1. Scope

This method aims to quantify ^{90}Sr in various environmental samples (e.g. river water) while accounting for the presence of ^{210}Pb, 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 (I. Gimenez, 2023). The primary objective was to apply an alternative strategy for determining ^{90}Sr in environmental samples using PS resins for separation and subsequent measurement of ^{90}Sr by LSC. Additionally, the focus was on reducing potential ^{210}Pb interference in the sample before loading it onto the PS resin, as this can affect the quantification of ^{90}Sr. To achieve this, the method incorporated a sample treatment involving lead precipitation as iodate, followed by loading the sample onto the PS resin and directly measuring ^{90}Sr by LSC. This method was compared to the routine method currently in use, showing comparable results and offering an alternative approach that reduces processing time and minimizes the production of mixed waste.

3. Significance of use

This method proposes an alternative approach for the quantification of ^{90}Sr in various environmental samples, leading to shorter procedural times and reduced production of mixed waste.

4. Interferences

Given the nature of the extractant used in the PS resin and the chemical similarity between Sr and Pb, ^{210}Pb presented a significant interference that needed to be removed before loading the sample onto the PS resin. Therefore, a mixture of sodium iodate and calcium nitrate was used to precipitate the lead on the samples.

5. Apparatus

a. Hot plate and stirrer
b. Analytical balance -0.0001 g sensitivity
c. Peristaltic pump
d. Büchner funnel + filter Whatman GF/C (1.2 µm)
e. pH meter

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f. Centrifuge machine
g. OPTIMA 8300 ICP-OES detector or ELAN-6000 ICP-MS detector to quantify strontium and lead concentrations
h. Vacuum box
i. 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.

- Lead standard (1000mg/L) (Inorganic Ventures, USA)
- Strontium standard (1000mg/L) (Inorganic Ventures, USA)
- HNO_3 69% from PanReac (Castellar del Valles, Spain)
- Ammonia 25% from PanReac (Castellar del Valles, Spain)
- Oxalic acid from PanReac (Castellar del Valles, Spain)
- The crown ether 4' ,4''(5'')-di-tert-butyldicyclohexane-18-crown-6, 1-octanol, sodium iodate, di-ammonium hydrogen phosphate, potassium iodate and calcium nitrate were supplied by Sigma Aldrich (USA)
- Working solution of ^{210}Pb of 160 ±4.8 Bq/g prepared from a standard purchased in CEA/DAMRI, Gif-Sur-Yvette CEDEX, France 22 years of ingrowth
- Working solution of $^{90}Sr/^{90}Y$ of 38.45 ±0.29 Bq/g prepared from a standard containing both strontium and yttrium (each 100 µg/g) supplied by Amersham International, Buckinghamshire, England
- Gold XR liquid scintillation cocktail

b. Preparation of solutions

- Fe solution (5 mg/L): For 100 mL solution dissolve approximately 2.16 mg of iron nitrate (from Sigma Aldrich, USA) in water. 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.

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- 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*

- River water sample
- MAPEP water samples (interlaboratory)
- CSN interlaboratory water samples

7. Procedure

a. *Sample preparation*

Precipitation method prior to TK-SrScint separation

1. Sample preparation : 5 mg of lead and 5 mg of strontium in 25 mL tubes
2. Precipitation : add 1 g of sodium iodate and 0.41 g of calcium nitrate to sample. Mixture was warmed in a water bath (15-20 min) at 45°C.
3. pH adjustment of the solution using nitric acid (pH 1-8) – lead precipitation
4. Centrifuge samples for 5 min at 5000 rpm. Strontium present on the supernatant. Filter and:
5. Dissolve solid (lead) with nitric acid and warm for 10 min in a water bath
6. Keep solution (supernatant) where strontium should remain
7. Add 0.5 g of di-ammonium hydrogen phosphate to the supernatant. Then, add ammonia until pH 10 → strontium precipitation
8. Centrifuge the mixture and separate the supernatant from the precipitate
9. Dissolve the precipitate in 11.5 mL of 8 M HNO_3.

b. *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)

1. Conditioning: 2 mL 8 M HNO_3
2. Loading: from the 11.5 mL, take 10 mL and pass them through the TK-SrScint Resin (using a vacuum box, settle the pressure to 5 inHg)
3. 1st rinsing: 2 times 2 mL 8 M HNO_3
4. 2nd rinsing: 2 times 2 mL 6 M $LiNO_3$
5. Leave for 5 min at 20 inHg to remove remaining solution on the cartridge

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c. *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. After 21 days, measure again the samples for 12 h with 10 min for SQP(E) parameter
5. 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 and Pb of 5 mg/L
    - Sr recoveries around 72% can be achieved by using iodiate precipitation and TK-SrScint Resin separation method while only 5% Pb

8. References

I. Gimenez, J. R. (2023). A new method based on selective fluorescent polymers (PSresin) for the analysis of ^{90}Sr in presence of ^{210}Pb in environmental samples. *Applied Radiation and Isotopes*, 199.

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