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Designation: C1507 – 07

Standard Test Method for Radiochemical Determination of Strontium-90 in Soil[1]

This standard is issued under the fixed designation C1507; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 C998 and C999. 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]

C859 Terminology Relating to Nuclear Materials
C998 Practice for Sampling Surface Soil for Radionuclides
C999 Practice for Soil Sample Preparation for the Determination of Radionuclides
D1193 Specification for Reagent Water

3. Terminology

3.1 For definitions of terms used in this standard, refer to Terminology C859.

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*.

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[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.

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7.4 *Muffle Furnace*.
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.[3] 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 D1193, Type III.

8.3 *Strontium Carrier*—Dissolve 10.00 grams of $Sr(NO_3)_2$ in 0.1*M* HNO_3 and dilute to one liter with 0.1*M* HNO_3[10 mg $Sr(NO_3)_2$ 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*—A2 mL strontium-specific extraction chromatographic column including funnel reservoir.[4]

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 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 effect is a

[3] *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*.

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

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