

Designation: D 4192 – 03

Standard Test Method for Potassium in Water by Atomic Absorption Spectrophotometry¹

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1. Scope*

1.1 This test method covers the determination of low amounts of potassium in waters ² having low solids content. The applicable range of this test method is 0.20 to 4.0 mg/L when using the 766.5-nm resonance line. The range may be extended upward by dilution of an appropriate aliquot of sample or by using the less-sensitive 404.4-nm resonance line. Many workers have found that this test method is reliable for potassium levels to 0.02 mg/L, but use of this test method at this low level is dependent on the configuration of the aspirator and nebulizer systems available in the atomic absorption spectrophotometer as well as the skill of the analyst. The precision and bias data presented are insufficient to justify use of this test method in the 0.02-mg/L range.

1.2 This test method has been used successfully with spiked reagent water. It is the analyst's responsibility to ensure the validity of this 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. For a specific precautionary statement, see Note 3.

2. Referenced Documents

- 2.1 ASTM Standards:
- D 1066 Practice for Sampling Steam³
- D 1129 Terminology Relating to Water³
- D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits³
- D 1193 Specification for Reagent Water³

- D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water³
- D 3370 Practices for Sampling Water from Closed Conduits³
- D 5810 Guide for Spiking into Aqueous Samples³

D 5847 Practice for the Writing Quality Control Specifications for Standard Test Methods for Water Analysis³

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 Potassium is determined by flame atomic absorption spectrophotometry. The potassium content is determined by aspirating the low solids sample directly with no sample pretreatment.

5. Significance and Use

5.1 Potassium occurs in rocks in a form that is not easily solubilized; therefore, the potassium content of natural waters is usually low. Most natural waters contain less than 20 mg/L of potassium, but waters containing several hundred milligrams per litre are occasionally found. Potassium is essential to animal nutrition, but a concentration of 1000 to 2000 mg/L in stock water is regarded as the extreme limit permissible.

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 766.5 nm.

NOTE 1—The manufacturer's instructions should be followed for all instrumental parameters. Wavelengths other than 766.5 nm may be used only if they have been determined to be equally suitable.

7.2 *Potassium 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.

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 Platte, J. A., and Marcy, V. M., "A New Tool for the Water Chemist," *Industrial Water Engineering*, May 1965.

³ Annual Book of ASTM Standards, Vol 11.01.

7.3 *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 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. ⁴ 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 Potassium Solution, Stock (1.0 mL = 1.0 mg K)—Dry potassium chloride to constant weight at 105°C. Dissolve 1.907 g of the dry potassium chloride (KCl) in water and dilute to 1 L with water.

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

8.4 *Potassium Solution, Standard (1.00 mL = 0.1 mg K)*— Dilute 100.0 mL of potassium stock solution to 1 L with water.

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

8.6 *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 carry over.

NOTE 3—Warning: "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.

9. Sampling

9.1 Collect the samples in accordance with the applicable ASTM standard 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 potassium concentration range of the samples to be analyzed by diluting the standard potassium solution 8.4 with water. Prepare the stan-

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

Amount Added, mg/L	Amount Found, mg/L	S_t	So	Bias %
0.15	0.164	0.037	0.014	+ 9.33
1.50	1.62	0.085	0.044	+ 8.00
3.00	3.03	0.179	0.062	+ 1.13

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 concentrations of potassium 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 potassium, in milligrams per litre, by the appropriate dilution factor.

13. Precision and Bias ⁵

13.1 The overall and single-operator precision of this test method for eight laboratories, which included 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 4—Only reagent water was used to obtain the precision statement since this test method is designated for the determination of trace amounts of potassium in low-solids water.

13.2 It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

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

13.4 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.

⁴ "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."

⁵ Supporting data are available from ASTM Headquarters. Request RR: D19-1081.