



Designation: D2036 – 09 (Reapproved 2022)

Standard Test Methods for Cyanides in Water¹

This standard is issued under the fixed designation D2036; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the determination of cyanides in water. The following test methods are included:

	Sections
Test Method A	12 – 18
Total Cyanides after Distillation	
Test Method B	19 – 25
Cyanides Amenable to Chlorination ² by Difference	
Test Method C	26 – 32
Weak Acid Dissociable Cyanides	
Test Method D	33 – 39
Cyanides Amenable to Chlorination without Distillation (Short-Cut Method)	

1.2 Cyanogen halides may be determined separately.

NOTE 1—Cyanogen chloride is the most common of the cyanogen halide complexes as it is a reaction product and is usually present when chlorinating cyanide-containing industrial waste water. For the presence or absence of CNCl, the spot test method given in [Annex A1](#) can be used.

1.3 These test methods do not distinguish between cyanide ions and metalocyanide compounds and complexes. Furthermore, they do not detect the cyanates. Cyanates can be determined using ion chromatography without digestion.

NOTE 2—The cyanate complexes are decomposed when the sample is acidified in the distillation procedure.

1.4 The cyanide in cyanocomplexes of gold, platinum, cobalt and some other transition metals is not completely recovered by these test methods. Refer to Test Method [D6994](#) for the determination of cyanometal complexes.

1.5 Cyanide from only a few organic cyanides are recovered, and those only to a minor extent.

1.6 Part or all of these test methods have been used successfully with reagent water and various waste waters. It is

the user's responsibility to assure the validity of the test method for the water matrix being tested.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in [5.1](#), [8.8](#), [8.18](#), [Section 9](#), [11.3](#), and [16.1.9](#).

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*³

[D1129 Terminology Relating to Water](#)
[D1193 Specification for Reagent Water](#)
[D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water](#)
[D5788 Guide for Spiking Organics into Aqueous Samples](#)
[D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis](#)
[D6696 Guide for Understanding Cyanide Species](#)
[D6888 Test Method for Available Cyanides with Ligand Displacement and Flow Injection Analysis \(FIA\) Utilizing Gas Diffusion Separation and Amperometric Detection](#)
[D6994 Test Method for Determination of Metal Cyanide Complexes in Wastewater, Surface Water, Groundwater and Drinking Water Using Anion Exchange Chromatography with UV Detection](#)
[D7284 Test Method for Total Cyanide in Water by Micro](#)

¹ These test methods are under the jurisdiction of ASTM Committee [D19](#) on Water and are the direct responsibility of Subcommittee [D19.06](#) on Methods for Analysis for Organic Substances in Water.

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² For an explanation of the term cyanides amenable to alkaline chlorination, see Lancy, L. E. and Zabban, W., "Analytical Methods and Instrumentation for Determining Cyanogen Compounds," *Papers on Industrial Water and Industrial Waste Water*, ASTM STP 337, 1962, pp. 32–45.

³ 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.

Distillation followed by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection
D7365 Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this standard, refer to Terminology **D1129** and Guide **D6696**.

3.2 Acronyms:

3.2.1 *FIA*, *n*—flow injection analysis

3.2.2 *HPLC*, *n*—high performance liquid chromatography

3.2.3 *IC*, *n*—ion chromatography

3.2.4 *PAD*, *n*—pulsed amperometric detection

4. Summary of Test Method

4.1 The cyanide as hydrocyanic acid (HCN) is released from compounds by means of reflux distillation and absorbed in sodium hydroxide solution. The conditions used for the distillation distinguish the type of cyanide. The sodium cyanide in the absorbing solution can be determined colorimetrically, by ion chromatography, titration, by selective ion electrode, or as described in Test Method **D6888** using flow injection with amperometric detection.

4.2 Test Method A, Total Cyanides, is based on the decomposition of nearly all cyanides in the presence of strong acid, magnesium chloride catalyst, and heat during a 1-h reflux distillation.

4.3 Test Method B, Cyanide Amenable to Chlorination, is based on chlorinating a portion of the sample under controlled conditions followed by the determination of total cyanide in both the original and chlorinated samples. Cyanides amenable to chlorination are calculated by difference.

4.3.1 This test method can be affected by compounds that are converted during chlorination to color-producing compounds or react with the reagents used, and cause interference in the procedure employed to determine cyanide in the absorption solution.

4.4 Test Method C, Weak Acid Dissociable Cyanides, is based on the decomposition of cyanides in the presence of weak acid, zinc acetate and heat during a 1-h reflux distillation.

4.5 Test Method D, Cyanide Amenable to Chlorination without Distillation, is a direct colorimetric procedure.

4.6 In the absence of interference, the minimum concentration of cyanide in the absorption solution that can be accurately determined colorimetrically is 0.005 mg/L, ion chromatography and Test Method **D6888** are 0.002 mg/L, titration is 0.4 mg/L and by selective ion electrode is 0.05 mg/L. Pretreatment including distillation tends to increase these concentrations to a degree determined by the amount of manipulation required and the type of sample.

4.7 Round-robin data indicate the following minimum concentrations: colorimetric 0.03 mg/L; titration 1.0 mg/L; and selective ion electrode 0.03 mg/L. Ion chromatography and Test Method **D6888** have a minimum levels equal to approximately 0.002 mg/L.

5. Significance and Use

5.1 Cyanide is highly toxic. Regulations have been established to require the monitoring of cyanide in industrial and domestic wastes and in surface waters (**Appendix X1**).

5.2 Test Method D is applicable for natural water and clean metal finishing or heat treatment effluents. It may be used for process control in wastewater treatment facilities providing its applicability has been validated by Test Method B or C.

5.3 The spot test outlined in **Annex A1** can be used to detect cyanide and thiocyanate in water or wastewater, and to approximate its concentration.

6. Interferences

6.1 Common interferences in the analysis for cyanide include oxidizing agents, sulfides, aldehydes, glucose and other sugars, high concentration of carbonate, fatty acids, thiocyanate, and other sulfur containing compounds.

6.2 It is beyond the scope of these test methods to describe procedures for overcoming all of the possible interferences that may be encountered. Refer to Practice **D7365** for potential interferences for the analysis of cyanide in water.

7. Apparatus

7.1 *Distillation Apparatus*—The reaction vessel shall be a 1 L round bottom flask, with provision for an inlet tube and a condenser. The inlet tube shall be a funnel with an 8 mm diameter stem that extends to within 6 mm of the bottom of the flask. The condenser, which is recommended, shall be a reflux-type, cold finger, or Allihn. The condenser shall be connected to a vacuum-type absorber which shall be in turn connected to a vacuum line which has provision for fine control. The flask shall be heated with an electric heater. Examples of the apparatus are shown in **Fig. 1**. Equivalent apparatus is acceptable provided cyanide recoveries of 100 % $\pm$ 4 % are documented.

7.1.1 Smaller distillation tubes such as 50 mL MIDI tubes or 6 mL MicroDist (trademarked) tubes described in Test Method **D7284** can be used if the quality control requirements in Section 40 are satisfied. The reagents should be added proportionately to those specified in this test method for smaller sample sizes. While the use of smaller distillation tubes is generally accepted, the interlaboratory study was conducted with 500 mL samples; therefore, the user is responsible to determine the actual precision and bias when using a different type of distillation apparatus.

7.2 *Spectrophotometer or Filter Photometer*, suitable for measurement in the region of 578 nm, using 1.0 cm, 2.0 cm, 5.0 cm, and 10.0 cm absorption cells. Filter photometers and photometric practices used in these test methods shall conform to Practice **E60**. Spectrophotometers shall conform to Practice **E275**.