



Designation: D857 – 12

Standard Test Method for Aluminum in Water¹

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1. Scope*

1.1 This test method covers the direct flame atomic absorption determination of aluminum in the nitrous oxide-acetylene flame.

1.2 This test method is applicable to waters containing dissolved and total recoverable aluminum in the range from 0.5 to 5.0 mg/L. Aluminum concentrations as high as approximately 50 mg/L can be determined using this test method without dilution. However, no precision and bias data are available for concentrations greater than 5.0 mg/L.

1.3 This test method was tested on reagent, natural, and potable waters. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.4 The same digestion procedure may be used to determine total recoverable nickel (Test Methods D1886), chromium (Test Methods D1687), cobalt (Test Methods D3558), copper (Test Methods D1688), iron (Test Methods D1068), lead (Test Method D3559), manganese (Test Method D858), and zinc (Test Methods D1691).

1.5 Precision and bias data have been obtained on reagent, natural, and potable waters. It is the responsibility of the user to ensure the validity of this test method on untested matrices.

1.6 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are mathematical conversions and may not be exact equivalents; therefore, each system shall be used independently of the other.

1.7 *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 specific hazard statements, see [Note 1](#), [Note 2](#), and [Note 4](#).

1.8 Former Test Methods A (Fluorometric) and B and C (Spectrophotometric) were discontinued. Refer to [Appendix X1](#) for historical information.

¹ These test methods are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

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2. Referenced Documents

2.1 ASTM Standards:²

D858 Test Methods for Manganese in Water
D1066 Practice for Sampling Steam
D1068 Test Methods for Iron in Water
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D1687 Test Methods for Chromium in Water
D1688 Test Methods for Copper in Water
D1691 Test Methods for Zinc in Water
D1886 Test Methods for Nickel in 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
D3558 Test Methods for Cobalt in Water
D3559 Test Methods for Lead in Water
D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
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 these test methods, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *total recoverable aluminum, n*—an arbitrary term relating to the forms of aluminum recovered in the acid digestion procedure specified in this test method.

4. Summary of Test Method

4.1 Aluminum is determined by direct atomic absorption with only the addition of an ionization suppressor and sensitivity enhancer (optional).

5. Significance and Use

5.1 Although there is little information available concerning the toxicological significance of aluminum in man, the American Water Works Association has established a water quality

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.

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

guideline or goal of a maximum of 0.05 mg/L. Under the National Pollution Discharge Elimination System (NPDES), some permits may set aluminum discharge limits. Some evidence does exist to indicate that low levels (5 mg/L) will interfere with activated sludge processes. For the above reasons monitoring of aluminum may be desirable.

5.2 Aluminum is monitored in boiler make-up water, where alum has been used, to determine whether aluminum is present after pretreatment. Residual aluminum may consume ion exchange capacity or consume boiler water treatment chemicals added to stoichiometrically chelate hardness ions (that is, calcium and magnesium) in boiler feed water.

5.3 Aluminum is monitored in cooling water make-up, since its presence may result in deactivation of anionic substances in scale or corrosion inhibitor treatment chemicals, or both. Deactivation may result in decreased performance of inhibitors.

6. Interferences

6.1 Aluminum ionizes slightly in the nitrous oxide-acetylene flame, but the addition of sodium chloride described in this test method suppresses this interference. By this technique, a maximum concentration of 9000 mg/L sodium, 9000 mg/L potassium, 4000 mg/L calcium, 4000 mg/L magnesium, 9000 mg/L sulfate, 9000 mg/L chloride, 9000 mg/L nitrate, and 9000 mg/L iron may be tolerated.

7. Apparatus

7.1 *Atomic Absorption Spectrophotometer*, for use at 309.3 nm.

7.2 *Aluminum Hollow-Cathode Lamp*—Multielement lamps are not recommended.

7.3 *Oxidant*—See 8.8.

7.4 *Fuel*—See 8.9.

7.5 *Pressure-Reducing Regulators*—The supplies of fuel and oxidant should be reduced by suitable regulators to the levels recommended by the manufacturer of the spectrophotometer.

NOTE 1—**Warning:** The nitrous oxide-acetylene flame is hazardous due to its flash-back potential. Follow the instrument manufacturer's recommended operating procedures closely.

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.³ 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, references 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 lessening the bias and precision of the determination. Type II water was specified at the time of round-robin testing of this test method.

8.3 *Aluminum Solution, Standard (1 mL = 0.1 mg Al)*—Dissolve 1.758 g of aluminum potassium sulfate ($\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$) in water. Add 1 mL of nitric acid and dilute to 1 L. A purchased aluminum stock solution of appropriate known purity is also acceptable.

8.4 *Bis-(2-Ethoxyethyl) Ether*.

NOTE 2—**Warning:** Avoid inhalation. Perform all manipulation in a well-ventilated hood. This ether can form dangerous peroxides and should be inspected regularly for their presence.

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

8.6 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO_3).

8.7 *Sodium Chloride Solution (25.4 g/L)*—Dissolve 25.4 g of sodium chloride (NaCl) in water and dilute to 1 L.

8.8 *Oxidant:*

8.8.1 *Air*, which has been suitably dried and filtered, is used to support combustion before switching to nitrous oxide.

8.8.2 *Nitrous Oxide* is the required oxidant.

8.9 *Fuel*—Standard commercially available acetylene is the required fuel. The cylinder should be replaced at a gage pressure of 517 kPa (75 psi) to minimize the carry-over of acetone. Since “purified” grades contain a solvent that softens poly(vinyl chloride) tubing, its use constitutes a safety hazard and is not recommended.

9. Sampling

9.1 Collect samples in accordance with Practices D1066 or D3370, as applicable. For dissolved aluminum, filter the samples at the time of collection through a 0.45- μm filter. Acidify the filtrate to pH 2 or less with nitric acid. For total recoverable aluminum, acidify the unfiltered sample to pH 2 or less at the time of collection. The holding time for samples may be calculated in accordance with Practice D4841.

NOTE 3—Alternatively, the pH may be adjusted in the laboratory if the sample is returned within 14 days. This could reduce hazards of working with acids in the field when appropriate.

9.2 Soak all glassware in hot HCl (1 + 1) for 2 h. Drain and rinse at least five times with water. Drain and flush with methyl alcohol, ethyl alcohol, or isopropyl alcohol.

10. Preparation of Apparatus

10.1 Soak all glassware in hot HCl (1 + 1) for 2 h. Drain and rinse at least five times with water. Drain and flush with methyl alcohol, ethyl alcohol, or isopropyl alcohol.

³ *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.

11. Calibration and Standardization

11.1 Prepare aluminum standards in the range from 0.0 to 5.0 mg/L by dilution of the aluminum standard solution (see 8.3) with water.

11.2 Add 1.0 mL of NaCl solution (8.7) to 10.0 mL of standard and mix thoroughly. If very low concentrations of aluminum are anticipated in the samples, 1 mL of *bis*-(2-ethoxyethyl) (8.4) ether may be added to the blank and each standard, which will act as a sensitivity enhancer. If this sensitivity enhancer is used, it must also be added to each sample (12.6).

11.3 Measure the absorbance of the standards and construct an analytical curve by plotting the absorbance of the standards versus the concentration of aluminum. Alternatively, calibrate the spectrophotometer and read directly in concentration.

12. Procedure

12.1 If dissolved aluminum is to be determined proceed to 12.6.

12.2 Measure a volume of well-mixed acidified sample containing less than 0.5 mg of aluminum (100 mL maximum) into a 125-mL beaker.

12.3 Add 0.5 mL of HNO₃ and 5 mL of HCl.

12.4 Heat the samples on a hot plate in a well-ventilated hood until the volume has been reduced to 10 to 15 mL, making certain the samples do not boil.

NOTE 4—For brines and samples with high levels of suspended matter, the amount of reduction in volume is left to the discretion of the analyst.

NOTE 5—Many laboratories have found block digestion systems a useful way to digest samples for trace metals analysis. Systems typically consist of either a metal or graphite block with wells to hold digestion tubes. The block temperature controller must be able to maintain uniformity of temperature across all positions of the block. For trace metals analysis, the digestion tubes should be constructed of polypropylene and have a volume accuracy of at least 0.5%. All lots of tubes should come with a certificate of analysis to demonstrate suitability for their intended purpose.

12.5 Cool and, if necessary, filter the samples through a filter (fine-texture, acid-washed, ashless paper) into a 100-mL volumetric flask. Wash the paper several times and bring the volume to 100-mL.

12.6 To a 10-mL aliquot of sample add 1.0 mL of NaCl and mix thoroughly. If 1 mL of *bis*-(2-ethoxyethyl) ether was added to the blank and standards as a sensitivity enhancer (11.2), an equal amount must be added to each sample.

12.7 Atomize each of the standards, samples, and blank and determine its absorbance or concentration. Atomize water between samples.

13. Calculation

13.1 Determine the concentration of aluminum in each sample by referring to the calibration curve in 13.3. Alternatively, calibrate the spectrophotometer and read directly in concentration mode.

13.2 Calculate the concentration of total recoverable aluminum in milligrams per litre using Eq 1:

TABLE 1 Overall (S_T) and Single-Operator (S_o) Interlaboratory Precision for Aluminum by Direct AAS

Reagent Water:			
Concentration (X), mg/L	0.848	2.54	4.11
S_T	0.167	0.19	0.19
S_o	0.128	0.18	0.23
Natural Water:			
Concentration (X), mg/L	0.772	2.48	4.07
S_T	0.194	0.19	0.21
S_o	0.166	0.16	0.28

$$\text{Aluminum, mg/L} = C \times (100/V) \quad (1)$$

where:

C = concentration from curve, mg/L, and

V = volume of aliquot, mL.

13.3 Calculate the concentration of dissolved aluminum directly from the calibration curve.

14. Precision and Bias⁴

14.1 Precision data for this test method were obtained on reagent, natural, and potable waters. It is the user's responsibility to assure the validity of this test method for waters of untested matrices.

14.2 The collaborative test of the direct atomic absorption test method for aluminum in reagent water was performed at three levels by four laboratories (seven operators) making a total of fifteen observations at each level. The test in matrix water at the same levels was performed by three laboratories (six operators) making a total of twelve observations at each level.

14.3 Precision and bias for this test method conform to Practice D2777 – 77, which was in place at the time of collaborative testing. Under the allowances made in 1.4 of Practice D2777 – 08, these precision and bias data do meet existing requirements for interlaboratory studies of Committee D19 test methods.

14.4 The overall and single-operator precision of this test method (S_T and S_o respectively) within its designated range for reagent water and selected water matrices varies with the quantity tested in accordance with Table 1.

14.5 Recoveries of known amounts of aluminum from reagent water, Type II, and selected water matrices were as shown in Table 2.

14.6 The sensitivity enhancer was not used in collaborative tests. It is the user's responsibility to determine its effect on precision and bias.

15. Quality Control

15.1 In order to be certain that analytical values obtained using this test method are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when analyzing aluminum by this test method.

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D19-1064 and RR:D19-1065. Contact ASTM Customer Service at service@astm.org.