



Standard Test Methods for Zinc in Water ¹

This standard is issued under the fixed designation D 1691; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 These test methods cover the determination of zinc in water. Two test methods are given as follows:

		Sections
Test Method A—Atomic Absorption, Direct	0.05 to 2 mg/L	8-16
Test Method B—Atomic Absorption, Chelation-Extraction	20 to 200 μ g/L	17-25

1.2 Either dissolved or total recoverable zinc may be determined.

1.3 These test methods have been used successfully with reagent grade water. See the specific test method for applicability to other matrices. It is the user's responsibility to assure the validity of these test methods in 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.* For specific hazard statements, see Section 6 and Note 5, Note 8, and Note 13.

1.5 Two former colorimetric test methods were discontinued. Refer to Appendix X1 for historical information.

2. Referenced Documents

2.1 ASTM Standards:

- D 858 Test Methods for Manganese in Water ²
- D 1066 Practice for Sampling Steam ²
- D 1068 Test Methods for Iron in Water ²
- D 1129 Terminology Relating to Water ²
- D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits ²
- D 1193 Specification for Reagent Water ²
- D 1687 Test Methods for Chromium in Water ²
- D 1688 Test Methods for Copper in Water ²
- D 1886 Test Methods for Nickel in Water ²
- D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water ²

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 3370 Practices for Sampling Water from Closed Conduits ²

D 3557 Test Methods for Cadmium in Water ²

D 3558 Test Methods for Cobalt in Water ²

D 3559 Test Methods for Lead in Water ²

D 4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents ²

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 zinc*—an arbitrary analytical term relating to the recoverable form of zinc that is determinable by the digestion method which is included in the Procedure.

4. Significance and Use

4.1 Zinc is an essential and beneficial element in body growth. Concentrations above 5 mg/L can cause a bitter astringent taste and opalescence in alkaline waters. The zinc concentration of U.S. drinking waters varies between 0.06 and 7.0 mg/L with a mean of 1.33 mg/L. Zinc most commonly enters the domestic water supply from deterioration of galvanized iron and dezincification of brass. Zinc in water also may result from industrial water pollution. ³

5. Purity of Reagents

5.1 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.

5.2 *Purity of Water*—Unless otherwise indicated, references

³ "Standard Methods for the Examination of Water and Wastewater," 16th edition, 1985, APHA, AWWA-WPCF.

⁴ *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.

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.

6. Hazards

6.1 Although zinc is nontoxic to man, these test methods require the use of certain other toxic and hazardous reagents and materials. Each should be used with care and exerting proper precautions.

7. Sampling

7.1 Collect the sample in accordance with Practice D 1066, Specification D 1192, and Practices D 3370, as applicable.

7.2 Samples shall be preserved with nitric acid (HNO₃) (sp gr 1.42) to a pH of 2 or less immediately at the time of collection, normally about 2 mL/L of HNO₃. If only dissolved zinc is to be determined, the sample, shall be filtered through a 0.45- μ m membrane filter before acidification. The holding time for samples may be calculated in accordance with Practice D 4841.

TEST METHOD A—ATOMIC ABSORPTION, DIRECT

8. Scope

8.1 This test method covers the determination of dissolved and total recoverable zinc in most waters and wastewaters.

8.2 This test method is applicable in the range from 0.05 to 2.0 mg/L of zinc. The range may be extended to concentrations greater than 2.0 mg/L by dilution of the sample.

8.3 This test method has been used successfully with reagent grade water, river water, wastewater, ground water, tap water, lake water, refinery effluent. The information on precision and bias may not apply to other waters.

9. Summary of Test Method

9.1 Zinc is determined by atomic absorption spectrophotometry. Dissolved zinc is determined by aspirating a portion of the filtered and preserved sample directly with no pretreatment. Total recoverable zinc is determined by aspirating the sample following hydrochloric-nitric acid digestion and filtration. The same digestion procedure is used to determine total recoverable cadmium (Test Methods D 3557), chromium, (Test Methods D 1687), cobalt (Test Methods D 3558), copper (Test Methods D 1688), iron (Test Methods D 1068), lead (Test Methods D 3559), manganese (Test Methods D 858), and nickel (Test Methods D 1886).

10. Interferences

10.1 Sodium, potassium, sulfate, and chloride (9000 mg/L each), calcium and magnesium (4000 mg/L each), nitrate (2000 mg/L), and cadmium, lead, copper, nickel, cobalt, and chromium (10 mg/L each) do not interfere.

10.2 Background correction or a chelation-extraction procedure (see Test Method B) may be necessary to determine low levels of zinc in some waters.

NOTE 1—Instrument manufacturers' instructions for use of the specific correction technique should be followed.

11. Apparatus

11.1 *Atomic Absorption Spectrophotometer*, for use at 213.9 nm.

NOTE 2—The manufacturer's instructions should be followed for all instrumental parameters. Wavelengths other than 213.9 nm may be used if they have been determined to be equally suitable.

11.1.1 *Zinc Light Source*—Hollow-cathode lamps or electrodeless discharge lamps have been found satisfactory.

11.2 *Oxidant*—See 12.6.

11.3 *Fuel*—See 12.7.

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

12. Reagents and Materials

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

NOTE 3—If the reagent blank concentration is greater than the method detection limit, distill the HCl or use a spectrograde acid. **Caution**—When HCl is distilled an azeotropic mixture is obtained (approximately 6 N HCl). Therefore, whenever concentrated HCl is specified in the preparation of a reagent or in the procedure, use double the amount specified if a distilled acid is used.

12.2 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO₃).

NOTE 4—If the reagent blank concentration is greater than the method detection limit, distill the HNO₃ or use a trace metal grade acid.

12.3 *Nitric Acid* (1 +499)—Add 1 volume of HNO₃(sp gr 1.42) to 499 volumes of water.

12.4 *Zinc Solution, Stock* (1 mL = 1.0 mg Zn)—Dissolve 1.245 g of zinc oxide (ZnO) in a mixture of 10 mL of HNO₃(sp gr 1.42) and 10 mL of water. Dilute to 1 L with water.

12.5 *Zinc Solution, Standard* (1 mL = 0.1 mg Zn)—Dilute 100.0 mL of the zinc stock solution and 1 mL of HNO₃(sp gr 1.42) to 1 L with water.

12.6 *Oxidant*:

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

12.7 *Fuel*:

12.7.1 *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).

NOTE 5—**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.

13. Standardization

13.1 Prepare 100 mL each of a blank and at least four standard solutions to bracket the expected zinc concentration range of the samples to be analyzed by diluting the standard zinc solution (12.5) with HNO₃(1 +499). Prepare the standards