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**Rapid Radiochemical Method for
Total Radiostrontium (Sr-90) In Building
Materials
for Environmental Remediation Following
Radiological Incidents**

U.S. Environmental Protection Agency

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Total Radiostrontium (⁹⁰Sr) in Water: Rapid Method for High-Activity Samples

Revision History

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**RAPID RADIOCHEMICAL METHOD FOR TOTAL RADIOSTRONTIUM (SR-90)
IN BUILDING MATERIALS
FOR ENVIRONMENTAL REMEDIATION FOLLOWING RADIOLOGICAL INCIDENTS**

1. Scope and Application

- 1.1. The method will be applicable to samples where the source of the contamination is either from known or unknown origins. The method provides a very rapid screen for total radiostrontium ($^{89}\text{Sr} + ^{90}\text{Sr}$) in building materials samples.
- 1.2. This method is specific for beta-emitting isotopes of strontium in building materials such as concrete and brick. It uses rapid digestion and precipitation steps to preconcentrate strontium isotopes, followed by final purification using Sr Resin (see footnote 1 on next page) to remove interferences.
- 1.3. This method uses rapid radiochemical separations techniques for the determination of beta-emitting strontium radioisotopes in concrete or brick samples following a nuclear or radiological incident.
- 1.4. The method is capable of satisfying a required method uncertainty for ^{90}Sr (total as ^{90}Sr) of 0.31 pCi/g at an analytical action level of 2.4 pCi/g. To attain the stated measurement quality objectives (MQOs) (see Step 9.2), a sample weight of 1.5 g and a count time of approximately 1.5 hours are recommended. The sample turnaround time and throughput may vary based on additional project MQOs, the time for analysis of the final counting form, and initial sample volume. The method must be validated prior to use following the protocols provided in *Method Validation Guide for Qualifying Methods Used by Radiological Laboratories Participating in Incident Response Activities* (Reference 16.1).
- 1.5. This method is intended to be used for building materials. The rapid ^{90}Sr method was evaluated following the guidance presented for “Level E Method Validation: Adapted or Newly Developed Methods, Including Rapid Methods” in *Method Validation Guide for Qualifying Methods Used by Radiological Laboratories Participating in Incident Response Activities* (Reference 16.1) and Chapter 6 of *Multi-Agency Radiological Laboratory Analytical Protocols Manual* (MARLAP 2004, Reference 16.2).
- 1.6. Other solid samples such as soil can be digested using the rapid sodium hydroxide fusion procedure as an alternative to other digestion techniques, but this procedure will have to be validated by the laboratory.

2. Summary of Method

- 2.1. Strontium is collected and purified from the building materials sample matrix using sodium hydroxide fusion (Reference 16.3) and purified from potentially interfering radionuclides and matrix constituents using a strontium-specific, rapid chemical separation method. The sample is equilibrated with strontium carrier, and preconcentrated by Sr/ CaCO_3 coprecipitation from the alkaline fusion matrix. The carbonate precipitate is dissolved in HCl and strontium is precipitated with calcium fluoride to remove silicates. The precipitate is dissolved in strong nitric acid and the

solution is passed through a Sr Resin extraction chromatography column¹ that selectively retains strontium while allowing most interfering radionuclides and matrix constituents to pass through to waste. If present in the sample, residual plutonium and several interfering tetravalent radionuclides are stripped from the column using an oxalic/nitric acid rinse. Strontium is eluted from the column with 0.05M HNO_3 and taken to dryness in a tared, stainless steel planchet. The planchet containing the strontium nitrate precipitate is weighed to determine the strontium yield.

- 2.2. The sample test source is promptly counted on a gas flow proportional counter to determine the beta emission rate, which is used to calculate the total radiostrontium activity.
 - 2.2.1. The same prepared sample test source can be recounted after ~10 days to attempt to differentiate ^{89}Sr from ^{90}Sr . If the initial and second counts agree (based on the expected ingrowth of ^{90}Y), this is an indication that ^{89}Sr is not present in significant amounts relative to ^{90}Sr (within the uncertainty of the measurement).
 - 2.2.2. Computational methods are available for resolving the concentration of ^{89}Sr and ^{90}Sr from two sequential counts of the sample. An example of an approach that has been used successfully at a number of laboratories is presented in Appendix B to this method. It is the responsibility of the laboratory, however, to validate this approach prior to its use.
 - 2.2.3. It is also possible to determine ^{89}Sr more rapidly using Cerenkov counting if significant amounts of ^{89}Sr are suspected; this method must be validated independently. The minimum detectable activity (MDA) levels with Cerenkov counting, however, will be higher than of determination with gas proportional counting and may or may not meet measurement quality objectives.

3. Definitions, Abbreviations, and Acronyms

- 3.1. Analytical Protocol Specification (APS). The output of a directed planning process that contains the project's analytical data needs and requirements in an organized, concise form.
- 3.2. Analytical Action Level (AAL). The term analytical action level is used to denote the value of a quantity that will cause the decision-maker to choose one of the alternative actions.
- 3.3. Discrete Radioactive Particles (DRPs or "hot particles"). Particulate matter in a sample of any matrix where a high concentration of radioactive material is contained in a tiny particle (μm range).

¹ Sr-Resin[™] is a proprietary extraction chromatography resin consisting of octanol solution of 4,4'(5')-bis (t-butyl-cyclohexanol)-18-crown-6-sorbed on an inert polymeric support. The resin can be employed in a traditional chromatography column configuration (gravity or vacuum) or in a flow cartridge configuration designed for use with vacuum box technology. Sr-Resin[™] is available from Eichrom Technologies, Lisle, IL. Throughout the remainder of the method, the terms Sr-Resin or Sr-cartridge will be used for Sr-Resin[™].

- 3.4. Laboratory Control Sample (LCS). A standard material of known composition or an artificial sample (created by fortification of a clean material similar in nature to the sample), which is prepared and analyzed in the same manner as the sample. In an ideal situation, the result of an analysis of the laboratory control sample should be equivalent to (give 100 percent of) the target analyte concentration or activity known to be present in the fortified sample or standard material. The result normally is expressed as percent recovery.
 - 3.5. Matrix Spike (MS). An aliquant of a sample prepared by adding a known quantity of target analytes to specified amount of matrix and subjected to the entire analytical procedure to establish if the method or procedure is appropriate for the analysis of the particular matrix.
 - 3.6. *Multi-Agency Radiological Analytical Laboratory Protocols (MARLAP) Manual* provides guidance for the planning, implementation, and assessment phases of those projects that require the laboratory analysis of radionuclides (Reference 16.2).
 - 3.7. Measurement Quality Objective (MQO). MQOs are the analytical data requirements of the data quality objectives and are project- or program-specific. They can be quantitative or qualitative. MQOs serve as measurement performance criteria or objectives of the analytical process.
 - 3.8. Radiological Dispersal Device (RDD), i.e., a “dirty bomb.” This device is an unconventional weapon constructed to distribute radioactive material(s) into the environment either by incorporating them into a conventional bomb or by using sprays, canisters, or manual dispersal.
 - 3.9. Required Method Uncertainty (u_{MR}). The required method uncertainty is a target value for the individual measurement uncertainties and is an estimate of uncertainty (of measurement) before the sample is actually measured. The required method uncertainty is applicable below an AAL.
 - 3.10. Relative Required Method Uncertainty (ϕ_{MR}). The relative required method uncertainty is the u_{MR} divided by the AAL and is typically expressed as a percentage. It is applicable above the AAL.
 - 3.11. Sample Test Source (STS). This is the final form of the sample that is used for nuclear counting. This form is usually specific for the nuclear counting technique in the method, such as a solid deposited on a filter for alpha spectrometry analysis.
 - 3.12. Total Radiostrontium (also called Total Strontium): A radiological measurement that does not differentiate between ^{89}Sr and ^{90}Sr . The assumption is that all of the strontium is in the form of ^{90}Sr . When it is certain that no ^{89}Sr is present, the total radiostrontium activity is equal to the ^{90}Sr activity and may be reported as such.
 - 3.13. Working Calibration Source (WCS): A prepared source, made from a certified reference material (standard), including those diluted or prepared by chemical procedure, for the purpose of calibrating an instrument.
4. Interferences
 - 4.1. Radiological

- 4.1.1. Count results should be monitored for detectable alpha activity and appropriate corrective actions taken when observed. Failure to address the presence of alpha emitters in the sample test source may lead to high result bias due to alpha-to-beta crosstalk.
- Elevated levels of radioisotopes of tetravalent plutonium, neptunium, cerium, and ruthenium in the sample may hold up on the column and co-elute with strontium. The method employs an oxalic acid rinse that should address low to moderate levels of these interferences in samples.
- The resin has a higher affinity for polonium than strontium at low nitric acid concentrations but only minimal retention in 8M HNO_3 .
- If there were any residual Po (IV) retained, it would likely be removed using the 3M HNO_3 -0.05M oxalic acid rinse
- 4.1.2. Significant levels of ^{89}Sr in the sample will interfere with the total radiostrontium analysis.
- 4.1.2.1. The absence of higher activities of interfering ^{89}Sr may be detected by counting the sample test source quickly after initial separation (minimizing ingrowth of ^{90}Y), and then recounting the sample test source after 1–21 days to verify that the calculated activity (based on the expected ingrowth of ^{90}Y) does not change significantly. The presence of only ^{89}Sr may be indicated when the calculated activity of the second count is less than that of the first count by an amount greater than that which can be attributed to statistical variation in the two analyses. However, the second count activity is a complex function of the amount of the ^{89}Sr present, the ingrowth of the ^{90}Y from ^{90}Sr and the time between the first and second counts.
- 4.1.2.2. Alternatively, Appendix B provides a numerical approach for the isotopic determination ^{89}Sr and ^{90}Sr from two sequential counts of the sample, one immediately following separation, and one after a delay to allow for ingrowth of ^{90}Y and decay of ^{89}Sr .
- 4.1.3. High levels of ^{210}Pb may interfere with low-level strontium analysis due to ingrowth of short-lived ^{210}Bi during chemical separations, where Pb is retained by Sr Resin but is not eluted. If ^{210}Pb is known to be present in samples, minimizing the time between the final rinse and the elution of strontium to less than 15 minutes will maintain levels of interfering ^{210}Bi to less than 0.1% of the ^{210}Pb activity present. The presence or absence of interfering ^{210}Bi may be determined by recounting the sample test source to verify the half-life of the nuclide present. Bi-214 also can grow in during the elution step if ^{214}Pb (half life-26.8 minutes) makes it to the Sr Resin. In this case, holding the samples until the ^{214}Bi decays (~2 hours) may be advisable.
- 4.1.4. High levels of ^{228}Th or its decay progeny ^{224}Ra and ^{212}Pb may interfere with low-level strontium determinations due to ingrowth of short-lived decay products during chemical separations. Monitoring count data for alpha activity may provide indications of interferences. Minimizing the time

between the final rinse and the elution of strontium from the column to 5 minutes should maintain levels of interfering ^{212}Pb and ^{208}Tl to less than 2% of the parent nuclide activity. The presence or absence of ^{212}Pb may be determined by recounting the sample test source to verify the half-life of the nuclide present.

- 4.1.5. Levels of radioactive cesium or cobalt in excess of approximately 10^3 times the activity of strontium being measured may not be completely removed and may interfere with final results. Column rinsing to remove interferences may be increased to minimize interference if high levels of cesium or cobalt are known to be present. Changing cartridge connector tips and/or column reservoirs prior to final elution of strontium from Sr Resin can facilitate removal of sample matrix interferences. It may also be possible to increase the nitric acid in the eluted Sr fraction to 8M HNO_3 and reprocess the sample through the column separation method again.
- 4.2. Non-Radiological
 - 4.2.1. Stable strontium present in the concrete, brick or other solid sample at levels that are significant relative to the stable Sr carrier added will increase the apparent gravimetric yield and cause a negative bias in the final results. If the quantity of native strontium in the sample aliquant exceeds ~5% of the expected strontium carrier mass, chemical yield measurements will be affected unless the native strontium is accounted for in the yield calculations.
 - 4.2.2. The native strontium content in the sample may be determined by an independent spectrometric measurement (such as inductively coupled plasma - atomic emission spectroscopy [ICP-AES], etc.) or by taking an aliquant and processing the sample without the addition of strontium carrier to obtain an estimate of the native strontium content of the sample.
 - 4.2.3. Sr Resin has a greater affinity for lead than for strontium. Lead will quantitatively displace strontium from the column when the two are present in combined amounts approaching or exceeding the capacity of the column. If the combined quantity of lead and strontium carrier in the sample exceeds the capacity of the column, decreased strontium yields will be observed. High lead levels are not typically seen in concrete samples. However, decreasing the sample size will help address samples with elevated levels of lead.
 - 4.2.4. High levels of calcium, barium or potassium may compete slightly with strontium for uptake on the resin, possibly leading to low chemical yield. If these interfering matrix constituents are present in the final sample test source, yield results will overestimate the true strontium yield and cause a low result bias.
 - 4.2.5. Rinsing Sr Resin with 8M HNO_3 minimizes retention of Ba, K ions which have more retention at lower nitric acid levels to optimize removal of interferences.

- 4.2.6. The final solids on the planchets containing strontium nitrate should be white to very light brown. A significant brown color could indicate formation of iron oxide solids from the stainless steel planchets. This can cause a positive bias in the gravimetric chemical yields. Annealing the planchets properly minimizes the formation of iron oxide solids (Step 6.8).
5. Safety
 - 5.1. General
 - 5.1.1. Refer to your safety manual for concerns of contamination control, personal exposure monitoring and radiation dose monitoring.
 - 5.1.2. Refer to your laboratory's chemical hygiene plan for general chemical safety rules.
 - 5.2. Radiological
 - 5.2.1. Hot Particles (DRPs)
 - 5.2.1.1. Hot particles, also termed "discrete radioactive particles" (DRPs), will be small, on the order of 1 mm or less. Typically, DRPs are not evenly distributed in the media and their radiation emissions are not uniform in all directions (anisotropic).
 - 5.2.2. For samples with detectable activity concentrations of these radionuclides, labware should be used only once due to potential for cross contamination.
 - 5.3. Procedure-Specific Non-Radiological Hazards

None noted.
 6. Equipment and supplies
 - 6.1. Analytical balance with 10⁻⁴ g readability or better.
 - 6.2. Centrifuge able to accommodate 225 mL and 50 mL centrifuge tubes.
 - 6.3. Centrifuge tubes, 50 mL and 225 mL.
 - 6.4. Hot plate.
 - 6.5. Low-background gas flow proportional counter.
 - 6.6. 100 µL, 200 µL, 500 µL and 1 mL pipets or equivalent and appropriate plastic tips.
 - 6.7. 1-10 mL electronic pipet.
 - 6.8. Stainless steel planchets or other sample mounts: ~2-inch diameter, annealed at 530-550 °C or higher in a furnace for ~3.5 to 4 hours with a volume of ~5 mL. Planchets annealed properly will typically have a bronze/brown color. Do not overheat or this will make the planchets more susceptible to acid degradation and iron oxide formation.
 - 6.9. Tips, white inner, Eichrom part number AC-1000-IT, or PFA 5/32" × 1/4" heavywall tubing connectors, natural, Ref P/N 00070EE, cut to 1 inch, Cole Parmer, or equivalent.
 - 6.10. Tips, yellow outer, Eichrom part number AC-1000-OT, or equivalent.

6.11. Vacuum box, such as Eichrom part number AC-24-BOX, or equivalent.

6.12. Vacuum pump or laboratory vacuum system.

7. Reagents and Standards

NOTES:

All reagents are American Chemical Society (ACS) reagent grade or equivalent unless otherwise specified.

Unless otherwise indicated, all references to water should be understood to mean Type I reagent water (ASTM D1193, Reference 16.5). All solutions used in microprecipitation should be prepared with water filtered through a 0.45 μm (or better) filter.

7.1. Type I reagent water as defined in ASTM Standard D1193 (Reference 16.5).

7.2. Aluminum nitrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$)

7.2.1. Aluminum nitrate solution, 2M ($\text{Al}(\text{NO}_3)_3$): Add 750 g of aluminum nitrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) to ~700 mL of water and dilute to 1 L with water.

7.3. Ethanol, reagent 95% ($\text{C}_2\text{H}_5\text{OH}$), available commercially.

7.4. Nitric Acid, HNO_3 (15.8M), concentrated, available commercially.

7.4.1. Nitric acid (8M): Add 506 mL of concentrated HNO_3 to 400 mL of water and dilute to 1 L with water.

7.4.2. Nitric acid (3M): Add 190 mL of concentrated HNO_3 to 800 mL of water and dilute to 1 L with water.

7.4.3. Nitric acid (0.1M): Add 6.4 mL of concentrated HNO_3 to 900 mL water. Dilute to 1 L with water.

7.4.4. Nitric acid (0.05M): Add 3.2 mL of concentrated HNO_3 to 900 mL water. Dilute to 1 L with water.

7.5. Nitric acid (3M)/oxalic acid solution (0.05M): Add 190 mL of concentrated HNO_3 (7.3) and 6.3 g of oxalic acid dihydrate ($\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$), to 800 mL of demineralized water and dilute to 1 L with de-ionized water.

7.6. Sr Resin columns, ~1.00 g resin, 3 mL, small particle size (50–100 μm), in appropriately sized column or stacked 2 mL+ 1 mL pre-packed cartridges. (Available from Eichrom Technologies, Inc., Lisle IL.)

7.7. Strontium carrier solution, 7 mg/mL in 0.1M HNO_3 , traceable to a national standards body such as NIST or standardized at the laboratory by comparison to independent standards.

7.7.1. Option 1: Dilute elemental strontium standard to a concentration of 7.00 mg/mL (or mg/g) in 0.1M HNO_3 . Verify per Step 7.7.3.

7.7.2. Option 2: To ~200 mL de-ionized water, add 6.3 mL HNO_3 and approximately 16.90 g of strontium nitrate ($\text{Sr}(\text{NO}_3)_2$ dried to constant mass and the mass being determined to at least 0.001 g). Dilute to 1000 mL with water. Calculate the amount of strontium nitrate/mL actually present and verify per Step 7.7.3.

- 7.7.3. Prior to use, verify the strontium carrier solution concentration by transferring at least five 1.00-mL portions of the carrier to tared stainless steel planchets. Evaporate to dryness on a medium heat on a hotplate using the same technique as that used for samples (Ex. Heat 5 minutes after dryness is reached to ensure complete dryness). Allow to cool and weigh as the nitrate to the nearest 0.1 mg. The relative standard deviation for replicates should be less than 5% and the average residue mass within 5% of the expected value.
 - 7.8. ⁹⁰Sr standard solution (carrier free), traceable to a national standards body such as NIST, in 0.5M HNO₃ solution.
8. Sample Collection, Preservation, and Storage
Not Applicable.
9. Quality Control
 - 9.1. Batch quality control results shall be evaluated and meet applicable Analytical Protocol Specifications (APS) prior to release of unqualified data. In the absence of project-defined APS or a project-specific quality assurance project plan (QAPP), the quality control sample acceptance criteria defined in the laboratory quality manual and procedures shall be used to determine acceptable performance for this method.
 - 9.1.1. A laboratory control sample (LCS) shall be run with each batch of samples. The concentration of the LCS should be at or near the action level or a level of interest for the project.
 - 9.1.2. One method blank shall be run with each batch of samples fused using the procedure *Rapid Method for Sodium Hydroxide Fusion of Concrete and Brick Matrices Prior to Americium, Plutonium, Strontium, Radium, and Uranium Analyses* (Reference 16.3). If analyte-free blank material is not available and an empty crucible is used to generate a reagent blank sample, it is recommended that 100 mg calcium be added as calcium nitrate to the empty crucible as blank simulant. This facilitates strontium carbonate precipitations from the alkaline fusion matrix.
 - 9.1.3. One laboratory duplicate shall be run with each batch of samples. The laboratory duplicate is prepared by removing an aliquant from the original sample container.
 - 9.1.4. A matrix spike sample may be included as a batch quality control sample if there is concern that matrix interferences, such as the presence of elemental strontium in the sample, may compromise chemical yield measurements, or overall data quality.
 - 9.2. This method is capable of achieving a u_{MR} of 0.31 pCi/g at or below an action level of 2.4 pCi/g. This may be adjusted if the event-specific MQOs are different.
 - 9.3. This method is capable of achieving a required relative method uncertainty, ϕ_{MR} , 13% above 2.4 pCi/g. This may be adjusted if the event-specific MQOs are different.

- 9.4. This method is capable of achieving a required minimum detectable concentration (MDC) of 0.41 pCi/g.

10. Calibration and Standardization

- 10.1. The effective detection efficiency for total radiostrontium (referenced to ^{90}Sr) is calculated as the weighted sum of the ^{90}Sr and ^{90}Y efficiencies that reflects the relative proportions of ^{90}Y and ^{90}Sr based on the ^{90}Y ingrowth after ^{90}Sr separation.
- 10.2. Set up, operate, and perform quality control for gas-flow proportional counters (GPC) in accordance with the laboratory's quality manual and standard operating procedures, and consistent with ASTM Standard Practice D7282, Sections 7-13 (Reference 16.4).
- 10.3. See Appendix A for details on calibration/standardization of the GPC specific to ^{90}Sr and ^{90}Y .

11. Procedure

11.1. Initial Sample Preparation for $^{89}\text{Sr} + ^{90}\text{Sr}$

- 11.1.1. $^{89,90}\text{Sr}$ may be preconcentrated from building material samples using the separate procedure (Reference 16.3), which fuses the samples using rapid NaOH fusion followed by carbonate and fluoride precipitations to preconcentrate $^{89,90}\text{Sr}$ from the hydroxide matrix.

NOTE: The fusion procedure provides a column load solution for each sample (consisting of 20 mL of 8M HNO_3 -0.5M $\text{Al}(\text{NO}_3)_3$), ready for column separation on Sr Resin.

- 11.1.2. This separation can be used with other solid sample matrices if the initial sample preparation steps result in a column load solution containing ~8M HNO_3 - 0.5M $\text{Al}(\text{NO}_3)_3$ is used.
- 11.1.3. A smaller volume of the total load solution may be taken and analyzed as needed for very high activity samples, with appropriate dilution factor calculations applied.

11.2. Rapid Sr Separation using Sr Resin

11.2.1. Set up vacuum box

- 11.2.1.1. Place the inner tube rack (supplied with vacuum box) into the vacuum box with the centrifuge tubes in the rack. Place the lid onto the vacuum box system.
- 11.2.1.2. Place the yellow outer tips into all 24 openings of the lid of the vacuum box. Fit in the inner white tip into each yellow tip.
- 11.2.1.3. For each sample solution, place the Sr Resin cartridges (2 mL+1 mL cartridges) on to the inner white tip.
- 11.2.1.4. Place reservoirs on the top end of the Sr Resin cartridge.
- 11.2.1.5. Turn the vacuum on (building vacuum or pump) and ensure proper fitting of the lid.

IMPORTANT: The unused openings on the vacuum box should be sealed. Yellow caps (included with the vacuum box) can be used to plug unused white tips to achieve good seal during the separation. Alternately, plastic tape can be used to seal the unused lid holes as well.

11.2.1.6. Add 5 mL of 8M HNO₃ to the column reservoir to precondition the Sr Resin cartridges.

11.2.1.7. Adjust the vacuum to achieve a flow-rate of ~1 mL/min.

NOTE: Unless otherwise specified in the procedure, use a flow rate of ~ 1 mL/min for load and strip solutions and ~ 2-3 mL/min for rinse solutions.

11.2.2. Sr Resin Separation

11.2.2.1. Transfer each sample solution from the fusion procedure (Reference 16.3) into the appropriate reservoir. Allow solution to pass through the Sr Resin cartridge at a flow rate of ~1 mL/min.

11.2.2.2. Add 5 mL of 8M HNO₃ to each beaker/tube (from Step 11.2.2.1) as a rinse and transfer each solution into the appropriate reservoir (the flow rate can be adjusted to ~2 mL/min).

11.2.2.3. Add 15 mL of 8M HNO₃ into each reservoir as second column rinse (flow rate ~3–4 mL/min).

11.2.2.4. Turn off vacuum and discard rinse solutions.

11.2.2.5. Add ~ 5 mL 3M HNO₃ - 0.05M oxalic acid solution to each column (flow rate ~1–2 mL/min).

11.2.2.6. Add 5 mL of 8M HNO₃ into each reservoir as second column rinse (flow rate ~3 mL/min).

11.2.2.7. Discard column rinses.

11.2.2.8. Record time and date of the end of last rinse to the nearest 15 minutes as t_1 , "time of strontium separation."

11.2.2.9. Place clean 50 mL centrifuge tubes beneath the columns to catch the strontium eluate before proceeding to the next step.

11.2.2.10. Elute strontium from the columns by adding 15 mL of 0.05M HNO₃ at ~1 mL/min.

11.2.2.11. Discard Sr Resin cartridges.

11.2.3. Preparation of the STS and determination of chemical yield

11.2.3.1. Clean and label a stainless steel planchet for each STS.

11.2.3.2. Weigh and record the tare mass of each planchet to the nearest 0.1 mg.

11.2.3.3. Transfer the strontium eluate from Step 11.2.2.10 to the planchet and take to dryness on a hotplate (medium heat) to produce a uniformly distributed residue across the bottom of the planchet.

NOTE: A few mL at a time typically is added to the planchet during evaporation. Do not evaporate all the way to dryness to prevent

splattering. After adding all 15 mL, take the planchet all the way to dryness.

- 11.2.3.4. Rinse tubes with ~ 2 mL 0.05M HNO_3 and add to the planchet.
- 11.2.3.5. Heat on hot plate for ~5-10 minutes after initial dryness is reached.
- 11.2.3.6. Allow planchets to cool.
- 11.2.3.7. Weigh and record the gross mass of each planchet to the nearest 0.1 mg.

NOTE: If gravimetric yields are unusually high with the possibility of moisture present, additional heating and reweighing should be performed.

- 11.2.3.8. Calculate the chemical yield as presented in Step 12 of this method.

11.3. Counting the Sample Test Source

- 11.3.1. On a calibrated gas-flow proportional detector that has passed all required daily performance and background checks, count the STS for a period as needed to satisfy MQOs.
 - 11.3.1.1. If the presence of ^{89}Sr cannot be excluded, and total radiostrontium is being determined as a screen for the presence of ^{89}Sr or ^{90}Sr , count the STS as soon as practicable after preparation to minimize the ingrowth of ^{90}Y into the STS.
 - 11.3.1.2. If the presence of ^{89}Sr can be excluded, total radiostrontium will provide isotopic ^{90}Sr results and the STS may be counted at any time after preparation (taking into account the appropriate increase in activity due to ^{90}Y ingrowth).
- 11.3.2. Calculate the total radiostrontium (^{90}Sr) sample results using calculations presented in Step 12.
- 11.3.3. Hold planchets for recounting as needed.

12. Data Analysis and Calculations

12.1. Calculation of Total Radiostrontium

- 12.1.1. When a sample is analyzed for total radiostrontium (equivalent ^{90}Sr), the effective efficiency is calculated as follows:

$$\varepsilon_{\text{Total Sr}} = \varepsilon_{\text{Sr90}} + \left(1 - e^{-\lambda_{\text{Y90}}(t_2 - t_1)}\right) \times \varepsilon_{\text{Y90}} \quad (1)$$

where

- $\varepsilon_{\text{Total Sr}}$ = effective detection efficiency for total radiostrontium referenced to ^{90}Sr
- $\varepsilon_{\text{Sr90}}$ = final ^{90}Sr detection efficiency
- ε_{Y90} = final ^{90}Y detection efficiency
- λ_{Y90} = decay constant for ^{90}Y , 3.005×10^{-6} seconds (s)⁻¹

t_1 = date and time of the Sr/Y separation (s)
 t_2 = date and time of the midpoint of the count (s)

NOTE: The elapsed time between the sample count and the reference date must be calculated using the same time units as the decay constant.

12.1.2. The standard uncertainty of the effective efficiency is calculated as follows:

$$u(\varepsilon_{\text{Total Sr}}) = \sqrt{u^2(\varepsilon_{\text{Sr90}}) + \left(1 - e^{-\lambda_{\text{Sr90}}(t_2-t_1)}\right)^2 u^2(\varepsilon_{\text{Y90}}) + 2\left(1 - e^{-\lambda_{\text{Sr90}}(t_2-t_1)}\right) u(\varepsilon_{\text{Sr90}}, \varepsilon_{\text{Y90}})} \quad (2)$$

where

$$u(\varepsilon_{\text{Sr90}}, \varepsilon_{\text{Y90}}) = r(\varepsilon_{\text{Sr90}}, \varepsilon_{\text{Y90}}) u(\varepsilon_{\text{Sr90}}) u(\varepsilon_{\text{Y90}})$$

NOTE: The terms $u(\varepsilon_{\text{Sr90}}, \varepsilon_{\text{Y90}})$ and $r(\varepsilon_{\text{Sr90}}, \varepsilon_{\text{Y90}})$ are derived during calibrations as shown in Appendix A, Step A4.

12.1.3. The total radiostrontium activity concentration ($AC_{\text{Total Sr}}$) equivalent to ⁹⁰Sr is calculated as follows:

$$AC_{\text{Total Sr}} = \frac{R_a - R_b}{2.22 \times \varepsilon_{\text{Total Sr}} \times Y \times W \times DF} \quad (3)$$

where

$$DF = e^{-\lambda_{\text{Sr90}}(t_1-t_0)}$$

and where

R_a = beta gross count rate for the sample (counts per minute [cpm])
 R_b = beta background count rate (cpm)
 $\varepsilon_{\text{Total Sr}}$ = effective efficiency of the detector for total strontium referenced to ⁹⁰Sr
 Y = fractional chemical yield for strontium
 W = weight of the sample aliquant (g)
 DF = correction factor for decay of the sample from its reference date until the midpoint of the total strontium count
 λ_{Sr90} = decay constant for ⁹⁰Sr, $7.642 \times 10^{-10} \text{ s}^{-1}$
 t_0 = reference date and time for the sample (s)
 t_1 = date and time of the Sr/Y separation (s)

NOTE: The elapsed time between the sample count and the reference date must be calculated using the same time units as the decay constant

12.1.4. The standard counting uncertainty of the total radiostrontium activity concentration, $u_{\text{CC}}(AC_{\text{Total Sr}})$ is calculated as follows:

$$u_{cC}(AC_{\text{Total Sr}}) = \frac{\sqrt{\frac{R_a}{t_a} + \frac{R_b}{t_b}}}{2.22 \times \varepsilon_{\text{Total Sr}} \times Y \times W \times DF} \quad (4)$$

where:

t_a = Duration of the sample count (min)
 t_b = Duration of the background subtraction count (min)

12.1.5. The combined standard uncertainty (CSU) for the total radiostrontium activity concentration, $u_c(AC_{\text{Total Sr}})$, is calculated as follows:

$$u_c(AC_{\text{Total Sr}}) = \sqrt{u_{cC}^2(AC_{\text{Total Sr}}) + AC_{\text{Total Sr}}^2 \left(\frac{u^2(\varepsilon_{\text{Total Sr}})}{\varepsilon_{\text{Total Sr}}^2} + \frac{u^2(Y)}{Y^2} + \frac{u^2(W)}{W^2} \right)} \quad (5)$$

where:

$u(Y)$ = standard uncertainty of fractional chemical yield for strontium
 $u(W)$ = standard uncertainty of the weight of the sample aliquant (g)

12.1.6. If the critical level concentration (L_c) or the minimum detectable concentration (MDC) are requested (at an error rate of 5%), they can be calculated using the following equations:²

$$L_c = \frac{\left[0.4 \times \left(\frac{t_s}{t_b} - 1 \right) + 0.677 \times \left(1 + \frac{t_s}{t_b} \right) + 1.645 \times \sqrt{(R_b t_b + 0.4) \times \frac{t_s}{t_b} \times \left(1 + \frac{t_s}{t_b} \right)} \right]}{t_s \times 2.22 \times \varepsilon_{\text{Total Sr}} \times Y \times V \times DF} \quad (6)$$

$$MDC = \frac{\left[2.71 \times \left(1 + \frac{t_s}{t_b} \right) + 3.29 \times \sqrt{R_b t_s \times \left(1 + \frac{t_s}{t_b} \right)} \right]}{t_s \times 2.22 \times \varepsilon_{\text{Total Sr}} \times Y \times W \times DF} \quad (7)$$

12.2. Chemical Yield for Strontium

12.2.1. Calculate the chemical yield for strontium using the gravimetric data collected in Step 11.2.3:

$$Y = \frac{m_s F_{\text{Sr(NO}_3)_2}}{c_c V_c + c_n W} \quad (8)$$

where:

Y = strontium yield, expressed as a fraction
 m_s = mass of Sr(NO₃)₂ recovered from the sample (mg)
 $F_{\text{Sr(NO}_3)_2}$ = gravimetric factor for strontium weighed as the nitrate, 0.414

² The formulations for the critical level and minimum detectable concentration are based on the Stapleton Approximation as recommended in MARLAP Section 20A.2.2, Equations 20.54 and 20A.3.2, and Equation 20.74, respectively. The formulations presented assume $\alpha = 0.05$, $\beta = 0.05$ (with $z_{1-\alpha} = z_{1-\beta} = 1.645$), and $d = 0.4$.

- c_c = Sr mass concentration in the strontium carrier solution (mg/mL)
 V_c = volume of strontium carrier added to the sample (mL)
 c_n = Sr mass concentration native to the sample – if determined (mg/g)
 W = weight of sample aliquant (g)

12.2.2. Calculate the standard uncertainty of the yield as follows:

$$u(Y) = Y \times \sqrt{u_r^2(m_s) + \frac{u^2(c_c)V_c^2 + c_c^2 u^2(V_c) + u^2(c_n)V^2 + c_n^2 u^2(W)}{(c_c V_c + c_n W)^2}} \quad (9)$$

where

- $u(x)$ = standard uncertainty of the quantity in parentheses,
 $u_r(x)$ = relative standard uncertainty of the quantity in parentheses.

12.3. Results Reporting

12.3.1. Unless otherwise specified in the APS, the following items should be reported for each result:

- 12.3.1.1. Result for total radiostrontium in scientific notation ± 1 combined standard uncertainty.
 12.3.1.2. Weight of sample aliquant and any dilutions used.
 12.3.1.3. Yield of tracer and its uncertainty.
 12.3.1.4. Case narrative.

13. Method Performance

13.1. Results of method validation performance are to be archived and available for reporting purposes.

13.2. Expected turnaround time per sample or per batch (See Step 17.5 for typical processing times (assumes samples are not from RDD)).

13.2.1. Preparation and chemical separations for a batch of 20 samples can be performed by using a vacuum box system (24 ports each) simultaneously, assuming 24 detectors are available. For an analysis of a 1-g sample aliquant, sample preparation and digestion should take ~2.5 h.

13.2.2. Purification and separation of the strontium fraction using cartridges and vacuum box system should take ~2.5 h.

13.2.3. Sample test source preparation takes ~1.5 h.

13.2.4. A 60–90-minute counting time is sufficient to meet the MQO in Step 9.2, assuming 1.5-g aliquant, a background of 1 cpm, detector efficiency of 0.4–0.5, and radiochemical yield of at least 0.5.

13.3. Total radiostrontium (⁸⁹Sr+⁹⁰Sr) data reduction should be achievable between 5 and 8.5 hours after the beginning of the analysis, depending on batch size and count time.

- 13.4. The sample may be recounted following a delay of 10–21 days to differentiate the ^{89}Sr and ^{90}Sr activities. If the source contains pure ^{90}Sr , the total radiostrontium activity calculated from the two counts should agree within the uncertainty of the measurements. Minimizing the time between the chemical separation of Sr and the initial count, longer count times, and increasing the delay between the two counts, will minimize the overall uncertainty of the data and provide more sensitive and reliable measures of the radiochemical purity of the STS.

NOTE: The ^{89}Sr and ^{90}Sr may be determined from two consecutive counts of the source – calculations are presented in Appendix B. This approach must be validated prior to use.

14. Pollution Prevention

- 14.1. The use of Sr Resin reduces the amount of acids and hazardous metals that would otherwise be needed to co-precipitate and purify the sample and prepare the final counting form.

15. Waste Management

- 15.1. Nitric acid and hydrochloric acid wastes should be neutralized before disposal and then disposed in accordance with prevailing laboratory, local, state and federal requirements.
- 15.2. Final precipitated materials may contain radiostrontium and should be treated as radioactive waste and disposed in accordance with the restrictions provided in the facility's radioactive materials license and any prevailing local restrictions.
- 15.3. Used resins and columns should be considered radioactive waste and disposed of in accordance with restriction provided in the facility's radioactive materials license and any prevailing local restrictions.

16. References

Cited References

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- 16.5. ASTM D1193, "Standard Specification for Reagent Water," ASTM Book of Standards 11.02, current version, ASTM International, West Conshohocken, PA.

Other References

- 16.6. Maxwell, S. 2006. "Rapid Column Extraction Method for Actinides and ^{89/90}Sr in Water Samples," *J. Radioanalytical and Nuclear Chemistry*. 267(3): 537-543.
- 16.7. Maxwell, S., Culligan, B. and Noyes, G. 2010. Rapid method for actinides in emergency soil samples, *Radiochimica Acta*. 98(12): 793-800.
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- 16.12. U.S. Environmental Protection Agency (EPA). 2014. Rapid Radiochemical Method for Americium-241 in Building Materials for Environmental Remediation Following Radiological Incidents. Revision 0, EPA 402-R14-007. Office of Air and Radiation, Washington, DC. Available at: www.epa.gov/narel.
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- 16.15. U.S. Environmental Protection Agency (EPA). 2014. Rapid Radiochemical Method for Total Radiostrontium (Sr-90) in Building Materials for Environmental Remediation Following Radiological Incidents. Revision 0, EPA 402-R14-001. Office of Air and Radiation, Washington, DC. Available at: www.epa.gov/narel.
- 16.16. U.S. Environmental Protection Agency (EPA). 2014. Rapid Radiochemical Method for Isotopic Uranium in Building Materials for Environmental Remediation Following Radiological Incidents. Revision 0, EPA 402-R14-005. Office of Air and Radiation, Washington, DC. Available at: www.epa.gov/narel.

17. Tables, Diagrams, Flow Charts and Validation Data

17.1. Validation Data

This section intentionally left blank.

17.2. Nuclide Decay and Radiation Data

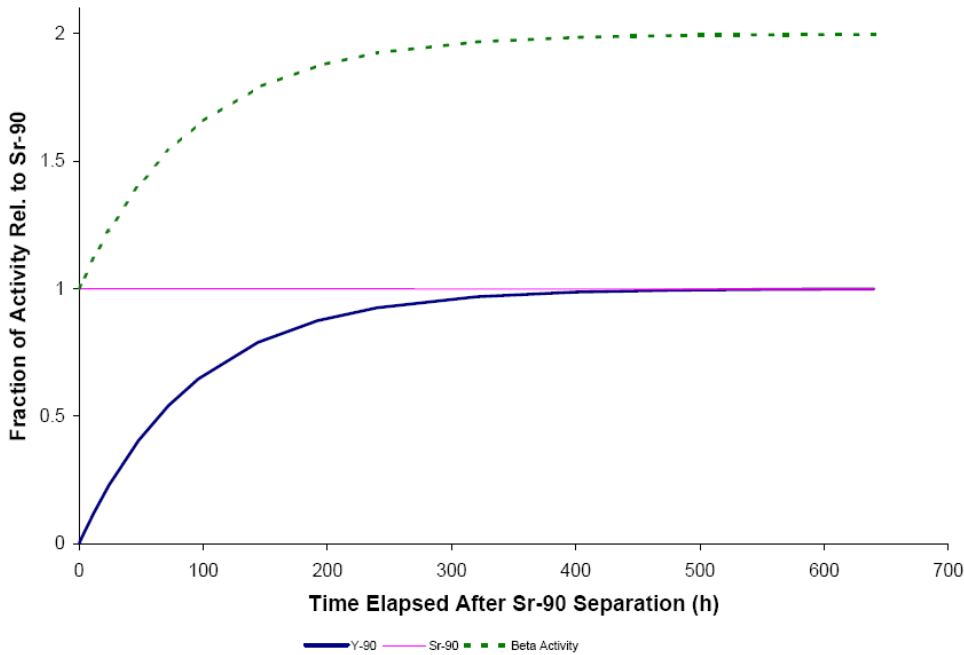
Table 17.2 – Decay and Radiation Data

Nuclide	Half-life (days)	λ (s ⁻¹)	Abundance	β_{max} (MeV ^[1])	β_{avg} (MeV)
⁹⁰ Sr	1.052E+04	7.642×10 ⁻¹⁰	1.00	0.546	0.196
⁹⁰ Y	2.6667	3.005×10 ⁻⁶	1.00	2.280	0.934
⁸⁹ Sr	50.53	1.587×10 ⁻⁷	1.00	1.495	0.585

[1] MeV – mega electron volts

17.3. Ingrowth and Decay Curves and Factors

In-Growth Curve for ⁹⁰Y in ⁹⁰Sr

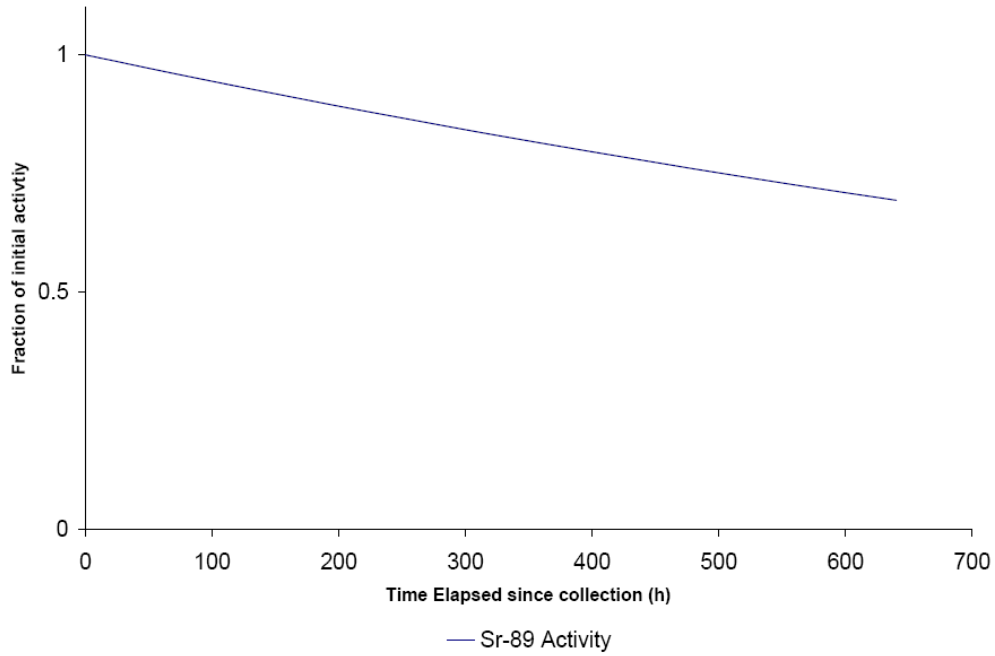


Total Beta Activity Ingrowth Factors for ⁹⁰Y in ⁹⁰Sr

Ingrowth time elapsed (hours)	0.25	2	4	12	24	48	72	96
Factor	0.003	0.021	0.042	0.122	0.229	0.405	0.541	0.646
Ingrowth time elapsed (hours)	144	192	240	320	400	480	560	640
Factor	0.790	0.875	0.926	0.969	0.987	0.994	0.998	0.999

Factor = (⁹⁰Y activity/⁹⁰Sr activity at zero hours of ingrowth)

Decay Curve for ^{89}Sr



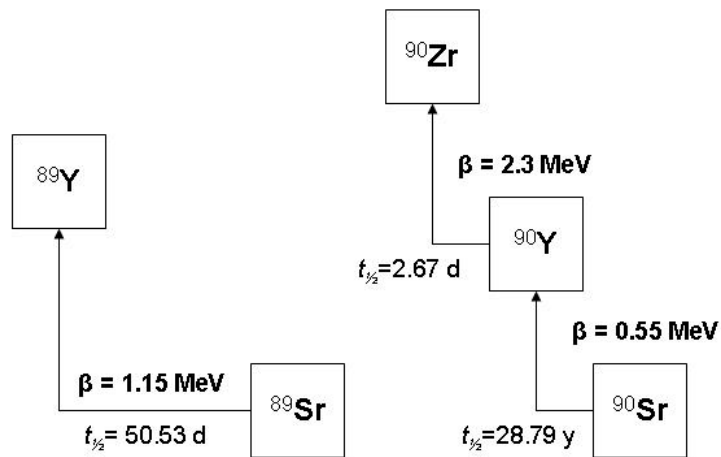
Decay Factors for ^{89}Sr

Decay time elapsed (hours)	0.25	2	4	12	24	48	72	96
Factor	1.000	0.999	0.998	0.993	0.986	0.973	0.960	0.947
Decay time elapsed (hours)	144	192	240	320	400	480	560	640
Factor	0.921	0.896	0.872	0.833	0.796	0.760	0.726	0.694

Factor = (^{89}Sr activity/ ^{89}Sr activity at zero hours of ingrowth)

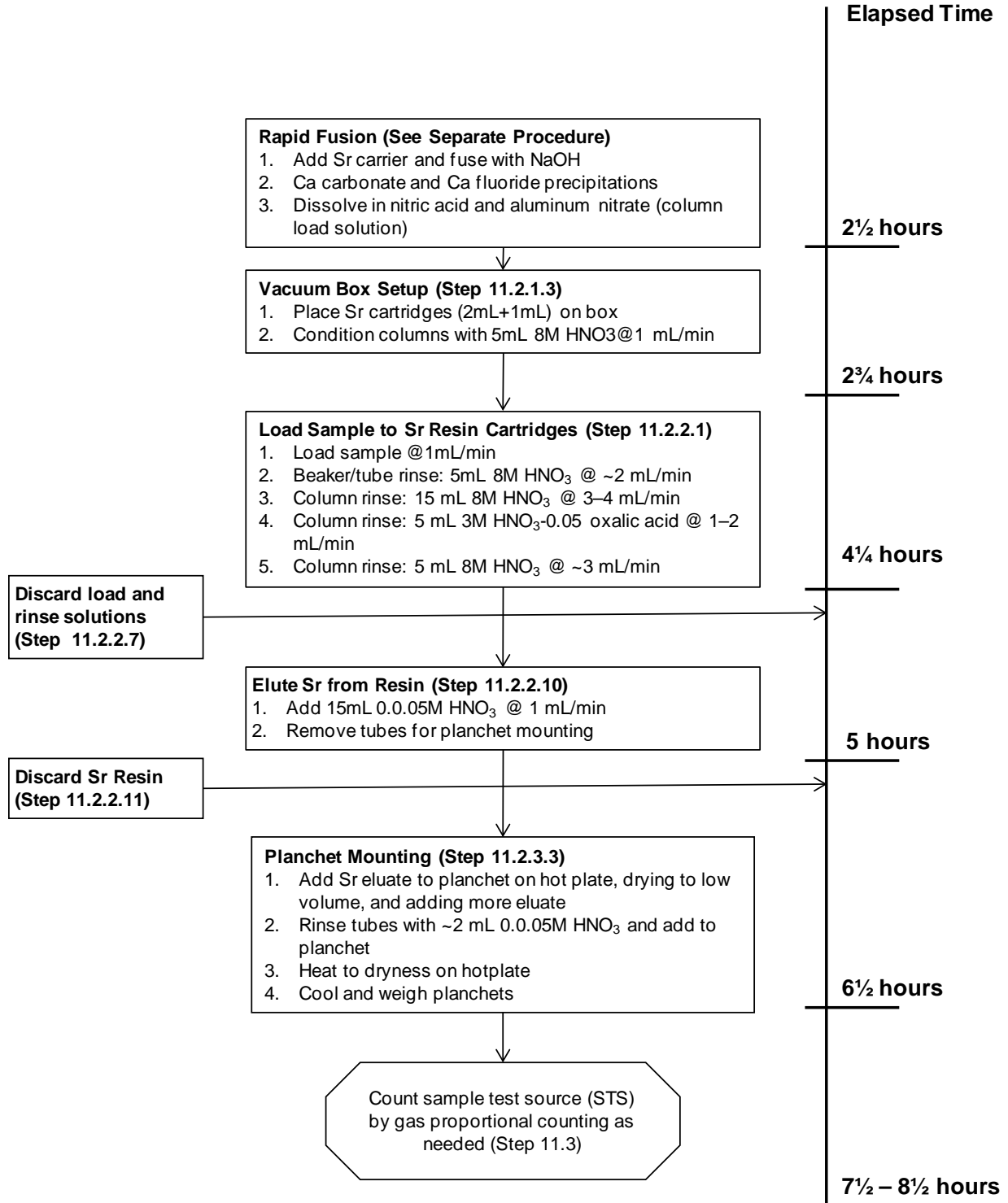
17.4. Decay Schemes for ^{89}Sr and ^{90}Sr

^{89}Sr and ^{90}Sr Decay Scheme



17.5. Process Flow with Typical Processing Times

Separation Scheme and Timeline for Determination of Strontium Isotopes in Building Materials Samples



Appendix A:

Method and Calculations for Detector Calibration

- A1. The effective detection efficiency for total radiostrontium (referenced to ^{90}Sr) is calculated as the weighted sum of the ^{90}Sr and ^{90}Y efficiencies that reflects the relative proportions of ^{90}Y and ^{90}Sr based on the ^{90}Y ingrowth after strontium separation.

NOTE: While ^{89}Sr efficiency calibration is not needed unless ^{89}Sr analysis will be performed, instructions for preparation are provided to support the two count approach should this option be desired.

- A1.1. Due to the low mass of carrier used for this method, self-absorption effects may be assumed to be constant. Calibrate each detector used to count samples according to ASTM Standard Practice D7282, Section 16, "Single Point Efficiency or Constant Test Mass for a Specific Radionuclide" and the instructions below.
- A1.2. Prepare a blank and at least three working calibration sources (WCS) for ^{90}Sr and ^{90}Y , and ^{89}Sr (if needed) as follows:
- A1.2.1. The ^{90}Sr and ^{89}Sr radioactive standard solutions used to prepare WCSs shall be traceable to a national standards body such as NIST and shall originate from a standards supplier (or lot) different from standards used for calibration verification and batch quality controls. The standards should be diluted in nitric acid.
- A1.2.2. The planchets used for the sources shall be of the same size, materials and type as those used for the analysis of STSs.
- A1.2.3. Preparation of ^{89}Sr WCSs (if needed): ^{89}Sr standard solution (in 0.5M HNO_3) is evaporated to dryness in a stainless steel planchet as follows:
- A1.2.3.1. For each ^{89}Sr WCS to be prepared, and for the associated blank, add strontium carrier to 15 mL of 0.05M HNO_3 in a disposable 50 mL centrifuge tube. The amount of carrier should be adjusted to approximate the amount expected to be recovered from routine samples.

NOTES:

If the average recovery has not been determined, the laboratory may assume 85% chemical yield for determining the amount of carrier to use in Step A1.2.3.1.

If the ^{89}Sr standard contains residual chloride, it will attack the surface of the planchet and compromise the quality of the calibration standard. In such cases, convert the aliquant of standard solution to a nitrate system by adding 1 mL concentrated HNO_3 and taking to dryness 2 times prior to quantitatively transferring the solution to the planchet.

- A1.2.3.2. For each WCS, add a precisely known amount of traceable ^{89}Sr solution to a 50 mL centrifuge tube. Sufficient activity must be present at the point of the count to permit accumulation of greater than 10,000 net counts in a counting period deemed to be reasonable by the laboratory. The minimum activity used, however, should produce WCS count rates at least 20 times the

- background signal but not greater than 5000 counts per second (cps).
- A1.2.3.3. Mix the solution and quantitatively transfer each WCS and the blank to respective clean stainless steel counting planchets using three rinses of 0.05M HNO₃.
- A1.2.3.4. Evaporate to dryness using the same techniques used for sample test sources.
- A1.2.3.5. For each detector to be calibrated, count three ⁸⁹Sr WCSs for sufficient time to accumulate at least 10,000 net counts.
- A1.3. Preparation of ⁹⁰Sr and ⁹⁰Y WCSs: Separate WCSs for ⁹⁰Sr and ⁹⁰Y are prepared by chemically separating ⁹⁰Y from a standard solution of ⁹⁰Sr.
- A1.3.1. For each ⁹⁰Sr WCS to be prepared, and for the associated blank, add 1 mL of 7 mg/mL strontium carrier to a disposable 50-mL centrifuge tube containing 10 mL 8M HNO₃. The amount of carrier added should correspond to that expected to be recovered from a routine sample.
- Note: If the average recovery has not been determined, the laboratory may assume 85% chemical yield for determining the amount of carrier to use for Step A1.3.1. 3M HNO₃ may be used instead of 8M HNO₃, however Sr yields may be slightly less.**
- A1.3.2. For each ⁹⁰Sr WCS, add a precisely known amount of traceable ⁹⁰Sr solution to a 50-mL centrifuge tube containing 10 mL of 8M HNO₃. Sufficient activity should be present at the point of the count to permit accumulation of greater than 10,000 ⁹⁰Sr and 10,000 ⁹⁰Y net counts in the respective sources in a counting period deemed to be reasonable by the laboratory. The minimum activity used, however should produce WCS count rates at least 20 times the background signal but not greater than 5000 cps.
- A1.3.3. Set up one (2+1mL)Sr Resin column for each ⁹⁰Sr WCS and for the associated blank. Condition each column with 5 mL of 8M HNO₃. Column effluents are discarded to waste.
- A1.3.4. Place a clean centrifuge tube under each column to catch all combined ⁹⁰Y effluents.
- Note: Unless otherwise specified in the procedure, use a flow rate of ~ 1 mL/min for load and strip solutions and ~ 3 mL/min for rinse solutions.**
- A1.3.5. Load the ⁹⁰Sr solution onto the column at 1 drop/second or less (~1 mL/min). The load solution effluent containing ⁹⁰Y is retained.
- A1.3.6. Rinse the centrifuge tube with three successive 2-mL portions of 8M HNO₃ adding each of the rinses to the column after the previous rinse has reached the upper surface of the resin. These effluents also contain ⁹⁰Y and are retained.
- A1.3.7. Rinse the column with 10 mL of 8M HNO₃ and retain the column effluents containing ⁹⁰Y. Record the date and time that the final rinse solution leaves the column to the nearest 5 minutes as t_1 , "Time of ⁹⁰Y Separation." Remove the centrifuge tube that has the combined ⁹⁰Y

effluents. Place a clean tube under the column to catch the strontium eluate in subsequent steps.

NOTE: From this point, ^{90}Sr must be eluted, and the ^{90}Sr WCS must be prepared and counted as expeditiously as possible to minimize ^{90}Y ingrowth and necessary corrections to the efficiency. Counting of the ^{90}Sr WCS should be completed, if possible, within 3–5 hours but no longer than 10 hours from the time of ^{90}Y separation. If processing or counting capacity is limited, concentrate resources on ^{90}Sr WCS and counting first. The ^{90}Y WCS are not compromised by ingrowth but must only be counted promptly enough to minimize decay and optimize counting statistics.

- A1.3.8. Strip strontium from each column by adding 15 mL of 0.05-M HNO_3 to each column, catching the effluents containing ^{90}Sr in the centrifuge tube.
- A1.3.9. Quantitatively transfer ^{90}Sr and ^{90}Y fractions to respective tared planchets using three portions of 0.05-M HNO_3 .
- A1.3.10. Evaporate to dryness using the same techniques for sample test sources, with the same heat time applied after dryness is reached.

Note: Gravimetric measurements may be performed following the counting to minimize elapsed time between separation and counting.

- A1.4. Weigh the ^{90}Sr and ^{90}Y WCS sources and calculate the net residue mass.
 - A1.4.1. The net mass of the strontium nitrate precipitate shall indicate near quantitative yield of strontium of 95–103%. If strontium yield falls outside this range, determine and address the cause for the losses and repeat the process. The known activity of ^{90}Sr in the standard is corrected for losses based on the measured chemical yields of the strontium carrier.

Note that no correction shall be applied for values greater than 100% because this will produce a negative bias in the calibrated efficiency.
 - A1.4.2. The net residue mass of the ^{90}Y should be low. Higher residue mass may indicate the breakthrough of strontium and will result in high bias in the ^{90}Y efficiency, but it may simply be the result of corrosion of the stainless planchet during evaporation of 8M HNO_3 , forming a small amount of iron oxide. Lower Sr carrier yields on the Sr planchet would indicate the present of Sr breakthrough.

NOTE: Formation of a small amount of iron oxide on the planchet during evaporation may result in a slight mass on the ^{90}Y planchet (~1 mg) but does not affect the ^{90}Y counting significantly.
 - A1.4.3. Count three ^{90}Sr WCS on each detector to be calibrated, for sufficient time to accumulate at least 10,000 net counts.
 - A1.4.4. Count three ^{90}Y WCS on each detector to be calibrated, for sufficient time to accumulate at least 10,000 net counts.
 - A1.4.5. Count the associated blanks as a gross contamination check on the process. If indications of contamination are noted, take appropriate corrective actions to minimize spread and prevent cross-contamination of other samples in the laboratory.

- A1.5. Verify the calibration of each detector according to ASTM Standard Practice D7282, Section 16, and the laboratory quality manual and standard operating procedures.
- A1.6. Calculations and data reduction for ⁹⁰Sr and ⁹⁰Y calibrations and calibration verifications are presented in Sections A2, A3, and A4. Calculations for total radiostrontium are in Section 12 of the method.

A2. Calculation of Detection Efficiency for ⁹⁰Sr

A2.1. Calculate the following decay and ingrowth factors for each WCS:

$$DF_s = e^{-\lambda_{Sr90}(t_1-t_0)} \quad (A1)$$

$$IF_{Y90} = 1 - e^{-\lambda_{Y90}(t_2-t_1)} \quad (A2)$$

where

- DF_s = decay factor for decay of the ⁹⁰Sr standard from its reference date until the ⁹⁰Sr/⁹⁰Y separation
- IF_{Y90} = ingrowth factor for ingrowth of ⁹⁰Y after the ⁹⁰Sr/⁹⁰Y separation
- λ_{Sr90} = decay constant for ⁹⁰Sr, $7.642 \times 10^{-10} \text{ s}^{-1}$
- λ_{Y90} = decay constant for ⁹⁰Y, $3.005 \times 10^{-6} \text{ s}^{-1}$
- t_0 = reference date and time for the ⁹⁰Sr standard (s)
- t_1 = date and time of the Sr/Y separation (s)
- t_2 = date and time of the midpoint of the ⁹⁰Sr count (s)

NOTE: The elapsed time between the sample count and the reference date must be calculated using the same time units as the decay constant

A2.2. Calculate the ⁹⁰Sr detection efficiency for each WCS:

$$\varepsilon_{Sr90,i} = \frac{R_{s,i} - R_b}{AC_{Sr90 \text{ std}} \times V_{s,i} \times DF_{s,i}} - IF_{Y90,i} \times \bar{\varepsilon}_{Y90} = \frac{R_{n,i}}{AC_{Sr90 \text{ std}} \times V_{s,i} \times DF_{s,i}} - IF_{Y90,i} \times \bar{\varepsilon}_{Y90} \quad (A3)$$

where

- $\varepsilon_{Sr90,i}$ = ⁹⁰Sr detection efficiency for the i^{th} WCS
- $\bar{\varepsilon}_{Y90}$ = average ⁹⁰Y detection efficiency (from Step A3.2)
- $R_{s,i}$ = beta gross count rate for the i^{th} WCS (cpm)
- R_b = background count rate, in cpm
- $R_{n,i}$ = beta net count rate for the i^{th} WCS (cpm)
- $AC_{Sr90 \text{ std}}$ = activity concentration of the ⁹⁰Sr standard solution on its reference date (cpm/mL or cpm/g)
- $V_{s,i}$ = amount (volume or mass) of the standard solution added to the i^{th} WCS

A2.3. Average the efficiencies determined in Step A2.2 for all the WCSs to obtain the final detection efficiency for ⁹⁰Sr.

$$\varepsilon_{Sr90} = \bar{\varepsilon}_{Sr90} = \frac{1}{n} \sum_{i=1}^n \varepsilon_{Sr90,i} \quad (A4)$$

where

$$\begin{aligned}\varepsilon_{\text{Sr90},i} &= {}^{90}\text{Sr detection efficiency determined for the } i^{\text{th}} \text{ WCS in A2.2,} \\ n &= \text{number of WCSs prepared and counted.}\end{aligned}$$

A2.4. Calculate the standard uncertainty of the average ⁹⁰Sr detection efficiency as follows:

$$u(\bar{\varepsilon}_{\text{Sr90}}) = \sqrt{\left[\frac{1}{n^2} \sum_{i=1}^n \frac{u^2(R_{n,i}) + R_{n,i}^2 u_r^2(V_{s,i})}{AC_{\text{Sr90 std}}^2 V_{s,i}^2 DF_{s,i}^2} \right] + \left(u^2(\bar{\varepsilon}_{\text{Y90}}) - \bar{\varepsilon}_{\text{Y90}}^2 u_r^2(AC_{\text{Sr90 std}}) \right) \bar{IF}_{\text{Y90}}^2 + \bar{\varepsilon}_{\text{Sr90}}^2 u_r^2(AC_{\text{Sr90 std}})} \quad (\text{A5})$$

where

$$\bar{IF}_{\text{Y90}} = \frac{1}{n} \sum_{i=1}^n IF_{\text{Y90},i} = \text{average value of } {}^{90}\text{Y ingrowth factors} \quad (\text{A6})$$

and

$$\begin{aligned}u(x) &= \text{standard uncertainty of the value in parentheses,} \\ u_r(x) &= \text{relative standard uncertainty of the value in parentheses.}\end{aligned}$$

A3. Detection Efficiency for ⁹⁰Y

A3.1. Calculate the ⁹⁰Y detection efficiency, $\varepsilon_{\text{Y90},i}$, for each WCS,

$$\varepsilon_{\text{Y90},i} = \frac{R_{s,i} - R_b}{AC_{\text{Sr90 std}} V_{s,i} DF_{s,i}} = \frac{R_{n,i}}{AC_{\text{Sr90 std}} V_{s,i} DF_{s,i}} \quad (\text{A7})$$

where

$$DF_{s,i} = e^{-\lambda_{\text{Sr90}}(t_1-t_0)} e^{-\lambda_{\text{Y90}}(t_2-t_1)} \quad (\text{A8})$$

and

$$\begin{aligned}\varepsilon_{\text{Y90},i} &= {}^{90}\text{Y detection efficiency determined for the WCS} \\ R_{s,i} &= \text{beta gross count rate for the } i^{\text{th}} \text{ WCS (cpm)} \\ R_b &= \text{background count rate, in cpm} \\ R_{n,i} &= \text{beta net count rate for the } i^{\text{th}} \text{ WCS (cpm)} \\ AC_{\text{Sr90 std}} &= \text{activity concentration of the } {}^{90}\text{Sr standard solution on its reference} \\ &\quad \text{date (disintegrations per minute [dpm]/mL or dpm/g)} \\ V_{s,i} &= \text{amount of the standard solution added to the } i^{\text{th}} \text{ WCS (mL or g)} \\ DF_{s,i} &= \text{combined correction factor for decay of the } {}^{90}\text{Sr standard in the } i^{\text{th}} \\ &\quad \text{WCS from its reference date until } {}^{90}\text{Y separation, and for the decay} \\ &\quad \text{of } {}^{90}\text{Y from its separation until the midpoint of the count} \\ \lambda_{\text{Sr90}} &= \text{decay constant for } {}^{90}\text{Sr, } 7.642 \times 10^{-10} \text{ s}^{-1} \\ \lambda_{\text{Y90}} &= \text{decay constant for } {}^{90}\text{Y, } 3.005 \times 10^{-6} \text{ s}^{-1} \\ t_0 &= \text{reference date and time for the } {}^{90}\text{Sr standard (s)} \\ t_1 &= \text{date and time of the } {}^{90}\text{Y separation (s)} \\ t_2 &= \text{date and time at the midpoint of the } {}^{90}\text{Y count (s)}\end{aligned}$$

NOTE: The elapsed time between the sample count and the reference date must be calculated using the same time units as the decay constant

A3.2. Average the efficiencies determined in Step A3.1 to obtain the final detection efficiency for ⁹⁰Y.

$$\varepsilon_{Y90} = \bar{\varepsilon}_{Y90} = \frac{1}{n} \sum_{i=1}^n \varepsilon_{Y90,i} \quad (\text{A9})$$

where

$\varepsilon_{Y90,i}$ = ⁹⁰Y detection efficiency determined for the i^{th} WCS in Step A3.1
 n = number of WCS prepared and counted

A3.3. The combined standard uncertainty of the average efficiency for ⁹⁰Y including uncertainty associated with the preparation of the calibration standards is calculated as follows:

$$u(\bar{\varepsilon}_{Y90}) = \sqrt{\left[\frac{1}{n^2} \sum_{i=1}^n \frac{u^2(R_{n,i}) + R_{n,i}^2 u_r^2(V_{s,i})}{AC_{Sr90\text{ std}}^2 V_{s,i}^2 DF_{s,i}^2} \right] + \bar{\varepsilon}_{Y90}^2 u_r^2(AC_{Sr90\text{ std}})} \quad (\text{A10})$$

where

$u(x)$ = standard uncertainty of the value in parentheses,
 $u_r(x)$ = relative standard uncertainty of the value in parentheses.

NOTE: The uncertainty of the net count rate, $u_{Rn,i}$, includes the uncertainty of the background or $u^2(R_{n,i}) = u^2(R_s) + u^2(R_b) = (R_s/t_s) + (R_b/t_b)$

A4. Calculate the covariance and correlation coefficient for the ⁹⁰Sr efficiency and the ⁹⁰Y efficiency:

$$u(\bar{\varepsilon}_{Sr90}, \bar{\varepsilon}_{Y90}) = \bar{\varepsilon}_{Sr90} \bar{\varepsilon}_{Y90} u_r^2(AC_{Sr90\text{ std}}) - \left(u^2(\bar{\varepsilon}_{Y90}) - \bar{\varepsilon}_{Y90}^2 u_r^2(AC_{Sr90\text{ std}}) \right) \overline{IF}_{Y90} \quad (\text{A11})$$

and

$$r(\bar{\varepsilon}_{Sr90}, \bar{\varepsilon}_{Y90}) = \frac{u(\bar{\varepsilon}_{Sr90}, \bar{\varepsilon}_{Y90})}{u(\bar{\varepsilon}_{Sr90}) u(\bar{\varepsilon}_{Y90})} \quad (\text{A12})$$

where

$u(x, x)$ = estimated covariance of the two quantities in parentheses,
 $r(x, x)$ = estimated correlation coefficient of the two quantities in parentheses,
 $u(x)$ = standard uncertainty of the quantity in parentheses,
 $u_r(x)$ = relative standard uncertainty of the quantity in parentheses.

A5. Detection Efficiency for ⁸⁹Sr (if needed for Appendix B Calculations)

A5.1. Calculate the detection efficiency, $\varepsilon_{Sr89,i}$, for each WCS as follows:

$$\varepsilon_{Sr89,i} = \frac{R_{s,i} - R_b}{AC_{Sr89\text{ std}} V_{s,i} DF_{s,i}} = \frac{R_{n,i}}{AC_{Sr89\text{ std}} V_{s,i} DF_{s,i}} \quad (\text{A13})$$

where

$$DF_{s,i} = e^{-\lambda_{Sr89}(t_1 - t_0)} \quad (\text{A14})$$

and

$\varepsilon_{Sr89,i}$ = ⁸⁹Sr detection efficiency for the i^{th} WCS

$R_{s,i}$	=	beta gross count rate for the i^{th} WCS (cpm)
R_b	=	background count rate, in cpm
$AC_{\text{Sr89 std}}$	=	activity concentration of the ⁸⁹ Sr standard solution on the reference date (dpm/mL or dpm/g)
$V_{s,i}$	=	amount (volume or mass) of the standard solution added to the i^{th} WCS (mL or g)
$DF_{s,i}$	=	correction factor for decay of the ⁸⁹ Sr standard for the i^{th} WCS from its reference date until the midpoint of the sample count
λ_{Sr89}	=	decay constant for ⁸⁹ Sr, $1.587 \times 10^{-7} \text{ s}^{-1}$
t_0	=	reference date and time for the ⁸⁹ Sr standard (s)
t_1	=	date and time at the midpoint of the ⁸⁹ Sr count (s)

A5.1.1. Average the efficiencies determined in Step A5.1 to obtain the final detection efficiency for ⁸⁹Sr.

$$\varepsilon_{\text{Sr89}} = \bar{\varepsilon}_{\text{Sr89}} = \frac{1}{n} \sum_{i=1}^n \varepsilon_{\text{Sr89},i} \quad (\text{A15})$$

where

$\varepsilon_{\text{Sr89},i}$	=	⁸⁹ Sr detection efficiency determined for the i^{th} WCS in Step A5.1,
n	=	number of WCSs prepared and counted.

A5.1.2. The combined standard uncertainty of the average efficiency for ⁸⁹Sr including uncertainty associated with the preparation of the calibration standards is calculated as follows:

$$u(\bar{\varepsilon}_{\text{Sr89}}) = \sqrt{\left[\frac{1}{n^2} \sum_{i=1}^n \frac{u^2(R_{n,i}) + R_{n,i}^2 u_r^2(V_{s,i})}{AC_{\text{Sr89 std}}^2 V_{s,i}^2 DF_{s,i}^2} \right] + \bar{\varepsilon}_{\text{Sr89}}^2 u_r^2(AC_{\text{Sr89 std}})} \quad (\text{A16})$$

where

$u(x)$	=	standard uncertainty of the value in parentheses,
$u_r(x)$	=	relative standard uncertainty of the value in parentheses.

Appendix B:

Calculations for Isotopic ^{89}Sr and ^{90}Sr Results

A numerical approach for determining ^{89}Sr and ^{90}Sr activity from a single sample is performed by a number of laboratories. This presentation, however, allows a more rigorous evaluation of uncertainties than commonly employed. Lacking this treatment, many labs have found that the traditional approach (evaluating counting uncertainty for a single count only) has led to overestimation of the quality of results, and to poor decisions regarding the presence or absence of low activities of one radioisotope of strontium in the presence of elevated activities of the second.

These calculations may be valuable to laboratories who wish to determine isotopic ^{89}Sr and ^{90}Sr in a large number of samples with a minimum of additional effort beyond the initial preparation and counting of total radiostrontium. Specifically, it involves performing a second count of the same radiostrontium sample test source (STS) and mathematically resolving the activity of the two isotopes. Although the STS may be recounted as soon as 1–2 days after the initial count, resolution is optimized if the two counts span as large a range of the ^{90}Y ingrowth as practicable. The time elapsed between the chemical separation and the first count should be minimized, while the second count should optimally proceed as ^{90}Y approaches secular equilibrium with ^{90}Sr but before significant decay of ^{89}Sr has occurred, for example, after 3–5 half-lives of ^{90}Y have elapsed (1–2 weeks).

This section may not be employed without complete validation of the approach by the laboratory, including testing with samples containing ratios of ^{90}Sr relative to ^{89}Sr varying from pure ^{90}Sr to pure ^{89}Sr .

B1. The equations in this section are used to calculate the ^{90}Sr and ^{89}Sr activity of a sample from data generated from two successive counts of the same radiostrontium sample test source.

B1.1. For each of the two counting measurements ($i = 1, 2$), calculate the following decay and ingrowth factors:

$$DF_{\text{Sr}89,i} = e^{-\lambda_{\text{Sr}89}(t_i - t_0)} \quad (\text{B1})$$

$$DF_{\text{Sr}90,i} = e^{-\lambda_{\text{Sr}90}(t_i - t_0)} \quad (\text{B2})$$

$$F_{\text{Y}90,i} = e^{-\lambda_{\text{Sr}90}(t_{\text{sep}} - t_0)} \left(1 - e^{-\lambda_{\text{Y}90}(t_i - t_{\text{sep}})} \right) \quad (\text{B3})$$

where:

$DF_{\text{Sr}89,i}$ = decay factor for decay of ^{89}Sr from the collection date to the midpoint of the i^{th} count of the STS

$DF_{\text{Sr}90,i}$ = decay factor for decay of ^{90}Sr from the collection date to the midpoint of the i^{th} count of the STS

$F_{\text{Sr}90,i}$ = combined decay and ingrowth factor for decay of ^{90}Sr from the collection date to the Sr/Y separation and ingrowth of ^{90}Y from the separation to the midpoint of the i^{th} count of the STS

$\lambda_{\text{Sr}89}$	=	decay constant for ⁸⁹ Sr = $1.587 \times 10^{-7} \text{ s}^{-1}$
$\lambda_{\text{Sr}90}$	=	decay constant for ⁹⁰ Sr = $7.642 \times 10^{-10} \text{ s}^{-1}$
t_0	=	collection date and time for the sample (s)
t_{sep}	=	date and time of the Sr/Y separation (s)
t_i	=	date and time of the midpoint of the i^{th} count of the STS (s)

NOTE: The elapsed time between the sample count and the reference date must be calculated using the same time units as the decay constant

B1.2. For $i = 1, 2$, use the results from Section A5.1 in Appendix A to calculate the following sensitivity factors:

$$a_i = DF_{\text{Sr}89,i} \varepsilon_{\text{Sr}89,i} \quad (\text{B4})$$

$$b_i = DF_{\text{Sr}90,i} \varepsilon_{\text{Sr}90,i} + F_{\text{Y}90,i} \varepsilon_{\text{Y}90,i} \quad (\text{B5})$$

where

a_i	=	sensitivity of the count rate in the i^{th} measurement to ⁸⁹ Sr activity
b_i	=	sensitivity of the count rate in the i^{th} measurement to ⁹⁰ Sr activity
$\varepsilon_{\text{Y}90,i}$	=	⁹⁰ Y efficiency of the detector for the i^{th} count of the STS
$\varepsilon_{\text{Sr}90,i}$	=	⁹⁰ Sr efficiency of the detector for the i^{th} count of the STS

B1.3. Calculate the standard uncertainties of the sensitivity factors using the equations:

$$u(a_i) = DF_{\text{Sr}89,i} u(\varepsilon_{\text{Sr}89,i}) \quad (\text{B6})$$

$$u(b_i) = \sqrt{DF_{\text{Sr}90,i}^2 u^2(\varepsilon_{\text{Sr}90,i}) + F_{\text{Y}90,i}^2 u^2(\varepsilon_{\text{Y}90,i}) + 2DF_{\text{Sr}90,i} F_{\text{Y}90,i} u(\varepsilon_{\text{Sr}90,i}, \varepsilon_{\text{Y}90,i})} \quad (\text{B7})$$

where the estimated covariance of the ⁹⁰Sr and ⁹⁰Y efficiencies is calculated as follows:

$$u(\varepsilon_{\text{Sr}90,i}, \varepsilon_{\text{Y}90,i}) = r(\varepsilon_{\text{Sr}90,i}, \varepsilon_{\text{Y}90,i}) u(\varepsilon_{\text{Sr}90,i}) u(\varepsilon_{\text{Y}90,i}) \quad (\text{B8})$$

and where the estimated correlation coefficient $r(\varepsilon_{\text{Sr}90,i}, \varepsilon_{\text{Y}90,i})$ was determined during the calibration.

B1.4. Calculate the covariances $u(a_1, a_2)$ and $u(b_1, b_2)$ as follows:

$$u(a_1, a_2) = \begin{cases} u(a_1) u(a_2), & \text{if only one detector is used} \\ a_1 a_2 u_r^2(AC_{\text{Sr}89 \text{ std}}), & \text{if two detectors are used} \end{cases} \quad (\text{B9})$$

$$u(b_1, b_2) = \begin{cases} (DF_{Sr90,1} F_{Y90,2} + DF_{Sr90,2} F_{Y-90,1}) u(\varepsilon_{Sr90,1}, \varepsilon_{Y90,1}) \\ + DF_{Sr90,1} DF_{Sr90,2} u^2(\varepsilon_{Sr90,1}) + F_{Y90,1} F_{Y90,2} u^2(\varepsilon_{Y90,1}), & \text{using only one detector} \quad (B10) \\ b_1 b_2 u_r^2(AC_{Sr90 \text{ std}}), & \text{using two detectors} \end{cases}$$

where

$AC_{Sr89 \text{ std}}$ = activity concentration of the ⁸⁹Sr standard used for calibration

$AC_{Sr90 \text{ std}}$ = activity concentration of the ⁹⁰Sr standard used for calibration

$u_r(\cdot)$ = relative standard uncertainty of the quantity in parentheses

B1.5. For $i = 1, 2$, calculate the net beta count rates, $R_{n,i}$, and their standard uncertainties:

$$R_{n,i} = R_{a,i} - R_{b,i} \quad (B11)$$

$$u(R_{n,i}) = \sqrt{\frac{R_{a,i}}{t_{a,i}} + \frac{R_{b,i}}{t_{b,i}}} \quad (B12)$$

where:

$R_{n,i}$ = net beta count rate for the i^{th} count of the STS (cpm)

$R_{a,i}$ = beta gross count rate for the i^{th} count of the STS (cpm)

$R_{b,i}$ = beta background count rate for the i^{th} count of the STS (cpm)

$t_{a,i}$ = sample count time for the i^{th} count of the STS (min)

$t_{b,i}$ = background count time for the i^{th} count of the STS (min)

B1.6. Using the values calculated in A5.1 – A5.5, calculate the ⁸⁹Sr and ⁹⁰Sr activity concentrations:

$$AC_{Sr89} = \frac{b_2 R_{n,1} - b_1 R_{n,2}}{2.22 \times X \times V \times Y} \quad (B13)$$

$$AC_{Sr90} = \frac{a_1 R_{n,2} - a_2 R_{n,1}}{2.22 \times X \times V \times Y} \quad (B14)$$

where:

$$X = a_1 b_2 - a_2 b_1 \quad (B15)$$

and where:

2.22 = conversion factor from dpm to pCi

Y = chemical yield for strontium

W = sample weight (g)

B2. The standard counting uncertainties for ⁸⁹Sr ($u_{cc}(AC_{Sr89})$) and ⁹⁰Sr ($u_{cc}(AC_{Sr90})$) are calculated in units of pCi/g as follows:

$$u_{cc}(AC_{Sr89}) = \frac{\sqrt{b_2^2 u^2(R_{n,1}) + b_1^2 u^2(R_{n,2})}}{2.22 \times X \times W \times Y} \quad (B16)$$

$$u_{\text{cC}}(AC_{\text{Sr90}}) = \frac{\sqrt{a_1^2 u^2(R_{n,2}) + a_2^2 u^2(R_{n,1})}}{2.22 \times X \times W \times Y} \quad (\text{B17})$$

B3. The combined standard uncertainties (CSU) for ^{89}Sr and ^{90}Sr are calculated as follows:

$$u_{\text{c}}(AC_{\text{Sr89}}) = \left[u_{\text{cC}}^2(AC_{\text{Sr89}}) + AC_{\text{Sr89}}^2 \left(\frac{u^2(W)}{W^2} + \frac{u^2(Y)}{Y^2} + \frac{b_2^2 u^2(a_1) + b_1^2 u^2(a_2) - 2b_1 b_2 u(a_1, a_2)}{X^2} \right) + AC_{\text{Sr90}}^2 \frac{b_2^2 u^2(b_1) + b_1^2 u^2(b_2) - 2b_1 b_2 u(b_1, b_2)}{X^2} \right]^{1/2} \quad (\text{B18})$$