



Standard Test Method for Aluminum in Water¹

This standard is issued under the fixed designation D 857; 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 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 D 1886), chromium (Test Methods D 1687), cobalt (Test Methods D 3558), copper (Test Methods D 1688), iron (Test Methods D 1068), lead (Test Method D 3559), manganese (Test Method D 858), and zinc (Test Methods D 1691).

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 *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 3.

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

2. Referenced Documents

2.1 ASTM Standards:

- D 858 Test Method for Manganese in Water²
- D 1066 Practice for Sampling Steam²
- D 1068 Test Methods for Iron in Water²
- D 1129 Terminology Relating to Water²
- D 1193 Specification for Reagent Water²

¹ These test methods are under the jurisdiction of ASTM Committee D-19 on Water and are 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.

- D 1687 Test Methods for Chromium in Water²
- D 1688 Test Methods for Copper in Water²
- D 1691 Test Methods for Zinc in Water²
- D 1886 Test Methods for Nickel in Water²
- D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water²
- D 3370 Practices for Sampling Water from Closed Conduits²
- D 3558 Test Methods for Cobalt in Water²
- D 3559 Test Method for Lead in Water²
- D 4841 Practice for Estimation of Holding Times for Water Samples Containing Organic and Inorganic Constituents²

3. Terminology

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

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *total recoverable aluminum*—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 guideline or goal of a maximum of 0.05 mg/L. Under the National Pollution Discharge Elimination System, 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 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 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.

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).

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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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:*

8.9.1 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 D 1066 or D 3370, 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 D 4841.

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.

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 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) 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, read directly in concentration if this capability is provided with the spectrophotometer.

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 HNO_3 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 3—For brines and samples with high levels of suspended matter, the amount of reduction in volume is left to the discretion of the analyst.