Designation: ~~C1507 – 12~~ C1507 – 20

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 ^{90}~~strontium-90~~Sr in soil at levels of detection dependent on count time, sample size, detector efficiency, background, and chemical yield.

1.2 This test method is designed for the analysis of ~~ten grams~~ 10 g 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 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.4 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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
D7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements

3. Terminology

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

3.1 *Definitions:*

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

[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.

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 ^{90}~~strontium-90~~Sr 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 ^{90}~~strontium-90~~Sr 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 may bias the reported ^{90}~~strontium-90~~Sr results using this method.

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~~Subsection 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~~ 35 % for ^{90}~~strontium-90~~Sr with a background of less than a few counts per minute. Practice D7282 may contain other useful information on the set-up, calibration, and usage of such instrumentation. The measurement of ^{90}~~strontium-90~~Sr and ^{90}~~yttrium-90~~Y 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.*

7.5 ~~*Whatman #2*~~*Cotton Cellulose Filter Paper or* ~~*equivalent.*~~*Equivalent.*

7.6 *Borosilicate Glass Erlenmeyers Flasks and Beakers.*

7.7 ~~*PTFE*~~ *Polytetrafluoroethylene (PTFE) Beakers.*

7.8 *Stir/Hot Plate.*

7.9 ~~*Polytetrafluoroethylene (PTFE)*~~ *PTFE 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~~g 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*—A 2 mL extraction chromatographic column (including funnel reservoir) containing ~~4,4(5)-di-t-butylcyclohexane 18-crown-6~~4,4′(5′)-di-t-butylcyclohexano 18-crown-6 (crown ~~Ether)~~ether) in ~~1-octanol~~1-octanol on an inert chromatographic support.[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*~~ *^{90}Sr*—This calibration should be carried out in triplicate for each volume of carrier pipetted.

9.2.1 Pipette 0.500, 1.000, ~~1.500~~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.

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[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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[4] The sole source of supply of the Sr Resin prepackaged columns ~~from~~ known to the committee at this time is Eichrom Technologies, LLC., Lisle, ~~IL, have been found to be satisfactory for this purpose. The Eichrom Technologies Sr Resin is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative to this patented item~~ IL. If you are aware of alternative suppliers, please provide this information to ASTM International ~~headquarters.~~Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

9.2.2 To each beaker, add a known amount (approximately 2 Bq) of a ^{90}~~strontium-90~~Sr 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~~ 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~~ 8 *M* nitric acid, draining after each addition. Discard the column effluent and washes, which contains the ^{90}~~yttrium-90.~~Y.

9.2.7 Record the end of the third rinse as ^{90}~~strontium-90/yttrium-90~~Sr/^{90}Y 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 eluate, by using a heat lamp or other suitable heat source, on to a previously cleaned and weighed counting dish by adding small portions (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~~ 100-min intervals overnight. Typically, this would result in ten separate measurements.

9.2.13 Collect the ~~100 minute~~ 100-min 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 ^{90}~~yttrium-90~~Y ingrowth.

9.2.14 Plot the counting efficiency of the ^{90}~~strontium-90~~Sr 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 and toxic acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid ~~is similar to other acids in that the initial extent of a 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~~ Familiarization and compliance with the Safety Data Sheet is essential.

11. Sampling

11.1 Collect the sample in accordance with Practice C998.

11.2 Prepare the sample for analysis in accordance with Practice C999.

12. Procedure

12.1 The soil sample is analyzed for ^{90}~~strontium-90~~Sr in duplicate. To account for the stable strontium in the soil, the second

aliquot of the same soil is analyzed without carrier. The analyst 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 flask and label accordingly. As an alternative for determining the chemical yield, ^{85}~~strontium-85~~Sr may be used as an internal standard, 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 ~~500ºC~~ 500 ºC in the Erlenmeyer flasks.

12.3 Cool, add 75 mL concentrated nitric acid and then ~~25 mL~~ 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 PTFE-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~~ cotton cellulose 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 Place the filter in a 400 mL beaker, dry the filter in a low temperature oven and ash overnight at 500º 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~~ 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~~cotton cellulose 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 representing ~~five grams~~ 5 g of the original soil sample.

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* HNO_3.