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.



Designation: D5811 – 20

Standard Test Method for Strontium-90 in Water¹

This standard is issued under the fixed designation D5811; 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 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.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 9.

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
- D1129 Terminology Relating to Water
- D1193 Specification for Reagent Water
- D1890 Test Method for Beta Particle Radioactivity of Water
- D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
- D3370 Practices for Sampling Water from Flowing Process Streams

D3648 Practices for the Measurement of Radioactivity D4448 Guide for Sampling Ground-Water Monitoring Wells D5673 Test Method for Elements in Water by Inductively

- Coupled Plasma—Mass Spectrometry
- D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
- D6001 Guide for Direct-Push Groundwater Sampling for Environmental Site Characterization
- D7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements
- D7902 Terminology for Radiochemical Analyses
- 2.2 Other Documents:
- ANSI N42.22 Traceability of Radioactive Sources to the National Institute of Standards and Technology (NIST) and Associated Instrument Quality Control³

BIPM-5 Decay Data Evaluation Project (DDEP)⁴ NUDAT2⁵

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.1.2 For definitions of terms used in this standard relating to radiochemical analysis, refer to Terminology D7902.

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 (8 M), selectively sorbed on a solid phase extraction column, eluted with dilute nitric acid, dried on a planchet. The sample test source (STS) is counted for beta radiation.

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

¹ This test method is under the jurisdiction of ASTM Committee 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* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from Institute of Electrical and Electronics Engineers, Inc. (IEEE), 445 Hoes Ln., Piscataway, NJ 08854-4141, http://www.ieee.org.

⁴ Available from BIPM, Sèvres Cedex, France, https://www.bipm.org.

⁵ Available from National Nuclear Data Center at Brookhaven National Laboratory, W Princeton Ave, Yaphank, NY 11980, http://www.nndc.bnl.gov.

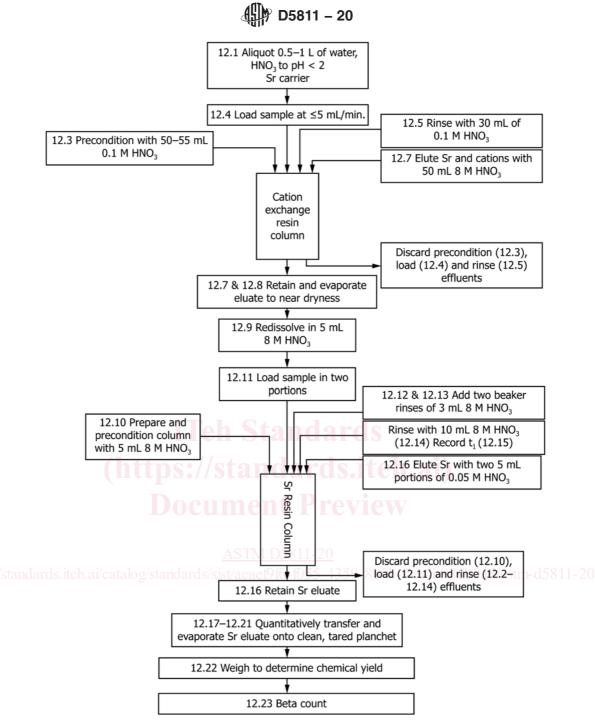


FIG. 1 Flow Diagram for the Procedure

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 native to the sample will interfere with the yield determination. If natural strontium is known or suspected to be present in the sample at levels that will compromise the determination of the chemical

yield, analyze sample aliquots to which no strontium carrier has been added to determine the natural strontium content. Alternatively, the elemental strontium concentration may be determined using an appropriate method such as ICP-MS, Test Method D5673. Account for the amount of natural strontium contained in the sample when calculating the chemical 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 of the chemical yield.

7. Apparatus

7.1 Analytical Balance, 0.0001 g.

7.2 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 to match calibration source.⁶

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.⁷ 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. Some reagents, even those of high purity, may contain naturallyoccurring radioactivity, such as isotopes of uranium, radium, actinium, thorium, rare earths, potassium compounds, or artificially produced radionuclides, or combinations thereof. Consequently, when such reagents are used in the analysis of low-radioactivity samples, the activity of the reagents should be determined under analytical conditions that are as close as practicable to those used for the test sample. The activity contributed by the reagents should be accounted for and applied as a correction when calculating the test sample result.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D1193, Type III.

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

8.4 Nitric Acid (sp gr 1.42)—Concentrated HNO₃.

8.5 *Nitric Acid* (8 *M* HNO₃)—Add 500 mL of concentrated HNO₃ to 400 mL of water. Dilute to 1 L with water.

8.6 *Nitric Acid* (0.1 HNO_3)—Add 6.4 mL of concentrated HNO₃ to 600 mL of water. Dilute to 1 L with water.

8.7 *Nitric Acid* (0.05 *M* HNO_3)—Add 3.2 mL of concentrated HNO₃ to 600 mL of water. Dilute to 1 L with water.

8.8 *Strontium Carrier (10 g/L)*—Preferably use 10 000 μ g/mL ICP standard. Alternatively, dissolve 24.16 g dry strontium nitrate (Sr(NO₃)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 chemical yield equation in 11.12 and 13.1. This value should be within 3 % of 12.08 mg/0.5 mL.

8.9 Strontium Extraction Chromatography Column, 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.⁸

8.10 *Strontium-90 Standardizing Solution*—Solution with a final concentration typically ranging from 85 to 125 Bq/mL and less than 0.1 mg of stable strontium, traceable to the SI through a national metrology institute (NMI), such as National Institute of Standards and Technology (NIST).

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.

10. Sampling

10.1 Collect a sample in accordance with Practices D3370, Guide D4448, and Guide D6001, or other documented procedure.

11. Calibration

11.1 Calibrate the low background gas proportional beta counting system in accordance with Practice D7282, Sections 14, 15, and 16. Prepare a set of three working calibration standards (WCS) according to the 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 ⁹⁰Sr solution and evaporate to near dryness on a hot plate.

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

11.5 Follow the steps described in 12.10 through 12.23.

11.6 Count to 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 WCS 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 WCS at time t_2 ($R_{n(2)}$) by subtracting the instrument background count rate from the gross count rate.

⁶ Stainless steel planchets available commercially have been found satisfactory.

⁷ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, 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.

⁸ Sr resin available commercially have been found satisfactory.

11.8 Store the WCS for at least 7 days to allow for 90 Y ingrowth.

11.9 Recount the WCS 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 t_3 ($R_{n(3)}$) by subtracting the instrument background count rate from the gross count rate.

11.11 Calculate the ⁹⁰Sr detection efficiency, ε_{Sr} , and the ⁹⁰Y detection efficiency, ε_Y , for each WCS using the equations presented below. Calculate the mean and standard deviation of the three ε_{Sr} and ε_Y values. Use the relative standard deviation of these parameters to estimate the relative uncertainty of the ingrowth efficiency factor, (defined in Eq 5), $u_r(\varepsilon_I)$ and used in Eq 7.

11.12 *Efficiency Calculations*—⁹⁰Sr detection efficiency ε_{Sr} :

$$\varepsilon_{\rm Sr} = \frac{\left(R_{\rm n(2)} \times IF_3\right) - \left(R_{\rm n(3)} \times IF_2\right)}{Y_{\rm Sr} \times A_{C(2)} \times \left(IF_3 - IF_2\right)} \tag{1}$$

 90 Y detection efficiency $\epsilon_{\rm Y}$:

$$\varepsilon_{\rm Y} = \frac{R_{\rm n(3)} - R_{\rm n(2)}}{Y_{\rm Sr} \times A_{\rm C(2)} \times (IF_3 - IF_2)}$$
(2)

where:

 $A_{C(2)}$ = activity of ⁹⁰Sr in becquerels (Bq) at the time of the first count of the calibration mount,

$$IF_2$$
 = ingrowth factor for ⁹⁰Y at the midpoint of the count
at time t_2 , $1 - e^{-\lambda_v(t_2 - t_1)}$,

$$IF_3$$
 = ingrowth factor for ⁹⁰Y at the midpoint of the count
at time t_3 , $1 - e^{-\lambda_Y(t_3 - t_1)}$,

 $\lambda_{\rm Y}$ = decay constant for ⁹⁰Y (0.2599 d⁻¹),⁹

 $R_{n(2)}$ = net count rate of the calibration test source at the midpoint of the first count, in counts per second, D5

 $R_{n(3)}$ = net count rate of calibration test source at the midpoint of the second count, in counts per second,

 t_1 = date and time of ⁹⁰Y separation,

 t_2 = date and time of midpoint of first count,

 t_3 = date and time of midpoint of second count.

 $Y_{\rm Sr}$ = fractional chemical yield of strontium carrier (see Eq 4).

Note 1—The time differences $(t_2 - t_1)$ and $(t_3 - t_1)$ are expressed in days.

12. Procedure

12.1 Add 0.5 mL of strontium carrier to a maximum of 1 L of sample. Add 1 mL of 8 M HNO₃ per 100 mL of sample and mix. Bring sample to a boil for 30 min and then cool.

12.2 Prepare a cation exchange column containing 10 mL of cation exchange resin.

12.3 Precondition the column by passing 50 to 55 mL of 0.1 M HNO₃ through the column.

12.4 Pass the sample through the column at a rate of not more than 5 mL/min.

12.5 Rinse the column with 25 to 30 mL of 0.1 M HNO₃.

12.6 Properly dispose of the feed and rinse.

12.7 Elute the strontium (and other cations) with 50 mL of 8 M HNO₃ into a 150 mL beaker.

12.8 Evaporate the eluate to near dryness on a hot plate in a fume hood. The residue will dissolve more easily in the next step if the evaporation is stopped just as the sample starts to go dry.

12.9 Dissolve the residues in 5 mL of 8 M HNO_3 . If necessary, cover with a watchglass and heat gently to facilitate complete dissolution.

12.10 Prepare a strontium extraction chromatography column by removing the bottom plug and the cap. Press the top frit down snugly to the resin surface using a glass rod (or equivalent) and let the water drain out. Add 5 mL of HNO_3 (8 M) and let the solution drain by gravity.

12.11 Carefully transfer the sample solution to the reservoir of the column. Add half and let the solution drain before adding the second half.

12.12 Rinse the beaker with 3 mL of 8 M HNO_3 and add to the column after the feed has passed through.

12.13 Repeat 12.12.

12.14 Rinse the column with 10 mL of 8 M HNO₃.

12.15 Record the end time of the last rinse as the time of 90 Y separation (start of 90 Y ingrowth, t_1).

12.16 Elute the strontium with two 5 mL portions of 0.05 M HNO₃ into a suitable container (for example, a liquid scintillation counting vial or centrifuge tube).

12.17 Clean a planchet with a paper towel moistened with alcohol. Wipe the planchet and let it dry.

12.18 Weigh the planchet to the nearest 0.0001 g and record the weight. 869b-7ea4b98db0a8/astm-d5811-20

12.19 Place the planchet under a heat lamp in a fume hood.

12.20 Evaporate the strontium eluate (see 12.16) onto the planchet by adding small portions (approximately 3 mL) to the planchet and allowing each portion to evaporate to near dryness between additions.

12.21 Rinse the liquid scintillation counting vial or centrifuge tube with approximately 3 mL of 0.05 M HNO_3 , add to the planchet and evaporate.

12.22 After all the solution has dried, cool the STS planchet to room temperature and reweigh the planchet. Record the mass to the nearest 0.0001 g.

12.23 Beta count the STS as soon as possible after preparation on a low background gas proportional counting system. Count an empty planchet fas a background subtraction sample for a duration that is as long or longer than associated STS to determine the instrument's beta background count rate. (See Test Method D1890 and Practices D3648.)

13. Calculation

13.1 Strontium-90 Radioactivity Concentration (AC_{Sr}) :

$$AC_{\rm Sr} = \frac{R_{\rm a} - R_{\rm b}}{\varepsilon_{\rm t} \times V_{\rm a} \times Y_{\rm Sr} \times e^{-[\lambda_{\rm Sr} \times (t_{\rm 1} - t_{\rm 0})]}}$$
(3)

⁹ See BIPM-5. Other sources of nuclear data (such as NUDAT2) may be used at the user's discretion. Regardless of which reference is used, the source should be clearly documented.