STRONTIUM-89/90 IN WATER

1. SCOPE

- 1.1. This is a method for the separation and measurement of strontium-89/90 in water.
- 1.2. This method does not address all aspects of safety, quality control, calibration or instrument set-up. However, enough detail is given for a trained radiochemist to achieve accurate and precise results for the analysis of the analyte(s) from the appropriate matrix, when incorporating the appropriate agency or laboratory safety, quality and laboratory control standards.

2. SUMMARY OF METHOD

2.1. Strontium is separated using Eichrom Sr Resin prior to gas flow proportional counting, liquid scintillation counting or Cerenkov counting. Cation exchange resin or calcium phosphate precipitation 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 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 HNO₃ 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 3M HNO₃-0.05M oxalic acid.

5. APPARATUS

- Beta detector, gas flow proportional, liquid scintillation or Cerenkov counter and appropriate counting planchet or vial.
- Column rack, Eichrom Part: AC-103
- Extension funnels, 25 mL, Eichrom Part: AC-120
- Fume hood
- Heat lamp
- Hot plate
- Plastic bottles or Glass Beakers, 1L
- Volumetric flask or cylinder, 1L

For Ion Exchange Preconcentration Option:

- Column reservoirs 250mL to 1L volume, Eichrom Part: AC-20X-20M
- Ion exchange columns 1 to 1.5 cm diameter, 10 mL resin volume, Eichrom Part: AC-20E-M

For Calcium Phosphate Precipitation Preconcentration Option:

- Centrifuge, with rotor and carriers for 50mL and 250mL tubes
- Centrifuge tubes, 50mL and 250mL

For Sr-85 yield tracer option:

Gamma spectrometry system

6. REAGENTS

Note: Analytical grade or ACS grade reagents are recommended. Evaluation of key reagents, such as aluminum nitrate and ammonium hydrogen phosphate, for contribution to method background levels from naturally occurring radioactive materials is recommended.

| Deionized water, all reagents are prepared using deionized water | |
|-----------------------------------------------------------------------------------------|--|
| Nitric acid (70%), concentrated HNO ₃ | |
| Oxalic acid dihydrate, $C_2H_2O_4\cdot 2H_2O$ | |
| Strontium nitrate, Sr(NO ₃) ₂ -or- ⁸⁵ Sr yield tracer | |
| <i>Sr[®] resin,</i> 2mL prepacked column, 100-150µm, Eichrom Part SR-C50-A | |
| If Counting Radiostrontium by Liquid Scintillation: | |
| in counting Radiostronitality Elquid Scintination. | |

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Liquid scintillation cocktail

If Preconcentrating Sr by Cation Exchange:

Cation exchange resin, hydrogen form, 100 to 200 mesh, Eichrom Part C8-B500-M-H

If Preconcentrating Sr by Calcium Phosphate Precipitation: Aluminum nitrate Nonahydrate, Al(NO₃)₃·9H₂O Ammonium hydrogen phosphate, (NH₄)₂HPO₄ Sodium Hydroxide, NaOH Calcium nitrate - Ca(NO₃)₂ Phenolphthalein pH indicator Isopropyl alcohol, C3H₇OH

- 6.1. *Nitric acid (3M)* Add 188mL of concentrated HNO₃ to 800mL of water. Dilute to 1L with water.
- 6.2. *Nitric acid (3M) oxalic acid solution (0.05M)-* Add 188mL of concentrated HNO₃ and add 6.3g of oxalic acid dihydrate to 800 mL of water and dilute to 1 liter with water.
- 6.3. *Nitric acid solution (0.05M)* Add 3.1mL of concentrated HNO₃ to 900mL of water. Dilute to 1 liter with water.
- 6.4. *Nitric acid solution (8M)* Add 500mL of concentrated HNO₃ to 400mL of water. Dilute to 1L with water.
- 6.5. Strontium (Sr) carrier (5 mg/mL), gravimetric Dissolve 12.1g $Sr(NO_3)_2$ 800mL of water. Dilute to 1L with water.
- 6.6. Prepare Only if Using Cation Exchange Preconcentration:
 - 6.6.1. *Nitric acid solution (0.1M)* Add 6.3mL of concentrated HNO₃ to 900mL of water. Dilute to 1L with water.
- 6.7. Prepare Only if Using Calcium Phosphate Precipitation Preconcentration:
 - 6.7.1. Ammonium hydrogen phosphate (3.2M)- Dissolve 106g of (NH₄)₂HPO₄ in 200mL of water. Heat gently to dissolve. Dilute to 250mL with water.
 - 6.7.2. *Calcium nitrate (1.25M)-* Dissolve 51g of Ca(NO₃)₂ in 100mL of water. Dilute to 250mL with water.
 - 6.7.3. *Phenolphthalein solution* Dissolve 1g phenolphthalein in 100mL 95% isopropyl alcohol. Dilute with 100mL of water.



6.7.4. Aluminum Nitrate (1M) - Dissolve 375g of Al(NO₃)₃⋅9H₂0 in 600mL of water. Dilute to 1L with water.

7. PROCEDURE

- 7.1. Water Sample Precipitation
 - 7.1.1. If samples larger than 1L are analyzed, evaporate the sample to approximately 1L.
 - 7.1.2. Measure the sample volume using a standard graduated cylinder (or equivalent) and transfer volume to an appropriate size plastic bottle (cation exchange) or glass beaker (calcium phosphate).
 - 7.1.3. Acidify the sample to pH 2 using concentrated nitric acid.
 - 7.1.4. Add 1 mL of 5mg/mL strontium carrier (for gravimetric yield option) or strontium-85 tracer (for gamma yield determination option) into each sample aliquot.
- 7.2. Preconcentration of Sr:
 - 7.2.1. Cation Exchange to Concentrate Strontium from Water Samples:

Note: Alternatively, evaporation to dryness (when insoluble residues such as calcium sulfate do not form) and calcium phosphate precipitation (7.2.2.) can be used to concentrate strontium.

- 7.2.1.1. Prepare a cation exchange column containing 10mL of C8-B500-M-H, 100-200 mesh in a 20mL plastic chromatography column for each sample analyzed. Slurry resin in ~20mL of deionized water. Pour slurry into empty column. Allow resin to settle and water to drain from each column.
- 7.2.1.2. Place columns on rack with large volume reservoirs (250mL to 1L).
- 7.2.1.3. Ensure that a suitable container is below each column.
- 7.2.1.4. Add 20mL of 0.1M HNO₃ to each column to precondition columns. Allow fluid to drain to the top of the column via gravity.
- 7.2.1.5. Load each sample onto the appropriate column and allow to drain. (Strontium will be retained by the column).

- 7.2.1.6. Rinse each column with 25mL of 0.1 M HNO₃.
- 7.2.1.7. Discard the feed and rinse the solution collected.
- 7.2.1.8. Ensure that a labeled 150mL beaker is below each column.
- 7.2.1.9. Add 50mL of 8M HNO₃ to each column to elute strontium.
- 7.2.1.10. Place each beaker on a hot plate in a fume hood and evaporate to dryness.
- 7.2.1.11. Dissolve residue in 10mL of 8M HNO₃.
- 7.2.2. Calcium Phosphate Precipitation Preconcentration Option:
 - 7.2.2.1. Add 2mL of 1.25M Ca $(NO_3)_2$ to each sample.
 - 7.2.2.2. If sample volume is greater than 200mL then place samples on a hotplate.
 - 7.2.2.3. Heat to 60-80°C.
 - 7.2.2.4. Add 0.75mL of phenolphthalein indicator and 5mL of 3.2M (NH4)₂HPO₄ solution per liter of sample.
 - 7.2.2.5. While stirring, slowly add enough 12M NaOH to reach the phenolphthalein end point (pH 9-10) and form a calcium phosphate precipitate. Continue heating and mixing for 15-20 minutes.
 - 7.2.2.6. Cool to room temperature. Allow precipitate to settle until solution can be decanted (30 minutes to 2 hours) or centrifuge in 250mL aliquots.
 - 7.2.2.7. Decant supernate and discard to waste.
 - 7.2.2.8. Dissolve precipitate in 7mL 70% HNO₃ and 8mL of 1M $AI(NO_3)_3$.
- 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, push the top frit down to the top of the resin bed, and allow each column to drain. (Save caps for later use during Y ingrowth).

- 7.3.4. Attach column reservoirs to each column or cartridge.
- 7.3.5. Add 5mL of 8M HNO_3 into each column reservoir to condition the Sr Resin. Allow solution to drain.
- 7.4. Sr Resin Column Separation:
 - 7.4.1. Transfer each dissolved sample into the appropriate Sr Resin column reservoir. Allow solution to drain.
 - 7.4.2. Add 5mL of 8M HNO_3 to rinse to each sample beaker or tube. Transfer the rinse solution into the appropriate Sr Resin column reservoir. Allow solution to drain.
 - 7.4.3. Rinse each column with 5mL of 3M HNO_3 0.05M oxalic acid. Allow solution to drain.

Note: The 3M HNO_3 - 0.05M oxalic acid removes Pu(IV), Np(IV), Zr(IV), or Ce(IV), which are retained by Sr Resin. If these interference's are known to be absent, this step may be skipped.

7.4.4. Add 5mL of 8M HNO₃ to each column reservoir. Allow the rinse solution to drain through each column.

Note: This additional 8M HNO₃ rinse removes any residual oxalic acid and ensures full removal of K^{\dagger} and Ba²⁺ that may be present.

- 7.4.5. Record the time when the last rinse completely drains through each column as the start of yttrium ingrowth.
- 7.4.6. Place clean, labeled centrifuge tubes below each column.
- 7.4.7. Add 15mL of 0.05M HNO $_3$ to each column reservoir to elute Sr. Allow solution to drain.
- 7.4.8. Ensure that calibration standards are prepared per step 7.5 and GOTO 7.6 or 7.7 to count samples.
- 7.4.9. Add 5mL of 0.05 M HNO₃ 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 in-growth is complete. Columns will be used again in section 7.9.
- 7.5. Preparation of Pure ⁹⁰Sr and Pure ⁹⁰Y for Counter Calibration Sources:
 - 7.5.1. Add an appropriate volume of calibrated ⁹⁰Sr standard solution (in equilibrium with ⁹⁰Y) to a small beaker, add 1mL of Sr carrier and evaporate the solution to dryness.

- 7.5.2. Redissolve the residue in 5mL of 8M HNO₃.
- 7.5.3. Place a waste vial below each column.
- 7.5.4. Add 5mL of 8M \mbox{HNO}_3 into each column to condition resin and allow to drain.
- 7.5.5. Ensure that a clean, labeled centrifuge tube is below the column for ⁹⁰Y collection in the following steps.
- 7.5.6. Transfer the redissolved residue into the appropriate Sr Resin column. Allow solution to drain.
- 7.5.7. Add 5mL of 8M HNO_3 to rinse the beaker. Transfer the beaker rinse into the appropriate Sr Resin column. Allow solution to drain.
- 7.5.8. Repeat step 7.5.7.
- 7.5.9. Add 5mL of 8M $\rm HNO_3$ to the Sr Resin column. Allow solution to drain.
- 7.5.10. Set centrifuge tube aside for ⁹⁰Y source preparation. Place a clean, labeled centrifuge tube below each column for ⁹⁰Sr recovery.
- 7.5.11. Add 10mL of 0.05M $\rm HNO_3$ to the column to strip the Sr-90. Allow solution to drain.
- 7.5.12. Prepare the ⁹⁰Sr fraction as appropriate for use as a calibration standard by evaporation on planchet or addition to liquid scintillation cocktail.
- 7.5.13. Prepare the ⁹⁰Y fraction as appropriate for use as a calibration standard by evaporation on a planchet, addition to liquid scintillation cocktail or rare earth fluoride micro precipitation (Eichrom Method SPA01).
- 7.6. Gas Proportional Counting Option for Sr Fractions:

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

7.6.1. For each sample analyzed, clean a cupped planchet with a paper towel moistened with ethanol. Let counting dishes dry.



Note: Planchets can also be annealed in an oven at 450°C for 1.5 hours. Properly annealed planchets will appear bronze/brown in color. Do not overheat planchets or they could become more susceptible to acid degradation.

- 7.6.2. Weigh the planchets on an analytical balance and record the weight.
- 7.6.3. Place each planchet on a hot plate in a hood. Arrange planchets so that each planchet is heated to approximately the same temperature (i.e. do not place some on outside edges of the hotplate with others in the middle).
- 7.6.4. Add 3-5mL of the column strip solution to the appropriate planchet, and evaporate onto each dish in successive 3-5mL volumes.
- 7.6.5. Allow each 3-5mL volume to evaporate to near dryness between additions.

Note: Adding more eluate before the planchet goes to complete dryness helps to prevent spattering.

- 7.6.6. Rinse the vial containing the column strip solution with 2mL of 0.05M HNO₃ and transfer to the counting dish.
- 7.6.7. After all the solution has evaporated to dryness, place planchets in an oven at 105-110°C for 30-60 minutes to ensure uniform drying. 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 ⁹⁰Y in-growth period.
- 7.7. Cerenkov Counting Option for ⁸⁹Sr:

Note: 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 ⁸⁹Sr and ⁹⁰Sr. It has been reported that high ratios of ⁸⁹Sr/⁹⁰Sr 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 7.4.7. into a liquid scintillation vial.
- 7.7.2. Count the samples in a liquid scintillation counter (without liquid scintillation cocktail) for a 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 ⁸⁵Sr on the counting dish or in the Sr strip solution using gamma spectrometry 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 (approximately 1 week)

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

7.9.1. Add 5mL of 0.05M HNO₃ into each column from step 7.4.9. Allow the liquid to drain from the column. Add 5mL of 8M HNO₃ to precondition each column. Allow the liquid drain.

Note: The 0.05M HNO₃ removes bismuth-210 in-growth from any lead-210 that may be tightly bound to the resin.

- 7.9.2. Ensure that a clean, labeled centrifuge tube is below the column.
- 7.9.3. Add 5mL of concentrated HNO₃ to the Sr strip solution (Cerenkov counting option) or redissolve the evaporated Sr residue (gas proportional counting option) in up to 15mL of 8M HNO₃. Transfer the solution into the appropriate Sr Resin column and allow to drain.

Note: This dissolution may be performed as follows: Place the planchet in a clean, dry 150mL glass beaker. Add 5mL of 8M HNO₃ 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 5mL volumes of 8M HNO₃. Warm the beaker gently if required to dissolve the residue.



- 7.9.4. Add 5mL 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 in-growth.
- 7.9.6. Prepare the combined eluate from 7.9.3. and 7.9.4. for ⁹⁰Y counting as appropriate for the counting method used (evaporation on planchet, addition to liquid scintillation cocktail or rare earth fluoride micro precipitation via Eichom Method SPA01). Assume a 100% yield of yttrium.

8. CALCULATIONS

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

Calculate the *Sr-85 tracer yield*:

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

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

Calculate the Gravimetric Yield: 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

Eichrom Technologies, LLC Analytical Procedure May 1, 2014 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.

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:

- $S_v = \text{count rate for yttrium, cpm}$
- $B_v =$ background count rate for yttrium, cpm
- $E_v = Y-90$ counting efficiency
- V' = sample volume, L
- $Y_{sr} = strontium yield$
- $Y_v =$ yttrium yield = assumed to be 1.0 (100%)
- I_v = yttrium in-growth factor = 1 exp [(-ln2/2.6708) x (T₂-T₁)]
- $\dot{D}_v = \text{decay correction for Y-90} = \exp [(-\ln 2/2.6708) \times (T_4 T_2)]$
- $T_0 = time of sample collection$
- $T_1 =$ 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:

$$pCi/L = (dpm/L) /2.22$$
$$pCi/L = 37 mBq/L$$

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, ⁸⁹Sr and ⁹⁰Sr can also be measured simultaneously by setting windows judiciously using liquid scintillation counting, except when high levels of ⁸⁹Sr relative to ⁹⁰Sr are present.

Sr-89 cpm =
$$\frac{\mathbf{S}_{t} - \mathbf{B} - \mathbf{S}_{Sr90cpm} - (\mathbf{S}_{Sr90dpm} \times \mathbf{E}_{y} \times \mathbf{I'}_{y})}{\mathbf{Y}_{Sr}}$$

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

where:

| S _t B Y _{sr} E _y E _{sr-89} S _{Sr90cpm} S _{sr90dpm} I _y | = = = | total Sr-89 + Sr-90 cpm background strontium count rate, cpm strontium yield Y-90 counting efficiency Sr-89 counting efficiency Y-90 dpm x Sr-90 counting efficiency Y-90 dpm determined by measuring Y^{90} after ingrowth yttrium in-growth factor = 1 - exp [(-ln2/2.6708) x (T ₃ -T ₁)] |
|--------------------------------------------------------------------------------------------------------------------------------------------------|-------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| T ₀ T ₁ | | time of sample collection start time f or yttrium ingrowth (last rinse-initial Sr separation) |
| T ₂ T ₃ | = | stop time for yttrium ingrowth (last rinse-Sr removal after ingrowth) midpoint of strontium sample measurement volume, L |
| v | _ | |

Calculate ⁸⁹Sr activity using Cerenkov counting and ⁸⁵Sr tracer:

Note 12: ⁸⁹Sr can be measured directly by Cerenkov counting with very little interference from ⁹⁰Sr. This calculation is used for ⁸⁹Sr by Cerenkov counting with a correction for beta emission from ⁸⁵Sr 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 |
| Υ _{sr} | = | strontium yield |
| E _{ys} | = | Y-90 counting efficiency |
| l _y | = | yttrium in-growth 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 $T_3 = CSr-90 \times exp(-ln2/28.6) \times (T_3-$ |
| 01 00 | | T ₀)/365.25 |
| T _o | = | 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, |
| 0.00 | | corrected for chemical recovery of strontium. |
| E _{Sr-85} | = | Sr-85 counting efficiency |
| | | |

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% \pm 8% (Sr-90) and 95% \pm 8% (Sr-89) have been reported.

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