

Designation: D 4191 – 03

# Standard Test Method for Sodium in Water by Atomic Absorption Spectrophotometry<sup>1</sup>

This standard is issued under the fixed designation D 4191; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope\*

1.1 This test method covers the determination of low amounts of sodium in waters<sup>2</sup> having low solids content. The applicable range of this test method is from 0.20 to 3.0 mg/L when using the 589.6-nm resonance line. This range may be extended upward by dilution of an appropriate aliquot of sample or by use of the less-sensitive 330.2-nm resonance line (see Test Method D 3561). Many workers have found that this test method is reliable for sodium levels to 0.005 mg/L, but use of this test method at this low level is dependent on the configuration of the aspirator and nebulizer system available in the atomic absorption spectrophotometer as well as the experience and skill of the analyst. The precision and bias data presented are insufficient to justify use of this test method in the 0.005 to 0.20-mg/L range.

1.2 This test method has been used successfully with spiked reagent water. It is the analyst's responsibility to assure the validity of the test method to other low dissolved solids matrices.

1.3 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:

- D 1066 Practice for Sampling Steam<sup>3</sup>
- D 1129 Terminology Relating to Water<sup>3</sup>
- D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits<sup>3</sup>
- D 1193 Specification for Reagent Water<sup>3</sup>
- D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water<sup>3</sup>
- D 3370 Practice for Sampling Water from Closed Conduits<sup>3</sup>
- D 3561 Test Method for Lithium, Potassium, and Sodium

Ions in Brackish Water, Seawater, and Brines by Atomic Absorption Spectrophotometry<sup>4</sup>

D 5810 Guide for Spiking into Aqueous Samples<sup>3</sup>

D 5847 Practice for the Writing Quality Control Specifications for Standard Test Methods for Water Analysis<sup>3</sup>

## 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 Sodium is determined by flame atomic absorption spectrophotometry. The sodium content is determined by aspirating the low solids sample directly with no sample pretreatment.

# 5. Significance and Use

5.1 Sodium salts are very soluble, and sodium leached from soil and rocks tends to remain in solution. Water with a high ratio of sodium to calcium is deleterious to soil structure.

5.2 Sodium is not particularly significant in potable water except for those persons having an abnormal sodium metabolism, but water supplies in some areas contain sufficient sodium to be a factor in the planning of sodium-free diets.

5.3 The use of sodium salts is common in industry; therefore, many industrial wastewaters contain significant quantities of sodium. For high-pressure boiler feed-water even trace amounts of sodium are of concern.

# 6. Interferences

6.1 In the analysis of low-solids water, interferences are usually negligible.

# 7. Apparatus

7.1 *Atomic Absorption Spectrophotometer* for use at 589.6 nm.

NOTE 1—The manufacturer's instructions should be followed for all instrumental parameters. Wavelengths other than 589.6 nm may be used only if they have been determined to be equally suitable.

7.2 *Sodium Hollow-Cathode Lamps*—Multielement hollow cathode lamps are available and also have been found satisfactory.

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

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> Platte, J. A., and Marcy, V. M., "A New Tool for the Water Chemist," *Industrial Water Engineering*, May 1965.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 11.02.

7.3 *Oxidant: Air*, which has been passed through a suitable filter to remove oil, water, and other foreign substances is the usual oxidant.

7.4 *Fuel:* Acetylene—Standard, commercially available acetylene is the usual fuel. Acetone, always present in acetylene cylinders can affect analytical results. The cylinder should be replaced at 50 psig (345 kPa) to avoid acetone carryover.

7.4.1 **Caution**—"Purified" grade acetylene containing a special proprietary solvent rather than acetone should not be used with poly(vinyl chloride) tubing as weakening of the tubing walls can cause a potentially hazardous situation.

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

### 8. Reagents

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.<sup>5</sup> 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. 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 bias and precision of the test method. Type II water was specified at the time of round robin testing of this test method.

8.3 Sodium Solution, Stock (1.0 mL = 1.0 mg Na)—Dry sodium chloride to constant weight at 105°C. Dissolve 2.5418 g of the dry sodium chloride (NaCl) in water and dilute to 1 L with water.

NOTE 2—Certified sodium stock solutions are commercially available through chemical supply vendors.

8.4 Sodium Solution, Standard (1.00 mL = 0.1 mg Na)— Dilute 100.0 mL of sodium stock solution to 1 L with water.

### 9. Sampling

9.1 Collect the samples in accordance with the applicable ASTM test method as follows: Practices D 3370, Practice D 1066, and Specification D 1192.

### **10. Standardization**

10.1 Prepare 100 mL each of a blank and at least four standard solutions to bracket the expected sodium concentration range of the samples to be analyzed by diluting the standard sodium solution (8.4) with water. Prepare the stan-

TABLE 1 Determination of Bias for Sodium in Reagent Water by Atomic Absorption

Amount Added, mg/L	Amount Found, mg/L	$S_t$	S <sub>o</sub>	Bias %
0.20	0.184	0.053	0.024	-8.0
1.20	1.25	0.094	0.028	+ 4.17
2.70	2.81	0.129	0.081	+ 4.07

dards each time the test is to be performed. Select the standards to give zero, middle, and maximum points for an analytical curve.

10.2 Aspirate the blank and the standards and record the instrument readings. Aspirate water between standards.

10.3 Prepare an analytical curve by plotting the absorbance versus concentration for each standard on linear graph paper. Alternatively, read directly in concentration if this capability is provided with the instrument.

# 11. Procedure

11.1 Aspirate each sample and determine its absorbance or concentration. Aspirate water between samples.

### 12. Calculation

12.1 Calculate the concentration of sodium in the samples, in milligrams per litre, by either referring the absorbance obtained for each sample to a prepared analytical curve (10.3) or reading directly in concentration if the capability is provided with the instrument.

12.2 If an aliquot of diluted sample was analyzed, multiply the concentration of sodium, in milligrams per litre, by the appropriate dilution factor.

# 13. Precision and Bias<sup>6</sup>

13.1 The overall and single-operator precision of this test method for eight laboratories, which include a total of twelve operators analyzing each sample on three consecutive days, within its range for reagent water varies with the quantity being measured according to Table 1.

NOTE 3—Only reagent water was used to obtain the precision statement since this test method is designated for the determination of low amounts of sodium in low-solids water.

13.2 Recoveries of known amounts of sodium (from sodium chloride) in the series of prepared standards for the same laboratories and operators were as given in Table 1.

13.3 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 Practice D 2777 – 98, these precision and bias data do meet existing requirements for interlaboratory studies of Committee D19 test methods.

#### 14. Quality Control

14.1 In order to be certain that analytical values obtained using these test methods are valid and accurate within the

<sup>&</sup>lt;sup>5</sup> "Reagent Chemical, American Chemical Society Specifications," Am. Chemical Soc., 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."

<sup>&</sup>lt;sup>6</sup> Supporting data are available from ASTM Headquarters. Request RR: D19-1080.