Strontium

EPA APPROVED

Sr-01-RC

STRONTIUM-89 *(see Volume II)*

EPA Approved

Sr-02-RC

STRONTIUM-90

(see Volume II)

Sr-03-RC

STRONTIUM-90 IN ENVIRONMENTAL MATRICES

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APPLICATION

This procedure is applicable to the preparation, separation, and analysis of vegetation, water, air filters and soil.

Strontium is separated from calcium, other fission products and natural radioactive elements. Fuming HNO_3 separations remove the calcium and most of the other interfering ions. Radium, lead and barium are removed with barium chromate. Traces of other fission products are scavenged with iron hydroxide. After the ${}^{90}Sr + {}^{90}Y$ equilibrium has been attained, the ${}^{90}Y$ is precipitated as the hydroxide and converted to the oxalate for counting on a low-background gas proportional beta counter. Chemical yield is determined with ${}^{85}Sr$ tracer by counting in a gamma well detector.

SPECIAL APPARATUS

- 1. Teflon filter holder or filter funnel and sample mount see Specification 7.12.
- 2. Rings and discs see Specification 7.2.
- 3. Magnetic stirrers with Teflon-coated magnet bars.
- 4. Mylar film see Specification 7.3.
- 5. Glass fiber filters see Specification 7.8.
- 6. Fisher filtrator, Fisher Chemical Company, Pittsburgh, PA 15219-4785.

7. Polyethylene reference bottles, 30-mL narrow mouth to fit in a gamma well detector.

SPECIAL REAGENTS

- 1. Strontium carrier, 20 mg Sr mL⁻¹ dissolve 48.4 g Sr(NO₃)₂ in 1 L of 1:99 HNO₃.
- Yttrium carrier, 10 mg Y mL⁻¹ dissolve 12.7 g of highest purity Y₂O₃ in a minimal amount of HNO₃; use heat if necessary. Filter, if necessary, and add water to make 1 L of solution. See the APPENDIX for the yttrium carrier counting check.
- 3. Iron carrier, 5 mg Fe mL⁻¹ dissolve 5 g Fe wire in 1:1 HCl and dilute to 1 L with 1:99 HCl, or dissolve 34.7 g Fe(NO₃)₃·9H₂O in 1 L of 1:99 HNO₃.
- 4. Barium carrier, 10 mg Ba mL⁻¹ dissolve 9.5 g Ba(NO₃)₂ in water and dilute to 0.5 L.
- Barium buffer solution 500 mL 6<u>M</u> acetic acid (glacial HOAC) plus 1 L of 6<u>M</u> NH₄OAc plus 0.5 L Ba carrier (10 mg mL⁻¹).
- 6. Calcium carrier, 200 mg Ca mL⁻¹ dissolve 500 g calcium carbonate (CaCO₃) in a minimum of HCl and dilute to 1 L with water.
- ⁸⁵Sr tracer, about 7x10⁵ Bq L⁻¹, in a well counter, this tracer should provide about 150 counts sec⁻¹ mL⁻¹.
- 8. Sodium carbonate solution, 1M dissolve 106 g Na₂CO₃ in H₂O and dilute to 1L.
- 9. Sodium carbonate solution, $2\underline{M}$ dissolve 212 g Na₂CO₃ in H₂O and dilute to 1L.
- 10. Sodium chromate solution, 0.3M dissolve 50 g Na₂CrO₄ in H₂O and dilute to 1L.
- 11. 50% sodium hydroxide solution slowly dissolve 500 g NaOH in 500 mL of H_2O in a plastic liter bottle immersed in a beaker of ice water.

SAMPLE PREPARATION

A. Water

- 1. Transfer an aliquot of sample water to an appropriate sized beaker (use deionized water for the blank).
- 2. Add 1 mL (20 mg) strontium carrier to the blank and the sample.
- 3. Add exactly 1.00 mL of ⁸⁵Sr tracer to three 30-mL plastic bottles, the blank and the sample.
- 4. Fill the plastic bottles with equal volumes of 1<u>M</u> HCl. Set bottles aside. They will serve as references when determining the strontium yield.
- 5. Evaporate the samples to dryness. Add 10-mL volumes of concentrated nitric acid to the dried residue, and evaporate repeatedly to remove any trace of HCl due to the reagents added.
- Dissolve the final residue in 8<u>M</u> HNO₃. If the sample is not in a 400-mL beaker, quantitatively transfer the sample with water to a 400-mL beaker containing a Teflon-coated magnetic stirring bar. Dilute the sample to 200 mL with additional water.
- Place the beaker on a magnetic stirrer/hot plate and stir. Adjust the pH to 5-6 with 50% NaOH. While continuing to stir, add 15 mL of 2<u>M</u> Na₂CO₃. Heat the sample to just below boiling and stir for 30 min.
- 8. Remove the sample from the hot plate and allow the precipitate to settle overnight.
- 9. Place a small glass fiber filter backed with a Whatman No. 42 filter paper of the same size into a Büchner funnel. Mount the funnel on a 500 mL filter flask.
- 10. Filter the sample by vacuum through the funnel. Wash the carbonates retained on the filter with 1M Na₂CO₃ solution. Discard the filtrate.

- 11. Release the vacuum and transfer the funnel to a clean filter flask. Apply the vacuum. Dissolve the carbonates on the filter with hot 8M HNO₃. Wash the filter with water.
- 12. Transfer the filtrate to a 400 mL beaker, rinsing with 8M HNO₃. Evaporate the solution to dryness.
- 13. Proceed to Determination, Fuming HNO₃ Separation

B. Air Filters

- 1. Place the air filter in a 400-mL beaker (use a dry beaker for the blank).
- 2. Add exactly 1.00 mL of ⁸⁵Sr to three 30-mL plastic reference bottles, the blank and the sample.
- 3. Fill the plastic bottles with equal volumes of $1\underline{M}$ HCl. Set bottles aside. (The same three reference bottles may be used for water and air filters if the analyses are done simultaneously.)
- 4. Add 20 mg (1 mL) strontium carrier to the blank and the sample.
- 5. Add 150 mL HNO₃ and 50 mL HCl. Reflux on a hot plate until clear and colorless. Evaporate to a volume of \sim 100 mL.
- 6. Add 100 mL of deionized water. Cool the sample to room temperature. Filter by gravity through a Whatman No. 42 filter. Wash the filter with 8<u>M</u> HNO₃.
- 7. Evaporate the filtrate to dryness. Add 20 mL volumes of concentrated nitric acid to the dried residue, and evaporate repeatedly to remove HCl. Continue with Step 6, Section A, Water.

C. Soil (NaOH-HCl method) - see Note 1

- 1. Weigh out enough soil to generate an activity at least 10 times background (ideally 100 times) into an appropriate sized beaker containing a Teflon-coated magnetic stirring bar (see chart below). Add water to about a quarter of the beaker's volume and add 5-10 mL (100-200 mg) strontium carrier solution. Place the beaker on a magnetic stirrer.
- To each of three 30-mL plastic reference bottles and to the sample add 1.00 mL of ⁸⁵Sr tracer solution. Fill the reference bottles to the same level with 1<u>M</u> HCl.
- 3. Stir the sample. While continuing to stir, add a sufficient amount of 50% NaOH to make the solution 1<u>N</u> NaOH. (see chart below)
- 4. Cover with a watch glass and stir for 10 min. Reflux overnight on a warm hot plate.
- 5. Remove the beaker from the hot plate and allow to cool. While stirring, cautiously add HCl, 1 mL at a time until the reaction slows, to make the solution 6<u>M</u> acidic (see chart below). If analyzing highly calcareous soils, an additional quantity of HCl should be added to replace the acid required to decompose the carbonates. If necessary, add a few drops of n-octyl alcohol to reduce foaming.

Activity (Bq kg ⁻¹)	Sample size (g)	Beaker size (mL)	Water (mL)	Carrier (mL)	50% NaOH (mL)	HCl (mL)
~500	5-10	250	70	5	4	90
~100	15-20	400	100	5	6	130
~50	30-40	800	200	5	11	260
~10	100	1000	250	5	14	320

- 6. Digest the sample overnight on a warm hot plate. Remove the beaker from the hot plate and cool.
- 7. Filter the sample under vacuum using a Whatman No. 42 filter paper backed by a glass fiber filter. Wash with approximately 100 mL hot 6M HCl, followed by 60 mL hot H₂O.

- 8. Turn off the vacuum. Return the soil residue and the filter paper to the original beaker.
- 9. Quantitatively transfer the filtrate and washes to an appropriate sized beaker, rinsing with water, and place on a warm hot plate to reduce the volume.
- 10. Remove the filtrate from the hot plate. Add 20-50 mL of HNO₃. Cover with a watch glass and place back on the hot plate. Turn the heat up to high. Continue adding HNO₃ until the conversion is complete, as indicated by the absence of brown fumes after the addition of HNO₃.
- 11. Add water to about a quarter of the beaker's volume to the beaker containing the filter paper and soil residue. Repeat Steps 3-7.
- 12. Transfer the second filtrate to the beaker containing the original filtrate. Convert to HNO_3 as in Step 10.
- 13. Reduce the volume of the combined filtrates to \sim 50 mL or until salting out begins to occur. Add \sim 100 mL (or twice the volume) of water, stir and cool to room temperature. If cloudy, filter under vacuum through two glass fiber filters, washing with hot 1-2<u>M</u> HNO₃. Discard the filters.
- 14. Quantitatively transfer the filtrate to a large (800-1500 mL) beaker containing a magnetic stirring bar, rinsing with water. Place the beaker on a magnetic stirrer/hot plate and stir while warming the solution.
- 15. Add approximately 5 g of $H_2C_2O_4$ (oxalic acid) L⁻¹ and continue to stir until the salt completely dissolves.
- 16. While stirring, adjust the pH to 5.5-6.0 with NH₄OH. If the mixture turns brown due to the presence of FeO(OH), add just enough $H_2C_2O_4$ to bring back the green color and readjust the pH. Repeat this process, using decreasing quantities of $H_2C_2O_4$, until the last pH adjustment does not result in the brown color. (**Note**: At this point, there should be enough $H_2C_2O_4$ to precipitate the insoluble white oxalates and to complex the Fe⁺³ ion, but not enough to cause crystallization of the (NH₄)₂C₂O₄ upon cooling.) Finish by adding several grams of oxalic acid as excess and adjust the pH again. Stir for 30 min on a warm hot plate.

- 17. Turn off the stirrer, remove the beaker from the hot plate, and allow the precipitate to settle overnight.
- 18. Add 5 mL of Ca carrier solution (1g Ca), stir the supernatant very gently and allow the fresh precipitate to settle for 15-20 min.
- 19. Filter the sample by gravity through a large Whatman No. 42 filter paper. Wash the beaker with H₂O, adding the washes to the funnel. Wash the precipitate with water until the filtrate is colorless.
- 20. Transfer the filter paper and precipitate to a 600-mL beaker. Add 100 mL of HNO₃. Cover with a watch glass and wet ash the oxalates until clear and colorless or oxidation seems complete, adding more HNO₃ as necessary.
- 21. Add an equal volume of water and stir on a magnetic stirrer. If cloudy, filter through two glass fiber filters washing with 8M HNO₃, followed by water. Evaporate to dryness.
- 22. Proceed to Determination, Fuming HNO₃ Separations.

Note: This method was developed at the U.S. Department of Agriculture Soil Survey Laboratory, Soil Conservation Service, Beltsville, MD. Comparative soil analyses at EML showed that the ⁸⁵Sr tracer could be completely equilibrated with ⁹⁰Sr present in the soils when consecutively treated with NaOH and HCl. The NaOH-HCl method yielded results equal to those obtained with the complete dissolution method.

D. Vegetation (dry ashing).

 Weigh an aliquot of up to 10 g of vegetation into a tared 250-mL porcelain crucible. (Note: After ashing, several aliquots can be combined to provide the desired sample size.) Place each crucible in a muffle furnace with the crucible cover slightly ajar. Increase the temperature of the furnace at a rate of 0.80°C min⁻¹ to 250°C. Maintain this temperature for 30 minutes. Increase the temperature at a rate of 10°C min⁻¹ to 600°C. Maintain this temperature for 960 min to completely ash the sample. Cool the crucible and weigh it to determine the percent ash. Ash content for replicate crucibles should vary by not more than 4%.

- 2. Transfer the ashed vegetation to a beaker using 8M HNO₃ to dissolve the ash and rinse the crucible. Add 1 mL of Sr carrier (20 mg).
- 3. Add 1.00 mL of ⁸⁵Sr tracer to the blank, the sample and each of three 30-mL plastic reference bottles. Fill the bottles to the same level with 1<u>M</u> HCl.
- 4. Cover with a watch glass and reflux on a hot plate until there is no evidence of remaining organic matter, adding HNO_3 or H_2O_2 as necessary.
- 5. Evaporate to near dryness. Add 50 mL of 8<u>M</u> HNO₃. Filter by gravity through a Whatman No. 42 filter paper into a beaker, washing with 8<u>M</u> HNO₃. Continue with Step 6 below.

E. Vegetation (wet ashing).

- 1. Weigh an aliquot of vegetation into an appropriate sized beaker. (For 100-300 g, use a 3000-mL beaker.) Add 1 mL of Sr carrier (20 mg).
- 2. Add 1.00 mL of ⁸⁵Sr tracer to the blank, the sample and each of three 30-mL plastic reference bottles. Fill the bottles to the same level with 1<u>M</u> HCl.
- 3. Slowly add 500 mL of 8<u>M</u> HNO₃. Control the foaming, if necessary, by adding a few drops of n-octyl alcohol. Cover with a watch glass and place on a low temperature hot plate overnight to maintain a slow reaction, stirring as necessary to break up the foam. Gradually increase the temperature of the hot plate, adding HNO₃ and continuing to reflux until the reaction is complete, as indicated by the absence of brown nitrogen oxide gas.
- 4. Remove from the hot plate. Slowly add enough HCl to equal one third the volume of HNO_3 still in the beaker. Allow the mixture to react at room temperature for 15 min, then cover with a watch glass and heat on a low temperature hot plate overnight with occasional stirring.
- Remove the sample from the hot plate and add an equal volume of water. Allow the sample to cool to room temperature. Filter by gravity through a large Whatman No. 42 filter paper into a beaker. Wash with 8<u>M</u> HNO₃.

- 6. Evaporate the filtrate to dryness. Dissolve the residue in a minimum of 8M HNO₃. Quantitatively transfer the solution to a 400-mL beaker containing a Teflon-coated magnetic stirring bar, rinsing with H₂O. Dilute the solution to 200 mL with additional H₂O.
- Place the beaker on a magnetic stirrer/hot plate and stir. Adjust the pH to 5-6 with 50% NaOH. While continuing to stir, add 15 mL of 2<u>M</u> Na₂CO₃. Heat the sample to just below boiling and stir for 30 min.
- 8. Remove the sample from the hot plate and allow the precipitate to settle overnight.
- 9. Place a small glass fiber filter backed by a Whatman No. 42 filter paper of the same size into a Büchner funnel. Mount the funnel on a 500 mL filter flask.
- 10. Filter the sample by vacuum through the funnel. Wash the carbonates retained on the filter with 1M Na₂CO₃ solution. Discard the filtrate.
- 11. Release the vacuum and transfer the funnel to a clean filter flask. Apply the vacuum. Dissolve the carbonates on the filter with hot 8M HNO₃. Wash the filter with water.
- 12. Transfer the filtrate to a 400 mL beaker, rinsing with 8M HNO₃. Evaporate to dryness.
- 13. Proceed to Determination, Fuming HNO₃ Separations.

DETERMINATION

A. Fuming HNO₃ separations.

Dissolve the residual salt in H₂O and some fuming HNO₃, while stirring on a magnetic stirrer. When dissolved, add additional fuming HNO₃ to precipitate Sr(NO₃)₂. The first two separations require concentrations of > 75% HNO₃, subsequent separations require less HNO₃ (see chart below). Water and air filters usually require two separations. Large quantities of soils with a high Ca content may require up to five or more fuming HNO₃ separations.

Separation	Water (mL)	Fuming HNO ₃ (mL)	% HNO ₃	final volume (mL)
1st	40	25 + 195	80.1	260
2nd	60	25 + 195	76.0	280
3rd	40	25 + 115	75.4	180
4th	30	105	75.4	135
5th	23	77	74.9	100

- Place a <u>dry</u> (very important to prevent sample loss) 5.5-cm glass fiber filter (for smaller volumes a 4.25-cm filter) in a <u>dry</u> Büchner funnel and mount the funnel in a 1 L filter flask.
- 3. Suction filter the sample into the flask. Turn off the vacuum. Transfer the funnel to a Fisher filtrator, placing an appropriate sized beaker underneath (for the last filtration, use a 40-mL heavy-wall conical centrifuge tube (C-tube)). Apply a vacuum while dissolving the precipitate on the filter with water into the beaker. Use additional water to complete the transfer of any residue in the original beaker to the funnel and subsequently into the beaker or C-tube. Proceed with Step 4 or 5.
- 4. Evaporate the sample solution to dryness if another fuming HNO₃ separation is desired, and repeat Steps 1 to 3 using smaller volumes as indicated in the chart.

5. If the sample solution is now in a C-tube, place the tube in a hot water bath and adjust the volume to ~20 mL. Proceed with **First Milking**.

B. First milking.

- 1. Add 1 mL of iron carrier solution to the separated strontium fraction in the centrifuge tube. Stir the solution and place the tube in a 90°C water bath to warm.
- 2. While stirring, adjust the pH of the sample to 8 with NH_4OH . Remove the stirring rod, rinsing with H_2O . Remove the centrifuge tube from the water bath and cool to room temperature in a cold water bath.
- 3. Centrifuge the sample at 2000 rpm for 5 min. Decant the supernate into a second 40mL centrifuge tube. Reserve the supernate for Step 6 and note the hour and date of this initial OH⁻¹ precipitation as **first milk separation time.**
- 4. Dissolve the precipitate in the first centrifuge tube in a few drops of HCl and dilute to 10 mL with H₂O. Stir the solution and warm the tube in the hot water bath.
- 5. While stirring, adjust the pH of the sample to 8 with NH_4OH . Remove the stirring rod, rinsing with H_2O . Remove the centrifuge tube from the water bath and cool to room temperature in a cold water bath.
- 6. Centrifuge the sample at 2000 rpm for 5 min. Decant and combine the supernate with the supernate reserved from Step 3. Evaporate to reduce the volume to 20 mL. Discard the precipitate.
- 7. While stirring, add 4 mL of barium buffer solution to the sample. If necessary, adjust the pH of the sample to 5.5 with either 6M HCl or NH₄OH (see **Note 1**).
- 8. Return the centrifuge tube to the hot water bath. While stirring vigorously, add 1 mL of 0.3M Na₂CrO₄ dropwise to the sample (see **Note 2**). Allow the sample to digest in the hot water bath for 10 min or longer to allow a good precipitate to form.
- 9. Remove the stirring rod, rinsing with H_2O . Remove the sample tube from the hot water bath and cool in a cold water bath.

- 10. Centrifuge the tube at 2000 rpm for 5 min. Decant the supernate into a 30-mL polyethylene bottle. Discard the precipitate.
- 11. Add 10-15 drops of HCl and exactly 1.00 mL of yttrium carrier solution to the sample in the polyethylene bottle and enough water to bring the volume of the solution to the same level as in the reference bottles.
- 12. Proceed to Strontium-85 yield determination.

Notes:

- 1. The pH of the solution is critical at this point. Complete precipitation of BaCrO₄ will not occur in a more acidic solution and strontium will partially precipitate in more basic solutions.
- 2. If large quantities of barium are present in the sample, only a partial precipitation of the Ba as $BaCrO_4$ may occur. The sample is centrifuged and the supernate decanted into another 40-mL centrifuge tube. The precipitation is completed by the dropwise addition of 0.3M Na₂CrO₄ to the supernate and the analysis is continued with Step 10.

C. Strontium-85 yield measurement.

- 1. Measure the activity of the three reference aliquots, the blank and the sample with a NaI(Tl) crystal gamma detector, collecting at least 10⁴ counts.
- 2. After subtracting the background counts, calculate the ⁸⁵Sr yield of the sample by dividing the sample counts by the average of the three reference counts.
- 3. Store the sample for 2 weeks to allow ⁹⁰Y to reach secular equilibrium with ⁹⁰Sr (see **Note**).

Note: The **first milk separation time** noted in Step 3, **First milking**, is the start of the yttrium ingrowth period. In order to correct for less than complete buildup of 90 Y, a correction factor is included in the calculations.

D. Second milking.

- 1. Quantitatively transfer the sample from the polyethylene bottle to a 40-mL, heavywalled, conical centrifuge tube with a minimum of H_2O . Stir the solution and place the tube in a 90°C water bath to warm.
- 2. While stirring, adjust the pH of the sample to 8 with NH_4OH . Add six drops of H_2O_2 and heat for 1 h. Remove and rinse the stirring rod. Remove the centrifuge tube from the water bath and cool to room temperature in a cold water bath.
- 3. Centrifuge the sample for 5 min at 2000 rpm. Decant the supernate into another 40-mL centrifuge tube. Record the hour and date of the precipitation as **second milk separation time.**
- 4. Dissolve the precipitate in the centrifuge tube with a few drops of HCl and stir. Dilute the sample to 15 mL with H₂O. Stir the solution and warm the tube in the hot water bath.
- 5. While stirring, adjust the pH of the sample to 8 with NH₄OH. Remove and rinse the stirring rod. Remove the centrifuge tube from the water bath and cool to room temperature in a cold water bath.
- 6. Centrifuge the sample for 5 min. Decant and combine the supernate with the supernate reserved from Step 3. Return the combined supernates to the hot water bath and reduce the volume to 20 mL. Transfer to a 30-mL polyethylene bottle and set aside for possible future milking.
- 7. Add four drops of HCl to the precipitate and stir until it dissolves. Add 25 mL of H₂O, stir and heat in the hot water bath.
- 8. Add 1 mL of strontium carrier (20 mg Sr) to serve as a holdback carrier. While stirring, adjust the pH to 8 with NH₄OH.
- 9. Remove and rinse the stirring rod. Remove the sample tube from the hot water bath and cool in a cold water bath.
- 10. Centrifuge the tube at 2000 rpm for 5 min. Decant and discard the supernate.

- 11. Add three drops of HCl to dissolve the precipitate, and 25 mL of H_2O .
- 12. Stir the sample and place the tube in a hot water bath. Add 1 mL of saturated $H_2C_2O_4$ (oxalic acid) solution.
- Adjust the pH to 2-3 by the dropwise addition of NH₄OH with vigorous stirring. Digest the sample in the hot water bath for 1 h.
- 14. Rinse and remove the stirring rod from the tube, and cool it to room temperature in cold water.
- 15. Centrifuge the tube at 2000 rpm for 10 min. Decant and discard most of the supernate.
- 16. Dry a 2.8-cm Whatman No. 42 filter paper on a 110°C hot plate or in an 110°C oven, cool and weigh to the nearest 0.1 mg.
- 17. Using a Teflon filter funnel assembly, filter the precipitate by suction through the weighed 2.8-cm Whatman No. 42 filter paper, backed by a 2.8-cm glass fiber filter, completing the transfer with a minimum amount of water. Wash the precipitate with 95% ethanol.
- 18. With the suction on, remove the filter funnel. Carefully separate the Whatman filter with the precipitate from the glass fiber filter backing. Place the filter paper with the precipitate on a 110°C hot plate. Turn off the vacuum and discard the glass fiber filter. Discard the filtrate.
- 19. Dry the filter with precipitate to a constant weight to determine the gravimetric yield.
- 20. Carefully mount the filtered precipitate on a nylon disc, cover with Mylar, and fasten the assembly with a nylon ring.
- 21. Measure the sample in a low-level gas flow proportional counter, recording the hour and date of the beginning of the measurement period.
- 22. Collect at least 10,000 counts, disregarding the first 200 min (2 cycles) of counting to eliminate possible interference from any ²²²Rn progeny present due to the filtration process. Proceed to calculations.

E. Gravimetric yttrium yield measurement.

- 1. Standardize triplicate 10-mL aliquots of the original yttrium carrier solution each time a fresh batch is made by precipitating the oxalate as described above and filtering through a fine (grade F), tared, sintered, glass filter crucible that has been dried to a constant weight.
- 2. Determine the weight of the yttrium oxalate precipitated from the sample as described in Steps 15-19 of **Second Milking**. The yttrium yield is the ratio of the weight of the sample oxalate to the standardized weight of the oxalate precipitated from the carrier solution.

CALCULATIONS

The β counting data obtained from the ⁹⁰Y precipitate must be corrected to give the activity of the ⁹⁰Sr in the sample. The corrections include those for ingrowth of ⁹⁰Y, counter background, ⁹⁰Y efficiency, strontium yield, yttrium yield, and ⁹⁰Y decay. ⁹⁰Y beta emissions are very energetic and are always counted with approximately the same mass of precipitate, so no correction for self-absorption is necessary.

The strontium yield is ordinarily determined by measuring the recovery of ⁸⁵Sr tracer added to the sample. Since the ratio of sample counts to counts from an aliquot of the original ⁸⁵Sr tracer solution is used to determine yield, there is no need to know the radioactivity rate of the tracer or to apply decay corrections for ⁸⁵Sr.

The activity of a sample of ⁹⁰Sr over a time interval, t, is

$$\int_0^{t} A dt' = \int_0^{t} A_0 e^{-\lambda t'} dt'$$

where A_0 is the initial activity of the sample. Integrating and rearranging to solve for A_0 yields:

$$A_0 = \left(\int_0^{t} A dt' \right) \cdot \frac{\lambda}{1 - e^{-\lambda t}}$$

The half-life of ⁹⁰Sr is quite large (29 y), so A_0 is essentially constant throughout the period of chemical separation and analysis. After 2 weeks, a sample of ⁹⁰Sr will be in secular equilibrium with its daughter, ⁹⁰Y, and the activities of the two nuclides will be equal. The quantity under the integral sign in the last equation above is the (corrected) measured activity of the separated ⁹⁰Y over the time period from separation to end of counting:

$$\int_{0}^{t} \mathbf{A} dt' = \frac{\mathbf{N}_{y} - \mathbf{B} \cdot dt_{c}}{\mathbf{R}_{y} \cdot \mathbf{R}_{sr} \cdot \mathbf{I}_{y} \cdot \mathbf{D}_{y} \cdot \mathbf{E}_{c}}$$

To obtain A₀, this quantity is multiplied by the factor

$$\frac{\lambda_{Y}}{1-\mathrm{e}^{-\lambda_{Y}\cdot dt_{\mathcal{C}}}}$$

where:

- $\lambda_{\rm Y}$ = decay constant of ⁹⁰Y (0.0108 h⁻¹)
- $dt_c = total count time minus two 100-minute cycles (see Note)$
- $N_v =$ total counts from all cycles except the first two (see Note)
- B = counter background for the matrix used (⁹⁰Y-oxalate)
- $R_v =$ yttrium yield fraction
- R_{sr} = strontium yield fraction
- $I_y = {}^{90}Y$ ingrowth fraction = $1 e^{-0.0108 \cdot dt1 2}$ = fraction of ${}^{90}Y$ produced during the time from extraction of ${}^{90}Sr$ ("1st milk") to separation of ${}^{90}Y$ from ${}^{90}Sr$ ("2nd milk")
- $D_v = {}^{90}Y \text{ decay fraction} = e^{-0.0108 \cdot dt^2 c} {}_0$
- E_c = counter efficiency for ⁹⁰Y-oxalate (counts min⁻¹ dpm⁻¹)

$dt1 \rightarrow 2 = 1$ st milk to 2nd milk time

 $dt_{2} - c_0 = 2nd$ milk time to start of counting plus two 100-min cycles (see Note)

Note: The first two cycles are ignored to allow for the decay of short-lived beta-emitting daughters from any radon-222 that may have attached to the Y-oxalate mount during preparation.

The calculated activity of the blank is subtracted from the calculated activity of the sample. The result is converted to appropriate units and divided by the sample size to obtain the activity concentration of the sample.

To check the radiochemical purity of the ⁹⁰Y-oxalate precipitate, a weighted linear regression analysis is done on the counting data, with Ln (counts-background counts) plotted against time. The weighting factor is the variance of the dependent variable:

Weighting factor =
$$Var(In(c-c_{bkg})) = \frac{c + (\sigma_{c_{bkg}})^2}{(c-c_{bkg})^2}$$

where:

The slope of the weighted regression line is equal to λ_Y , the decay constant of 90 Y. The value for λ_Y obtained from the regression analysis is compared to the known value of 0.0108 h⁻¹.

All calculations are done by computer.

Counter Efficiency Counter Background Yield (Sr) Yield (Y) Blank	(%) (cps) (%) (%) (cps)	40 0.005 80 95
LLD (400 min)	(Bq)	0.007
LLD (1000 min)	(Bq)	0.004

LOWER LIMIT OF DETECTION (LLD)

APPENDIX

YTTRIUM CARRIER COUNTING CHECK

To varify that the carrier solution contains only stable yttrium, complete the following procedure:

- 1. Pipette 1 mL of ytttrium carrier into each of three 40-mL centrifuge tubes. Dilute to 20 mL with H_2O .
- 2. Heat in a water bath to about 90°C. While stirring, adjust the pH to 8 with NH_4OH . Digest for 10 min and cool in a cold water bath.
- 3. Centrifuge for 5 min. Decant and discard the supernate. Proceed with Steps 11-22 of **Second Milking**.

Sr-04-RC

STRONTIUM-90 IN WATER CONTAINING OTHER RADIOISOTOPES BY CERENKOV COUNTING

Contact Person(s) : Salvatore C. Scarpitta

APPLICATION

The following procedure is used in the EML Quality Assessment Program (QAP; Sanderson and Greenlaw, 1996) for water or gross alpha/beta samples containing ⁹⁰Sr. This procedure was developed by Jimmy Chang, Institute of Nuclear Energy Research, Taiwan, and was tested at EML using NIST ⁹⁰Sr/Y reference materials and ⁹⁰Sr contained in QAP water samples. It allows for the rapid determination of ⁹⁰Sr ($\beta_{max} = 0.546$ MeV) via its progeny, ⁹⁰Y ($\beta_{max} = 2.27$ MeV) in aqueous solutions by Cerenkov counting (see Procedure Ba-01-R; Scarpitta and Fisenne, 1996).

Cerenkov counting is applicable for β particles with maximum energies > 0.263 MeV. Alpha and gamma emitting nuclides are not detected. Immediately following separation of ⁹⁰Y from ⁹⁰Sr, a baseline count rate, C_b, is obtained to quantify any Cerenkov contribution by other nuclides that may be present in the sample.

Yield recovery is determined by adding 30 mg of Sr^{+2} carrier to the sample or by gamma counting the added ⁸⁵Sr tracer. The sample is Cerenkov counted at any two time intervals, t_1 and t_2 , to measure the ingrowth of ⁹⁰Y above C_b . Strontium-90 is calculated from the ingrowth of ⁹⁰Y. The Cerenkov counting efficiency for ⁹⁰Y in water is about 65-70%, whereas that of ⁹⁰Sr is < 0.3% for a 0-15 keV Cerenkov counting window. For a 20-min count time, the detection limit is about 6 mBq mL⁻¹ (0.16 pCi mL⁻¹) or 0.35 dpm mL⁻¹ with a relative standard deviation < 3%.

SPECIAL APPARATUS

Packard Tri-Carb 2250-CA liquid scintillation spectrometer

SPECIAL REAGENTS

- 1. NIST traceable ⁹⁰Sr/Y reference standard of known activity (about 1000 dpm g⁻¹) to determine the ⁹⁰Y Cerenkov counting efficiency.
- 2. TRU Resin Eichrom Industries, 8205 Cass Ave., Suite 107, Darien, IL 606651.

DETERMINATION

- 1. Dispense about 8-16 Bq of ⁹⁰Sr gravimetrically into either a 20 mL low ⁴⁰K borosilicate glass or plastic scintillation vial (see **Note 1**).
- 2. Add 10 mL of deionized water.
- 3. Prepare a blank using 10 mL of water.
- 4. Count both samples three times for 10-min each using the net average count rate to determine the ⁹⁰Y counting efficiency, E ⁹⁰Y (counts min⁻¹ dpm⁻¹).
- Preset the Packard Tri-Carb to Protocol 4 for Cerenkov determinations (Scarpitta and Fisenne, 1996). (Note: The Cerenkov counting window is typically 0-15 keV, although the full window, 0-2000 keV, may be used with a 50% increase in background.)
- 6. Obtain a sample containing an unknown amount of 90 Sr.
- 7. Add 30 mg of Sr^{++} carrier (as nitrate) to the sample for yield recovery.
- 8. Prepare an identical vial containing water as a Sr^{++} reference standard.
- 9. Reduce a premeasured amount, M_g (g), of sample to be tested to 10 mL to improve counting statistics.

Note :

 A wavelength shifter, ANSA (7-Amino 1,3 Naphthalene di-Sulfonic Acid) can be used to enhance the Cerenkov counting efficiency but is not recommended if strontium yield recovery is to be determined gravimetrically. Nuclides that produce a Cerenkov signal in 25 mM ANSA are shown in Figure 1 (see Scarpitta and Fisenne, 1996).

SEPARATION

- Separate the ⁹⁰Y from the ⁹⁰Sr by either oxalate precipitation (see Procedure Sr-03-RC) or EiChrom's TRU Spec extraction chromatographic resin. Record the separation date and time, t_o.
- Obtain a net baseline count rate for C_b immediately following ⁹⁰Y separation, using Protocol No. 4 on the Packard Tri-Carb 2250 CA counter and the Cerenkov counting window (0-15 keV).

CALCULATIONS

- 1. Recount the ⁹⁰Sr fraction three times a day over a 2-day period using the count rates (counts min⁻¹) CT_1 , CT_2 and CT_3 to calculate the ⁹⁰Sr activity in Step 2. The times t_1 , t_2 and t_3 are the number of hours after ⁹⁰Y separation in Step 1 of **Separation**.
- 2. Use the ⁹⁰Sr calculation as follows when ⁸⁹Sr is not present in the sample. (**Note**: A Basic computer program is provided in the Appendix to perform the ⁹⁰Sr calculations.)

$$A_{1}^{90} \operatorname{Sr} \left(\operatorname{Bq} \operatorname{kg}^{-1} \right) = \frac{\left(\operatorname{CT}_{2} - \operatorname{CT}_{1} \right) - \operatorname{C}_{b}}{60 \operatorname{x} \operatorname{M}_{s} \operatorname{x} \operatorname{E}^{90} \operatorname{Y} \operatorname{x} \left[\exp \left\{ -\lambda \left(t_{1} - t_{0} \right) \right\} - \exp \left\{ -\lambda \left(t_{2} - t_{0} \right) \right\} \right]}$$
(1)

$$A_{2}^{90} Sr \left(Bq kg^{-1} \right) = \frac{\left(CT_{3} - CT_{1} \right) - C_{b}}{60 x M_{s} x E^{90} Y x \left[exp \left\{ -\lambda \left(t_{1} - t_{0} \right) \right\} - exp \left\{ -\lambda \left(t_{3} - t_{0} \right) \right\} \right]}$$
(2)

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where:

$$\begin{split} \lambda &= \ ^{90}\text{Y decay constant - 0.01083 h}^{-1} \\ \text{E} \ ^{90}\text{Y} &= \ ^{90}\text{Y Cerenkov counting efficiency (counts min}^{-1} \text{ dpm}^{-1}) \\ M_{s} &= \ \text{mass of sample (kg)} \end{split}$$

- Obtain the average of the two ⁹⁰Sr activity concentrations, A₁ and A₂ from Step 2.
 (Note: A third count could be obtained with Equation 2 if modified accordingly.)
- 4. Using the sample vial and the Sr⁺² reference standard, precipitate the strontium as the carbonate, filter, dry and weigh each to obtain the yield recovery. Correct the value obtained in Step 3, dividing by the yield recovery factor. (Note: Gamma emitting ⁸⁵Sr can be added to the sample in Step 7 of Determination instead of Sr⁺².)

REFERENCES

Sanderson, C. G. and P. Greenlaw "Semi-Annual Report of the Department of Energy, Office of Environmental Management, Quality Assessment Program" USDOE Report EML-581, July (1996)

Scarpitta, S. C. and I. M. Fisenne "Cerenkov Counting as a Complement to Liquid Scintillation Counting" Appl. Radiat. Isot., <u>47</u>, 795-800 (1996)

Scarpitta, S. C. and I. M. Fisenne "Calibration of a Liquid Scintillation Counter for Alpha, Beta and Cerenkov Counting" USDOE Report EML-583, July (1996)

Cerenkov Efficiency in 10 ml of 25 mM ANSA (Plastic Vials)

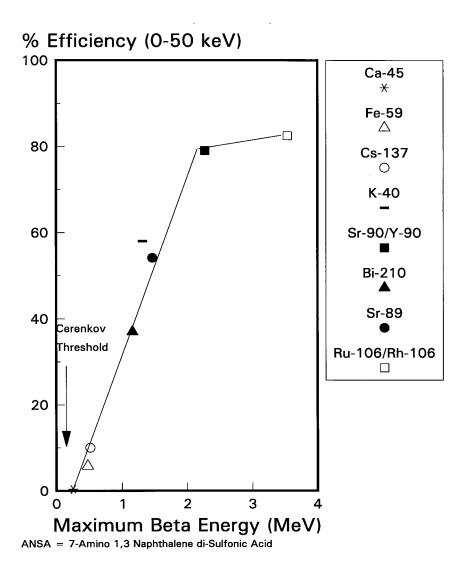


Figure 1. Cerenkov efficiency in 10 mL of 25 mM ANSA (plastic vials).

APPENDIX

Basic Computer Program

10	REM "SR90CER.BAS" BY J.CHANG METHOD
	REM CALCULATE 90SR ACTIVITY(DPM) FOR A SAMPLE USING CERENKOV COUNTING
	TITLE\$"Test of Cerenkov Method Using 895 dpm of NIST 90-Sr Standard"
	COUNTIMES="10 min"
	REM ENTER TIME DATA HERE
	DELTI=.75 ' HRS
	DELT2=19.22
	DELT3=111.6
	REM ENTER COUNT RATES HERE
	CT1=36.5
	CT2 = 159.1
	CT3=514, 4001
	LAMBD2 = $.01083$ ' HR-1
	EFFY90=.82 ' CPM/DPM
200	REM CALCULATE SR90 ACTIVITY(DPM) HERE
205	DF1 = EXP ((-LAMBD2*DELT1)) - EXP ((-LAMBD2*DELT2))
	A1SR90 = (CT2-CT1) / (DF1*EFFY90)
255	DF2 = EXP ((-LAMBD2*DELT1)) - EXP ((-LAMBD2*DELT3))
260	A2SR90 = (CT3-CT1)/(DF2*EFFY90)
500	REM PRINT RESULTS
510	CLS: SCREEN 2: KEY OFF
520	PRINT DATES;" ";
525	PRINT "STRONTIUM-90 RESULTS BY Sr-SPEC and CERENKOV COUNTING" : PRINT
530	PRINT TAB(10);TITLE\$: PRINT
535	PRINT TAB (15); "Count Time = ";COUNTIME\$; TAB (40) ;"Y-90 Effiency = ";EFFY90:
P	RINT : PRINT
540	PRINT "Data" : PRINT
550	PRINT TAB (1); "Del Ti" ;TAB (10); "Cnt Ti" ;TAB (30); "Del T2" ;TAB (40); "Cnt T2" ;TA (60) ;"Del T3" ;TAB (70);
	t T3"
555	PRINT TAB (1);" (hr) ";TAB (10);" (cpm) ";TAB (30);" (hr) ";TAB (40);" (cpm) ";TAB (60); (hr) ";TAB(70);" (cpm)":
PRI	NT
	PRINT TAB(1) ;DELT1;TAB(10) ;CT1;TAB(30) ;DELT2;TAB(40) ;CT2;TAB(60) ;DELT3;TAB(0) ;CT3
	PRINT : PRINT : PRINT "Sr-90 Results";
	PRINT TAB (30) ;A1SR90;" dpm" ;TAB(60) ;A2SR90;" dpm"
	PRINT TAB(1);"Obs/Exp";TAB(30);A1SR90/895;" ";TAB(60);A2SR90/895;
580	PRINT : PRINT : PRINT

02-21-1995 STRONTIUM-90 RESULTS BY Sr-SPEC and CERENKOV COUNTING

Test of. Cerenkov Method Using 895 dpm of NIST 90-Sr Standard

		Count Time 10 mi	n	Y-90 Effiency82		
Data						
Del T1 (hr)	Cnt T1 (cpm)		Del T2 (hr)	Cnt T2 (cpm)	Del T3 (hr)	Cnt T3 (cpm)
.75	36.5		19.22	159.1	111.6	514.4001
Sr-90 Resu Obs/Exp Ok	lts		831.421 dj .928962	pm	840.6198 .9392401	dpm

Sr-05-RC STRONTIUM-90 IN ENVIRONMENTAL WATER SAMPLES

Contact Person: Raymond J. Lagomarsino

APPLICATION

This procedure is applicable to the radiochemical analysis of ⁹⁰Sr in tap water, surface water, and rain water samples.

After addition of strontium carrier and ⁸⁵Sr tracer, strontium is separated from ¹³⁷Cs and other base soluble fission products by carbonate precipitation. The ⁹⁰Y and traces of activation and other fission products are scavenged with iron hydroxide. Radium, lead and barium are removed with barium chromate. The excess chromate from the barium precipitation is removed by a second strontium carbonate precipitation. Yttrium carrier is then added. After ⁹⁰Sr + ⁹⁰Y equilibrium has been attained, the ⁹⁰Y is precipitated as the hydroxide and converted to the oxalate for counting in a low-background gas proportional beta counter. Strontium recovery is determined by counting the ⁸⁵Sr tracer in a NaI(TI) gamma well detector.

The procedure provides strontium yields of > 80%. Gravimetric yields of yttrium oxalate are > 90%.

SPECIAL APPARATUS

- 1. Teflon filter holder or filter funnel and sample mount see Specification 7.12.
- 2. Rings and discs see Specification 7.2.
- 3. Mylar film see Specification 7.3.
- 4. Polyethylene bottles, 30 mL narrow mouth to fit in the gamma well detector (Wheaton No. 209045SP or equivalent).

- 5. Petri dish, glass, 4 quadrant (VWR, Bridgewater, NJ 08014 or equivalent).
- 6. Ionizing unit with positioner (static remover, VWR, Bridgewater, NJ 08014 or equivalent).

SPECIAL REAGENTS

- 1. Strontium carrier, 20 mg Sr^{+2} mL⁻¹ dissolve 48.4 g $Sr(NO_3)_2$ in 1 L of 1:99 HNO₃.
- 2 Yttrium carrier, 10 mg Y⁺³ mL⁻¹ (see Procedure Y-01-RC, Standardization of Yttrium Carrier).
- 3. Iron carrier, 5 mg Fe⁺³ mL⁻¹ dissolve 5 g of iron wire in 1:1 HCl and dilute to 1 L with 1:99 HCl, or dissolve 34.7 g Fe(NO₃)₃•9H₂O in 1 L of 1:99 HNO₃.
- 4. Barium carrier, 10 mg Ba⁺² mL⁻¹ dissolve 9.5 g Ba(NO₃)₂ in water and dilute to 0.5 L.
- Barium buffer solution mixture of 0.5 L 6<u>M</u> acetic acid (HOAc) plus 1 L of 6<u>M</u> NH₄OAc plus 0.5 L Ba carrier (10 mg mL⁻¹).
- 7. ⁸⁵Sr tracer, about 7 x 10^4 Bq L⁻¹ this tracer should provide about 25 counts sec⁻¹ mL⁻¹ in a NaI(Tl) gamma well detector with a ⁸⁵Sr efficiency of ~ 35.0%.
- 8. Sodium chromate solution, 0.3M dissolve 50 g Na₂CrO₄ in water and dilute to 1 L.
- 9. Sodium carbonate solution, saturated.
- 10. Ammonium oxalate solution, saturated.
- 11. Meta cresol purple indicator solution.
- 12. Aerosol OT solution 1.0%.

SAMPLE PREPARATION

A. Initial preparation and purification.

- 1. Transfer a measured quantity of water sample to an appropriate sized and labeled beaker. Prepare an equal volume of blank using deionized water (see **Note 1**).
- 2. Add 1.0 mL (20 mg) strontium carrier to the blank and each water sample.
- 3. Add 1.0 mL of ⁸⁵Sr tracer to three 30-mL plastic bottles, the blank and each sample.
- 4. Fill the plastic bottles with equal volumes of 1<u>M</u> HCl. Cap, shake to mix and set aside. They will serve as references when determining the strontium yield.
- Place a speedy-vap watch glass on the beakers, place the beakers on a hot plate and evaporate to reduce the volume to ~ 20 mL. Transfer each solution to a labeled 40-mL centrifuge tube using deionized water as a rinsing agent.
- 6. Adjust the pH of the solution to >8.5 with NH₄OH, while stirring with a glass rod.
- 7. Warm the tubes in a hot water bath and slowly add 10 mL of saturated Na₂CO₃ with stirring.
- 8. Digest the precipitate for ~ 20 min and cool to room temperature. Add several drops of 1.0 % Aerosol OT solution and centrifuge at 2000 rpm for ~ 5 min.
- 9. Decant and discard the supernate in an appropriate manner.
- 10. Add 10 mL of $3\underline{M}$ HNO₃ to dissolve the SrCO₃ precipitate in the centrifuge tube (stir well to break up the precipitate).
- 11. Boil the solution over a flame while stirring with a glass rod for ~ 1 min to evolve all CO₂, cool the tube, and proceed to **First Milking**.

B. First milking.

- 1. Add 1 mL (5 mg mL⁻¹) of iron carrier solution to the separated strontium fraction in the centrifuge tube.
- 2. While stirring with a glass stirring rod, adjust the pH of the sample to > 8.5 with NH₄OH. Digest the hydroxide precipitate in a hot water bath with occasional stirring for ~ 15 min. Remove the centrifuge tube from the hot water bath and cool to room temperature. Remove and rinse the stirring rod with water.
- Add several drops of 1.0% Aerosol OT solution, and centrifuge the sample at 2000 rpm for ~ 5 min. Decant the supernate into a second 40 mL centrifuge tube. (Note: Record the time and date of the decantation as the first milk separation time.)
- 4. Dissolve the Fe(OH)₃ precipitate in a few drops of HCl and dilute to 10 mL with water.
- 5. Repeat Step 2 of First Milking.
- 6. Add several drops of 1.0% Aerosol OT solution and centrifuge the sample at 2000 rpm for \sim 5 min.
- 7. Decant and combine the supernate with the supernate from Step 3; discard the precipitate.
- 8. Evaporate the combined supernates in a hot air bath to reduce the volume to $\sim 20 \text{ mL}.$
- 9. Add 4 mL of barium buffer solution to the combined supernates from Step 6.
- 10. Adjust the pH of the sample to 5.5 with either 6M HCl or NH₄OH, if necessary (see **Note 2**).
- 11. Heat the centrifuge tube in a hot water bath.
- Add 1 mL of 0.3<u>M</u> Na₂CrO₄ dropwise to the sample, while stirring vigorously (see Note 3).

- 13. Allow the precipitate to digest in the hot water bath for ~ 10 minutes.
- 14. Remove the sample tube from the hot water bath and cool to room temperature.
- 15. Remove and rinse the stirring rod with water.
- 16. Add several drops of 1.0% Aerosol OT solution, and centrifuge the sample tube at 2000 rpm for \sim 5 min.
- 17. Decant the supernate into a labeled 40 mL centrifuge tube and discard the precipitate.
- 18. Adjust the pH of the supernate to >8.5 with NH₄OH, heat in a hot water bath and slowly add 10 mL of saturated Na₂CO₃ solution with stirring.
- 19. Digest, with occasional stirring with a glass rod, in a hot water bath for ~ 15 min.
- 20. Remove the centrifuge tube from the hot water bath and cool to room temperature.
- 21. Remove and rinse the stirring rod with water.
- 22. Add several drops of 1% Aerosol OT solution, centrifuge at 2000 rpm for ~ 5 min, decant and discard the supernate in an appropriate manner.
- 23. Wash the precipitate with 10 mL of water, add several drops of 1.0% Aerosol OT solution, centrifuge at 2000 rpm for ~ 5 min and discard the wash.
- 24. Add 5 mL of $3\underline{M}$ HNO₃, boil the solution over a flame while stirring with a glass rod for about 1 min to evolve all the CO₂. Cool the solution to room temperature.
- 25. Add 1 mL of standardized yttrium carrier solution (10 mg Y⁺³ mL⁻¹) using a Class A pipette. Transfer the solution to a 30 mL polyethylene bottle using water as a rinsing agent.
- 26. Add enough water to bring the volume of the solution to the same liquid level as the ⁸⁵Sr reference bottles.
- 27. Cap the bottle and shake well.

28. Proceed to Strontium-85 yield determination.

C. Strontium-85 yield determination.

- 1. Measure the activity of the three reference aliquots, the blank and the sample with a NaI(Tl) gamma well detector. Count each bottle for total of 10⁴ counts.
- 2. Calculate the ⁸⁵Sr yield (strontium recovery) of the sample by dividing the sample counts by the average of the three reference counts, after subtracting the background counts.
- 3. Store the sample for 2 weeks to allow ⁹⁰Y to reach secular equilibrium with ⁹⁰Sr (see **Note 4**).

D. Second milking.

- 1. Transfer the sample from the polyethylene bottle to a 40 mL centrifuge tube.
- 2. Rinse the bottle with water and combine with the sample in the centrifuge tube.
- 3. Add 3 to 4 drops of meta-cresol purple indicator solution.
- 4. Precipitate $Y(OH)_3$ by the drop-wise addition of $6N NH_4OH$ until the indicator color changes from yellow to purple at pH 7.8 (see **Note 5**).
- Digest the precipitate in a hot water bath with occasional stirring for 15 min, cool to room temperature, add 3 to 4 drops 1.0% Aerosol OT solution and centrifuge at 2000 rpm for ~ 5 min.
- 6. Decant the supernate containing the strontium fraction into a clean 40 mL centrifuge tube and reserve for remilk.
- 7. Record the date and time of the decantation as the second milk separation time.
- 8. Dissolve the Y(OH)₃ precipitate in the centrifuge tube by adding a few drops of HCl, stir and dilute to 10 mL with water.

- 9. Add approximately 1 mL of strontium holdback carrier (20 mg Sr) and 3 to 4 drops of meta-cresol purple indicator solution and reprecipitate $Y(OH)_3$ with 6<u>M</u> NH₄OH (as in Step 4).
- 10. Heat the tube in a hot water bath with occasional stirring using a glass rod for ~ 10 min. Cool, add 3 to 4 drops of 1% Aerosol OT solution, centrifuge at 2000 rpm for ~ 5 min and combine the supernate with the strontium supernate from Step 6.
- 11. Repeat Steps 8 to 10 for a third time.
- 12. Acidify the combined supernates containing the strontium fraction with HCl and reduce to a volume of ~ 20 mL in a hot air bath. Transfer the solution to a 30 mL polyethylene bottle and set aside for possible remilking.
- 13. Add 2 mL of $1\underline{M}$ HCl and 1.5 mL of $6\underline{M}$ HNO₃ to the Y(OH)₃ and stir with a glass rod to completely dissolve the precipitate.
- 14. Heat to boiling over a flame while stirring and add 5 mL of saturated $(NH_4)_2C_20_4$ solution with stirring.
- 15. Stir for ~ 1/2 minute and gradually add an additional 10 mL of saturated (NH₄)₂C₂O₄.
- 16. Digest in a hot water bath for 20 min with intermittent stirring.
- 17. Remove the centrifuge tube from the hot water bath and cool to room temperature.
- Condition a 2.8 cm Whatman No. 42 filter paper by washing with ~ 10 mL of water and ~ 15 mL of 95% ethanol, using a Teflon filter funnel assembly.
- Place the filter paper in a labeled quadrant of the Petri dish, dry in an oven at 105°C for ¹/₂ hour and cool in a desiccator for ¹/₂ h.
- 20. Weigh the filter paper and record the weight to the nearest 0.1 mg.
- Filter the yttrium oxalate precipitate onto the previously washed, dried and weighed Whatman No. 42 filter paper using ~ 10 mL of water and and ~ 10 mL of 95% ethanol as transfer agents.

- 22. Place the filter paper and precipitate in a quadrant of a Petri dish, dry in an oven at 105° C for $\frac{1}{2}$ hour and cool to room temperature in a desiccator ($\frac{1}{2}$ h).
- 23. Weigh the filtered precipitate and record the weight to the nearest 0.1 mg.
- 24. Carefully mount the filtered precipitate on a nylon disc, cover with Mylar (after removing any static electricity from the Mylar with an ionizing unit), fasten the assembly with a nylon ring, trim the excess Mylar, and beta count.
- 25. Determine the yttrium yield from the ratio of the weight of the sample yttrium oxalate to the expected weight of yttrium oxalate as determined from the yttrium carrier standardization. [Note: The gravimetric factor for conversion of yttrium to yttrium oxalate is 3.397].
- 26. Calculate the sample ⁹⁰Y activity as in Procedure Sr-03-RC.

Notes:

- 1. The quantity of deionized water for the blank determination should be equivalent to the largest sample volume used.
- 2. The pH of the solution is critical at this point. Complete precipitation of $BaCrO_4$ will not occur in a more acidic solution and strontium will partially precipitate in more basic solutions.
- 3. If large quantities of barium are present in the sample, only a partial precipitation of the Ba as $BaCrO_4$ may occur. The sample is centrifuged and the supernate decanted into another 40 mL centrifuge tube. The precipitation is completed by the dropwise addition of 0.3 <u>M</u> Na₂CrO₄ to the supernate until no additional precipitate forms and the analysis is continued with Step16.
- 4. The **first milk separation time** noted in Step 3, **First milking**, is the start of the yttrium ingrowth period. A correction factor for the time between the first milk and second milk is applied in the calculation to correct for the ingrowth of ⁹⁰Y.
- 5. The indicator changes from red to yellow at pH 7.0 and from yellow to purple at pH 7.8.

LOWER LIMIT OF DETECTION (LLD)

Counter Efficiency	(%)	40
Counter Background	(cps)	0.005
Yield (Sr)	(%)	80
Yield (Y)	(%)	95
Blank	(cps)	-
LLD (400 min)	(Bq)	0.007
LLD (1000 min)	(Bq)	0.004