



Designation: D5811 – 08 (Reapproved 2013)

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 ⁹⁰Sr 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

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 Closed Conduits

D3648 Practices for the Measurement of Radioactivity

D4448 Guide for Sampling Ground-Water Monitoring Wells

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

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.

D7282 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 D1129.

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 test method.

5. Significance and Use

5.1 This test method was developed to measure the concentration of ⁹⁰Sr in non-process water samples. This test method may be used to determine the concentration of ⁹⁰Sr 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 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 reflected when calculating the yield correction factor.

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

6.3 Strontium nitrate (Sr(NO₃)₂) is hygroscopic. This chemical property may add uncertainty in the gravimetric yield determination.

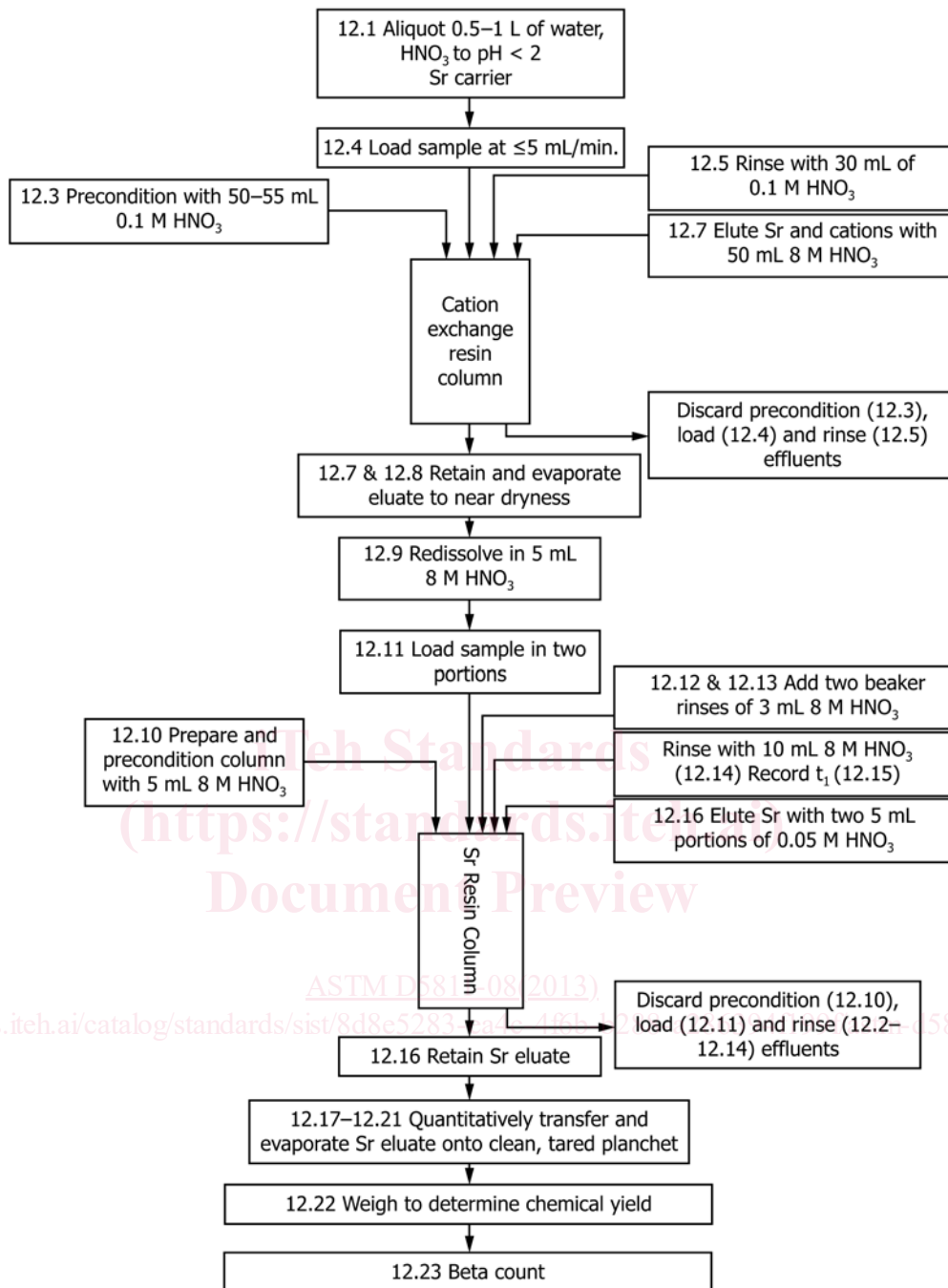


FIG. 1 Flow Diagram for the Procedure

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.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

³ Stainless steel planchets available commercially have been found satisfactory.

all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical 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 **D1193**, Type III.

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

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

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

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

8.7 *Strontium Carrier (10 g/L)*—Preferably use 10 000 µg/mL ICP standard. Alternatively, dissolve 24.16 g 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 recovery equation in **11.12** and **13.1**. This value should be within 3 % of 12.08 mg/0.5 mL.

8.8 *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.⁵

8.9 *Strontium-90 Standardizing Solution*—Traceable to a national standard body such as National Institute of Standards and Technology 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.

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 *Annual 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 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,¹ which you may attend.

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 Practice **D3370**, **D4448**, **D6001**, or other documented procedure.

11. Calibration

11.1 Calibrate the low background gas proportional beta counting system in accordance with Practice **D7282**. 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 ⁹⁰Sr solution and evaporate to near dryness on a hot plate.

11.4 Redissolve the residual in 5 mL of 8M 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 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 count at time 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 ⁹⁰Y 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 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 calibration mount 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), $u_r(\epsilon_r)$ and used in Eq 7.

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

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

⁹⁰Y detection efficiency ϵ_Y :

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

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 , $e^{-[\lambda_Y \times (t_2 - t_1)]}$