



Standard Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion¹

This standard is issued under the fixed designation D4282; 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 determination of free cyanides in waters and wastewaters. Free cyanide is here defined as the cyanide which diffuses as cyanide (HCN), at room temperature, from a solution at pH 6.²

1.2 This test method does not include complexes that resist dissociation, such as hexacyanoferrates and gold cyanide, nor does it include thiocyanate and cyanohydrin.

1.3 This test method may be applied to water and wastewater samples containing free cyanide from 10 to 150 $\mu\text{g/L}$. Greater concentrations may be determined by appropriate dilution.

1.4 This test method has been fully validated by collaborative testing as specified by Practice D2777.

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

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 8.6, 8.9, Section 9, and 12.2.1.

2. Referenced Documents

2.1 ASTM Standards:³

D1129 Terminology Relating to Water

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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² The paper by J. M. Kruse and L. E. Thibault “Determination of Free Cyanide in Ferro- and Ferricyanides,” *Analytical Chemistry*, 45(13): 2260–2261; 1973 Nov., recommends a diffusion at pH 7. The ANSI modification (ANSI PH 4.41-1978) uses pH 6. Using the conditions of the ANSI method, diffusion is completed within 4 hours at pH 6. Longer diffusion time was required at pH 7 on the samples analyzed.

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.

D1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits (Withdrawn 2003)⁴

D1193 Specification for Reagent 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

D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water

D4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data (Withdrawn 2002)⁴

D5788 Guide for Spiking Organics into Aqueous Samples

D5789 Practice for Writing Quality Control Specifications for Standard Test Methods for Organic Constituents (Withdrawn 2002)⁴

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

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.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *free cyanide*—those simple cyanides or loosely held cyanide complexes that diffuse at pH 6, at room temperature.

4. Summary of Test Method

4.1 The reactions are carried out in a microdiffusion cell.

4.2 The sample is treated with cadmium ion to precipitate the hexacyanoferrates.

4.3 The sample is buffered at pH 6 and allowed to stand for 4 h.

4.4 The HCN diffuses into sodium hydroxide solution.

4.5 An aliquot of the sodium hydroxide solution is treated with chloramine-T, and the cyanogen chloride formed is

⁴ The last approved version of this historical standard is referenced on www.astm.org.