~~Designation:D 5811–00~~ Designation: D 5811 – 08

An American National Standard

Standard Test Method for Strontium-90 in Water[1]

This standard is issued under the fixed designation D 5811; 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 covers the determination of radioactive ^{90}Sr in environmental water samples (for example, non-process and effluent waters) in the range of 0.037 Bq/L (1.0 pCi/L) or greater.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 This test method has been used successfully with tap water. It is the user's responsibility to ensure the validity of this test method for samples larger than 1 L and for waters of untested matrices.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1129 Terminology Relating to ~~Water~~ Water

D 1193 Specification for Reagent ~~Water[2]~~ Water

D 1890 Test Method for Beta Particle Radioactivity of ~~Water~~ Water

D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee ~~D-19~~D19 on ~~Water[2]~~ Water

D 3370 Practices for Sampling Water from Closed ~~Conduits[2]~~ Conduits

D 3648~~Practices for the Measurement of Radioactivity[3]~~ Practices for the Measurement of Radioactivity

D 4448 Guide for Sampling Ground-Water Monitoring Wells

D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

D 6001 Guide for Direct-Push Ground Water Sampling for Environmental Site Characterization

D 7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129.

4. Summary of Test Method

4.1 This test method is based on the utilization of solid phase extraction of strontium from water samples with detection of the radioactive strontium by gross beta gas proportional counting.

4.2 An aliquant of the sample is measured into a beaker, strontium carrier added, digested with nitric acid, sorbed on an ion exchange column, eluted, evaporated to dryness, dissolved in nitric acid (8M), selectively sorbed on a solid phase extraction column, eluted with dilute nitric acid, dried on a planchet, and counted for beta radiation.

4.3 Fig. 1 shows a flow diagram for this method.

5. Significance and Use

5.1 This test method was developed to measure the concentration of ^{90}Sr in non-process water samples. This test method may be used to determine the concentration of ^{90}Sr in environmental samples.

6. Interferences

6.1 Significant amounts of stable strontium present in the sample will interfere with the yield determination. If it is known or

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[1] This test method is under the jurisdiction of ASTM Committee ~~D-19~~D19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

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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* ~~, Vol 11.01.~~volume information, refer to the standard's Document Summary page on the ASTM website.

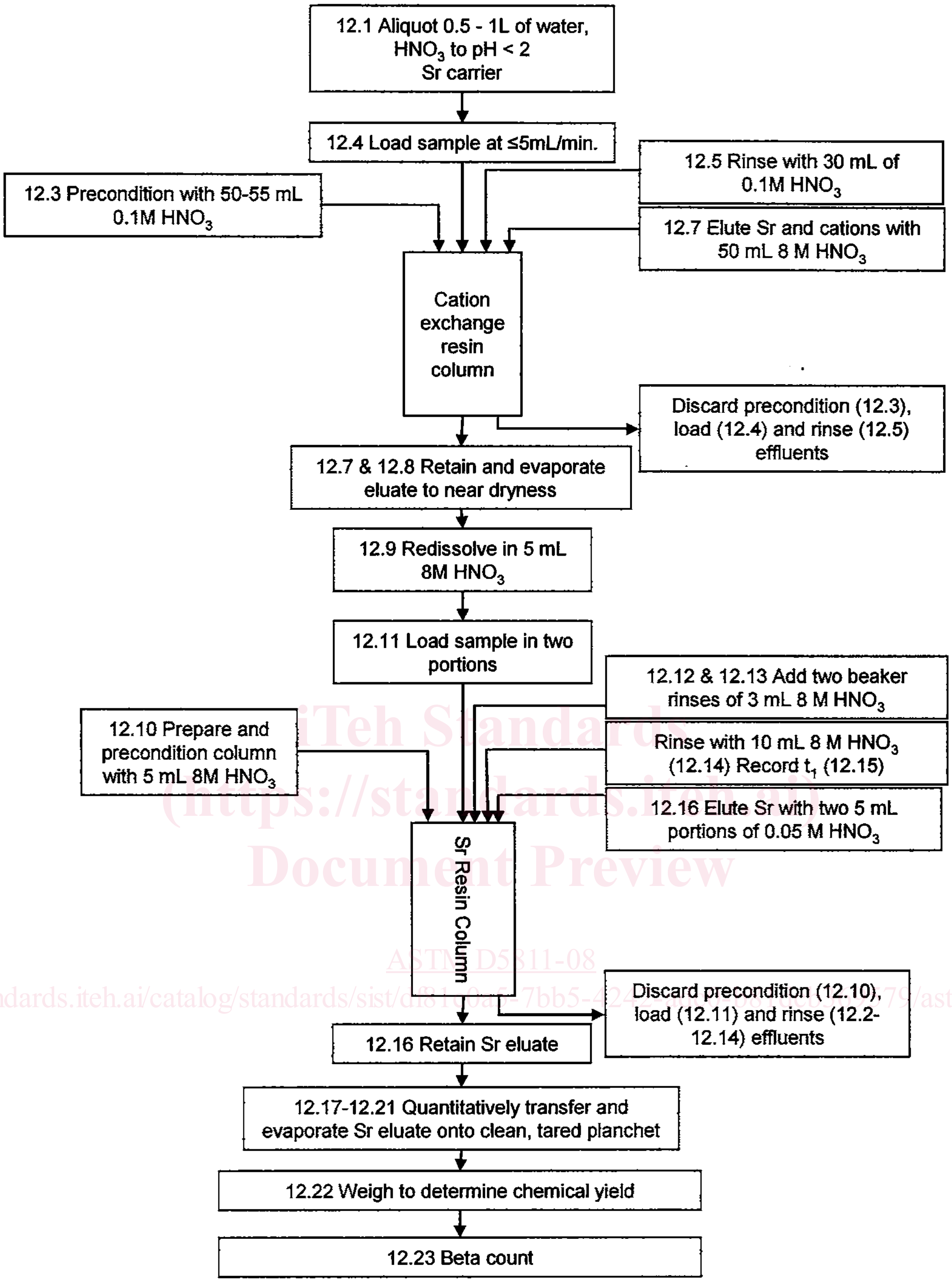

FIG. 1 Flow Diagram for the Procedure

suspected that natural strontium is present in the sample at levels that will compromise the determination of the chemical yield, blank sample aliquots to which no strontium carrier is added shall be analyzed to determine the natural strontium content. The amount of natural strontium contained in the sample shall be ~~used to correct~~reflected when calculating the yield correction factor.

6.2 Strontium-89 present in the sample will cause a high bias in proportion to the ^{89}Sr/^{90}Sr ratio. This technique is not applicable when it is suspected or known that ^{89}Sr is present in the sample.

6.3 Strontium nitrate ($Sr(NO_3)_2$) is hygroscopic. This chemical property may add uncertainty in the gravimetric yield determination.

7. Apparatus

7.1 *Analytical Balance*, 0.0001 g.

7.2 ~~*Gas Proportional Beta Counting System*, (<1.0 cpm beta), low background.~~ Low Background Gas Proportional Beta Counting System.

7.3 *Ion Exchange Columns*, 10 mL resin capacity, glass or acid-resistant plastic. An attached reservoir of at least 50 mL is desirable.

7.4 *Planchets*~~, stainless steel, 5.1 cm (2 in.) diameter with 6 mm (¼ in.) high outer lip.~~ , stainless steel to match calibration source.[3]

8. Reagents and Materials

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 specifications of the Committee on Analytical Reagents of the American Chemical ~~Society.~~ Society.[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. Reagent blanks shall be run with all determinations.

8.2 *Purity of Water*— Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193, Type III.

8.3 ~~*Radioactive Purity*—Radioactive purity shall be such that the measured radioactivity of blank samples does not exceed the calculated probable error of the measurement.~~

~~8.4~~*Cation Exchange Resin*~~, 100 to 200 mesh, hydrogen form.~~ , 100 to 200 mesh, hydrogen form. 8% cross linked, analytical grade.

8.4 *Nitric Acid (8M HNO$_3$)* — Add 500 mL of concentrated HNO$_3$ to 400 mL of water. Dilute to 1L with water.

8.5 ~~*Nitric Acid (1+1)* —Combine one part by volume concentrated nitric acid, HNO~~ Nitric Acid (0.1 HNO$_3$)—Add 6.4 mL of concentrated HNO$_3$ ~~(sp gr 1.42) and one part by volume water. This solution is 8 *M* HNO$_3$.~~ to 600 mL of water. Dilute to 1L with water.

8.6 ~~*Nitric Acid (1+159)* —Combine one part by volume concentrated nitric acid, HNO~~ Nitric Acid (0.05M HNO $_3$)—Add 3.2 mL of concentrated HNO$_3$ ~~(sp gr 1.42) and 159 parts by volume water. This solution is 0.1 *M* HNO$_3$.~~ to 600 mL of water. Dilute to 1L with water.

8.7 ~~*Nitric Acid (1+319)* —Combine one part by volume concentrated nitric acid, HNO $_3$(sp gr 1.42) and 319 parts by volume water. This solution is 0.05 *M* HNO$_3$.~~

~~8.8~~*Strontium Carrier (10 g/L)* ~~—Preferably use 10000 µg Sr/mL ICP standard. Alternatively, dissolve 24.16 g strontium nitrate (Sr (NO~~ —Preferably use 10 000 µg/mL ICP standard. Alternatively, dissolve 24.16 g strontium nitrate (Sr(NO$_3$)$_2$~~) in water and dilute to 1 L. Use the following procedure to standardize the prepared strontium carrier. Carefully pipet a 5.0 mL portion of the strontium carrier solution onto a clean, dried, and tared planchet. Dry the planchet under a drying lamp. Cool the planchet in a desiccator and weigh. Divide the net weight by 10. This result is the amount of strontium nitrate actually added. Use an average of three values in the denominator of the recovery equation in 11.2~~) in water, add 20 mL concentrated nitric acid, and dilute with water to 1 L. Use the following procedure to standardize the prepared strontium carrier: Carefully pipet a 5.0 mL portion of the strontium carrier solution onto a clean, dried, and tared planchet. Dry the planchet under the same conditions used for the final evaporation in 12.20. Allow the planchet to cool to room temperature and reweigh the planchet to the nearest 0.0001 g. Divide the net weight by 10. This result is the amount of strontium nitrate actually added. Use an average of three values in the denominator of the recovery equation in 11.12 and 13.1. This value should be within 3 % of 12.08 mg/0.5 mL.

~~8.9~~

8.8 *Strontium Extraction Chromatography Column*~~, 2 mL bed volume.~~

~~8.10~~*, 2 mL bed volume consisting of an octanol solution of 4,4'(5')-bis (t-butyl-cyclohexano)-18-crown-6-sorbed on an inert polymeric support.*[5]

8.9 *Strontium-90 Standardizing Solution*—Traceable to a national standard body such as National Institute of Standards and Technology ~~(NIST) traceable~~or National Physical Laboratory solution with less than 0.1 mg of stable strontium per mL of final solution with a typical concentration range from 85 to 125 Bq/mL.

9. Hazards

9.1 Use extreme caution when handling all acids. They are extremely corrosive and skin contact could result in severe burns.

9.2 When diluting concentrated acids, always use safety glasses and protective clothing, and add the acid *to* the water.

~~[3] Annual Book of ASTM Standards, Vol 11.02.~~

[3] Stainless steel planchets available commercially have been found satisfactory.

~~[4] Stainless steel planchets available commercially have been found satisfactory.~~

[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 *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.

~~[5] 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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.~~

[5] The sole source of supply of the apparatus known to the committee at this time is Sr Resin available from Eichrom Technologies, Inc. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

10. Sampling

10.1 Collect a sample in accordance with Practice D 3370~~.~~, D 4448, D 6001, or other documented procedure.

11. ~~Calibration~~

~~11.1Prepare a set of three calibration samples according to the calibration procedure outlined in the subsequent steps.~~
~~11.2Pipet 0.5 mL of strontium carrier into a small beaker.~~
~~11.3Add 1 mL of NIST traceable~~ Calibration

11.1 Calibrate the low background gas proportional beta counting system in accordance with Practice D 7282. Prepare a set of three calibration samples according to the calibration procedure outlined in the subsequent steps.

11.2 Pipet 0.5 mL of strontium carrier into a small beaker.

11.3 Add 1 mL of traceable ^{90}Sr solution and evaporate to near dryness on a hot plate.

11.4 Redissolve the residual in 5 mL of 8M nitric ~~acid (8M).~~ acid.

11.5 Follow the steps described in 12.10 through ~~12.22~~12.23.

11.6 Count to ~~amass 10000~~ accumulate 10 000 net counts in the counting period. Counting should be completed within 3 h of column elution. Record the time and date of the midpoint of this counting period as t_2. Count each sample mount twice, once for this step having a counting date designated as t_2 and a second time as specified below.

11.7 Calculate the net count rate of the ~~first~~ count ~~(~~at time ~~cps~~t_2($R_{n(2)}$) by subtracting the instrument background count rate from the gross count rate.

11.8 Store the calibration mount for at least 7 days to allow for ^{90}Y ingrowth.

11.9 Recount the calibration mount to amass 10 000 counts in a counting period. Record the time and date of the midpoint of this count period as t_3.

11.10 Calculate the net count rate of the second count ~~(~~at time ~~cps~~t_3($R_{n(3)}$) by subtracting the instrument background count rate from the gross count rate.

11.11 Calculate the ^{90}~~Sr detection efficiency, E_S~~Sr detection efficiency, ϵ_{Sr}, and the ^{90}Y detection efficiency, ~~E~~ϵ_Y, for each calibration mount using the equations presented below. Calculate the mean and standard deviation of the three ~~E~~ϵ_{Sr} and ~~E~~ϵ_Y values. Use the relative standard deviation of these parameters to estimate the relative uncertainty of the ingrowth efficiency factor, ~~U~~(defined in Eq 5), u~~IEr~~ ~~of~~ (ϵ_I) and used in Eq 7.

11.12 *Efficiency Calculations*:

^{90}Sr detection efficiency ~~E~~ ϵ_{Sr}—

$$E_{Sr} = \frac{(R_{C(2)} \times IF_2) - (R_{C(3)} \times IF_1)}{Y_{Sr} \times A_{C(2)} \times (IF_2 - IF_1)]} \quad (1)$$

$$\epsilon_{Sr} = \frac{(R_{n(2)} \times IF_3) - (R_{n(3)} \times IF_2)}{Y_{Sr} \times A_{C(2)} \times (IF_3 - IF_1)]} \quad (1)$$

2)

^{90}Y detection efficiency ~~E~~ ϵ_Y—

$$E_Y = \frac{R_{C(3)} - (Y_{Sr} \times A_{C(2)} \times E_{Sr})}{Y_{Sr} \times A_{C(2)} \times IF_2} \quad (2)$$

$$\epsilon_Y = \frac{R_{n(3)} - R_{n(2)}}{\frac{Y_{Sr} \times A_{C(2)} \times E_{Sr})}{Y_{Sr} \times A_{C(2)} \times IF_2}} \quad (2)$$

C(2) × (IF3 – IF 2)
AC(2)

where:

$A_{C(2)}$ = activity of ^{90}Sr in becquerels (Bq) at the time of the first count of the calibration mount,
IF~~1~~2 = ~~$1 - e^{-\lambda_Y(t_2 - t_1)}$~~ ingrowth factor for ^{90}Y at the midpoint of the count at time t_2, $1 - e^{-[\lambda_Y \times (t_2 - t_1)]}$
IF~~2~~3 = ~~$1 - e^{-\lambda_Y(t_3 - t_1)}$~~ ingrowth factor for ^{90}Y at the midpoint of the count at time t_3, $1 - e^{-[\lambda_Y \times (t_3 - t_1)]}$
λ_Y = decay constant for ^{90}~~Y (0.2595 d^{-1}),~~ ^{9}Y (0.2600 d^{-1}),
~~$R_{C(2)}$~~ $R_{n(2)}$ = ~~net count rate of calibration mount at first count, in counts per second,~~ net count rate of the calibration test source at the midpoint of the first count, in counts per second,
~~$R_{C(3)}$~~$R_{n(3)}$ = ~~net count rate of calibration mount at second count, in counts per second,~~ net count rate of calibration test source at the midpoint of the second count, in counts per second,
t_1 = date and time of ^{90}Y separation,
t_2 = date and time of midpoint of first count,
t_3 = date and time of midpoint of second count.
Y_{Sr} = fractional chemical yield of strontium carrier (see Eq 4).

NOTE 1—The time differences ($t_2 - t_1$) and ($t_3 - t_1$~~) shall be expressed in days.~~