



Designation: D8273 – 20

Standard Practice for Determination of Total and Available Cyanide in Solid Waste and Soil after Alkaline Extraction¹

This standard is issued under the fixed designation D8273; 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 practice is used for the determination of total or available cyanide in solid waste, sediment and soil samples after alkaline extraction. Simple cyanide (CN^-) salts of group 1 and group 2 (alkali and alkaline earth) metals; soluble alkali and alkaline earth salts of zinc, copper, cadmium, mercury, nickel, silver, and iron cyanide complexes; and insoluble metal-metal cyanide complexes, such as Prussian blue, are quantitatively recovered. Gold, platinum group metals and cobalt cyanide complexes are not recovered during analysis.

1.2 Free cyanide cannot be determined due to the change of equilibrium conditions during the extraction process.

1.3 Cyanide complexes are extracted into an alkaline solution as described in this practice. Measure the total cyanide using Test Methods [D7511](#) or [D7284](#). Measure the available cyanide using Test Method [D6888](#). Calculate cyanide content in the soil or waste.

1.4 The method detection limit (MDL) is dependent on the test method used to measure the cyanide content. Based on the methods cited, it is approximately 1 mg/kg and the minimum level (ML) is 5 mg/kg. The applicable range is also dependent on the test method used to measure cyanide. Based on the methods cited, it is 5 to 100 mg/kg.

1.5 This practice should be used by analysts experienced with extractions and flow injection analysis (FIA), or working under the close supervision of such qualified persons.

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

1.7 This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which

the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title means only that the document has been approved through the ASTM consensus process.

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 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:²

- [D1129 Terminology Relating to Water](#)
- [D1193 Specification for Reagent Water](#)
- [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 \(Withdrawn 2002\)³](#)
- [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](#)
- [D7284 Test Method for Total Cyanide in Water by Micro Distillation followed by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection](#)

¹ This practice 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 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.

³ The last approved version of this historical standard is referenced on www.astm.org.

D7365 Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide
D7511 Test Method for Total Cyanide by Segmented Flow Injection Analysis, In-Line Ultraviolet Digestion and Amperometric Detection

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 Definitions of Terms Specific to This Standard:

3.2.1 *available cyanide, n*—cyanides that are free (HCN and CN⁻) and metal-cyanide complexes that are easily dissociated into free cyanide ions.

3.2.1.1 *Discussion*—The method does not detect the less toxic strong metal-cyanide complexes, cyanides that are not “amenable to chlorination.” Available cyanide determines the same cyanide compounds determined as weak acid dissociable (WAD) and as cyanide amenable to chlorination (CATC).

3.2.2 *total cyanide, n*—inorganic cyanide containing compounds in a sample, including free cyanide, available cyanide, strong metal cyanide complexes, and water and acid insoluble metal-metal cyanide complexes.

3.2.2.1 *Discussion*—Analytically, gold, platinum group metals, and cobalt cyanide complexes are not detected.

4. Summary of Practice

4.1 A 1.00 ± 0.05 g portion of the prepared sample is measured into a 50-mL screw cap vial and extracted with 10 mL of 1 M Sodium Hydroxide Solution and mixed by shaking. The sample extract is filtered and diluted to 200 mL with water. An aliquot of the extract is injected into a flow injection analyzer (FIA) as described in Test Methods **D6888**, **D7284**, or **D7511**. The cyanide extracted from the solid sample is calculated.

4.2 Calibration and data are processed with the instrument’s data acquisition software.

5. Significance and Use

5.1 Cyanide and hydrogen cyanide are highly toxic. Regulations have been established requiring the measurement of cyanide in soil and solid waste samples. This practice is also useful for performing material balances to account for the distribution of cyanides in cyanidation products from metallurgical processes.

5.2 This practice is applicable to the determination of available or total water soluble, or both, and water insoluble cyanides in soil and solid waste.

5.3 Water insoluble cyanide complexes, such as Prussian blue, are not completely recovered by distillation methods. This practice extracts all cyanides, including the water insoluble cyanides such as Prussian blue, and then the extract solution can be analyzed for cyanide with Test Methods **D6888**, **D7284**, or **D7511**.

6. Interference

6.1 Method interferences can be caused by contamination in the reagents, reagent water, glassware, etc., which may bias the results. Take care to keep all such items from contaminants.

6.2 Sulfide and sulfide-containing compounds interfere with direct distillation methods; however, the preliminary extraction described in this practice only extracts soluble sulfides and does not decompose metal-sulfide minerals. The reagents in Test Methods **D6888**, **D7284**, or **D7511** are capable of complexing up to 50 mg/L of sulfide.

6.3 Many compounds that are known to cause interference with distillation procedures do not interfere under the alkaline extraction conditions of this practice.

6.4 See Test Methods **D6888**, **D7284**, and **D7511** and Practice **D7365** for a more detailed discussion of interferences and interference removal.

7. Apparatus

7.1 Refer to Test Methods **D6888**, **D7284**, or **D7511** for description of required apparatus.

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 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean interference free reagent water conforming to Type I or Type II grade of Specification **D1193**.

8.3 *Sodium Hydroxide Solution (1 M)*—Dissolve 40 g of sodium hydroxide (NaOH) in laboratory water and dilute to 1 L.

9. Hazards

9.1 **Warning**—Because of the toxicity of cyanide, great care must be exercised in its handling. Acidification of cyanide solutions produces toxic hydrocyanic acid (HCN). All manipulations must be done in the hood so that any HCN gas that might escape is safely vented.

9.2 **Warning**—Many of the reagents used in these test methods are highly toxic. These reagents and their solutions must be disposed of properly.

9.3 All reagents and standards should be prepared in volumes consistent with laboratory use to minimize the generation of waste.

⁴ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.