



Standard Test Method for Radiochemical Determination of Strontium-90 in Soil¹

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1. Scope

1.1 This test method is applicable to the determination of strontium-90 in soil at levels of detection dependent on count time, sample size, detector efficiency, background, and recovery.

1.2 This test method is designed for the analysis of ten grams of soil, previously collected and treated as described in Practices C 998 and C 999. This test method may not be able to completely dissolve all soil matrices. The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

C 859 Terminology Relating to Nuclear Materials³

C 998 Practice for Sampling Surface Soil for Radionuclides

C 999 Practice for Soil Sample Preparation for the Determination of Radionuclides

D 1193 Specification for Reagent Water

3. Terminology

3.1 For definitions of terms used in this standard, refer to Terminology C 859.

4. Summary of Test Method

4.1 Strontium is extracted from soil with a mixture of nitric, hydrochloric, and hydrofluoric acids in the presence of strontium carrier. Strontium is isolated by extraction chromatography and evaporated on a planchet for recovery determination and subsequent beta counting. This test method describes one of the possible approaches to determine strontium-90 in soil.

The chemical yield is typically 95 % with a detection limit of about 0.004 Bq/g for a ten gram sample.

5. Significance and Use

5.1 Because soil is an integrator and a reservoir of long-lived radionuclides, and serves as an intermediary in several pathways of potential exposure to humans, knowledge of the concentration of strontium-90 in soil is essential. A soil sampling and analysis program provides a direct means of determining the concentration and distribution of radionuclides in soil. A soil analysis program has the most significance for the preoperational monitoring program to establish baseline concentrations prior to the operation of a nuclear facility. Soil analysis, although useful in special cases involving unexpected releases, may not be able to assess small incremental releases.

6. Interferences

6.1 The presence of strontium-89 in the sample will interfere with the determination of strontium-90.

6.2 Large concentrations of strontium, calcium, barium, or lead in the soil sample could interfere with the extraction chromatographic separation by loading the column with these elements. Section 12.1 discusses procedures for accounting for the stable strontium.

6.3 The final strontium form is a nitrate salt and it is hygroscopic. Care must be taken when determining the mass of the final precipitate to avoid mass fluctuations and changes in physical form or self-absorption due to water absorption from the atmosphere.

7. Apparatus

7.1 *Beta Particle Counter*—A shielded low-background proportional detector with appropriate electronics and computational capabilities to control operations. The efficiency of the system should be greater than 35 percent for strontium-90 with a background of less than a few counts per minute. The measurement of strontium-90 can also be conducted by liquid scintillation spectrometry provided equivalency is demonstrated.

7.2 *Counting Dishes*—Typically, 50 mm diameter, 6 mm deep, stainless steel counting dishes, although other sizes may be used that are compatible with the measurement instrumentation.

7.3 *Heat Lamp.*

7.4 *Muffle Furnace.*

¹ This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Withdrawn.

- 7.5 Whatman #2 Filter Paper.
 7.6 Borosilicate Glass Erlenmeyers Flasks and Beakers.
 7.7 PTFE Beakers.
 7.8 Stir/Hot Plate.
 7.9 Teflon Coated Magnetic Stir Bars.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined in Specification D 1193, Type III.

8.3 *Strontium Carrier*—Dissolve 10.00 grams of $\text{Sr}(\text{NO}_3)_2$ in 0.1M HNO_3 and dilute to one liter with 0.1M HNO_3 [10 mg $\text{Sr}(\text{NO}_3)_2$ per mL]. If insoluble material is observed, filter the carrier solution through 0.1-0.45 μm filter media.

8.4 *29 M Hydrofluoric Acid (48 %)*—Concentrated hydrofluoric acid.

8.5 *12 M Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid.

8.6 *16 M Nitric Acid (sp gr 1.42)*—Concentrated nitric acid.

8.7 *8 M Nitric Acid*—Mix one volume of concentrated nitric acid with one volume of water.

8.8 *0.1 M Nitric Acid*—Add 6.25 mL concentrated nitric acid to water and dilute to one liter.

8.9 *0.05 M Nitric Acid*—Add 3.10 mL concentrated nitric acid to water and dilute to one liter.

8.10 *Extraction Chromatographic Column*—A 2 mL strontium-specific extraction chromatographic column including funnel reservoir.⁵

9. Standardization and Calibration

9.1 *Standardization of Strontium Carrier*—The standardization of the strontium carrier should be conducted in triplicate. Standardization of the strontium carrier and yield calculations may also be performed by plasma spectrometry analysis provided equivalency is demonstrated.

9.1.1 Clean and weigh the counting dish.

9.1.2 Pipette 1.000 mL of strontium carrier solution into the counting dish.

9.1.3 Place the counting dish in a fume hood under a heat lamp until the sample is at constant weight.

9.1.4 Cool the sample counting dish and counting dish/residue and reweigh.

9.1.5 Average the three net residue weights and record the average as the amount of the strontium nitrate in the carrier.

9.2 *Calibration of Beta Counting System for Strontium-90*—This calibration should be carried out in triplicate for each volume of carrier pipetted.

9.2.1 Pipette 0.500, 1.000, 1.500 and 2.000 mL of strontium carrier into separate small beakers and label. If the samples are expected to contain significant amounts of stable strontium, larger volumes of strontium carrier should be used provided the resin volume is adjusted accordingly.

9.2.2 To each beaker, add a known amount (approximately 2 Bq) of a strontium-90 standard solution traceable to a national standards body.

9.2.3 Evaporate the solution to near dryness and redissolve it in 5 mL of the 8 M nitric acid.

9.2.4 Transfer the solution to a previously prepared and conditioned 2 mL strontium extraction chromatographic column which has been conditioned with 5 mL of 8 M nitric acid.

9.2.5 Rinse the beaker with 3 mL of 8 M nitric acid and add to the column after the feed has passed through.

9.2.6 Wash the column with three 3 mL portions of 8 M nitric acid, draining after each addition. Discard the column effluent and washes, which contains the yttrium-90.

9.2.7 Record the end of the third rinse as strontium-90/yttrium-90 separation time.

9.2.8 Elute the strontium with 10 mL of 0.05 M nitric acid and collect in a 25 mL properly labeled clean beaker.

9.2.9 Evaporate the strontium eluant, by using a heat lamp or other suitable heat source, on to a previously cleaned and weighed counting dish by adding small portions (ca. 3 mL) to the dish and allowing each portion to evaporate to near dryness between additions.

9.2.10 Evaporate all the solution under a heat lamp, or other suitable heat source, cool, and weigh to constant weight.

9.2.11 Calculate the residue weight and determine the chemical recovery.

9.2.12 Count each standard for 100 minute intervals overnight. Typically, this would result in ten separate measurements.

9.2.13 Collect the 100 minute count data as a function of time since separation. Use a computer program to plot the recovery corrected net count rate and estimate the extrapolation to separation time. Alternatively, determine the mean counting efficiency from each of the counts, correct for yttrium-90 ingrowth.

9.2.14 Plot the counting efficiency of the strontium-90 as a function of sample weight to obtain a counting efficiency curve. Fit the mass attenuated counting efficiency to a linear expression and use this expression for each sample to determine the counting efficiency.

10. Precautions

10.1 Strong acids are used during this analysis. Safety glasses and gloves must be worn when handling these solutions. Extreme care should be exercised in using hydrofluoric acid and other hot concentrated acids.

10.2 Hydrofluoric acid is a highly corrosive acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid is similar to other acids in that the initial extent of a

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Reagent Chemicals and Standards*, by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the *United States Pharmacopeia*.

⁵ Sr Resin prepackaged columns from Eichrom Technologies, LLC., Darien, IL, have been found to be satisfactory for this purpose.

burn depends on the concentration, the temperature, and the duration of contact with the acid. Hydrofluoric acid differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left untreated. Due to the serious consequences of hydrofluoric acid burns, prevention of exposure or injury of personnel is the primary goal. Utilization of appropriate laboratory controls (hoods) and wearing adequate personal protective equipment to protect from skin and eye contact is essential.

11. Sampling

11.1 Collect the sample in accordance with Practice C 998.

11.2 Prepare the sample for analysis in accordance with Practice C 999.

12. Procedure

12.1 The soil sample is analyzed for strontium-90 in duplicate. To account for the stable strontium in the soil, the second aliquot of the same soil is analyzed without carrier. The analysis must understand the limitations of using duplicate samples. This approach is based on the concept that “identical” chemical yields are obtained for both samples with and without stable strontium added. This assumption results in a potentially significant contribution to the uncertainty analysis, as discussed in 14.6. Place two 10.000 gram aliquots of dried soil into each of two 500 mL Erlenmeyer flasks. Add 2.000 mL of strontium carrier into one of the flasks and label. Add no carrier to the other aliquot. As an alternative for determining the chemical yield, strontium-85 may be used, but it would be up to the user to determine equivalency. If the indigenous strontium content of the sample has been previously determined, the amount of strontium carrier added may be adjusted and the analysis of the second aliquot may not be required.

12.2 Ash the samples overnight at 500°C.

12.3 Cool, add 75 mL concentrated nitric acid and then 25 mL of concentrated hydrochloric acid.

12.4 Cover the Erlenmeyer flask and heat on a hot plate in the fume hood for several hours with stirring using Teflon-coated magnetic stirring bars.

12.5 Cool and dilute with an equal volume of water.

12.6 Transfer the sample to a 250 mL centrifuge bottle with water and centrifuge.

12.7 Decant the supernate through Whatman #2 24 cm fluted filter paper and save the filtrate.

12.8 Transfer the residue remaining in the centrifuge bottle with a mixture of 75 mL concentrated nitric acid and 25 mL concentrated hydrochloric acid to the original Erlenmeyer flask and repeat 12.4 and 12.5.

12.9 Filter the solution through Whatman #2 filter paper used in 12.7 and combine the filtrate, without centrifugation, with the original supernate from 12.7.

12.10 Dry the filter in a low temperature oven and ash overnight at 500° C in a 400 mL beaker.

12.11 Cool and transfer the ash to a 250 mL PTFE beaker with 15 mL concentrated nitric acid. Add 50 mL concentrated hydrofluoric acid to the PTFE beaker.

12.12 Cover the beaker and digest overnight on low heat.

12.13 Evaporate to dryness and repeat the acid addition and digestion in 12.11 and 12.12 one more time if a residue remains.

12.14 When there is no residue, add 15 mL concentrated nitric acid and evaporate to dryness.

12.15 Add 15 mL 8 M nitric acid, cover, and heat to boiling for 5 minutes.

12.16 Cool and add 50 mL water.

12.17 Filter through Whatman #2 filter paper and combine the filtrate with the original supernate and first filtrate, 12.9. Split the sample in two by volume. This results in two samples with carrier and two samples without carrier, each with five grams of soil.

12.18 Carefully evaporate to less than 5 mL. Do not allow the samples to go dry.

12.19 Slowly add concentrated nitric acid to bring the volume up to 5 mL and slowly add an additional 5 mL water to achieve a final acid concentration of 8 M HNO₃.

12.20 Prepare a 2 mL extraction column and condition with 5 mL of 8 M nitric acid.

12.21 Transfer the sample to the column incrementally and drain to the top of the column.

12.22 Rinse the beaker with 3 mL of 8 M nitric acid and add to the column.

12.23 Rinse the column three times with 3 mL portions of 8 M nitric acid, draining completely before the next addition. Discard the rinses.

12.24 Record this time as the strontium-90/yttrium-90 separation time.

12.25 Elute the strontium with 10 mL of 0.05 M nitric acid and collect in a clean labeled beaker.

12.26 Evaporate the strontium eluant onto a cleaned and weighed counting dish by adding small portions (ca. 3 mL) to the dish in a hood under a heat lamp and allowing each portion to evaporate to near dryness between additions.

12.27 Evaporate completely, cool, and reweigh to constant weight.

13. Calculations

13.1 Calculate the residue weight by subtracting the tare weight of the counting dish from the weight of the dish plus residue for all samples.

13.2 Calculate the net residue weight by subtracting the residue weight of the sample without carrier from the residue weight of the sample with carrier.

13.3 Calculate the chemical recovery by dividing the net residue weight (in mg) by the amount of carrier added (normally 20 mg).

14. Strontium-90 Measurements

14.1 Start the count of the samples within four hours of the separation.

14.2 Count the sample long enough to meet the facility statistical measurement requirements. Some samples may require overnight counts. Confirmation of the presence of strontium-90 may be accomplished by an additional count after yttrium-90 ingrowth.

14.3 Subtract the background count rate from the sample count rate to obtain the net count rate, i.e., perform the following calculation:

$$R_n = R_a - R_b = \frac{C_a}{t_a} - \frac{C_b}{t_b}$$

where:

- R_n = the net count rate,
- R_a = the gross sample count rate,
- R_b = is the background count rate,
- C_a = the sample aliquant counts,
- t_a = the sample aliquant count duration,
- C_b = the background counts, and
- t_b = the background count duration.

14.4 Calculate the 1-sigma standard deviation of the net count rate as:

$$\sigma_{R_n} = \sqrt{\frac{R_a}{t_a} + \frac{R_b}{t_b}} = \sqrt{\frac{C_a}{t_a^2} + \frac{C_b}{t_b^2}}$$

14.5 Calculate the activity concentration of strontium-90 in the sample at the time of the chemical separation, i.e., activity per unit mass, as:

$$A_{Sr} = \frac{R_n}{E \cdot y \cdot W \left[1 + \left(\frac{\lambda_y}{\lambda_y - \lambda_{Sr}} \right) \left(e^{-\lambda_{Sr} \frac{T_1 + T_2}{2}} - e^{-\lambda_y \frac{T_1 + T_2}{2}} \right) \right]}$$

where:

- T_1 = the elapsed time between chemical separation (12.24) and the beginning of the count time,
- T_2 = the elapsed time between chemical separation (12.24) and the end of the count time,
- λ_y = the decay constant of yttrium-90,
- λ_{Sr} = the decay constant of strontium-90,
- E = the counting efficiency obtained from the counting efficiency curve generated in 9.2.14,
- y = the chemical yield, and
- W = the mass of the sample, or the mass the sample represents.

If the counting is conducted within four hours of separation, the equation reduces to:

$$A_{Sr} = \frac{R_n}{E \cdot y \cdot W}$$

14.6 Calculate the uncertainty of the activity concentration of strontium-90 as:

$$\sigma_{A_{Sr}} = A_{Sr} \sqrt{\left(\frac{\sigma_{R_n}}{R_n} \right)^2 + \left(\frac{\sigma_E}{E} \right)^2 + \left(\frac{\sigma_y}{y} \right)^2 + \left(\frac{\sigma_W}{W} \right)^2}$$

where:

- σ_E = the 1-sigma uncertainty of the counting efficiency,
- σ_y = the 1-sigma uncertainty of the chemical yield, and
- σ_W = the 1-sigma uncertainty of the sample mass.

For simplicity, we have assumed that there is no uncertainty associated with the times (T_1 and T_2) or with the decay constants. The uncertainty from other parameters should be included if they can be measured or estimated. An examination of the coefficient of variation (COV, standard deviation/mean) of the fifteen pairs of laboratory duplicate-aliquot results in Table 1 (Fall 1994 results excluded) shows that estimates of the overall COV encompasses the relative one-sigma uncertainties

in estimates of chemical yield, counting efficiency, and sample mass, and suggests that on average the COV attributable to chemical yield for samples of the same soil is likely to be less than or equal to 0.020. If sufficient activity is present in the sample, another option is to confirm the determination by following the ingrowth of the yttrium-90 daughter.

14.7 An estimate of the a priori Minimum Detectable Amount (MDA) associated with this method can be calculated using:

$$MDA = \frac{4.65 \sqrt{C_b} + 2.71}{t_b \cdot E \cdot Y \cdot W \left[\left(1 + \left(\frac{\lambda_y}{\lambda_y - \lambda_{Sr}} \right) \right) \left(\frac{e^{-\lambda_{Sr} T_1} - e^{-\lambda_{Sr} T_2}}{\lambda_{Sr}} \right) - \left(\frac{\lambda_y}{\lambda_y - \lambda_{Sr}} \right) \left(\frac{e^{-\lambda_y T_1} - e^{-\lambda_y T_2}}{\lambda_y} \right) \right]}$$

15. Precision and Bias

15.1 To estimate the precision and bias of this test method, soil samples from the Department of Energy-Environmental Measurements Laboratory-Quality Assurance Program were analyzed for strontium-90 by a single laboratory using this method. The source of the soil is from near nuclear facilities and the strontium-90 determined by repeated analyses by the Environmental Measurements Laboratory (EML), which is used as a reference value. The uncertainty in the EML result is the standard deviation of the mean of repeated analyses, typically six. The results of 16 different concentration samples analyzed in duplicate were conducted by a single laboratory and the comparison to the EML result are presented in Table 1. The stated uncertainty from the lab results is based only on counting statistics.

15.2 Analysis of the measurements collected in Table 1 shows that the average of the 32 ratios of the lab to EML values is 1.01, a difference of 1 % relative to the EML values. This 1 % is an estimate of possible bias. The standard deviation of the ratio values is 0.12, 12 % relative to the EML values. This 12 % is an estimate of precision. Employing a two-sided one-sample t-test on the average ratio showed no statistically significant difference from one (at the five percent significant level), meaning no statistically significant indication of bias was observed in the data set.

15.3 The 2 results from fall 94 however have extremely low ratio values (0.61 and 0.68 when no other ratio values are less than 0.92). This indicates that there may have been an unknown problem with the measurements from that period. Analysis of the measurements collected in Table 1 excluding the fall 94 values shows that the average of the 30 ratios of the lab to EML values is 1.04, a difference of four percent relative to the EML values. This four percent is an estimate of possible bias. The standard deviation of the ratio values is 0.08, eight percent relative to the EML values. This eight percent is an estimate of precision. Employing a two-sided one-sample t-test on the average ratio showed a statistically significant difference from one (at the five percent significant level), meaning a statistically significant indication of bias was observed in the data set when the possible outlying values were removed from consideration.

TABLE 1 Comparison of Single Lab and EML Results

Sample	Lab Results (Bq/kg)	Error (1 σ) ^A	EML Result (Bq/kg)	Uncertainty ^B	Ratio Lab/EML
Spring 1993	40.8	0.2	41.7	2.1	0.98
Spring 1993	41.1	0.2			0.99
Fall 1993	5.50	0.17	5.4	2.2	1.02
Fall 1993	5.50	0.17			1.02
Spring 1994	9.67	0.17	8.79	0.53	1.10
Spring 1994	10.00	0.17			1.14
Fall 1994	2.02	0.07	3.30	0.33	0.61
Fall 1994	2.25	0.06			0.68
Spring 1995	13.27	0.17	11.3	0.17	1.17
Spring 1995	13.47	0.18			1.19
Fall 1995	7.20	0.17	7.81	0.28	0.92
Fall 1995	7.35	0.17			0.94
Spring 1996	1309	100	1340	113	0.98
Spring 1996	1314	100			0.98
Fall 1996	72.5	0.3	69.9	5.1	1.04
Fall 1996	71.0	0.3			1.02
Spring 1997	46.4	0.3	40.3	0.4	1.15
Spring 1997	43.7	0.3			1.08
Fall 1997	36.2	0.3	34.8	1.0	1.04
Fall 1997	35.2	0.3			1.01
Spring 1998	13.6	0.2	13.1	0.3	1.04
Spring 1998	12.9	0.2			0.98
Fall 1998	45.4	0.3	39.6	0.3	1.15
Fall 1998	44.1	0.3			1.11
Spring 1999	32.3	0.2	32.4	0.5	1.00
Spring 1999	33.5	0.2			1.03
Fall 1999	14.9	0.2	13.0	0.5	1.15
Fall 1999	14.3	0.2			1.10
Spring 2000	19.0	0.2	20.2	0.2	0.94
Spring 2000	18.7	0.2			0.93
Fall 2000	46.7	0.3	50.4	2.0	0.93
Fall 2000	47.3	0.3			0.94
					All Results
					Mean 1.01
					Std. Dev 0.12
					N 32
					Without Fall 94 Results
					Mean 1.04
					Std. Dev 0.08
					N 30

^A The 1 σ error of the lab results is based on counting statistics for that measurement.

^B The stated uncertainty of the EML value is the standard deviation of the mean of (usually) six repeated measurements.

16. Keywords

16.1 beta counting; extraction chromatography; soil analysis; strontium-90; strontium-90 determination

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