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Rapid Radiochemical Method for Total Radiostrontium (Sr-90) In Water for Environmental Remediation Following Homeland Security Events

U.S. Environmental Protection Agency

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

Revision History

Revision 0	Original release.	02/23/2010
Revision 0.1	<ul style="list-style-type: none">• Corrected typographical and punctuation errors.• Improved wording consistency with other methods.• Corrected specification of analytical balance (6.1) to 10⁻⁴-g readability.• Added pH paper to list of equipment and supplies (6.6).• Added equations in 12.1.6 that allow theoretical calculation of the MDC and critical level for different decision error rates.• Updated footnote 3 to further clarify origin of critical value and minimum detectable concentration formulations.• Updated rounding example in 12.3.2 for clarity.• Deleted Appendix C (composition of Atlanta tap water) as irrelevant	10/28/2011

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TOTAL RADIOSTRONTIUM (SR-90) IN WATER: RAPID METHOD FOR HIGH-ACTIVITY SAMPLES

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. If any filtration of the sample is performed prior to starting the analysis, those solids should be analyzed separately. The results from the analysis of these solids should be reported separately (as a suspended activity concentration for the water volume filtered), but identified with the filtrate results.
- 1.2. The method provides a very rapid non-radioisotope-specific screen for total radiostrontium in drinking water and other aqueous samples.
- 1.3. This method uses rapid radiochemical separations techniques for the determination of beta-emitting strontium radioisotopes in water samples following a nuclear or radiological incident. Although this method can detect concentrations of ^{90}Sr on the same order of magnitude as methods used for the Safe Drinking Water Act (SDWA), this method is not a substitute for SDWA-approved methods for radiostrontium.
- 1.4. The method is capable of satisfying a required method uncertainty for ^{90}Sr (total as ^{90}Sr) of 1.0 pCi/L at an analytical action level of 8.0 pCi/L. To attain the stated measurement quality objectives (MQOs) (see Step 9.2), a sample volume of approximately 500 mL and a count time of approximately 1.25 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* (EPA 2009, reference 16.3).
- 1.5. This method is intended to be used for water samples that are similar in composition to drinking water. 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* (EPA 2009, reference 16.3) and Chapter 6 of *Multi-Agency Radiological Laboratory Analytical Protocols Manual* (MARLAP 2004, reference 16.4). Multi-radionuclide analysis using sequential separation may be possible.
- 1.6. This method is applicable to the determination of soluble radiostrontium. This method is not applicable to the determination of strontium isotopes contained in highly insoluble particulate matter possibly present in water samples contaminated as a result of a radiological dispersal device (RDD) event.
- 1.7. Sequential, multi-radionuclide analysis may be possible by using this method in conjunction with other rapid methods.

2. Summary of Method

- 2.1. Strontium is isolated from the matrix 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 concentrated by Sr/BaCO₃ coprecipitation. If insoluble residues are noted during acid dissolution

steps, the residue and precipitate mixture is digested in 8 M HNO₃ to solubilize strontium. 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 0.05 M HNO₃ 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. This test assumes that it is reasonable to assume the absence of ⁸⁹Sr in the sample. In such cases, a total radiostrontium analysis will provide for a specific determination of ⁹⁰Sr in the sample. The same prepared sample test source can be recounted after ~1–21 days to verify the total radiostrontium activity. If the initial and second counts agree, this is an indication that ⁸⁹Sr is not present in significant amounts relative to ⁹⁰Sr (within the uncertainty of the measurement).
 - 2.2.2. Computational methods are available for resolving the concentration of ⁸⁹Sr and ⁹⁰Sr 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.

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 decisionmaker to choose one of the alternative actions.
- 3.3. Analytical Decision Level (ADL). The analytical decision level refers to the value that is less than the AAL based on the acceptable error rate and the required method uncertainty.
- 3.4. 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).
- 3.5. *Multi-Agency Radiological Analytical Laboratory Protocol Manual* (see Reference 16.4.)
- 3.6. Measurement Quality Objective (MQO). MQOs are the analytical data requirements of the data quality objectives and are project- or program-specific. They can be

¹ Sr-Resin™ is a proprietary extraction chromatography resin consisting of octanol solution of 4,4'(5')-bis (t-butyl-cyclohexano)-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.

quantitative or qualitative. MQOs serve as measurement performance criteria or objectives of the analytical process.

- 3.7. Radiological Dispersal Device (RDD), i.e., a “dirty bomb.” This 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.
 - 3.8. 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.
 - 3.9. 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 action level.
 - 3.10. 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.
 - 3.11. Total Radiostrontium (also called Total Strontium): A radiological measurement that does not differentiate between ⁸⁹Sr and ⁹⁰Sr. The assumption is that all of the strontium is in the form of ⁹⁰Sr. When it is certain that no ⁸⁹Sr is present, the total radiostrontium activity is equal to the ⁹⁰Sr activity and may be reported as such.
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.
 - 4.1.1.1. 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.
 - 4.1.1.2. The resin has a higher affinity for polonium than strontium. Under the conditions of the analysis, however, polonium is not expected to elute from the column.
 - 4.1.2. Significant levels of ⁸⁹Sr in the sample will interfere with the total radiostrontium analysis.
 - 4.1.2.1. The absence of higher levels of interfering ⁸⁹Sr may be detected by counting the sample test source quickly after initial separation (minimizing ingrowth of ⁹⁰Y), and then recounting the sample test source after 1–21 days to verify that the calculated activity does not change significantly. The presence of ⁸⁹Sr 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.

- 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 . Note that the approach in Appendix B must be validated prior to use.
- 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. 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.
- 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.
- 4.2. Non-Radiological
- 4.2.1. Chemical yield results significantly greater than 100% may indicate the presence of non-radioactive strontium native to the sample. If the quantity of native strontium in the sample aliquant exceeds ~5% of the expected strontium carrier mass, chemical yield measurements will be affected and chemical yield corrections lead to low result bias unless the native strontium is accounted for in the yield calculations. When problematic levels of strontium are encountered, the native strontium content of the sample can be determined by an independent spectrometric measurement (such as inductively coupled plasma atomic emission spectroscopy [ICP-AES] or atomic absorption spectroscopy [AAS], etc). If the laboratory does not have access to instrumentation processing a split of the sample without the addition of strontium carrier may be used to obtain an estimate of the native strontium content of the sample.
- 4.2.2. Sr-ResinTM 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. Decreasing the sample size will help address samples with elevated levels of lead.
- 4.2.3. High levels of calcium, barium, magnesium, or potassium may compete with strontium for uptake on the resin leading to low chemical yield. One should consider that yield results will overestimate the true strontium yield and cause a

low result bias if these interfering matrix constituents are present as significant contaminants in the final sample test source.

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 the laboratory 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). Filtration using a 0.45- μ m or finer filter will minimize the presence of these particles.
- 5.2.1.2. Care should be taken to provide suitable containment for filter media used in the pretreatment of samples that may have DRPs, because the particles become highly statically charged as they dry out and will “jump” to other surfaces causing contamination.
- 5.2.1.3. Filter media should be individually surveyed for the presence of these particles, and this information reported with the final sample results.

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 250-mL flasks and 50-mL centrifuge tubes.
- 6.3. Centrifuge flasks, 250 mL, disposable.
- 6.4. Centrifuge tubes, 50 mL, disposable.
- 6.5. Low-background gas flow proportional counter.
- 6.6. pH paper.
- 6.7. Stainless steel planchets or other sample mounts: ~2-inch diameter.
- 6.8. Vacuum box may be procured commercially, or constructed. Setup and use should be consistent with manufacturer instructions or laboratory SOP.
- 6.9. Vacuum pump or laboratory vacuum system.

7. Reagents and Standards:

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

Note: Unless otherwise indicated, all references to water should be understood to mean Type I Reagent water (ASTM D1193).

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- 7.1. Barium carrier solution (10 mg Ba/mL, standardization not required): Dissolve 19 g Ba(NO₃)₂ in water add 20 mL concentrated HNO₃ and dilute to 1 L with water.
- 7.2. Ethanol, reagent 95% (C₂H₅OH), available commercially.
- 7.3. Nitric Acid, HNO₃ (15.8M), concentrated, available commercially.
 - 7.3.1. Nitric acid (8 M): Add 506 mL of concentrated HNO₃ to 400 mL of water and dilute to 1 L with water.
 - 7.3.2. Nitric acid (3 M): Add 190 mL of concentrated HNO₃ to 800 mL of water and dilute to 1 L with water.
 - 7.3.3. Nitric acid (0.1 M): Add 6.3 mL of concentrated HNO₃ to 900 mL of water and dilute to 1 L with water.
 - 7.3.4. Nitric acid (0.05 M): Add 3.2 mL of concentrated HNO₃ to 900 mL water. Dilute to 1 L with water.
- 7.4. Nitric acid (3M)/oxalic acid solution (0.05 M): Add 190 mL of concentrated HNO₃ (7.3) and 6.3 grams of oxalic acid dihydrate (C₂H₂O₄·2H₂O), to 800 mL of demineralized water and dilute to 1 L with de-ionized water.
- 7.5. Sodium carbonate (2 M): Dissolve 212 g anhydrous Na₂CO₃ in 800 mL of water, then dilute to 1 L with water.
- 7.6. Sodium hydroxide (12 M): Dissolve 480 g of sodium hydroxide (NaOH) in 500 mL of water and dilute the solution to 1 L in water.

Caution: The dissolution of NaOH is strongly exothermic. Take caution to prevent boiling when preparing this solution. Use of a magnetic stirrer is recommended. Allow to cool prior to use.

- 7.7. Sr-Resin[™] columns,² ~0.7 g resin, small particle size (50–100 μm), in appropriately sized column or pre-packed cartridge.
- 7.8. Strontium carrier solution, 5.00 mg/mL in 0.1-M HNO₃, traceable to a national standards body such as NIST or standardized at the laboratory by comparison to independent standards.
 - 7.8.1. Option 1: Dilute elemental strontium standard to a concentration of 5.00 mg/mL (or mg/g) in 0.1-M HNO₃.
 - 7.8.2. Option 2: To 200 mL de-ionized water, add 6.3 mL HNO₃ and approximately 12.07 g of strontium nitrate (Sr(NO₃)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 7.8.3.
 - 7.8.3. Prior to use, verify the strontium carrier solution concentration as by transferring at least five 1.00-mL portions of the carrier to tared stainless steel planchets. Evaporate to dryness on a hotplate or under a heat lamp using the same technique as that used for samples. Cool in a desiccator 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.

² Available from Eichrom Technologies, Inc., Lisle IL.

- 7.9. ⁹⁰Sr standard solution (carrier free), traceable to a national standards body such as NIST, in 0.5 M HNO₃ solution.
8. Sample Collection, Preservation and Storage
 - 8.1. Samples should be collected in 1-L plastic containers.
 - 8.2. No sample preservation is required if sample analysis is initiated within 3 days of sampling date/time.
 - 8.3. If the sample is to be held for more than three days, HNO₃ shall be added until pH<2.
 - 8.4. If the dissolved concentration of strontium is sought, the insoluble fraction must be removed by filtration before preserving with acid.
9. Quality Control
 - 9.1. Batch quality control results shall be evaluated and meet applicable Analytical Project 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. The laboratory blank should consist of laboratory water.
 - 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 μ_{MR} of 1.0 pCi/L at or below an action level of 8.0 pCi/L. This may be adjusted if the event-specific MQOs are different.
 - 9.3. This method is capable of achieving a ϕ_{MR} 13% above 8 pCi/L. 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 1.0 pCi/L.
10. Calibration and Standardization
 - 10.1. The effective detection efficiency for total radiostrontium (referenced to ⁹⁰Sr) is calculated as the weighted sum of the ⁹⁰Sr and ⁹⁰Y efficiencies that reflects the relative proportions of ⁹⁰Y and ⁹⁰Sr based on the ⁹⁰Y ingrowth after ⁹⁰Sr 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 (see reference 16.5).

10.3. See Appendix A for details on calibration/standardization of the GPC specific to ^{90}Sr and ^{90}Y .

11. Procedure

11.1. For each sample in the batch, aliquant 0.5 L of raw or filtered water into a beaker. Add concentrated HNO_3 with mixing to bring the solution to a pH less than 2.0.

Note: Smaller or larger aliquants may be used if elevated sample activity is present or as needed to meet detection requirements or MQOs. Method validations must be conducted using a volume equivalent in size to the sample size to be used.

11.2. Add 1.00 mL (using a volumetric pipette) of 5 mg/mL strontium carrier and 0.5 mL barium carrier. Record the volume of strontium carrier added and the associated uncertainty of the mass of strontium added.

11.3. Place the beaker on a hotplate (for aliquants of 0.2 L a centrifuge cone in a hot water bath may also be used) and heat the solution to near boiling with occasional stirring.

11.4. Add ~0.4–0.5 mL (8–10 drops) 0.1% phenolphthalein indicator solution per 200 mL of sample. Add 12 M NaOH slowly with occasional stirring until a persistent pink color is obtained.

Note: Additional phenolphthalein solution may be used if needed to provide a clear indication that the pH is above ~8.3. A slight excess of NaOH may be added.

11.5. Add 30 mL of 2-M Na_2CO_3 to the sample and digest for 15 minutes with occasional stirring. Remove the sample from the hot plate and allow the solution to cool and the precipitate to settle.

Note: Samples may be placed in an ice bath to expedite the cooling process.

Note: If greater than a 0.2-L aliquant is used, the supernatant solution is decanted or an aspirator line used to remove as much supernatant solution as possible prior to transfer to a centrifuge tube.

11.6. Transfer the sample to a centrifuge tube and centrifuge for 3 to 5 minutes at 1500–2000 rpm. Discard supernatant solution.

11.7. Add 5 mL of 8-M HNO_3 to the centrifuge tube and vortex to dissolve the precipitate containing Sr.

11.8. If there are no undissolved solids visible in the sample and the sample is not from an RDD, or there is no reason to possibly suspect highly intractable material to be present (e.g., insoluble ceramics), proceed with Step 11.11.

11.9. If the sample contains undissolved solids or may contain intractable material, cover the tube to minimize evaporation of the solution and digest the solution on a hot water bath for 30 minutes. Allow to cool.

11.10. If solids persist, remove by filtering solution through a glass fiber filter (1 μm or finer). The filter containing the solids should be analyzed separately for gross beta activity (^{90}Sr efficiency) to determine whether the AAL may be exceeded

(screening ADLs apply). The solution containing soluble strontium is retained as load solution for Step 11.13.

Note: See Section 12.3.2 for reporting results when liquid and solid fractions are analyzed separately.

- 11.11. Set up a vacuum box for Sr-Resin™ columns or cartridges with minimum 10-15 mL reservoirs according the manufacturer's instructions or laboratory SOP. The initial configuration should permit column effluents during the preconditioning, sample loading and rinses (Steps 11.12 – 11.16) to be discarded to waste.
- 11.12. Add 5 mL of 8-M HNO₃ to precondition the column. Adjust the vacuum as necessary to maintain flow rates at ≤ 3 mL/min. Discard preconditioning solution effluent.

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.

- 11.13. Decrease the vacuum to obtain flow rates of ≤ 1 mL/min. Load the sample from Step 11.8 or 11.10 into the column reservoir. When the solution reaches the top surface of the resin proceed with the next step. Discard column effluent.
- 11.14. Adjust the vacuum as necessary to maintain flow rates at ≤ 3 mL/min. Rinse centrifuge tube with three successive 3 mL portions of 8-M HNO₃ adding the next one after the previous one reaches the top of the resin column. Discard column effluent.
- 11.15. If plutonium, neptunium, or radioisotopes of ruthenium or cerium may be present in the sample, add 10 mL 3-M HNO₃ – 0.05-M oxalic acid solution to each column. Allow the solution to completely pass through the column prior to proceeding. Adjust the vacuum as necessary to maintain flow rates at ≤ 3 mL/min. Discard column effluent.
- 11.16. Remove residual nitric/oxalic acid solution with two 3 mL rinses of 8-M HNO₃, allowing each rinse solution to drain before adding the next one. Adjust the vacuum as necessary to maintain flow rates at ≤ 3 mL/min. Record time and date of the end of last rinse to the nearest 15 minutes as t_1 , "time of strontium separation." Discard column effluent.
- 11.17. Place clean 50 mL centrifuge tubes beneath the columns to catch the strontium eluate before proceeding to the next step.
- 11.18. Decrease the vacuum as necessary to maintain flow rates at ≤ 1 mL/min. Elute strontium from the columns by adding 10 mL of 0.05-M HNO₃.
- 11.19. Preparation of the STS and determination of chemical yield
 - 11.19.1. Clean and label a stainless steel planchet for each STS.
 - 11.19.2. Weigh and record the tare mass of each planchet to the nearest 0.1 mg.
 - 11.19.3. Transfer the strontium eluate from Step 11.18 to the planchet and take to dryness on a hotplate or under a heat lamp to produce a uniformly distributed residue across the bottom of the planchet.

- 11.19.4. When dry, place the sample in an oven at 105–110 °C until shortly before sample test sources are ready for weighing. At that point, remove the STS from the oven and allow it to cool in a desiccator before weighing.
- 11.19.5. Weigh and record the gross mass of each planchet to the nearest 0.1 mg.

Note: If the laboratory cannot operationally ensure that the precipitate has been dried to constant mass, the mass stability of the precipitate should be demonstrated by reheating the precipitate in an oven at 105–110 °C and reweighing. Since sample self-attenuation is not a significant factor in the detection efficiency, the sample may be counted prior to completion of this step if desired.

- 11.19.6. Calculate the chemical yield as presented in Section 12 of this method.

11.20. Counting the Sample Test Source

- 11.20.1. 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.

11.20.1.1. If the presence of ⁸⁹Sr cannot be excluded, and total radiostrontium is being determined as a screen for the presence of ⁸⁹Sr or ⁹⁰Sr, count the STS as soon as practicable after preparation to minimize the ingrowth of ⁹⁰Y into the STS.

11.20.1.2. If the presence of ⁸⁹Sr can be excluded, total radiostrontium will provide isotopic ⁹⁰Sr results and the STS may be counted at any time after preparation.

- 11.20.2. Calculate the total radiostrontium (⁹⁰Sr) sample results using calculations presented in Section 12.

12. Data Analysis and Calculations

12.1. Calculation of Total Radiostrontium

- 12.1.1. When a sample is analyzed for total radiostrontium (equivalent ⁹⁰Sr), 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
 $\varepsilon_{\text{Sr90}}$ = final ⁹⁰Sr detection efficiency
 ε_{Y90} = final ⁹⁰Y detection efficiency
 λ_{Y90} = decay constant for ⁹⁰Y, $3.008 \times 10^{-6} \text{ s}^{-1}$
 t_1 = date and time of the Sr/Y separation
 t_2 = date and time of the midpoint of the count

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:

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

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, Section 4.

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

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

where

$$DF = e^{-\lambda_{\text{Sr90}}(t_1-t_0)} \quad (4)$$

and where

R_a	=	beta gross count rate for the sample (cpm)
R_b	=	beta background count rate (cpm)
$\varepsilon_{\text{Total Sr}}$	=	effective efficiency of the detector for total strontium referenced to ⁹⁰ Sr
Y	=	fractional chemical yield for strontium
V	=	volume of the sample aliquant (L)
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 ⁹⁰ Sr, $7.642 \times 10^{-10} \text{ s}^{-1}$
t_0	=	reference date and time for the sample
t_1	=	date and time of the Sr/Y separation

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:

$$u_{\text{cC}}(AC_{\text{Total Sr}}) = \frac{\sqrt{\frac{R_a}{t_a} + \frac{R_b}{t_b}}}{2.22 \times \varepsilon_{\text{Total Sr}} \times Y \times V \times DF} \quad (5)$$

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:

$$u_c(AC_{\text{Total Sr}}) = \sqrt{u_{\text{cC}}^2(AC_{\text{Total Sr}}) + AC_{\text{Total Sr}}^2 \left(\frac{u^2(\varepsilon_{\text{Total Sr}})}{\varepsilon_{\text{Total Sr}}^2} + \frac{u^2(Y)}{Y^2} + \frac{u^2(V)}{V^2} \right)} \quad (6)$$

where:

- $u(Y)$ = standard uncertainty of fractional chemical yield for strontium
- $u(V)$ = standard uncertainty of the volume of the sample aliquant (L)

12.1.6. If the critical level concentration (S_c) or the minimum detectable concentration (MDC) are requested (at an error rate of 5%), they can be calculated using the following equations:³

$$S_c = \frac{\left[d \times \left(\frac{t_s}{t_b} - 1 \right) + \frac{z_{1-\alpha}^2}{4} \times \left(1 + \frac{t_s}{t_b} \right) + z_{1-\alpha} \sqrt{\left(R_b t_b + d \right) \times \frac{t_s}{t_b} \times \left(1 + \frac{t_s}{t_b} \right)} \right]}{t_s \times 2.22 \times \varepsilon_{\text{Total Sr}} \times Y \times V \times DF} \quad (7)$$

When the Type I decision error rate, α , equals 0.05, $z_{1-\alpha} = 1.645$, and the constant, d , from the Stapleton approximation is set to 0.4, the expression above becomes:

$$S_c = \frac{\left[0.4 \times \left(\frac{t_s}{t_b} - 1 \right) + 0.677 \times \left(1 + \frac{t_s}{t_b} \right) + 1.645 \times \sqrt{\left(R_b t_b + 0.4 \right) \times \frac{t_s}{t_b} \times \left(1 + \frac{t_s}{t_b} \right)} \right]}{t_s \times 2.22 \times \varepsilon_{\text{Total Sr}} \times Y \times V \times DF} \quad (7a)$$

$$\text{MDC} = \frac{\left[\frac{\left(z_{1-\alpha} + z_{1-\beta} \right)^2}{4} \times \left(1 + \frac{t_s}{t_b} \right) + \left(z_{1-\alpha} + z_{1-\beta} \right) \times \sqrt{R_b t_s \times \left(1 + \frac{t_s}{t_b} \right)} \right]}{t_s \times 2.22 \times \varepsilon_{\text{Total Sr}} \times Y \times V \times DF} \quad (8)$$

³ The formulations for the critical level and minimum detectable concentrations are as recommended in MARLAP Section 20A.2.2, Equations 20.54 and Equation 20.74, respectively. For methods with very low numbers of counts, these expressions provide better estimates than do the traditional formulas for the critical level and MDC assuming that the observed variance of the background conforms to Poisson statistics. Consult MARLAP when background variance may exceed that predicted by the Poisson model or when other decision error rates may apply.

When the Type I decision error rate, α , equals 0.05, $z_{1-\alpha} = 1.645$, and the Type II decision error rate, β equals 0.05, $z_{1-\beta} = 1.645$, the expression above becomes:

$$MDC = \frac{\left[2.71 \times \left(1 + \frac{t_s}{t_b} \right) + 3.29 \times \sqrt{R_b t_s \times \left(1 + \frac{t_s}{t_b} \right)} \right]}{t_s \times 2.22 \times \varepsilon_{\text{Total Sr}} \times Y \times V \times DF} \quad (8a)$$

12.2. Chemical Yield for Strontium

12.2.1. Calculate the chemical yield for strontium using the gravimetric data collected in Step 11.18:

$$Y = \frac{m_s F_{\text{Sr}(\text{NO}_3)_2}}{c_c V_c + c_n V} \quad (9)$$

where:

- Y = strontium yield, expressed as a fraction
- m_s = mass of $\text{Sr}(\text{NO}_3)_2$ recovered from the sample (g)
- $F_{\text{Sr}(\text{NO}_3)_2}$ = gravimetric factor for strontium weighed as the nitrate, 414.0 mg Sr/g $\text{Sr}(\text{NO}_3)_2$
- 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/L)
- V = volume of sample aliquant (L)

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

$$u(Y) = Y \times \sqrt{u_r^2(m_s) + \frac{u^2(c_c)V_c^2 + c_c^2 u^2(V_c) + u^2(c_n)V^2 + c_n^2 u^2(V)}{(c_c V_c + c_n V)^2}} \quad (10)$$

where

- $u(\cdot)$ = standard uncertainty of the quantity in parentheses,
- $u_r(\cdot)$ = 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 (Step 12.1.3) in scientific notation ± 1 combined standard uncertainty.
- 12.3.1.2. Volume of sample aliquant and any dilutions used.
- 12.3.1.3. Yield of tracer and its uncertainty.
- 12.3.1.4. Case narrative
- 12.3.1.5. The APS may specify reporting requirements for samples originating from an RDD or other event where intractable

material (e.g., strontium titanate) may be present. If specific guidance is not provided, but intractable materials are likely present in samples, the results for soluble strontium (from the aqueous phase) should be reported per Step 12.3.2.

- 12.3.2. If solid material was filtered from the solution and analyzed separately, the gross beta results from the direct count of filtered solids should be calculated as “gross beta (⁹⁰Sr)” or “gross beta equivalent ⁹⁰Sr” and reported separately in terms of pCi/L of the original volume of sample.

For example:

⁹⁰Sr for Sample 12-1-99:

Filtrate result: $(1.28 \pm 0.15) \times 10^1$ pCi/L

Gross beta (⁹⁰Sr) filtered residue result: $(2.50 \pm 0.30) \times 10^0$ pCi/L

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 Figure 17.4 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 two vacuum box systems (12 ports each) simultaneously, assuming 24 detectors are available. For an analysis of a 500 mL sample aliquant, sample preparation and digestion should take ~3–4 h.
- 13.2.2. Purification and separation of the strontium fraction using cartridges and vacuum box system should take ~0.5–1.2 h.
- 13.2.3. Sample test source preparation takes ~0.75 – 1.5 h.
- 13.2.4. A 100-minute counting time is sufficient to meet the MQO listed in Step 9.2, assuming 0.5 L aliquant, a background of 1 cpm, detector efficiency of 0.3–0.4, and radiochemical yield of at least 0.5.
- 13.3. Total radiostrontium (⁹⁰Sr) data reduction should be achievable between 6 and 9 hours after the beginning of the analysis.
- 13.4. The sample may be recounted following a delay of 1–21 days to verify the radiochemical purity of ⁹⁰Sr. If the source contains pure ⁹⁰Sr, 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 ⁸⁹Sr and ⁹⁰Sr 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. Initial column effluents contain mg/mL levels of barium and should be disposed in accordance with prevailing laboratory, local, state and federal requirements.
 - 15.3. 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.
 - 15.4. 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
 - 16.1. SRW04-11, "Strontium 89, 90 in Water," Eichrom Technologies, Inc., Lisle, Illinois (February 2003).
 - 16.2. "Rapid Column Extraction Method for Actinides and 89/90Sr in Water Samples," S.L. Maxwell III. *Journal of Radioanalytical and Nuclear Chemistry* 267(3): 537-543 (Mar 2006).
 - 16.3. U.S. Environmental Protection Agency (EPA). 2009. *Method Validation Guide for Radiological Laboratories Participating in Incident Response Activities*. Revision 0. Office of Air and Radiation, Washington, DC. EPA 402-R-09-006, June. Available at: www.epa.gov/narel/incident_guides.html and www.epa.gov/erln/radiation.html.
 - 16.4. *Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)*. 2004. EPA 402-B-1304 04-001A, July. Volume I, Chapters 6, 7, 20, Glossary; Volume II and Volume III, Appendix G. Available at: www.epa.gov/radiation/marlap/index.html.
 - 16.5. ASTM D7282 "Standard Practice for Set-Up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements," ASTM Book of Standards 11.02, current version, ASTM International, West Conshohocken, PA.
 - 16.6. SR-04, "Radiochemical Determination of Radiostrontium in Water, Sea Water, and Other Aqueous Media," Eastern Environmental Radiation Facility (EERF) Radiochemistry Procedures Manual, Montgomery, AL, EPA 520/5-84-006 (August 1984).
 - 16.7. ASTM D1193, "Standard Specification for Reagent Water," ASTM Book of Standards 11.02, current version, ASTM International, West Conshohocken, PA
 - 16.8. Nuclear data from NUDAT 2.3 and the National Nuclear Data Center at Brookhaven National Laboratory; available at www.nndc.bnl.gov/nudat2/indx_dec.jsp, database version of 6/30/2009.

17. Tables, Diagrams, Flow Charts and Validation Data

17.1. Validation Data

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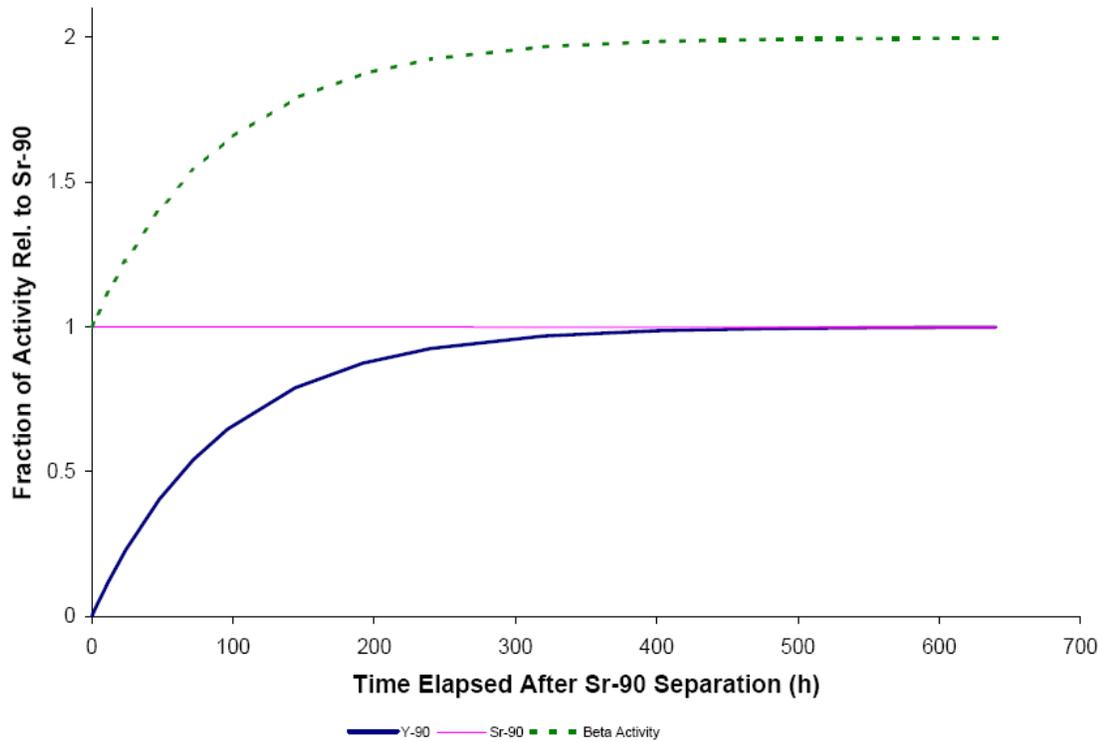
17.2. Nuclide Decay and Radiation Data

Table 17.1. Decay and Radiation Data

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

17.3. Ingrowth and Decay Curves and Factors

In-Growth Curve for ⁹⁰Y in ⁹⁰Sr



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

Table 17.2. 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 ⁸⁹Sr

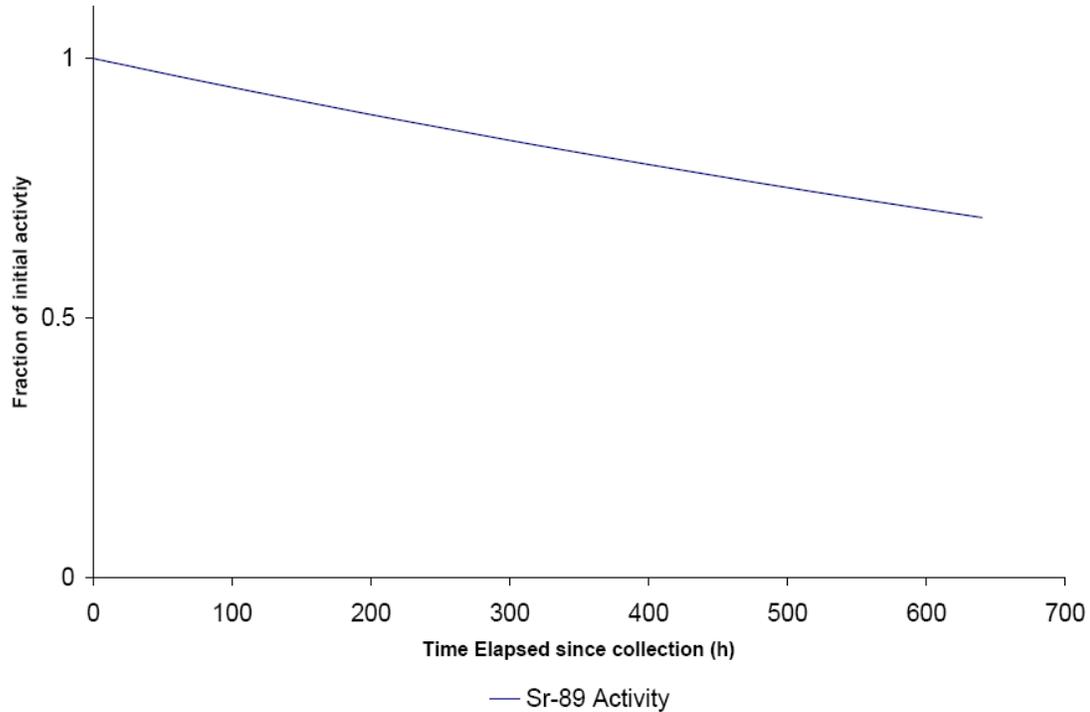


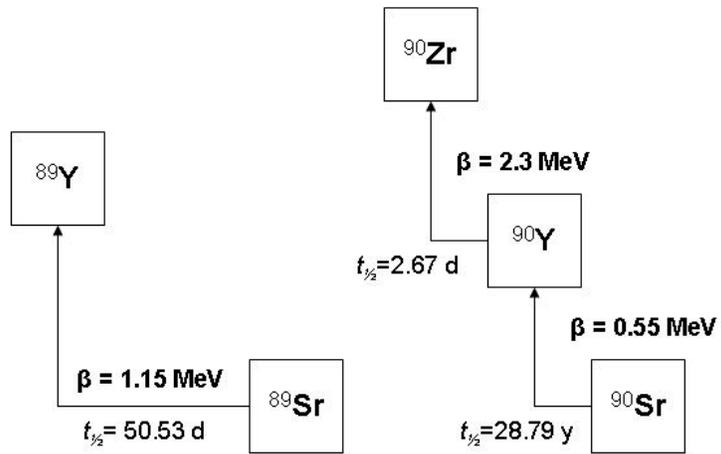
Table 17.3. Decay Factors for ⁸⁹Sr

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 = (⁸⁹Sr activity/⁸⁹Sr activity at zero hours of ingrowth)

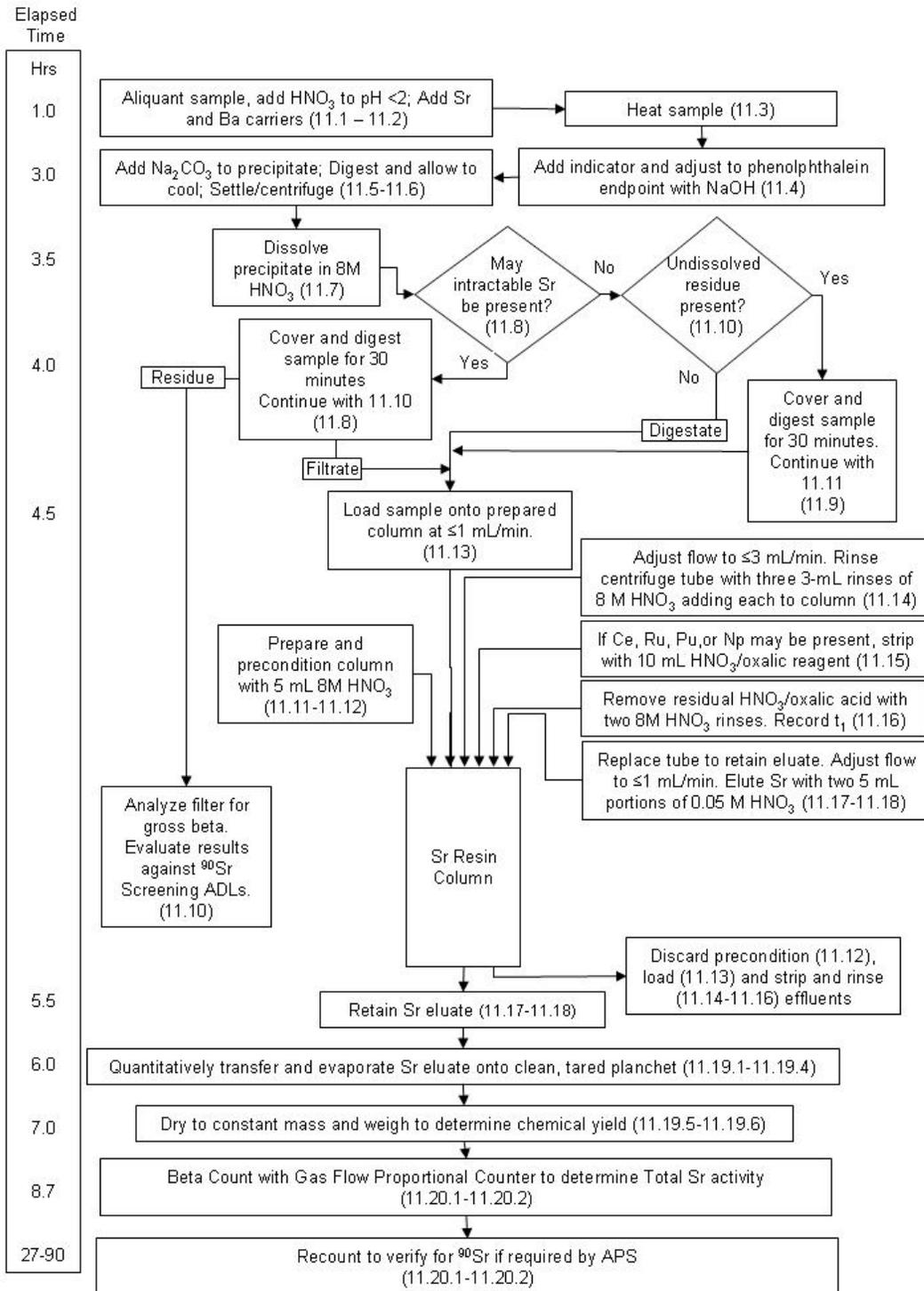
17.4. Decay Schemes for ^{89}Sr and ^{90}Sr

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



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

17.5. Process Flow with Typical Processing Times (assumes no filtration necessary)



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 0.5-M 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 a strontium carrier to 10 mL of 0.05-M 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.

Note: 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 1.2.3.1.

Note: 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 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 0.05-M HNO_3 .
- 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 ^{89}Sr WCSs for sufficient time to accumulate at least 10,000 net counts.

A1.3. Preparation of ^{90}Sr and ^{90}Y WCSs: Separate WCSs for ^{90}Sr and ^{90}Y are prepared by chemically separating ^{90}Y from a standard solution of ^{90}Sr .

A1.3.1. For each ^{90}Sr WCS to be prepared, and for the associated blank, add 1 mL of 5 mg/mL strontium carrier to a disposable 50-mL centrifuge tube. 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 1.3.1.

A1.3.2. For each ^{90}Sr WCS, add a precisely known amount of traceable ^{90}Sr solution to a 50-mL centrifuge tube. Sufficient activity should be present at the point of the count to permit accumulation of greater than 10,000 ^{90}Sr and 10,000 ^{90}Y 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 Sr Resin column for each ^{90}Sr WCS and for the associated blank. Condition each column with 5 mL of 3-M HNO_3 . Column effluents are discarded to waste.

A1.3.4. Place a clean centrifuge tube under each column to catch all combined ^{90}Y 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 ^{90}Sr solution onto the column. The load solution effluent containing ^{90}Y is retained.

A1.3.6. Rinse the centrifuge tube with three successive 2-mL portions of 3-M HNO_3 adding each of the rinses to the column after the previous rinse has reached the upper surface of the resin. These effluents also contain ^{90}Y and are retained.

A1.3.7. Rinse the column with 5 mL of 3 M HNO_3 and retain the column effluents containing ^{90}Y . Record the date and time that the final rinse solution leaves the column to the nearest 5 minutes as t_1 , "Time of ^{90}Y Separation." Remove

the centrifuge tube that has the combined ^{90}Y 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 10 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 used for sample test sources.

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 equivalent to that of the associated blank (i.e., ~0.0 mg). Higher residue mass may indicate the breakthrough of strontium and will result in high bias in the ^{90}Y efficiency. If blank corrected net residue mass exceeds 3% of the strontium carrier added, determine and address the cause for the elevated mass and repeat the process.
 - 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.

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
- t_1 = date and time of the Sr/Y separation
- t_2 = date and time of the midpoint of the ⁹⁰Sr count

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:

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

where

- $\epsilon_{Sr90,i}$ = ⁹⁰Sr detection efficiency for the i^{th} WCS
- $\bar{\epsilon}_{Y90}$ = average ⁹⁰Y detection efficiency (from Step A3.2)
- $R_{s,i}$ = beta gross count rate for the i^{th} WCS (in 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.

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

where

- $\epsilon_{Sr90,i}$ = ⁹⁰Sr detection efficiency determined for the i^{th} WCS in A2.2,
- n = number of WCSs prepared and counted.

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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

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

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

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

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.

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

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:

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

(A10)

where

- $u(\cdot)$ = standard uncertainty of the value in parentheses,
- $u_r(\cdot)$ = 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

- $u(\cdot, \cdot)$ = estimated covariance of the two quantities in parentheses,
- $r(\cdot, \cdot)$ = estimated correlation coefficient of the two quantities in parentheses,
- $u(\cdot)$ = standard uncertainty of the quantity in parentheses,
- $u_r(\cdot)$ = 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

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$AC_{Sr89\text{ std}}$	= 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, $1.372 \times 10^{-2} \text{ d}^{-1}$
t_0	= reference date and time for the ⁸⁹ Sr standard
t_1	= date and time at the midpoint of the ⁸⁹ Sr count

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

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

where

$\epsilon_{Sr89,i}$	= 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:

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

where

$u(\cdot)$	= standard uncertainty of the value in parentheses,
$u_r(\cdot)$	= 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 $^{89}\text{Sr} = 1.587 \times 10^{-7} \text{ s}^{-1}$
- $\lambda_{\text{Sr}90}$ = decay constant for $^{90}\text{Sr} = 7.642 \times 10^{-10} \text{ s}^{-1}$
- t_0 = collection date and time for the sample

t_{sep} = date and time of the Sr/Y separation
 t_i = date and time of the midpoint of the i^{th} count of the STS

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

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

$$u(b_1, b_2) = \begin{cases} (DF_{Sr90,1}F_{Y90,2} + DF_{Sr90,2}F_{Y90,1})u(\varepsilon_{Sr90,1}, \varepsilon_{Y90,1}) \\ + DF_{Sr90,1}DF_{Sr90,2}u^2(\varepsilon_{Sr90,1}) + F_{Y90,1}F_{Y90,2}u^2(\varepsilon_{Y90,1}), & \text{using only one detector} \quad (B10) \\ b_1b_2u_r^2(AC_{Sr90\text{ std}}), & \text{using two detectors} \end{cases}$$

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:

$$AC_{Sr89} = \frac{b_2R_{n,1} - b_1R_{n,2}}{2.22 \times X \times V \times Y} \quad (B13)$$

$$AC_{Sr90} = \frac{a_1R_{n,2} - a_2R_{n,1}}{2.22 \times X \times V \times Y} \quad (B14)$$

where:

$$X = a_1b_2 - a_2b_1 \quad (B15)$$

and where:

2.22 = conversion factor from dpm to pCi
 Y = chemical yield for strontium
 V = sample volume (L)

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

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

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

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

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

$$u_c(AC_{Sr90}) = \left[u_{cC}^2(AC_{Sr90}) + AC_{Sr90}^2 \left(\frac{u^2(V)}{V^2} + \frac{u^2(Y)}{Y^2} + \frac{a_2^2 u^2(b_1) + a_1^2 u^2(b_2) - 2a_1 a_2 u(b_1, b_2)}{X^2} \right) + AC_{Sr89}^2 \frac{a_2^2 u^2(a_1) + a_1^2 u^2(a_2) - 2a_1 a_2 u(a_1, a_2)}{X^2} \right]^{1/2} \quad (\text{B19})$$