Designation: D 3920 – 02

An American National Standard

Standard Test Method for Strontium in Water[1]

This standard is issued under the fixed designation D 3920; 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 dissolved and total recoverable strontium in water and wastewater by atomic absorption spectroscopy.

1.2 The test method is applicable in the range from 0.1 to 1 mg/L of strontium. The range may be extended by dilution of the original sample.

1.3 Round-robin data were obtained in natural and reagent water matrices. It is the user's responsibility to ensure the validity of the test method 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.* Specific precautionary statements are given in 8.4 and 8.9.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]

D 1193 Specification for Reagent Water[2]

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

D 4691 Practice for Measuring Elements in Water by Flame Atomic Absorption Spectrophotometry[2]

D 4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents[2]

D 5810 Guide for Spiking into Aqueous Samples[2]

D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis[3]

3. Terminology

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

3.2 *Definition of Term Specific to This Standard:*

3.2.1 *total recoverable strontium, n*—an arbitrary analytical term relating to the forms of strontium that are determinable by the digestion procedure described in this test method.

3.2.2 *laboratory control sample, n*—a solution with a certified concentration of the strontium.

4. Summary of Test Method

4.1 Strontium is determined by atomic absorption spectrophotometry. The sample is aspirated into an air-acetylene flame following the addition of lanthanum chloride/potassium chloride solution. Samples containing particulate matter that may clog the aspirator capillary or burner, thus producing inaccurate results, are filtered through a 0.45-µm membrane filter prior to testing.

5. Significance and Use

5.1 Although most potable supplies contain little strontium, some well waters in the midwestern part of the United States have levels as high as 39 mg/L.[4]

5.2 This test method affords a reliable means of accurately determining strontium and correcting calcium results obtained by the methods cited in 6.2.

6. Interferences

6.1 Chemical interference caused by silicon, aluminum, and phosphate is controlled by adding lanthanum chloride. Potassium chloride is added to suppress the ionization of strontium.

NOTE 1—A nitrous oxide-acetylene flame has been used successfully by some to remove chemical interferences.

6.2 Strontium chemically resembles calcium and causes a positive error in gravimetric and titrimetric methods for calcium determination.

7. Apparatus

7.1 *Atomic Absorption Spectrophotometer* for use at 460.7 nm. A general guide for the use of flame atomic absorption applications is given in Practice D 4691.

NOTE 2—The manufacturer's instructions should be followed for setting instrumental parameters.

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[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.

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

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

[4] *Standard Method for the Examination of Water and Wastewater,* 14th Ed. American Public Health Assn., Washington, DC 20005.

7.2 *Strontium Hollow-Cathode Lamp*.

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, where such specifications are available. [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.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193, Type I, II, and III water. Type I is perferred and more commonly used. 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 II water was specified at the time of round robin testing of this test method.

NOTE 3—The user must ensure the type of reagent water chosen is sufficiently free of interferences. The water should be analyzed using the test method.

8.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

8.4 *Lanthanum Chloride/Potassium Chloride Solution*—Dissolve 11.73 g of lanthanum oxide (La_2O_3) in a minimum amount of concentrated hydrochloric acid (approximately 50 mL.). Add 1.91 g of potassium chloride (KCl). Allow solution to cool to room temperature and dilute to 100 mL with water. (**Warning**—Add acid slowly and in small portion to control the reaction rate upon mixing.)

8.5 *Nitric Acid* (sp gr 1.42)—Concentrated HNO_3.

8.6 *Strontium Solution, Stock* (1.0 mL = 1.0 mg Sr)—Dissolve 2.415 g of strontium nitrate ($Sr(NO_3)_2$) in a 1-L flask containing 10 mL of concentrated HCl and 700 mL of water. Dilute to 1000 mL with water.

8.7 *Strontium Solution, Standard* (1 mL = 0.010 mg Sr)—Dilute 5.0 mL of strontium solution standard to 500 mL with water.

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

8.9 *Fuel*—Standard, commercially available acetylene is the usual fuel. Acetone, always present in acetylene cylinders, can be prevented from entering the burner system by replacing the cylinder when the pressure reaches 490 kPa (70 psig). (**Warning**—Purified grade acetylene containing a special proprietary solvent other than acetone should not be used with poly (vinyl chloride) tubing as weakening of the walls may result and cause a potentially hazardous situation.)

9. Sampling

9.1 Collect the samples in accordance with instructions in Practices D 3370. The holding time for the samples may be calculated in accordance with Practice D 4841.

9.2 To preserve the samples add concentrated HNO_3(sp gr 1.42) to a pH of 2 or less immediately at the time of collection; normally about 2 mL/L is required. If only dissolved strontium is to be determined, filter the samples at time of collection through a 0.45-µm membrane filter before acidification.

10. Standardization

10.1 Prepare a blank and at least four working standards to bracket the expected strontium concentration range of the samples to be analyzed by diluting the standard strontium solution (8.6) to the desired concentrations. Select concentrations that will give a zero, middle, and maximum points for the analytical curve.

10.2 Pipet 10.0 mL of each standard into a 50-mL beaker or flask and add 1.0 mL of lanthanum chloride/potassium chloride solution. Mix well by swirling.

10.3 To test the suitability of reagents used in the analysis, zero the instrument while aspirating reagent water. Aspirate the zero standard and record the response. If the zero standard produces a response sufficient to affect the detection limit or accuracy, or both, of the test method, the contaminated reagent should be identified and replaced with a grade of suitable quality before proceeding.

10.4 Aspirate the blank (zero standard) and adjust the instrument reading to zero. Aspirate standards and record the instrument reading for each. Aspirate reagent water between each standard.

NOTE 4—Best results have been obtained with a slightly fuel rich flame.

10.5 Construct an analytical curve by plotting the absorbance of standards versus milligrams of strontium per litre. Alternatively, read directly in concentration if this capability is provided in the instrument.

11. Procedure

11.1 For total recoverable strontium, add 5 mL of concentrated nitric acid to 100 mL of the sample in a 250-mL Erlenmeyer flask, and mix well. Heat the sample at 95°C on a steam bath/hot plate in a well-ventilated fume hood until the volume is reduced to 15 to 20 mL.

NOTE 5—When testing samples of brine or samples containing a large amount of solids, the amount of reduction in volume is left to the discretion of the analyst.

11.2 If color in the digested solution indicates the presence of partially oxidized materials, add additional nitric acid and approximately 90 mL of reagent water to the cooled solution and repeat the digestion as before. Repeat this step several times if necessary.

11.3 Cool and filter the digested solution through a suitable filter (such as fine-textured, acid-washed ashless paper) into a 100-mL volumetric flask. Wash the filter paper 2 to 3 times and make up to volume with reagent water. A reagent blank (100 mL of reagent water and 5 mL of concentrated nitric acid) should be carried through the digestion process and treated as

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[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

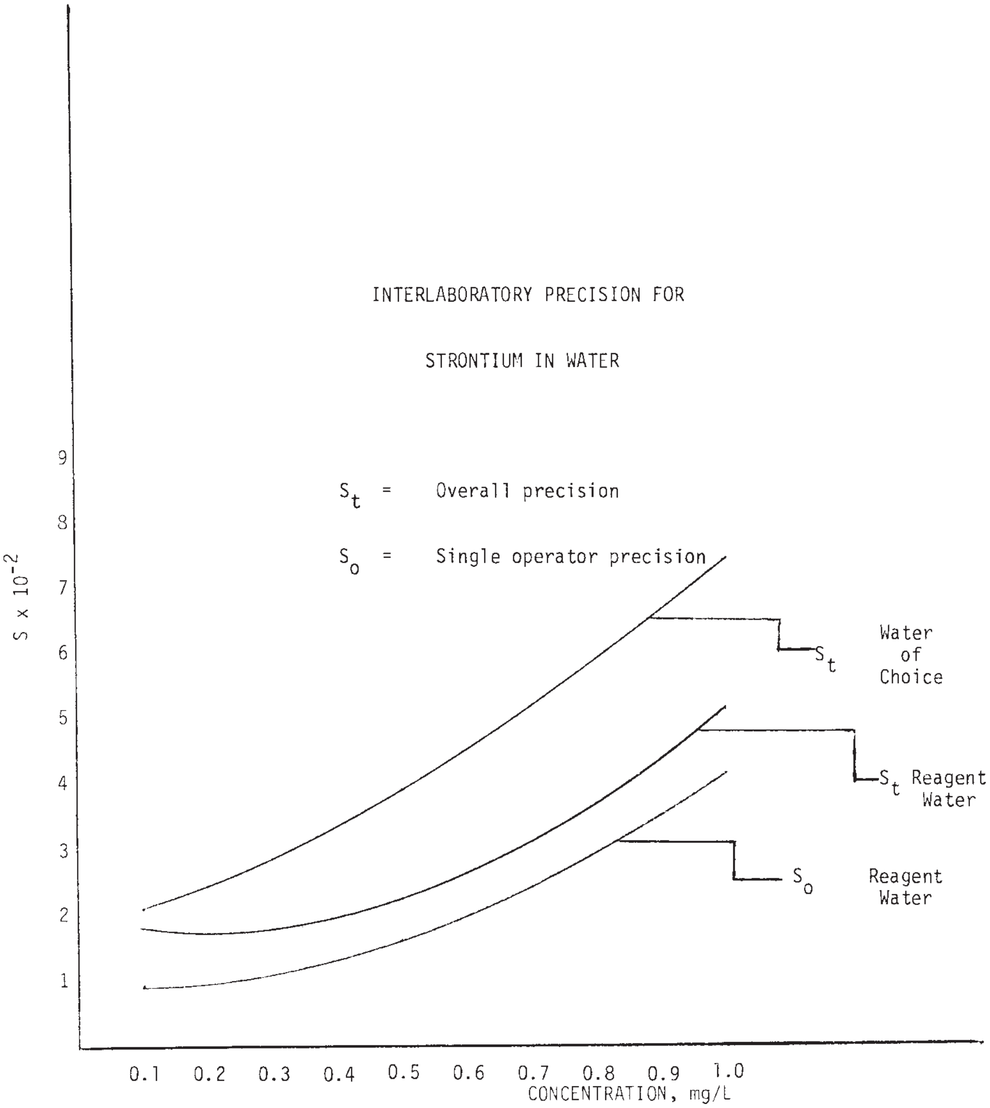

FIG. 1 Interlaboratory Precision for Strontium in Water

instructed in 10.3. An unacceptable high response could indicate that the nitric acid was of poor quality.

NOTE 6—If only dissolved strontium is to be determined, filter portion of the sample through a 0.45-µm membrane filter and proceed with 11.4.

11.4 Pipet 10.0 mL of sample, or a dilution of the sample, into a 50-mL beaker or flask.

11.5 Add 1.0 mL of lanthanum chloride/potassium chloride solution to each sample. Mix well by swirling.

11.6 Aspirate each sample and record its absorbance or concentration. Aspirate reagent water between each sample.

11.7 For instruments that do not readout directly in concentration, determine the strontium concentration of the aspirated solution from the analytical curve (10.5).

12. Calculation

12.1 Where dilutions have been made, calculate the concentration of strontium in the original sample as follows:

$$\text{Sr, mg/L} = A \times \frac{B}{C}$$

where:
A = Sr read from the analytical curve (10.5), mg/L,
B = total final volume of the diluted sample, and
C = volume of original sample diluted.

13. Precision and Bias [6]

13.1 The overall precision (reagent and waters of choice) and the single-operator precision for reagent water, based on data from eight participating laboratories involving twelve operators, varies with the quantity measured within the designated range of the test method as shown in Fig. 1.

13.2 The single-operator precision for waters of choice can be defined by the expression $0.023 \leq S_o \leq 0.026$, with the mean precision value of 0.024 mg/L over the designated range.

13.3 Recoveries of known amounts of strontium in a series of prepared standards were as given in Table 1.

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[6] Supporting data are available from ASTM International Headquarters. Request RR: D19-1054.

TABLE 1 Recovery

Amount Added, mg/L	Amount Found, mg/L	Bias	% Bias	Significant (95 % Confidence Level)
		Reagent Water Type II		
1.00	0.998	−0.002	−0.2	no
0.50	0.503	+ 0.003	+ 0.6	no
0.10	0.102	+ 0.002	+ 2	no
		Water of Choice		
1.00	1.03	+ 0.03	+ 3	no
0.50	0.504	+ 0.004	+ 0.8	no
0.10	0.086	−0.014	−14	

13.4 This test method was evaluated with reagent and natural water matrices. These data may not apply to waters of other matrices.

13.5 Precision and bias for this test method conforms to Practice D 2777-77, which was in place at the time of collaborative testing. Under the allowances made in 1.4 of D 2777-98, these precision and bias data do meet existing requirements for interlaboratory studies of Committee D19 test methods.

NOTE 7—The nitric acid digestion steps were not performed in the round-robin of this test method. It is an approved, recommended practice for determining total recoverable metals by atomic absorption spectrometry; however, its use can be expected to increase the variability of final results. The user should verify its suitability for a matrix of interest by evaluating recovery for spikes that have been taken through the digestion process (Guide D 5810).

14. Quality Control (QC)

14.1 The following quality control information is recommended for the determination of strontium in water.

14.2 For each method the instrument shall be calibrated using a minimum of four calibration standards and a calibration blank. The calibration correlation coefficient shall be equal to or greater than 0.990. In addition to the initial calibration blank, a calibration blank should be analyzed at the end of the batch run to ensure contamination was not a problem during the batch analysis.

14.3 An instrument check standard shall be analyzed at a minimum frequency of 10 % throughout the batch analysis. The value of the instrument check standard should fall between 80 % and 120 % of the true value.

14.4 Two method blanks shall be prepared ensuring that an adequate method blank volume is present for a minimum of seven repetitive analysis. The standard deviation of the method blank is used to determine the minimum detectable concentration of each sample and control in the batch.

14.5 A Laboratory Control Sample shall be analyzed with each batch of sample at a minimum frequency of 10 %.

14.6 If the QC for the sample batch is not within the established control limits, reanalyze the samples or qualify the results with the appropriate flags, or both.

14.7 Blind control samples should be submitted by an outside agency in order to determine the laboratory performance capabilities.

15. Keywords

15.1 atomic absorption; strontium; water