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Strontium 89, 90 in Water

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1. Scope

1.1 This procedure describes a method for separation and measurement of strontium 89, 90 in water.

2. Summary of Method

2.1 Radioactive strontium is separated using Sr Resin prior to gas proportional counting, liquid scintillation counting or Cerenkov counting. Cation exchange resin is used to concentrate strontium from water samples. Stable strontium and/or strontium-85 tracer are used to monitor method yields and correct results to improve precision and accuracy.

3. Significance of Use

3.1 This method is a rapid, reliable method for the measurement of strontium in water samples.

4. Interferences

4.1 The presence of elemental strontium in the sample may bias the gravimetric yield determination. If it is suspected that natural strontium is present in the sample, its concentration should be determined by a suitable means and the yield calculation appropriately modified.

4.2 Strontium must be separated from interfering isotopes of other elements to enable measurement by beta counting.

4.3 Sr Resin with an 8M HNO_3 load solution is used to effectively remove barium-140 and potassium-40 isotopes as well as other matrix interference's. Tetravalent plutonium, neptunium, cerium and ruthenium, however, are not removed using nitric acid. If necessary, these isotopes can be effectively removed by including an additional rinse of approximately four free column volumes of 3M HNO_3-0.05M oxalic acid.

5. Apparatus

5.1 *Beta detector* -gas proportional counter, liquid scintillation or Cerenkov counter
5.2 *Column rack* – Eichrom part number AC-103
5.3 *Column reservoirs* - 250 mL to 1 liter volume
5.4 *Column reservoirs* - 25 mL, Eichrom part number AC-120
5.5 *Counting dishes* - 50.8 mm diameter, 6.4 mm deep flat bottom, cupped planchet.
5.6 *Fume hood*
5.7 *Gamma pulse height analyzer* - Sr-85 tracer only
5.8 *Heat lamp*
5.9 *Hot plate*
5.10 *Ion exchange columns* - 1 to 1.5 cm diameter, 10 ml resin volume
5.11 *Liquid scintillation vials*
5.12 *Plastic bottle* - 1 liter
5.13 *Volumetric flask* - 1 liter

6. Reagents

6.1 *Cation exchange resin* - C8-B500-M-H, hydrogen form, 100 to 200 mesh. Available from Eichrom.
6.2 *Ethyl alcohol* - USP, 100%
6.3 *Liquid scintillation cocktail* (LSC counting only)
6.4 *Nitric acid (15.7M)* - concentrated nitric acid (sp gr 1.42)
6.5 *Nitric acid (3M)* - Add 191 mL of concentrated HNO_3 (sp gr 1.42) to 800 mL of water and dilute to 1 liter with water.
6.6 *Nitric acid (3M) - oxalic acid solution (0.05M)*- Add 191 mL of concentrated HNO_3 (sp gr 1.42) and add 6.3 grams of oxalic acid dihydrate to 800 mL of water and dilute to 1 liter with water.
6.7 *Nitric acid solution (0.05M)* - Add 3.2 mL of concentrated nitric acid (sp gr 1.42) to 900 mL of water and dilute to 1 liter with water.
6.8 *Nitric acid solution (0.1M)* - Add 6.4 mL of concentrated nitric acid (sp gr 1.42) to 900 mL of water and dilute to 1 liter with water.

6.9 *Nitric acid solution (8M)* - Add 510 mL of concentrated nitric acid (sp gr 1.42) to 400 mL of water and dilute to 1 liter with water.

6.10 *Sr Resin* - prepacked column, 0.7 grams resin, or small particle size (50-100 μm) in appropriate size column

6.11 *Sr-85 tracer and/or standards*

6.12 *Strontium (Sr) carrier (5 mg/mL), gravimetric* - Dissolve 12.1 grams $Sr(NO_3)_2$ in water and dilute to 1 liter with water.

7. Procedure

7.1. *Water Sample Precipitation*

7.1.1 If samples larger than 1 liter are analyzed, evaporate the sample to approximately 1 liter.

7.1.2 Measure the sample volume using a standard graduated cylinder (or equivalent) and transfer volume to an appropriate size plastic bottle or volumetric flask.

7.1.3 Acidify the sample to pH 2 using concentrated nitric acid.

7.1.4 Add 1 ml of 5 mg/ml strontium carrier (for gravimetric yield option) or strontium-85 tracer (for gamma yield determination option) into each sample aliquot.

7.2 *Cation Exchange to Concentrate Strontium from Water Samples:*

Note 1 – Alternatively, evaporation to dryness (when insoluble residues such as calcium sulfate do not form) and calcium carbonate precipitation can be used to concentrate strontium.

7.2.1 Prepare a cation exchange column containing 10 ml of C8-B500-M-H, 100-200 mesh for each sample analyzed.

7.2.2 Place columns on rack with large volume reservoirs (250 ml to 1 liter).

7.2.3 Ensure that a suitable container is below each column.

7.2.4 Add 20 ml of 0.1 M HNO_3 to each column to condition columns.

7.2.5 Load each sample onto the appropriate column and allow to drain.

7.2.6 Add 25 ml of 0.1 M HNO_3 to each column to rinse.

7.2.7 Discard the feed and rinse the solution collected.

7.2.8 Ensure that a labeled 150 ml beaker is below each column.

7.2.9 Add 50 ml of 8 M HNO_3 to each column to elute strontium.

7.2.10 Place each beaker on a hot plate in a fume hood and evaporate to dryness.

7.3 *Sr Resin Column Preparation:*

7.3.1 For each sample, place a Sr Resin column in the column rack.

7.3.2 Place a beaker below each column.

7.3.3 Remove the bottom plug and cap from each column and allow each column to drain. (Save caps for later use during Y ingrowth).

7.3.4 Attach column reservoirs to each column.

7.3.5 Pipette 5 mL of 8M HNO_3 into each column and allow to drain.

7.4 *Sr Resin Column Separation:*

7.4.1 Dissolve residue from step 7.2.10 in 10 ml of 8M HNO_3.

7.4.2 Transfer each redissolved sample into the appropriate Sr Resin column by pouring or by using a plastic transfer pipette and allow to drain.

7.4.3 Add 5 mL of 8M HNO_3 to rinse to each tube/beaker and transfer each solution into the appropriate Sr Resin column and allow to drain.

7.4.4 If Pu^{+4}, Np^{+4} or Ce^{+4} may be present, add 5 mL of 3M HNO_3 - 0.05M oxalic acid into each column and allow to drain.

Note 2: The 3M HNO_3 - 0.05M oxalic acid removes Pu^{+4}, Np^{+4} or Ce^{+4}, which are retained by Sr Resin. If these interference's are known to be absent, this step may be skipped.

7.4.5 Add 5 mL of 8M HNO_3 to each column and allow the rinse solution to drain through each column.

Note 3: This additional 8M HNO_3 rinse removes any residual oxalic acid and ensures full removal of K^+ and Ba^{+2} that may be present.

7.4.6 Record the time when the last rinse completely drains through each column as the start of yttrium ingrowth.

7.4.7 Ensure that labeled plastic vials are below each column.

7.4.8 Pipette 10 mL of 0.05M HNO_3 into each column and allow to drain to elute the strontium.

7.4.9 Ensure that calibration standards are prepared per step 7.5 and GOTO 7.6 or 7.7 to count samples.

7.4.10 Pipette 5 ml of 0.05 M HNO_3 into each column to keep the resin wet during ingrowth period. Immediately replace the top cap on the column and set aside in a safe place until Y-90 ingrowth is complete. Columns will be used again in section 7.9.

7.5 *Preparation of Pure Sr-90 and Pure Y-90 for Counter Calibration Sources:*

7.5.1 Add an appropriate volume of calibrated Sr-90 standard solution (in equilibrium with Y-90) to a small beaker, add 1 mL of Sr carrier and evaporate the solution to dryness.

7.5.2 Redissolve the residue in 5 mL of 8M HNO_3.

7.5.3 Place a beaker below each column.

7.5.4 Pipette 5 mL of 8M HNO_3 into each column to condition resin and allow to drain.

7.5.5 Ensure that a clean beaker or vial is below the column.

7.5.6 Transfer the redissolved residue into the appropriate Sr Resin column by pouring or by using a plastic transfer pipette and allow to drain.

7.5.7 Add 5 mL of 8M HNO_3 to rinse to the beaker and transfer each solution into the Sr Resin column and allow to drain.

7.5.8 Repeat step 7.5.7.

7.5.9 Add 5 mL of 8M HNO_3 to the Sr Resin column and allow to drain.

7.5.10 Add 10 mL of 0.05M HNO_3 to the column to strip the Sr-90.

7.5.11 Prepare the Sr-90 from step 7.5.10 as appropriate for use as a calibration standard (evaporation on planchet, etc.).

7.5.12 Prepare the Y-90 in the load plus rinse solutions as appropriate for use as a calibration standard.

7.6 *Gas Proportional Counting Option:*

Note 4: Gas proportional counting provides lower detection limits than liquid scintillation counting or Cerenkov counting.

7.6.1 For each sample analyzed, clean a counting dish by moistening a paper towel with ethanol, wiping the dish and letting it dry.

7.6.2 Weigh the counting dish(s) on an analytical balance and record the weight.

7.6.3 Place each counting dish on a hot plate under a heat lamp in a hood.

7.6.4 Evaporate the column strip solution from step 7.4.9 onto each dish in successive 3 mL volumes.

7.6.5 Allow each 3 mL volume to evaporate to near dryness between additions.

7.6.6 Rinse the vial containing the column strip solution with 2 mL of 0.05M HNO_3 and transfer to the counting dish.

7.6.7 After all the solution has evaporated to dryness, cool each dish.

7.6.8 Reweigh each counting dish, and record the weight.

7.6.9 Count samples sufficient time to achieve the desired counting statistics and minimum detectable concentration.

7.6.10 After total strontium has been counted, set planchets aside in a safe place during the Y-90 ingrowth period.

7.7 *Cerenkov Counting Option:*

Note 5: This option gives somewhat poorer detection limits because of the relatively higher backgrounds of Cerenkov counting. However, it is fast and has virtually no interference between Sr-89 and Sr-90. It has been reported that high ratios of Sr-89/Sr-90 may cause a high bias with the gas proportional counting option. It is advisable to use the Cerenkov counting option in these cases.

7.7.1 Pour the column strip solution from step 7.4.9 into the Cerenkov counting vial.

7.7.2 Count the samples sufficient time to achieve the desired counting statistics and minimum detectable concentration.

7.7.3 Measure a blank vial before and after each sample group.

7.8 *Gamma Counting of Strontium-85 Tracer Option:*

7.8.1 Measure the Sr-85 on the counting dish or in the Sr strip solution using gamma pulse height analysis after counting the sample for beta activity.

7.8.2 Count the samples sufficient time to achieve the desired counting statistics (typically <5% rsd).

7.9 *Y-90 Isolation after Ingrowth:*

Note 6: If Sr-89 is known to be absent, Sr-90 can be measured with a single count. However, if Sr-89 is present, Sr-90 is measured by isolating Y-90 using this section and counting the Y-90.

7.9.1 Pipette 5 mL of 0.05M HNO_3 into each column from step 7.4.11, allow to drain, add 5 mL of 8M HNO_3 and allow to drain to condition the same column used for initial strontium separation.

Note 7: The 0.05M HNO_3 removes bismuth-210 ingrowth from any lead-210 that may be tightly bound to the resin.

7.9.2 Ensure that a clean beaker or vial is below the column.

7.9.3 After yttrium ingrowth of approximately 1 week, add 5 mL of concentrated HNO_3 to the Sr strip solution (Cerenkov counting option) or redissolve the evaporated Sr residue (gas proportional counting option) in up to 15 mL of 8M HNO_3. Transfer the solution into the appropriate Sr Resin column and allow to drain.

Note 8: This dissolution may be performed as follows: Place the planchet in a clean, dry 150 mL glass beaker. Add 5 mL of 8M HNO_3 to redissolve the residue, warm gently, and swirl. Remove the planchet with a pair of tweezers and rinse any remaining residue into the beaker twice with additional 5 mL volumes of 8M HNO_3. Warm the beaker gently if required to dissolve the residue.

7.9.4 Add 5 ml of 8M HNO_3 to rinse the beaker and transfer each solution into the Sr Resin column and allow to drain.

7.9.5 Record the time of the completion of last rinse as the stop time for yttrium ingrowth.

7.9.6 Prepare the Y-90 solution for counting as appropriate depending on the counting method used (section 7.6 or 7.7) and assume a 100% yield of yttrium.

8. Calculations

8.1 *Calculate the strontium yield using stable Sr carrier added or Sr-85 tracer:*

8.1.1 *Sr-85 tracer*

$$\text{Yield} = \frac{(C_s - B_s)}{E_s \times A_s}$$

where:

C_S = measured Sr-85 tracer, cpm
B_S = background, cpm
E_S = counting efficiency for Sr-85 tracer
A_S = Sr-85 tracer activity, dpm

8.1.2 *Gravimetric: Sr carrier*

$$\text{Yield} = \frac{R_w - T_w - B_w}{C_w}$$

where:

R_w = residue + dish, mg
T_w = tare weight of dish, mg
B_w = blank weight, mg (extractant loss from column)
C_w = $Sr(NO_3)_2$ added, mg

Percent yield = Yield x 100

8.2 *Calculate Sr-90 activity based on Y-90 ingrowth:*

Note: If Sr-89 is known to be absent, Sr-90 can be measured with a single count. However, if Sr-89 is present, Sr-90 is measured by counting the isolated Y-90 after ingrowth. This calculation applies to measurement by gas proportional counting, liquid scintillation counting or Cerenkov counting.

$$\text{Sr-90 concentration (dpm/L)} = \frac{S_y - B_y}{E_y \times V \times Y_{Sr} \times Y_y \times I_y \times D_y}$$

where:

S_y = count rate for yttrium, cpm
B_y = background count rate for yttrium, cpm
E_y = Y-90 counting efficiency
V = sample volume, L
Y_{sr} = strontium yield
Y_y = yttrium yield = assumed to be 1.0 (100%)
I_y = yttrium ingrowth factor = 1 - exp [(-ln2/2.6708) x (T_2-T_1)]
D_y = decay correction for Y-90 = exp [(-ln2/2.6708) x (T_4-T_2)]
T_0 = time of sample collection
T_1 = start time for yttrium ingrowth (last rinse-initial Sr separation)
T_2 = stop time for yttrium ingrowth (last rinse-Sr removal after ingrowth)
T_4 = midpoint of yttrium sample measurement

Note 10: Convert time differences from hours to days + fractions of days since decay factors are given in days.

Conversion of dpm/L to pCi/liter:

pCi/L=(dpm/L) /2.22

8.3 *Calculate Sr-89 by subtracting Sr-90 from Total Sr (89+90):*

Note 11: This calculation is used for gas proportional or liquid scintillation counting. Alternately, Sr-89 and Sr-90 can also be measured simultaneously by setting windows judiciously using liquid scintillation counting, except when high levels of Sr-89 relative to Sr-90 are present.

$$\text{Sr-89 cpm} = \frac{S_t - B - S_{Sr90cpm} - \left(S_{Sr90dpm} \times E_y \times I'_y\right)}{Y_{Sr}}$$

$$\text{Sr-89 concentration (dpm/L)(corrected for Sr-89 decay)} = \frac{\text{Sr - 89 cpm}}{V \times E_{Sr89} \times D_{Sr89}}$$

where:

S_t	=	total Sr-89 + Sr-90 cpm
B	=	background strontium count rate, cpm
Y_{sr}	=	strontium yield
E_y	=	Y-90 counting efficiency
$E_{sr\text{-}89}$	=	Sr-89 counting efficiency
$S_{Sr90cpm}$	=	Y-90 dpm x Sr-90 counting efficiency
$S_{sr90dpm}$	=	Y-90 dpm determined by measuring Y^{90} after ingrowth
I_y	=	yttrium ingrowth factor = 1 - exp [(-ln2/2.6708) x (T_3-T_1)]
$D_{Sr\text{-}89}$	=	decay correction for Sr-89= exp [(-ln2/50.5) x (T_3-T_0)]
T_0	=	time of sample collection
T_1	=	start time f or yttrium ingrowth (last rinse-initial Sr separation)
T2	=	stop time for yttrium ingrowth (last rinse-Sr removal after ingrowth)
T_3	=	midpoint of strontium sample measurement
V	=	volume, L

8.4 *Calculate Sr-89 activity using Cerenkov counting and Sr-85 tracer:*

Note 12: Sr-89 can be measured directly by Cerenkov counting with very little interference from Sr-90. This calculation is used for Sr-89 by Cerenkov counting with a correction for beta emission from Sr-85 tracer illustrated.

$$\text{Sr-89 concentration (dpm/L)} = \frac{(S_{Sr89} - B_{Sr89}) - (E_{Sr85} \times A_{Sr85})}{V \times Y_{Sr} \times E_{Sr89} \times D_{Sr89}} - \frac{J_{Sr90} \times [E_{Sr90} + (E_{ys} \times I_y)]}{E_{Sr89} \times D_{Sr89}}$$

where:

S_{Sr89} = count rate for strontium, cpm
B_{Sr89} = background count rate for strontium, cpm
E_{Sr89} = Sr-89 counting efficiency
V = sample volume, L
Y_{Sr} = strontium yield
E_{ys} = Y-90 counting efficiency
I_y = yttrium ingrowth factor = 1 - exp (-ln2/2.6708) x (T_3-T_1)
D_{Sr-89} = decay correction for Sr-89 = exp (-ln2/50.5) x (T_3-T_0)
J_{Sr-90} = Sr-90 activity decayed T_0 to T_3 = CSr-90 x exp (-ln2/28.6) x (T_3-T_0)/365.25
T_0 = time of sample collection
T_1 = start time for yttrium ingrowth (last rinse-initial Sr separation)
T_3 = midpoint of strontium sample measurement
C_{Sr-89} = Sr-90 concentration (dpm/L) at time T_0.
A_{Sr-85} = Sr-85 concentration (dpm/L) at time of sample measurement, corrected for chemical recovery of strontium.
E_{Sr-85} = Sr-85 counting efficiency

Conversion of dpm/L to pCi/liter:

pCi/L = (dpm/L) /2.22

9. Precision and Bias

9.1 *Precision* - Relative standard deviations of 8% at the 500 dpm level (Sr-90) and 8% at the 2000 and 10,000 dpm levels (Sr-90) have been reported.

9.2 *Bias* - Mean tracer recoveries, corrected for chemical yield, of 104% ± 8% (Sr-90) and 95% ± 8% (Sr-89) have been reported.

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