1. Scope

This method developed by Sáez-Muñoz et al. 2018 [1] aims to quantify ^{90}Sr during emergency situations in mil samples using PS Resin for simultaneous separation and quantification of ^{90}Sr by liquid scintillation counting (LSC).

2. Summary

The reported method is based on the publication by Sáez-Muñoz et al. (2018) [1]. The main objective was to implement a rapid method for determining ^{90}Sr in milk samples using PS resins for separation and subsequent measurement by LSC. The sample preparation step was optimized to homogenize the sample, remove any proteins or fats in the matrix, and separate strontium (and other alkaline-earth elements) from alkali metals. After treatment, the precipitate (oxalate) is dissolved in a suitable acidic medium to load onto TK-SrScint Resin for the separation of elements such as Ca or Y. Following rinsing of the PS Resin, the cartridge is placed in an LS vial for LSC measurement. The solution collected during loading and rinsing is used for ICP measurement to quantify chemical yield. This method was compared with other separation methods in the literature, showing comparable results in chemical yield (with lower variability in this method) and a minimum detectable activity of 0.34 Bq/L, which meets the requirements for emergency situations.

3. Significance of use

This method offers a rapid approach for the quantification of ^{90}Sr in milk samples in emergency situations, minimizing mixed waste production and achieving activity levels suitable for this purpose.

4. Interferences

Regarding the quantification of ^{90}Sr, as this study focuses on milk samples with the goal of isolating strontium, the primary interferences evaluated were Ca and Ba, which are major elements in milk, as well as ^{90}Y (since the presence of ^{90}Sr leads to ^{90}Y as its daughter radionuclide). Additionally, biological compounds such as fats and proteins were considered, as they can impact chemical separation (e.g. through resin saturation).

5. Apparatus

a. Hot plate and stirrer

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b. Analytical balance -0.0001 g sensitivity
c. Büchner funnel + filter Whatman GF/C (1.2 µm)
d. Peristaltic pump
e. pH meter
f. Beckman J2-HS (Indianapolis, Indiana, USA)
g. OPTIMA 8300 ICP-OES detector or ELAN-6000 ICP-MS detector to quantify strontium concentrations
h. R-210 Rotavapor from Buchi (Flawil, Switzerland)
i. MS 3 digital vortex from IKA (Staufer, Germany)
j. Vacuum box
k. Wallac Quantulus 1220 liquid scintillation spectrometer

6. Reagents

a. *Reagents*

Unless otherwise indicated, all references to water should be understood to mean double deionized distilled water. All reagents should be at least of analytical grade.

- Strontium carrier prepared with $Sr(NO_3)_2$ (Merck, Darmstadt, Germany)
- The crown ether 4,40(50)-di-t-butylcyclohexano-18-crown-6 (DtBuCH18C6) ([95% purity), lithium nitrate ([99% purity), methanol (99% purity) and 1-octanol (98% purity) were supplied by Sigma-Aldrich (St. Louis, USA)
- ammonia (25% w/w), tricloroacetic acid ([99.5% purity), oxalic acid and nitric acid (69% w/w) were supplied by Panreac (Barcelona, Spain)
- PS Resin preparation: deposition of DtBuCH18C6 in 1-octanol on the surface of PS microspheres (median diameter 60 µm) (preparation procedure described in
- Working solution of $^{90}Sr/^{90}Y$ of 37.2 ±0.3 Bq/g containing both strontium and yttrium (each 100 µg/g) prepared from a standard supplied by Amersham International, Buckinghamshire, England
- Working solution of ^{89}Sr of 1.303 ±0.3 kBq/g containing 50 µg/g strontium prepared from a standard supplied by Eckert Ziegler (Berlin, Germany)
- Solid-phase extraction cartridges (SPE cartridges) and frits from Triskem International (Rennes, France)
- 20 mL polyethylene vials from PerkinElmer (Waltham, Massachusetts, USA)

b. *Preparation of solutions*

- Strontium carrier (5 mg/mL): For 100 mL solution dissolve approximately 1.208 g of iron nitrate (from Merck, Germany) in water. Mix thoroughly.

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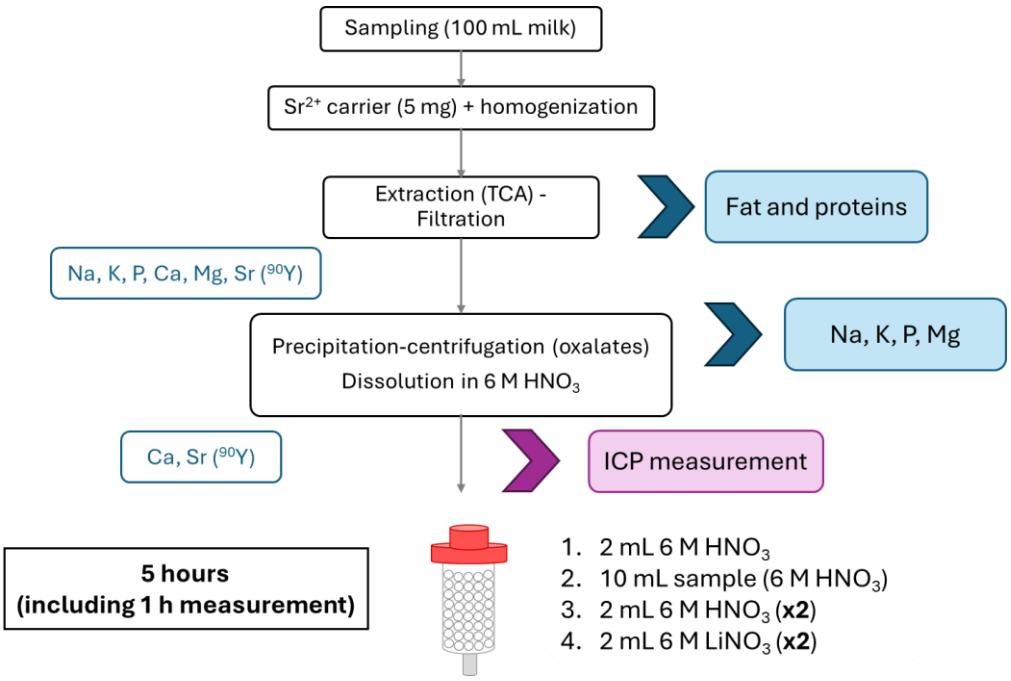
- 20% 2,2,2-trichloroacetic acid (TCA): For 100 mL solution add around 40 mL deionized water at the bottom of the 100 mL flask and add slowly 20.1 g of tricloroacetic acid (99.5% purity). Then, add water to the volumetric flask until the total volume. Mix thoroughly.
- 6 M HNO_3: For 100 mL solution add around 50 mL deionized water at the bottom of the 100 mL flask and add slowly 38.85 mL concentrated HNO_3 (69% HNO_3). Then, add water to the volumetric flask until the total volume. Mix thoroughly.
- 8 M HNO_3: For 100 mL solution add around 40 mL deionized water at the bottom of the 100 mL flask and add slowly 51.81 mL concentrated HNO_3 (69% HNO_3). Then, add water to the volumetric flask until the total volume. Mix thoroughly.
- 6 M $LiNO_3$: For 100 mL solution add around 40 mL deionized water at the bottom of the 100 mL flask and add slowly 41.83 g of $LiNO_3$ (99% purity). Then, add water to the volumetric flask until the total volume. Mix thoroughly.

c. *Samples used*

- Cow's, goat's and sheep's milk purchased in local supermarket. Different fat content milks were evaluated (whole (3.6 g/100 mL), semi-skimmed (1.6 g/100 mL) and skimmed (0.3 g/100 mL) cow's milk.
- Certified reference material (IAEA-473 milk powder) for method validation

7. Procedure

a. *Detailed procedure*



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Based on Sáez-Muñoz et al. (2018) *[1]*.

b. *Sample preparation*

Sample homogeneization

1. Homogenize milk sample on a hot plate by stirring with a magnetic stirrer for 15 min.
2. Use 100 mL of milk sample for the analysis. Weight the sample.
3. Add 1 mL strontium carrier solution (5 mg/mL) and stir for 30 min to homogenize the sample. Weight the carrier in a vial.

Protein denaturation

4. The milk is acidified using 100mL 20% 2,2,2-tricloroacetic acid (ratio 1:1 TCA:milk sample). Mix for 30 min.
5. Filter with a paper filter Whatman GF/C (1.2 µm) in a Büchner funnel. Wash the beaker with 5-10 mL 10% TCA. Proteins and fats are kept on the paper filter while strontium will remain on the filtrate solution.

Alkali metal group removal

6. Heat the solution and add 5 g oxalic acid to the warm filtrate solution of the previous step. Mix for 5 min.
7. Adjust the pH to 5–6 with ammonia, mixing for 5 minutes, and then allow it to reach room temperature.
8. Centrifuge the precipitate obtained after cooling at 5000 rpm for 15 min.
9. Dissolve the precipitate in 10 mL of 6 M HNO_3. Heat the centrifuge tube in a water bath to speed up the dissolution of the precipitate.
10. Take an aliquot to evaluate strontium chemical yield by ICP-OES.

c. *Radiochemical separation*

Preparation of Sr-PSresin cartridges (TK-SrScint Resin)

Filling with 1.4 g TK-SrScint resin an empty 2 mL cartridge. Sealing of the cartridge and placed on a vacuum chamber for homogenization passing though 10 mL water (flow rate 1 mL/min). Before use, cartridges were mixed using a vortex for 3 min (at 3000 rpm)

^{90}Sr separation from interferences (mostly Ca and Y)

1. Condition of the TK-SrScint resin with 2 mL 6 M HNO_3 (flow rate 1 L/min).

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2. Pass the sample throw the cartridge at 1 L/min.
3. Rinse the cartridge with 2x 2 mL 6 M HNO_3 at 1 L/min.
4. Rinse the cartridge with 2x 2 mL 6 M $LiNO_3$ at 1 L/min.
5. Collect all the solutions on the same 50 mL centrifuge tube. Take an aliquot to measure the strontium content by ICP-OES in order to evaluate the chemical yield
6. Leave for 5 min at 20 inHg to remove remaining solution on the cartridge

d. Sample measurement

1. Place the TK-SrScint Resin cartridge on a 20 mL polyethylene scintillation vial
2. Prepare a protocol in the low coincidence bias and ^{14}C configuration
3. Measurement of the samples must be performed immediately after separation to prevent ^{90}Y ingrowth using 3 cycles of 20 min with 10 min for SQP(E) parameter
4. Chemical recovery evaluation: a 50 mL tube is used to collect the solutions that passed through the resin (loading the sample and rinsing solutions). Dilution of the initial solution is made using 1% HNO_3 as final medium to measure a maximum concentration of Sr of 5 mg/L
    - Sr recoveries around 65% can be achieved by using this method with a relative bias of ^{90}Sr+^{89}Sr activity below 15%

8. References

[1] M. Sáez-Muñoz, H. Bagán, A. Tarancón, J. F. García, J. Ortiz, and S. Martorell, “Rapid method for radiostrontium determination in milk in emergency situations using PS resin,” *J Radioanal Nucl Chem*, vol. 315, no. 3, pp. 543–555, Mar. 2018, doi: 10.1007/s10967-017-5682-3.

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