



Standard Test Method for Strontium Ion in Brackish Water, Seawater, and Brines¹

This standard is issued under the fixed designation D3352; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope*

1.1 This test method covers the determination of soluble strontium ion in brackish water, seawater, and brines by atomic absorption spectrophotometry.

1.2 Samples containing from 5 to 2100 mg/L of strontium may be analyzed by this test method.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D1129 Terminology Relating to Water

D1193 Specification for Reagent 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

D5810 Guide for Spiking into Aqueous Samples

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

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 dependent on the fact that metallic elements, in the ground state, will absorb light of the same

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved Feb. 1, 2015. Published April 2015. Originally approved in 1974. Last previous edition approved in 2008 as D3352–08a. DOI: 10.1520/D3352-15.

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.

wavelength they emit when excited. When radiation from a given excited element is passed through a flame containing ground state atoms of that element, the intensity of the transmitted radiation will decrease in proportion to the amount of the ground state element in the flame. A hollow cathode lamp whose cathode is made of the element to be determined provides the radiation. The metal atoms³ to be measured are placed in the beam of radiation by aspirating the specimen into an oxidant-fuel flame. A monochromator isolates the characteristic radiation from the hollow cathode lamp and a photosensitive device measures the attenuated transmitted radiation.

4.2 Since the variable and sometimes high concentrations of matrix materials in the waters and brines affect absorption differently, it is difficult to prepare standards sufficiently similar to the waters and brines. To overcome this difficulty, the method of additions is used in which three identical samples are prepared and varying amounts of a standard added to two of them. The three samples are then aspirated, the concentration readings recorded, and the original sample concentration calculated.

5. Significance and Use

5.1 This test method⁴ can be used to determine strontium ions in brackish water, seawater, and brines.

6. Interferences

6.1 The chemical suppression caused by silicon, aluminum, and phosphate is controlled by adding lanthanum. The lanthanum also controls ionization interference.

7. Apparatus

7.1 *Atomic Absorption Spectrophotometer*—The instrument shall consist of atomizer and burner, suitable pressure-regulating devices capable of maintaining constant oxidant and

³ For additional information on atomic absorption, see the following references: Angino, E. E., and Billings, G. K., *Atomic Absorption Spectrophotometry in Geology*, Elsevier Publishing Co., New York, N.Y., 1967; Dean, J. A., and Rains, T. C., Editors, *Flame Emission and Atomic Absorption Spectrometry Vol 1 – Theory*, Marcel Dekker, New York, NY, 1969.

⁴ Additional information is contained in the following references: Fletcher, G. F., and Collins, A. G., "Atomic Absorption Methods of Analysis of Oilfield Brines: Barium, Calcium, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Potassium, Sodium, Strontium, and Zinc," *U.S. Bureau of Mines, Report of Investigations 7861*, 1974, 14 pp.; Collins, A. G., *Geochemistry of Oilfield Waters*, Elsevier Publishing Co., Amsterdam, Netherlands, 1975.

*A Summary of Changes section appears at the end of this standard

fuel pressure for the duration of the test, a hollow cathode lamp for each metal to be tested, an optical system capable of isolating the desired line of radiation, an adjustable slit, a photomultiplier tube or other photosensitive device as a light measuring and amplifying device, and a read-out mechanism for indicating the amount of absorbed radiation.

7.1.1 *Multi-Element Hollow Cathode Lamps* are available and have been found satisfactory.

7.2 *Pressure-Reducing Valves*—The supplies of fuel and oxidant shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

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 the specifications of the Committee on Analytical Reagents of the American Chemical Society,⁵ where such specifications are available. 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, reference to water shall be understood to mean reagent water conforming to Specification D1193, Type I. Other reagent water types may be used provided it is first ascertained that the water is of sufficiently high purity to permit its use without adversely affecting the precision and bias of the test method. Type III water was specified at the time of round robin testing of this test method.

8.3 *Filter Paper*—Purchase suitable filter paper. Typically the filter papers have a pore size of 0.45- μ m membrane. Material such as fine-textured, acid-washed, ashless paper, or glass fiber paper are acceptable. The user must first ascertain that the filter paper is of sufficient purity to use without adversely affecting the bias and precision of the test method.

8.4 *Lanthanum Solution (5 % La)*—Wet 58.65 g of lanthanum oxide (La₂O₃) with water. Add 250 mL of concentrated hydrochloric acid (sp gr 1.19) very slowly until the material is dissolved. Dilute solution to 1 litre with water.

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 *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.

TABLE 1 Compositions of Artificial Brine Samples

Sample No.	g/L			
	1	2	3	4
Sr	0.060	0.100	1.600	2.100
NaCl	24.0	170.0	80.0	200.0
KCl	0.5	2.0	1.5	3.0
KBr	1.0	2.0	2.0	2.0
KI	0.1	0.5	0.5	1.0
CaCl ₂	1.5	3.0	2.0	5.0
MgCl ₂	4.5	5.0	2.0	1.0
BaCl ₂	0.05	1.0	0.5	0.5

8.5 *Strontium Solution, Standard* (1 mL = 1 mg Sr)—Dissolve 2.415 g of strontium nitrate [Sr(NO₃)₂] in 10 mL of concentrated hydrochloric acid (sp gr 1.19) and about 700 mL of water. Dilute solution to 1 L with water. One millilitre of this solution contains 1 mg of strontium. A purchased strontium stock solution of appropriate known purity is also acceptable.

8.6 *Oxidant, for Atomic Absorption Spectrophotometer:*

8.6.1 *Air*, which has been cleaned and dried through a suitable filter to remove oil, water, and other foreign substances, is the usual oxidant.

8.6.2 *Nitrous Oxide* may be required as an oxidant for refractory-type metals.

8.7 *Fuel, for Atomic Absorption Spectrophotometer:*

8.7.1 *Acetylene*—Standard, commercially available acetylene is the usual fuel. Acetone, always present in acetylene cylinders, can be prevented from entering and damaging the burner head by replacing a cylinder which only has 689 kPa (100 psi) of acetylene remaining.

9. Sampling

9.1 Collect the sample in accordance with Practices D3370.

10. Procedure

10.1 Strontium is determined at the 460.7-nm wavelength with an air-acetylene flame.

10.2 *Preliminary Calibration*—Using micropipets prepare standard strontium solutions containing 1 to 10 mg/L of strontium using the standard strontium solution (8.5) and 50-mL volumetric flasks. Before making up to volume, add to each of these and to a blank, 5 mL of the lanthanum solution (8.4). Analyze at least three working standards containing concentrations of strontium that bracket the expected sample concentration, prior to analysis of samples, to calibrate the instrument. Aspirate these standards and the blank (for background setting) and adjust the curvature controls, if necessary, to obtain a linear relationship between absorbance and the actual concentration of the standards.

10.3 Transfer an aliquot of water or brine (previously filtered (8.3) through a 0.45- μ m filter) to a 50-mL volumetric flask. The specific gravity of the water or brine can be used to estimate the strontium content of the sample and, thereby, serve as a basis for selecting the aliquot size that will contain about 0.1 mg of strontium. Fig. 1 shows the relationship between strontium concentration and specific gravity for some oilfield brines from the Smackover formation. The concentrations of strontium in the Smackover brines will not necessarily correlate with the concentrations found in other formations.

TABLE 2 Determination of Precision and Bias of Strontium Ion

Amount Added, mg/L	Amount Found, mg/L	S _O	S _T	± Bias	Statistically Significant (95 % Confidence Level)
60	63.48	2.96	8.49	+ 5.8	yes
100	99.5	4.12	11.84	- 0.5	no
1600	1665.6	54.87	157.3	+ 4.1	no
2100	2167.2	71.12	203.9	+ 3.2	no

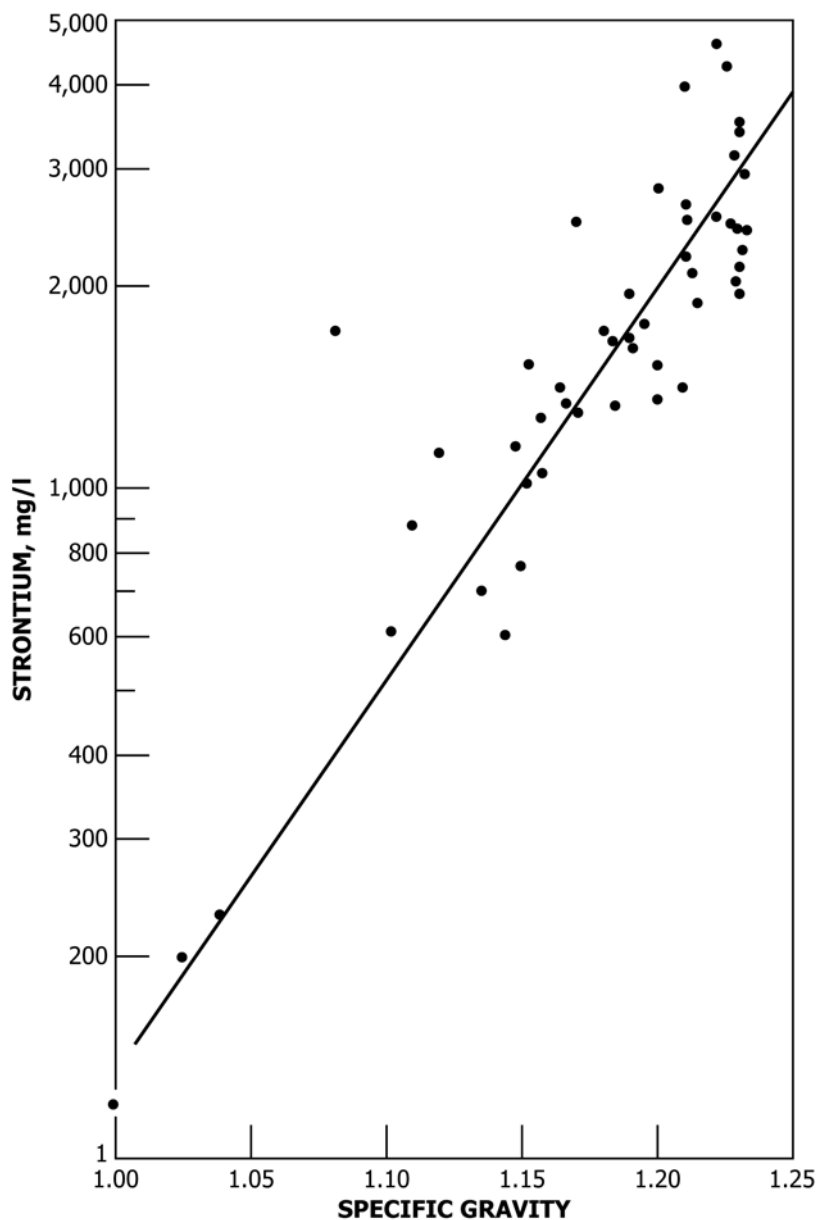


FIG. 1 Relationship of the Concentration of Strontium in Some Oilfield (Smackover) Brines to Specific Gravity

Therefore, the user of this test method may find it necessary to draw a similar curve for brine samples taken from other formations. Add 5 mL of the lanthanum stock solution (8.4), dilute to volume, and aspirate. Calculate the approximate sample concentration from the preliminary calibration readings, and determine the aliquot size that will contain about 0.1 mg of strontium.

10.4 Transfer equal aliquots containing about 0.1 mg of strontium to three 50-mL volumetric flasks. Add no strontium standard to the first flask. With a micropipet add 0.1 mg to the second and 0.2 mg to the third.

10.5 Add 5 mL of the lanthanum solution (8.4) to each of the three flasks and dilute to volume. Aspirate and record the

direct concentration of the sample, if this capability is provided with the instrument or record the absorbance readings for each sample.

11. Calculation

11.1 Calculate the concentration of strontium ion in the original sample in milligrams per litre as follows:

$$\text{Strontium concentration, mg/L} = \frac{V_1(A_s \times C_{\text{std}})}{V_2(A_{\text{std}} - A_s)}$$

where:

- V_1 = volume of the diluted samples, mL,
- V_2 = volume of the original sample, mL,

A_s = absorbance of dilute sample,
 A_{std} = absorbance of one of the standard additions, and
 C_{std} = concentration of the same standard addition as A_{std} ,
 mg/L.

Since there are two standard additions, calculate for each and average the two results.

12. Precision and Bias⁶

12.1 The precision of the test method within its designated range may be expressed as follows:

$$S_t = 0.0929X + 2.596$$

$$S_o = 0.0324X + 0.901$$

where:

S_t = overall precision,
 S_o = single-operator precision, and
 X = concentration of strontium determined, mg/L.

12.2 The bias of the test method determined from recoveries of known amounts of strontium in a series of prepared standards are given in [Table 2](#).

NOTE 1—The above precision and bias estimates are based on an interlaboratory study on four artificial brine samples containing various amounts of strontium and interfering ions as shown in [Table 1](#). One analyst in each of three laboratories and two analysts in each of six laboratories performed duplicate determinations on each of two days. Practice [D2777](#) was used in developing these precision and bias estimates.

12.3 Precision and bias for this test method conforms to Practice [D2777 – 77](#), which was in place at the time of collaborative testing. Under the allowances made in [1.4 of D2777 – 13](#), these precision and bias data do meet existing requirements for interlaboratory studies of Committee D19 test methods.

13. Quality Control (QC)

13.1 In order to be certain that analytical values obtained using these test methods are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when analyzing strontium.

13.2 Calibration and Calibration Verification:

13.2.1 Analyze at least three working standards containing concentrations of strontium that bracket the expected sample concentration, prior to analysis of samples, to calibrate the instrument (see [10.2](#)).

13.2.2 Verify instrument calibration after standardization by analyzing a standard at the concentration of one of the calibration standards. The absorbance shall fall within 4 % of the absorbance from the calibration. Alternately, the concentration of a mid-range standard should fall within ± 15 % of the known concentration. Analyze a calibration blank to verify system cleanliness.

13.2.3 If calibration cannot be verified, recalibrate the instrument.

13.2.4 It is recommended to analyze a continuing calibration blank (CCB) and continuing calibration verification

(CCV) at a 10 % frequency. The results should fall within the expected precision of the method or ± 15 % of the known concentration.

13.3 Initial Demonstration of Laboratory Capability:

13.3.1 If a laboratory has not performed the test before, or if there has been a major change in the measurement system, for example, new analyst, new instrument, etc., a precision and bias study must be performed to demonstrate laboratory capability.

13.3.2 Analyze seven replicates of a standard solution prepared from an Independent Reference Material containing a midrange concentration of strontium. The matrix and chemistry of the solution should be equivalent to the solution used in the collaborative study. Each replicate must be taken through the complete analytical test method including any sample preservation and pretreatment steps.

13.3.3 Calculate the mean and standard deviation of the seven values and compare to the acceptable ranges of bias in [12.1](#). This study should be repeated until the recoveries are within the limits given in [12.1](#). If a concentration other than the recommended concentration is used, refer to Practice [D5847](#) for information on applying the F test and t test in evaluating the acceptability of the mean and standard deviation.

13.4 Laboratory Control Sample (LCS):

13.4.1 To ensure that the test method is in control, prepare and analyze a LCS containing a mid-range concentration of strontium with each batch (laboratory defined or twenty samples). The laboratory control samples for a large batch should cover the analytical range when possible. The LCS must be taken through all of the steps of the analytical method including sample preservation and pretreatment. The result obtained for the LCS shall fall within ± 15 % of the known concentration.

13.4.2 If the result is not within these limits, analysis of samples is halted until the problem is corrected, and either all the samples in the batch must be reanalyzed, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

13.5 Method Blank:

13.5.1 Analyze a reagent water test blank with each laboratory-defined batch. The concentration of strontium found in the blank should be less than 0.5 times the lowest calibration standard. If the concentration of strontium is found above this level, analysis of samples is halted until the contamination is eliminated, and a blank shows no contamination at or above this level, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

13.6 Matrix Spike (MS):

13.6.1 To check for interferences in the specific matrix being tested, perform a MS on at least one sample from each laboratory-defined batch by spiking an aliquot of the sample with a known concentration of strontium and taking it through the analytical method.

13.6.2 The spike concentration plus the background concentration of strontium must not exceed the high calibration standard. The spike must produce a concentration in the spiked

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1022. Contact ASTM Customer Service at service@astm.org.

sample that is 2 to 5 times the analyte concentration in the unspiked sample, or 10 to 50 times the detection limit of the test method, whichever is greater.

13.6.3 Calculate the percent recovery of the spike (P) using the following formula:

$$P = 100 [A(V_s + V) - BV_s] / CV$$

where:

A = analyte known concentration (mg/L) in spiked sample,

B = analyte known concentration (mg/L) in unspiked sample,

C = known concentration (mg/L) of analyte in spiking solution,

V_s = volume (mL) of sample used, and

V = volume (mL) of spiking solution added.

13.6.4 The percent recovery of the spike shall fall within the limits, based on the analyte concentration, listed in Test Method D5810, Table 1. If the percent recovery is not within these limits, a matrix interference may be present in the sample selected for spiking. Under these circumstances, one of the following remedies must be employed: the matrix interference must be removed, all samples in the batch must be analyzed by a test method not affected by the matrix interference, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

NOTE 2—Acceptable spike recoveries are dependent on the concentra-

tion of the component of interest. See Test Method D5810 for additional information.

13.7 Duplicate:

13.7.1 To check the precision of sample analyses, analyze a sample in duplicate with each laboratory-defined batch. If the concentration of the analyte is less than five times the detection limit for the analyte, a matrix spike duplicate (MSD) should be used.

13.7.2 Calculate the standard deviation of the duplicate values and compare to the precision in the collaborative study using an F test. Refer to 6.4.4 of Practice D5847 for information on applying the F test.

13.7.3 If the result exceeds the precision limit, the batch must be reanalyzed or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

13.8 Independent Reference Material (IRM):

13.8.1 In order to verify the quantitative value produced by the test method, analyze an Independent Reference Material (IRM) submitted as a regular sample (if practical) to the laboratory at least once per quarter. The concentration of the IRM should be in the concentration mid-range for the method chosen. The value obtained must fall within the control limits established by the laboratory.

14. Keywords

14.1 brackish; brines; seawater; strontium ion

SUMMARY OF CHANGES

Committee D19 has identified the location of selected changes to this standard since the last issue (D3352 – 08a) that may impact the use of this standard. (Approved Feb. 1, 2015.)

(1) 1.3 was updated.

(2) Section 8 was modified to allow for commercial standards and filter paper information was added.

(3) Section 10 was modified to add reagent references, to add calibration information, and to allow for direct reading instruments.

(4) 13.2.4 and 13.6.3 were modified.

(5) The title of Fig. 1 was updated.

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