Sr-03-RC

STRONTIUM-90 IN ENVIRONMENTAL MATRICES

Contact Person : Marie Lawrence

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 $Sr(NO_3)_2$ in 1 L of 1:99 HNO_3.

2. Yttrium carrier, 10 mg Y mL^{-1} - dissolve 12.7 g of highest purity Y_2O_3 in a minimal amount of HNO_3; 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 $Fe(NO_3)_3 \cdot 9H_2O$ in 1 L of 1:99 HNO_3.

4. Barium carrier, 10 mg Ba mL^{-1} - dissolve 9.5 g $Ba(NO_3)_2$ in water and dilute to 0.5 L.

5. Barium buffer solution - 500 mL 6M acetic acid (glacial HOAC) plus 1 L of 6M NH_4OAc plus 0.5 L Ba carrier (10 mg mL^{-1}).

6. Calcium carrier, 200 mg Ca mL^{-1} - dissolve 500 g calcium carbonate ($CaCO_3$) in a minimum of HCl and dilute to 1 L with water.

7. ^{85}Sr tracer, about $7x10^5$ 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 Na_2CO_3 in H_2O and dilute to 1L.

9. Sodium carbonate solution, 2M - dissolve 212 g Na_2CO_3 in H_2O and dilute to 1L.

10. Sodium chromate solution, 0.3M - dissolve 50 g Na_2CrO_4 in H_2O 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 ^{85}Sr tracer to three 30-mL plastic bottles, the blank and the sample.

4. Fill the plastic bottles with equal volumes of 1M 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.

6. Dissolve the final residue in 8M HNO_3. 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.

7. 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 2M Na_2CO_3. 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 Na_2CO_3 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 HNO_3. Wash the filter with water.

12. Transfer the filtrate to a 400 mL beaker, rinsing with 8M HNO_3. Evaporate the solution to dryness.

13. Proceed to **Determination, Fuming HNO_3 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 ^{85}Sr to three 30-mL plastic reference bottles, the blank and the sample.

3. Fill the plastic bottles with equal volumes of 1M 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 HNO_3 and 50 mL HCl. Reflux on a hot plate until clear and colorless. Evaporate to a volume of ~ 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 8M HNO_3.

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.

2. To each of three 30-mL plastic reference bottles and to the sample add 1.00 mL of ^{85}Sr tracer solution. Fill the reference bottles to the same level with 1M HCl.

3. Stir the sample. While continuing to stir, add a sufficient amount of 50% NaOH to make the solution 1N 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 6M 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 H_2O.

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 HNO_3. Cover with a watch glass and place back on the hot plate. Turn the heat up to high. Continue adding HNO_3 until the conversion is complete, as indicated by the absence of brown fumes after the addition of HNO_3.

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 ~50 mL or until salting out begins to occur. Add ~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 1-2M HNO_3. 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 NH_4OH. 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 $(NH_4)_2C_2O_4$ 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 H_2O, 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 HNO_3. Cover with a watch glass and wet ash the oxalates until clear and colorless or oxidation seems complete, adding more HNO_3 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 HNO_3, followed by water. Evaporate to dryness.

22. Proceed to **Determination, Fuming HNO_3 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 ^{85}Sr tracer could be completely equilibrated with ^{90}Sr 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).

1. 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 HNO_3 to dissolve the ash and rinse the crucible. Add 1 mL of Sr carrier (20 mg).

3. Add 1.00 mL of ^{85}Sr tracer to the blank, the sample and each of three 30-mL plastic reference bottles. Fill the bottles to the same level with 1M 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 8M HNO_3. Filter by gravity through a Whatman No. 42 filter paper into a beaker, washing with 8M HNO_3. 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 ^{85}Sr tracer to the blank, the sample and each of three 30-mL plastic reference bottles. Fill the bottles to the same level with 1M HCl.

3. Slowly add 500 mL of 8M HNO_3. 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 HNO_3 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.

5. 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 8M HNO_3.

6. Evaporate the filtrate to dryness. Dissolve the residue in a minimum of 8M HNO_3. Quantitatively transfer the solution to a 400-mL beaker containing a Teflon-coated magnetic stirring bar, rinsing with H_2O. Dilute the solution to 200 mL with additional H_2O.

7. 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 2M Na_2CO_3. 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 Na_2CO_3 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 HNO_3. Wash the filter with water.

12. Transfer the filtrate to a 400 mL beaker, rinsing with 8M HNO_3. Evaporate to dryness.

13. Proceed to **Determination, Fuming HNO_3 Separations.**

DETERMINATION

A. Fuming HNO_3 separations.

1. Dissolve the residual salt in H_2O and some fuming HNO_3, while stirring on a magnetic stirrer. When dissolved, add additional fuming HNO_3 to precipitate $Sr(NO_3)_2$. The first two separations require concentrations of > 75% HNO_3, subsequent separations require less HNO_3 (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 HNO_3 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

2. Place a dry (very important to prevent sample loss) 5.5-cm glass fiber filter (for smaller volumes a 4.25-cm filter) in a dry 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 HNO_3 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 40-mL 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 H_2O. 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 NH_4OH (see **Note 1**).

8. Return the centrifuge tube to the hot water bath. While stirring vigorously, add 1 mL of 0.3M Na_2CrO_4 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 $BaCrO_4$ 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 Na_2CrO_4 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 10^4 counts.

2. After subtracting the background counts, calculate the ^{85}Sr 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 ^{90}Y to reach secular equilibrium with ^{90}Sr (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, heavy-walled, 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 H_2O. 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 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 H_2O, 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 NH_4OH.

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.

13. Adjust the pH to 2-3 by the dropwise addition of NH_4OH 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 ^{222}Rn 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 ß counting data obtained from the ^{90}Y precipitate must be corrected to give the activity of the ^{90}Sr in the sample. The corrections include those for ingrowth of ^{90}Y, counter background, ^{90}Y efficiency, strontium yield, yttrium yield, and ^{90}Y decay. ^{90}Y 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 ^{85}Sr tracer added to the sample. Since the ratio of sample counts to counts from an aliquot of the original ^{85}Sr 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 ^{85}Sr.

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

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

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

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

The half-life of ^{90}Sr 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 ^{90}Sr will be in secular equlibrium with its daughter, ^{90}Y, 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 ^{90}Y over the time period from separation to end of counting:

$$\int_0^t A dt' = \frac{N_y - B \cdot dt_c}{R_y \cdot R_{Sr} \cdot I_y \cdot D_y \cdot E_c}$$

To obtain A_0 , this quantity is multiplied by the factor

$$\frac{\lambda_Y}{1 - e^{-\lambda_Y \cdot dt_C}}$$

where:

λ_Y = decay constant of ^{90}Y (0.0108 h^{-1})

dt_c = total count time minus two 100-minute cycles (see **Note**)

N_y = total counts from all cycles except the first two (see **Note**)

B = counter background for the matrix used (^{90}Y-oxalate)

R_y = yttrium yield fraction

R_{sr} = strontium yield fraction

I_y = ^{90}Y ingrowth fraction = $1\text{-}e^{-0.0108 \cdot dt1 \rightarrow 2}$ = 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_y = ^{90}Y decay fraction = $e^{-0.0108 \cdot dt2 \rightarrow c}{}_0$

E_c = counter efficiency for ^{90}Y-oxalate (counts min^{-1} dpm^{-1})

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

$dt2 \rightarrow c_0$ = 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 ^{90}Y-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:

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

where:

C = sample counts

C_{bkg} = background counts

σ_{Cbkg} = standard deviation of background counts

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.

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

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 90ºC. 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**.