H_2O_2 and dissolved in HNO_3-$Al(NO_3)_3$. 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 by ICP-AES measurement. Average chemical recovery of strontium is 75 ± 17%. Measured values of ^{90}Sr agreed to within 3.2% and 0.5% of reference values for 20 minute count times and 60 minute count times, respectively. The lower limit of detection for 100mL samples with 20 minute count times is 0.5Bq/L and with 60 minute count times is 0.16Bq/L. A single operator can prepare batches of 12-24 samples for ^{90}Sr measurement in less than 8 hours.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Sr Resin, 1mL Cartridges (Eichrom SR1ML-R50-S)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Nitric Acid (70%)
Hydrogen Peroxide (30%)
Deionized Water
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
Strontium Carrier (10mg/mL)
2M $Al(NO_3)_3$
^{90}Sr standard
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 Glass Beakers
Vacuum Pump

Figure 1. Sample Preparation

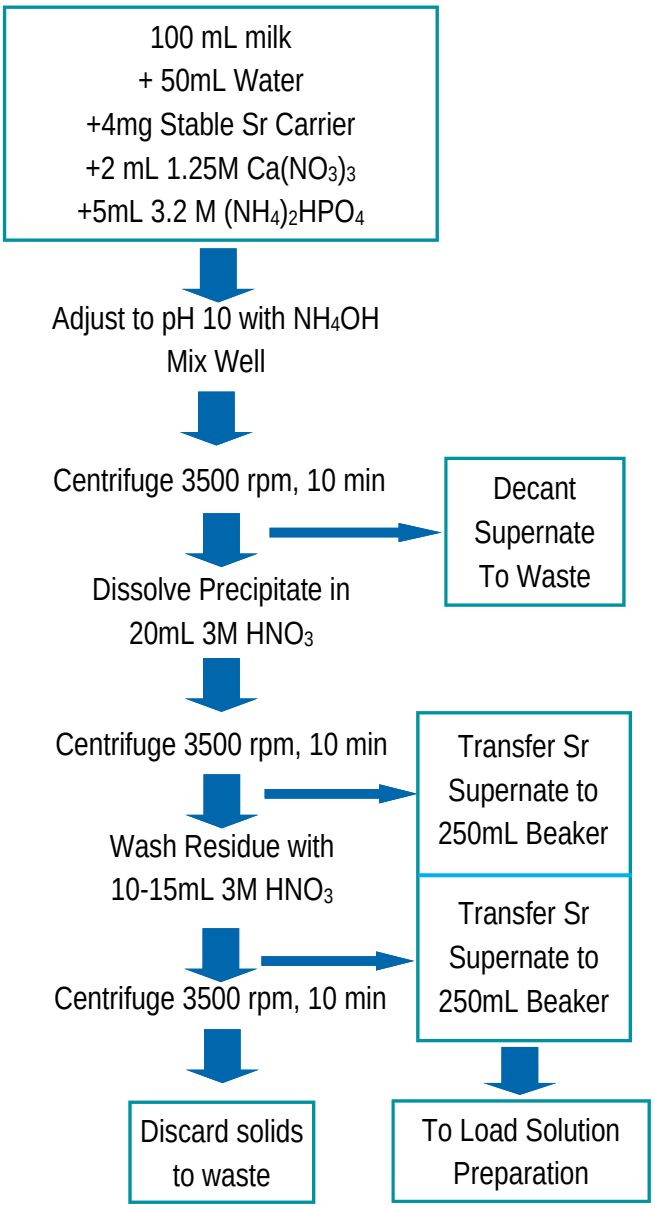

Figure 2. Load Solution Preparation and Strontium Separation

Evaporate Supernate to Dryness

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Wet ash with 5mL 70% HNO_3 and 5mL of 30% H_2O_2

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Muffle at 550°C for 30-60 min.

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Wet ash with 5mL 70% HNO_3 and 5mL of 30% H_2O_2

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Dissolve residue in 5mL 70% HNO_3 and 5mL 2M $Al(NO_3)_3$ (warm to facilitate dissolution)

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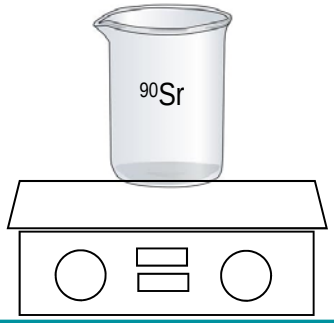
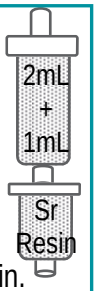
Cool to room temperature.



(1) Precondition Sr Resin with 5mL 8M HNO_3.

(2) Load sample.

(3) Rinse sample beaker with 5mL 8M HNO_3.



(4) Add beaker rinse to Sr Resin.

(5) Rinse Sr Resin sequentially with:
- 15 mL 8M HNO_3
- 5mL 3M HNO_3 - 0.05 oxalic acid
- 7mL 8M HNO_3

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

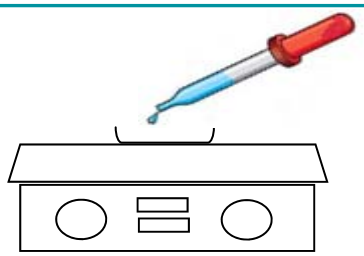
(7) Strip Sr with 13mL 0.05M HNO_3.

Gas Flow Proportional Counting*:

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 8M 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.

Performance of Radiostrontium in Milk Method

20 Minute Count Times			
^{90}Sr, reference (Bq/L)	^{90}Sr,measured (Bq/L)	Uncertainty %, k = 2	% Bias
0	0.26	98.9	N/A
0	0.26	81.9	N/A
2.86	2.66	24.1	-7.0
2.86	3.96	24.7	38
2.86	3.31	20.2	15.7
2.86	2.67	18.7	-6.6
5.7	6.11	16.7	7.2
5.7	5.71	13.1	0.2
5.7	5.16	13.9	-9.5
14.3	12.8	9.1	-11
14.3	15.2	8.5	6.3
14.3	14.1	8.6	-1.4

60 Minute Count Times			
^{90}Sr, reference (Bq/L)	^{90}Sr,measured (Bq/L)	Uncertainty %, k = 2	% Bias
0	0.11	130	N/A
0	0.27	59	N/A
2.86	3.09	13.2	8.0
2.86	3.11	16.7	8.7
2.86	2.67	13.6	-6.6
2.86	2.67	11.3	-6.6
5.7	5.85	10.4	2.6
5.7	5.75	8.3	0.9
5.7	6.04	8.2	5.9
14.3	13.6	6.1	-4.9
14.3	14.0	6.1	-2.1
14.3	14.2	6.1	-0.7

References

1) Sherrod L. Maxwell, Brian K. Culligan, "Rapid method for the determination of radiostrontium in emergency milk samples," *J. Radioanal. Nucl. Chem., 279(3), 757-760* (2009).