Designation: D 5811 – ~~95~~00

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 strontium-90~~ 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.~~3~~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:*

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

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D 1129 Terminology Relating to Water [2]
D 1193 Specification for Reagent Water [2]
D 1890 Test Method for Beta Particle Radioactivity of Water [3]
D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water [2]
D 3370 Practices for Sampling Water from Closed Conduits [2]
D 3648 Practices for the Measurement of Radioactivity [3]

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 aliqu~~o~~ant 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 ~~(1 + 1),~~ (8M), selectively sorbed on a solid phase extraction column, eluted with dilute nitric acid, dried on a planchet, and counted for beta radiation.

5. Significance and Use

5.1 This test method was developed to measure the concentration of ~~strontium-90~~ ^{90}Sr in non-process water samples. This test method may be used to determine the concentration of ~~Sr-90~~ ^{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 suspected that natural strontium is present in the sample, 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 the yield.

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.

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.[4] ~~stainless steel, conical, 2 in. diameter by 0.25 in. deep.~~

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.[5] 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*,[6] 100 to 200 mesh, hydrogen form.

8.~~4~~5 *Nitric Acid (1 + 1)*—Combine one part by volume concentrated nitric acid, HNO_3 (sp gr 1.42) and one part by volume water. This solution is 8 *M* HNO_3.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

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

[4] Stainless steel planchets available ~~from A. F. Murphy Die & Machine Co., 430 Hancock St., North Quincy, MA 02171,~~ commercially have been found satisfactory.

[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.

~~[6] Biorad AG 50W-X8, available from Biorad Laboratories, 2000 Alfred Nobel Dr., Hercules, CA 94547, or equivalent.~~

[6] 8 % cross-linked cation resin, analytical grade has been found satisfactory.

8.~~5~~6 *Nitric Acid (1 + 159)*—Combine one part by volume concentrated nitric acid, HNO_3(sp gr 1.42) and 159 parts by volume water. This solution is 0.1 *M* HNO_3.

8.~~6~~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.~~7~~8 *Strontium Carrier (10 g/L)*—Preferably use 10 000 µg Sr/mL ICP standard.[7] 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 and 13.1. This value should be within 3 % of 12.08 mg/0.5 mL.

8.~~8~~9 *Strontium Extraction Chromatography Column*, 2 mL bed volume.[8]

8.~~9~~10 *Strontium-90 Standardizing Solution*—National Institute of Standards and Technology (NIST) traceable 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.

10. Sampling

10.1 Collect a sample in accordance with Practice D 3370.

11. ~~Calibration and Standardization~~ Calibration

11.1 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 NIST traceable ~~Sr-90~~ ^{90}Sr solution and evaporate to near dryness on a hot plate.

11.4 Redissolve the residual in 5 mL of nitric acid ~~(1 + 1).~~ (8M).

11.5 Follow the steps described in 12.10 through 12.22.

11.6 Count to amass 10 000 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 ~~after an approximate 7 day ingrowth date designated~~ as ~~t_3.~~ specified below.

11.7 Calculate the net count rate of the first count (cps_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 (cps_3) by subtracting the instrument background count rate from the gross count rate.

11.11 Calculate the ^{90}Sr ~~counting~~ detection efficiency, E_S, and the ^{90}Y ~~counting~~ detection efficiency, E_Y, for each calibration mount using the equations presented below. Calculate the mean and standard deviation of the three E_{Sr} and E_Y values. Use the relative standard deviation of these parameters to estimate the relative uncertainty of the ingrowth efficiency factor, ~~F_I;~~ U_{IE}, of Eq 7.

11.12 *Efficiency Calculations*:

^{90}Sr detection efficiency E_{Sr}—

$$^{90}\text{Sr counting efficiency } (E_S) = \frac{(cps_2 \times I_2) - (cps_3 \times I_1)}{[R \times A_S \times (I_2 - I_1)]} \quad (1)$$

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

^{90}Y detection efficiency E_Y—

$$^{90}\text{Y counting efficiency } (E_Y) = \frac{(cps_3 - R \times A_S \times E_S)}{R \times A_S \times I_2} \quad (2)$$

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

~~[7] Available from Aldrich Chemical Co., 1001 W. St. Paul Ave., Milwaukee, WI 53233, catalog No. 35, 658-1, or equivalent,~~

[7] ICP Sr standard available commercially has been found satisfactory.

~~[8] Sr·Spec® column, 2-mL~~

[8] Strontium resin column– 2 mL resin bed volume ~~manufactured by Eichrom Industries, Inc., 8205 S. Cass Ave., Darien, IL 60561, or equivalent, has been found satisfactory.~~ consisting of an octanol solution of 4,4'(5')-bis (t-butyl-cyclohexano)-18-crown-6 sorbed on an inert polymeric support is required.

IF1 IF2

where:

$A_{C(2)}$ = activity of ^{90}Sr in Bq at the time of the first count of the calibration mount,
IF_1 = $1 - e^{-\lambda_Y(t_2 \cancel{- t} - t_1)}$
IF_2 = $1 - e^{-\lambda_Y(t_3 - t_1)}$
λ_Y = decay constant for ~~Y-90 (0.2596/day),~~ ^{90}Y (0.2595 d^{-1}),[9]
$R_{C(2)}$ = net count rate of calibration mount at first count, in counts per second,
$R_{C(3)}$ = net count rate of calibration mount at second count, in counts per second,
t_1 = date and time of ~~Y-90~~ ^{90}Y separation,
t_2 = date and time of midpoint of first ~~count, and~~ count,
t_3 = date and time of midpoint of second count.

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

~~cps_2~~
~~Y_{Sr}~~ = ~~net count rate of calibration mount at first count (*cps*),~~
~~cps_3~~ = ~~net count rate of calibration mount at second count (*cps*),~~
~~R~~ = ~~fractional~~fractional chemical yield of strontium ~~(recovery, see~~ carrier (see Eq ~~4), and~~
~~A_S~~ = ~~activity of 90Sr in Bq at the time of the first count.~~ 4).

12. Procedure

12.1 Add 0.5 mL of strontium carrier to a maximum of 1 L of sample. Add 1 mL of HNO_3~~(1 + 1)~~ (8M) 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 HNO_3~~(1 + 159)~~ (0.1M) 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 HNO_3~~(1 + 159).~~ (0.1M).

12.6 Properly dispose of the feed and rinse.

12.7 Elute the strontium (and other cations) with 50 mL of HNO_3~~(1 + 1)~~ (8M) 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 salts in 5 mL of HNO_3~~(1 + 1).~~ (8M). If necessary, cover with a watchglass and heat gently.

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 ~~(1 + 1)~~ (8M) 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 HNO_3~~(1 + 1)~~ (8M) 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 HNO_3~~(1 + 1).~~ (8M).

12.15 Record the end time of the last rinse as the ~~yttrium-90~~ 90 $_y$ separation (start of ~~Y-90~~ 90$_y$ ingrowth, t_1).

12.16 Elute the strontium with two 5 mL portions of HNO_3~~(1 + 319)~~ 3(0.5M) into a plastic container (for example, a liquid scintillation counting vial).

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

12.18 Weigh the ~~counting dish~~ planchet to 0.0001 g and record the weight.

12.19 Place the ~~dish~~ 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 ~~dish~~ planchet and allowing each portion to evaporate to near dryness between additions.

12.21 After all the solution has dried, cool the planchet to room temperature and reweigh the planchet. Record the weight to 0.0001 g.

12.22 Count the sample as soon as possible after preparation on a low background beta gas proportional counting system. Also count an empty planchet for an equal length of time to measure the instrument's background count rate. (See Test Method D 1890 and Practices D 3648.)

13. Calculation

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

$$\text{Concentration (Bq/L)} = \frac{(C_s - C_b)}{V \times R \times I \times e^{-\lambda s(t_I - t_0)}} \quad (3)$$

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[9] Firestone, R.B., and Shirley, V.S., *Table of Isotopes* (Eighth Edition), John Wiley and Sons, Inc., New York, 1995.

$$AC_{Sr}\ (\text{Bq/L}) = \frac{R_a - R_b}{IE \times V_a \times Y_{Sr} \times e^{-\lambda_{Sr}(t_1 - t_0)}} \tag{3}$$

~~$$\text{Recovery}\ \ (R) = \frac{\text{mg Sr(NO}_3)_2\ (\text{sample}) - \text{mg Sr(NO}_3)_2\ (\text{blank})}{\text{mg Sr carrier added (as nitrate)}}$$~~

$$Y_{Sr} = \frac{\text{mg Sr(NO}_3)_2\ (\text{sample aliquant}) - \text{mg Sr(NO}_3)_2\ (\text{reagent blank})}{\text{mg Sr carrier added (as nitrate)}} \tag{4}$$

~~$$\text{Ingrowth Factor}\ (I) = E_S + E_Y\,(1 - e^{-\lambda y(t_m - t_l)})$$~~

$$IE = E_{Sr} + E_Y\,(1 - e^{-\lambda y(t_m - t_l)}) \tag{5}$$

EYIE λSr λYRaR bt0t1t m

where:

~~t_0~~ E_{Sr} = ~~collection time~~ the mean of ~~sample,~~ the values calculated using Eq 1,
~~t_1~~ E_Y = ~~Y-90 separation time,~~ the mean of the values calculated using Eq 2,
~~t_m~~ IE = ~~midpoint of sample count,~~ ingrowth efficiency factor,
~~T_s~~ λ_{Sr} = ~~sample count duration (seconds), used in Eq 6,~~ decay constant for ^{90}Sr (6.594 E – 5 d^{-1}),[9]
~~C_s~~ λ_Y = ~~cps of sample,~~
~~C_b~~ = ~~*cps* of background,~~ decay constant for ^{90}Y (0.2595 d^{-1}),[9]
~~λ_s~~ R_a = ~~decay constant for Sr-90 (6.64E-05/day),~~ count rate of sample aliquant, in counts per second,
~~λ_y~~ R_b = ~~decay constant for Y-90 (0.2596/day),~~ count rate of instrument background, in counts per second,
~~R~~ t_0 = ~~fractional chemical yield (recovery),~~ date and time of sample collection,
~~V~~ t_1 = ~~aliquot volume (litres),~~ date and time of ^{90}Y separation, and
~~I~~ t_m = ~~ingrowth-efficiency factor,~~ midpoint of count of sample aliquant (date and time).

NOTE 2—The time differences (t_1– t_0) and (t_m– t_1) shall be expressed in days.

~~E_S~~ V_a = ~~Sr-90 efficiency,~~volume of sample aliquant, in litres, and
~~E_Y~~ Y_{Sr} = ~~Y-90 efficiency.~~fractional chemical yield (recovery) of strontium carrier.

13.2 ~~C~~The sample aliquant has an uncertainty in its net count rate (“counting ~~U~~ uncertainty ~~(CU~~”)~~,~~. The uncertainty in the ~~68 % confidence level, assuming equal~~ ^{90}Sr radioactivity concentration in the sample ~~and instrument background~~ from the counting ~~times:~~ uncertainty, U_R, is given at a 68 % confidence level by:

~~$$CU\ (\text{Bq/L}) = \frac{\frac{\sqrt{C_s + C_b}}{T_s}}{V \times R \times I \times e^{-\lambda s(t_1 - t_0)}} \tag{6}$$~~

$$U_R\ (\text{Bq/L}) = \frac{\frac{\sqrt{R_a + R_b}}{\sqrt{t_a}}}{IE \times V_a \times Y_{Sr} \times e^{-\lambda_{Sr}(t_l - t_0)}} \tag{6}$$

where:
t_a = count duration, in seconds, of the sample aliquant,
and the other terms are as defined in 13.1.

13.3 *Total Propagated Uncertainty (TPU)*:

~~$$TPU\ (\text{Bq/L}) = A \times \sqrt{(CU/A)^2 + F_I^{\,2} + F_V^{\,2} + F_R^{\,2} + F_P^{\,2}}$$~~

$$TPU\ (\text{Bq/L}) = AC_{Sr}\sqrt{\left(\frac{U_R}{AC_{ST}}\right)^2 + U_{IE}^{\,2} + U_{Va}^{\,2} + U_{YSr}^{\,2} + U_o^{\,2}} \tag{7}$$

UVaUYSrU o

where:

~~A~~ U_{IE} = ~~concentration of~~ fractional uncertainty in the ~~sample, Bq/L,~~ ingrowth efficiency factor,
~~CU~~ = ~~counting~~fractional uncertainty ~~of~~ in the ~~sample,~~ volume measurement,
U_{Va}
~~F_I~~ U_{YSr} = ~~relative fractional random~~fractional uncertainty ~~associated with~~ in the ~~counting efficiency,~~ chemical yield of the strontium carrier,

~~F_V~~U_o = ~~relative~~any additional fractional~~ random~~ uncertainty~~ associated with the volume measurement,~~
~~F_R = relative fractional random uncertainty associated with the fractional chemical yield (recovery), and~~
~~F_P = relative fractional systematic uncertainty associated with the procedure (matrix dependent).~~ that has been determined or estimated,

and the other terms are as defined in 13.1 and 13.2.

13.4 *“A Priori” Minimum Detectable Radioactivity Concentration (MDC)*:

$$\cancel{MDC\ (\text{Bq/L}) = \frac{4.65\sqrt{B} + (2.71)}{V \times T_S \times R \times I \times e^{-\lambda s_{(t_1 - t_0)}}}} \tag{8}$$

$$MDC\ (\text{Bq/L}) = \frac{4.65\sqrt{C_b} + 2.71}{IE \times t_a \times V_a \times Y_{Sr} \times e^{-\lambda_{Sr}(t_1 - t_0)}} \tag{8}$$

where:

~~B~~ C_b = ~~background~~ number of counts accumulated during the counting of the sample~~ count.~~ aliquant due to instrument background,

and the other terms are as defined previously.

NOTE~~ 2—For the~~ 3—The counting~~ uncertainty and minimum detectable concentration calculations to be correct,~~ times of the sample aliquant and of instrument background~~ count duration shall~~ must be~~ the same as the sample count duration (T_s).~~ of equal length for Eq 6-8 to be valid. The values used for~~ E_s, E_y, V, T_s, R, t_1, and I should~~ the terms in Eq 8 should be typical for the laboratory.

14. Quality Control

14.1 The project leader, as part of the external quality control program, shall submit blind quality control samples to the analyst along with routine samples. These external quality control samples, which usually include duplicate and blank samples, shall test sample collection and preparation as well as sample analysis whenever possible. In addition, analysts are expected to run internal quality control samples that will indicate to them whether the analytical procedures are in control. Both the external and internal quality control samples need to be prepared in such a way as to duplicate the chemical matrix of the routine samples, insofar as this is practical. The quality control samples that are routinely used consist of~~ five~~ four basic types: blank samples, replicate samples, reference materials,~~ control samples,~~ and “spiked” samples.

15. Precision and Bias

15.1 The overall precision, *S(T)*, and the single-operator precision, *S(O)*, of this test method, within the designated range, have been found to vary with levels according to Table~~ 1.~~ 1.

15.2 The collaborative test conducted on this test method included eleven laboratories, each with one operator. Three radioactivity levels, 0.21 Bq/L (5.8 pCi/L), 1.52 Bq/L (41.1 pCi/L), and 4.05 Bq/L (109.5 pCi/L), were tested with three replicates per level. The determination of the precision and bias statements were made in accordance with Practice D 2777. Two laboratories’ data were omitted as statistical outliers.

15.3 These collaborative test data were obtained using 1 L of tap water available at each laboratory site. For other matrices, these data may not apply.

15.4 The bias of this test method, based upon the collaborative test data, was found to vary with levels according to Table 1.

16. Keywords

16.1 extraction chromatography; radioactive strontium; radioactivity; radiochemistry; strontium–90; water

TABLE 1 Precision and Bias

Amount Added, Bq/L	Mean Found, Bq/L	± Bias	± % Bias	Statistically Significant (5 % *CL*)	Precision	
					S(T)	S(O)
0.21	0.22	0.01	3.54	no	0.02	0.02
1.52	1.55	0.03	1.80	no	0.09	0.06
4.05	4.05	0.00	−0.06	no	0.26	0.21