



# Standard Test Method for Low Level Sodium in High Purity Water by Graphite Furnace Atomic Absorption Spectroscopy<sup>1</sup>

This standard is issued under the fixed designation D 6071; 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 trace sodium in high purity water. The method range of concentration is 1 to 40  $\mu\text{g/L}$  sodium. The analyst may extend the range once its applicability has been ascertained.

NOTE 1—It is necessary to perform replicate analysis and take an average to accurately determine values at the lower end of the stated range.

1.2 This test method is a graphite furnace atomic absorption spectrophotometric method for the determination of trace sodium impurities in ultra high purity water.

1.3 This test method has been used successfully with a high purity water matrix.<sup>2</sup> It is the responsibility of the analyst to determine the suitability of the test method for other 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.*

## 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 Test Methods of Committee D-19 on Water<sup>3</sup>
- D 3370 Practices for Sampling Water from Closed Conduits<sup>3</sup>
- D 3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry<sup>3</sup>
- D 4453 Practice for Handling of Ultra-Pure Water Samples<sup>3</sup>

## 3. Terminology

### 3.1 Definitions:

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.11 on Water for Power Generation and Process Use.

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<sup>2</sup> RP2712 Sub Program—Grab Sample Method Validation Report Results, Electric Power Research Institute, Palo Alto, CA 1987.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

3.1.1 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 atomic absorption utilizing a graphite furnace for sample atomization.

4.2 A sample volume of several microlitres, depending upon the concentration of the analyte, is deposited on a graphite tube housed within an electrical furnace, and the system is heated in an inert gas environment. The sample is evaporated to dryness, ashed (charred or pyrolyzed), and atomized.

4.3 Ground-state atoms are produced during the atomization stage of the temperature program. The ground-state atoms absorb the energy at a specific wavelength produced from a source as they are bombarded by the energy. The amount of energy absorbed is proportional to the concentration of the analyte in the sample.

4.4 The absorption signal produced during atomization is recorded on a chart recorder or stored in microprocessor memory and compared to those standards taken through the same process by means of an analytical curve.

4.5 A general guide for graphite furnace applications is given in Practice D 3919.

## 5. Significance and Use

5.1 Small quantities of sodium, 1 to 10  $\mu\text{g/L}$ , can be significant in high pressure boiler systems and in nuclear power systems. Steam condensate from such systems must have less than 10  $\mu\text{g/L}$ . In addition, condensate polishing system effluents should have less than 1  $\mu\text{g/L}$ . Graphite furnace atomic absorption spectroscopy (GFAAS) represents technique for determining low concentrations of sodium.

## 6. Interferences

6.1 No known interferences from other constituents are found in high purity waters.

6.2 For a complete discussion on interferences with graphite furnace procedures, refer to Practice D 3919.

6.3 Sodium is a common contaminant in many reagents. The analyst must ensure that the quality of the reagent used in the procedure is sufficiently high to determine trace levels of sodium.

6.4 All plasticware and apparatus must be cleaned and maintained to eliminate high background levels of sodium

when determining trace levels.

6.5 Airborne particulates are a potential interference with the analysis of sodium by GFAAS. The user must ensure that all plasticware and other equipment is capped or stored in air tight containers.

## 7. Apparatus

7.1 *Atomic Absorption Spectrophotometer*, with the capability of setting the following instrumental parameters:

Metal	Wavelength, nm	Slit width (SBW), nm
Sodium	589.0	0.5

NOTE 2—The manufacturer's instructions should be followed for all instrument parameters.

7.2 *Hollow Cathode Lamp*, for sodium.

7.2.1 Multielement hollow cathode lamps may be used if the analyst ensures the necessary sensitivity is available for the low level determination.

7.3 *Graphite Furnace*, capable of reaching temperatures sufficient to atomize the elements of interest. Maximum sensitivity will be obtained when atomization temperatures are reached rapidly.

7.4 *Graphite Tubes*, compatible with the furnace device. Standard graphite tubes of uncoated graphite should be used. When maximum sensitivity is required, the analyst may choose to use pyrolytically coated graphite tubes.

7.5 *Autosampler*, compatible with the graphite furnace device may be used to increase the precision of the injection or dispensing the sample into the graphite tube.

7.6 *Pipets*, microlitre with disposable plastic tips. Sizes may vary from 1 to 100 µL, as required.

7.7 *Flasks*, plastic, volumetric. Sizes may vary from 100 to 1000 mL.

7.8 *Strip Chart Recorder (Computing Device with Display)*—The user must keep a permanent record of the data in addition to instrument problems (drift, incomplete atomization, changes in sensitivity, etc.).

7.8.1 The strip chart recorder with a response of 0.2 s or less for full scale deflection is recommended to ensure accuracy.

## 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.<sup>4</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*—All references to water in this test method shall be understood to mean reagent water conforming to Specification D 1193, Type I.

8.3 *Sodium Solution, stock (1 mL = 1000 µg Na)*—Dissolve 2.542 g of sodium chloride in water and dilute to 1 L.

8.4 *Sodium Solution, intermediate (1 mL = 10.0 µg Na)*—Dilute 10.0 mL of stock sodium solution from 8.3 to 1 L with water.

8.5 *Sodium Solution, standard (1 mL = 0.05 µg Na)*—Dilute 5 mL of intermediate sodium solution from 8.4 to 1 L with water.

NOTE 3—Alternatively, the analyst may purchase a commercially available standard (1 mL = 1000 µg Na). Additional dilution will be necessary to obtain the stock sodium solution concentration in 8.3.

8.6 *Argon*, 99.99 % pure.

## 9. Sampling

9.1 Collect the sample in accordance with Practices D 1066 and D 3370 or Specification D 1192.

9.2 Samples should be collected in polystyrene, TFE-fluorocarbon or polypropylene bottles only. Do not use glass or polyethylene containers. The containers should be rinsed with Type I water. The container should be stored prior to use by either air drying and capping or filling with Type I water and capping. For further details, see Practice D 4453.

9.3 To avoid the possibility of contamination, samples should not be acidified.

## 10. Calibration

10.1 Prepare standards for the analytical calibration containing 0.0, 1.0, 5.0, 10.0, and 25.0 µg/L Na by diluting 0, 2, 10, 20, and 50 mL sodium standard solution in 8.5 to 100 mL with water. The 50.0 µg/L intermediate stock solution in 8.5 will be used as the high standard concentration.

10.2 Perform an instrument zero without making an injection.

10.3 Set the temperature program (dry, ash/char/pyrolyze, atomize) in accordance with the manufacturer's instructions.

10.3.1 *Sample Size*—Use 10 to 100 µL depending on the graphite tube size, the concentration, and desired detection level required. Use the same injection volume for the blank, standards, and all samples.

10.3.2 *Temperature Program*:

Program stage	Temperature, °C	Time, s
Dry	100	1 to 3 s/µL
Ash/char/pyrolyze	800	20.0
Atomize	2300	4.9

10.4 Condition the graphite tube surface prior to initiating the analysis. Condition the furnace by conducting the following steps.

10.4.1 Determine the furnace blank by initiating the atomization program without making an injection. Repeat until the furnace blank reproduces within 10 % of the initial absorbance value obtained.

10.4.2 Condition the graphite tube surface by injecting the 10 µg/L standard. Repeat until the absorbance reproduces within 10 %.

10.4.3 Inject each of the calibration standards. Reproducibility must be within 10 % for each standard concentration.

10.5 Prepare a calibration curve by plotting peak height or peak area versus concentration as µg/L on linear paper if direct concentration readout is unavailable on the instrument.

<sup>4</sup> *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.

**11. Procedure**

11.1 Use volumetric plasticware for the preparation of all standards and samples.

11.2 Rinse all the plasticware and equipment with water prior to use. The analyst may choose to store all plasticware in water or air dry and store in covered containers.

11.3 Analyze the samples as described in Section 10.

NOTE 4—The analyst should cover the sample containers or use a covered autosampler to minimize the possibility of airborne contamination between injections of the samples into the graphite tube.

**12. Calculation**

12.1 Read the sample concentration directly from the instrument where applicable or from the calibration curve generated in 10.5.

**13. Precision and Bias**<sup>5</sup>

13.1 The precision and bias for this test method were obtained in accordance with Practice D 2777.

13.2 *Precision*—The precision of this test method was determined using high purity water in eight laboratories. The

precision may be expressed as follows:

$$S_t = 0.22(X) + 0.88$$

$$S_o = 0(X) + 0.91$$

where:

$S_t$  = overall precision,

$S_o$  = single operator precision, and

$X$  = determined concentration of sodium,  $\mu\text{g/L}$ .

13.3 *Bias*—Mean recoveries of known amounts of sodium in prepared series of high purity water were as follows:

Amount added, $\mu\text{g/L}$	Amount found, $\mu\text{g/L}$	$\pm$ Bias	$\pm$ % Bias	Statistically significant
0.00	0.16	+ 0.16	—	No
0.00	0.40	+ 0.40	—	No
1.07	1.03	-0.04	-4 %	No
1.42	1.36	-0.06	-4 %	No
5.65	5.56	-0.09	-2 %	No
7.08	7.14	+ 0.06	+ 1 %	No
28.40	28.54	+ 0.14	0 %	No
35.40	40.41	+ 5.01	+ 14 %	No

**14. Keywords**

14.1 graphite furnace atomic absorption spectroscopy (GFAAS); high purity water; sodium

<sup>5</sup> Supporting data are available from ASTM Headquarters. Request RR:D19-1160.

**APPENDIX**

**(Nonmandatory Information)**

**X1. TECHNIQUES FOR REDUCING CONTAMINATION**

X1.1 Always wear uncolored, unpowdered plastic gloves during all operations. If gloves come in contact with sources of contamination, they need to be replaced.

X1.2 Wash flasks with HCl (1 + 1) immediately before use. If glass pipets are used, rinse with HCl (1 + 1) and rinse several times with water immediately before use.

X1.3 If using an automatic sampler for sample introduction, rinse the sample cups several times with water immediately prior to use. Do not load more than five cups on the sampler at one time. Contamination appears to be a problem if samples are allowed to sit for a period of time prior to analysis.

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