



Designation: D7572 – 15 (Reapproved 2022)

Standard Guide for Recovery of Aqueous Cyanides by Extraction from Mine Rock and Soil¹

This standard is issued under the fixed designation D7572; 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 guide is applicable for the collection, extraction, and preservation of extracts from mine rock and soil samples for the analysis of cyanide in the extracts. Responsibilities of field sampling personnel and the laboratory are indicated.

1.2 The sampling, preservation, and extraction procedures described in this guide are recommended for the analysis of total cyanide, available cyanide, weak acid dissociable cyanide, and free cyanide by Test Methods [D2036](#), [D4282](#), [D4374](#), [D6888](#), [D6994](#), [D7237](#), and [D7284](#). The information supplied in this guide can also be applied to other analytical methods for cyanide, for example, US EPA Method 335.4.

1.3 The procedure options methods appear in the following order:

Procedure Option	Sections
Option A Laboratory Processing of Field Preserved Samples	11 and 12
Option B Laboratory Processing of Moist Field Samples	13 and 14

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

1.5 *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.*

1.6 *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.*

¹ This guide 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.

Current edition approved May 1, 2022. Published May 2022. Originally approved in 2009. Last previous edition published 2015 as D7572 – 15. DOI: 10.1520/D7572-15R22.

2. Referenced Documents

2.1 ASTM Standards:²

- [D1129 Terminology Relating to Water](#)
- [D1193 Specification for Reagent Water](#)
- [D1293 Test Methods for pH of Water](#)
- [D2036 Test Methods for Cyanides in Water](#)
- [D3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents](#)
- [D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water](#)
- [D4282 Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion](#)
- [D4374 Test Methods for Cyanides in Water—Automated Methods for Total Cyanide, Weak Acid Dissociable Cyanide, and Thiocyanate \(Withdrawn 2012\)³](#)
- [D4840 Guide for Sample Chain-of-Custody Procedures](#)
- [D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents](#)
- [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](#)
- [D7237 Test Method for Free Cyanide and Aquatic Free Cyanide with 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](#)
- [D7365 Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide](#)

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.

D7511 Test Method for Total Cyanide by Segmented Flow Injection Analysis, In-Line Ultraviolet Digestion and Amperometric Detection

2.2 U.S. EPA Methods:⁴

EPA OIA-1677 Available Cyanide in Water

EPA Method 335.2 Cyanide, Total (Titrimetric; Spectrophotometric)

EPA Method 335.4 Determination of Total Cyanide by Semi-Automated Colorimetry

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology **D1129** and Guide **D6696**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *mine rock, n*—ore, waste rock or overburden excavated in order to construct an ore-processing site, or recover metals or minerals during mining operations; or coarse processed ore such as heap-leach spoils.

3.2.2 *nominal size, n*—in sampling, for a screen of the standard series, the opening that would pass 95 % of a representative sample.

3.2.3 *refrigeration, n*—storing the sample between its freezing point and 6 °C.

4. Summary of Guide

4.1 Samples are collected in appropriate containers at the sampling site, optionally field preserved, refrigerated, and transported to the laboratory where they are weighed, optionally sub-sampled, the moisture is determined or aqueous mass estimated, and cyanides are extracted prior to analysis. Results of the analysis of the extract are applied to the original solid sample to determine the apparent content of cyanides on the basis of dry weight.

5. Significance and Use

5.1 This guide is intended as a means for obtaining an extract from mine rock and soil samples to measure cyanide content in the aqueous portion of the sample on a dry weight basis. Cyanide is analyzed in mine rock and soil extracts for measurement of cyanide concentration; however, improper sample collection and extraction can result in significant positive or negative bias.

5.2 This guide is designed to mobilize aqueous cyanides present in the solids, so that the resulting extract can be used to assess leachate that could potentially be produced from mine rock or soil.

5.3 This guide is not intended to simulate actual site leaching conditions.

5.4 This guide produces extracts that are amenable to the determination of trace cyanides. When trace cyanides are being determined, it is especially important that precautions be taken

in sample preservation, storage, and handling to avoid possible contamination of the extracts.

5.5 This guide uses a comparative test method and is intended for use as a routine method for monitoring mine rock and soils. It is assumed that all who use this guide will be trained analysts capable of performing it skillfully and safely. It is expected that work will be performed in a properly equipped laboratory applying appropriate quality control practices such as those described in Guide **D3856**.

5.6 This guide identifies proper methods for obtaining mine rock and soil samples for the specific purpose of measuring cyanide concentrations.

6. Interferences

6.1 Many interferences are known for the analysis of cyanide and could effect the results of the analysis of extracts produced using this guide. Refer to Practice **D7365** for proper handling of the extracts during sampling, mitigation of interferences, and preservation prior to cyanide analysis.

6.2 Unless otherwise specified, samples must be extracted as soon as possible after sampling and the extracts must be analyzed within 14 days; however, it is recommended to estimate the actual holding time for each new sample matrix as described in Practice **D4841**. Certain sample matrices may require immediate analysis to avoid cyanide degradation due to interferences. A holding time study is required if there is evidence that cyanide degradation occurs from interferences which would cause the holding time to be less than specified in this guide or Practice **D7365**. Potential interferences for cyanide analytical methods are shown in **Table 1**.

7. Apparatus

7.1 *Agitation Equipment*, of any type that rotates the extraction vessel in an end-over-end fashion at a rate of 30 ± 2 r/min such that the axis of rotation is horizontal and it passes through the center of the bottle (see **Fig. 1**).

7.2 *Drying Pans or Dishes*, for moisture content determinations, 500 g to 8 kg capacity.

7.3 *Drying Oven*—Any thermostatically controlled drying oven capable of maintaining a steady temperature of ± 2 °C in a range of 100 °C to 110 °C.

7.4 *Extraction Vessels*, cylindrical, wide-mouth, of a composition suitable to the nature of the mine rock or soil and cyanide analyses to be performed, constructed of materials that will not allow sorption of the constituents of interest, and sturdy enough to withstand the impact of the falling sample fragments. The size of the container should be selected so that the sample, plus extraction fluid occupy approximately 50–95 % of the container in order to provide good mixing without overfilling. The containers must have water-tight closures of sufficient diameter to fill with the samples.

7.5 *Filtration Device*, pressure or vacuum of a composition suitable to the nature of the analyses to be performed and equipped with a pre-washed glass wool or equivalent filter. An assembly for pre-filtration or a centrifuge may be required if filtration is difficult. (**Warning**—Avoid passing excessive

⁴ Available from United States Environmental Protection Agency (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

TABLE 1 Examples of Potential Interferences if not Mitigated in Standard Cyanide Methods

NOTE 1—Alkyl halides were identified as a potential interference for the distillation methods, however they are not normally found in soil and mine rock.

Method	Description	Measurement	Interferences	Number
Total Cyanide	Automated UV	Colorimetric	Aldehydes Color Fatty Acids Ketones Mercury Nitrate Nitrite Oxidants Sulfides Turbidity Sulfur Compounds Thiocyanate	CFR Kelada-01 D4374
Total Cyanide	Manual Distillation MgCl ₂	Amperometric	Aldehydes Carbonates Ketones Nitrite Nitrate Oxidants Sulfide Sulfur Compounds Thiocyanate	D7284 D2036 , Test Method A
Total Cyanide	Manual Distillation MgCl ₂	Manual or Automated Colorimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Color Turbidity	D2036 , Test Method A Standard Methods 4500-CN C/E EPA Method 335.2 EPA Method 335.4
Total Cyanide	Manual Distillation MgCl ₂	ISE	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sulfide Sulfur Compounds Thiocyanate Color Turbidity	D2036 , Test Method A
Total Cyanide	Manual Distillation MgCl ₂	Titrimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Turbidity	D2036 , Test Method A
Total Cyanide	Micro distillation	Amperometric	Carbonates Oxidants Sulfide	D7284
Total Cyanide	UV/FIA	Amperometric	Carbonates Oxidants Sulfide	D7511

TABLE 1 *Continued*

Method	Description	Measurement	Interferences	Number
Available Cyanide	Flow Injection Ligand Exchange	Amperometric	Carbonates Oxidants Sulfide	D6888 EPA OIA-1677
Cyanide Amenable to Chlorination	Alkaline Chlorination and Manual Distillations	Manual Colorimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sulfide Sulfur Compounds Thiocyanate Color Turbidity Unknowns that cause negative results	D2036 , Test Method B
Weak Acid Dissociable Cyanide	Buffered Distillation	Manual Colorimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Turbidity	D2036 , Test Method C
Weak Acid Dissociable Cyanide	Automated Method	Automated Colorimetric	Aldehydes Color Fatty Acids Ketones Mercury Nitrate Nitrite Oxidants Sulfides Turbidity	D4374
Weak Acid Dissociable Cyanide	Buffered Distillation	ISE	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Turbidity	D2036 , Test Method C
Weak Acid Dissociable Cyanide	Buffered Distillation	Titrimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Turbidity	D2036 , Test Method C

amounts of air through the sample during filtration to prevent liberation of toxic hydrogen cyanide or cyanogen chloride gas.)

7.6 *Laboratory Balance*, capable of weighing to 1.0 g.

TABLE 1 *Continued*

Method	Description	Measurement	Interferences	Number
Weak Acid Dissociable Cyanide		Manual Colorimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrite Nitrate Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Volatile Compounds	D2036, Test Method B
Metal Cyanide Complexes	Ion Chromatography	UV	Carbonate Dissolved Solids Metal Anions Metal Cations Oxidants Photodecomposition	D6994
Free Cyanide	Flow Injection	Amperometric	Carbonate Oxidants Sulfide	D7237
Free Cyanide	Microdiffusion	Colorimetric	Aldehydes Ketones Oxidants Sulfide Sulfur Compounds	D4282

7.7 *pH Meter*, with a readability of 0.01 units and an accuracy of at least ± 0.1 units at 25 °C.

7.8 *Riffle Splitter*—A stationary sampler comprising an even number of equally-sized, adjacent chutes discharging in opposite directions. For use with this practice, there must be a minimum of twelve contained chutes (not bars) with an opening width of at least 3 times the nominal size.

NOTE 1—For riffle splitting finer materials (<3 mm) the 3 times nominal size should be increased to the point where the plugging of chutes is eliminated. For riffle splitting coarser materials (>12.5 mm) it is recommended not to exceed 3½ times nominal size as it is required that the full width of the riffle be used since the accuracy of the split increases with the number of chutes. For free-flowing materials, the 3 times top size may be reduced to 1½ times provided it is ascertained that there is no chute plugging for a particular material.

7.9 *Rotary Sample Divider*—A rotating sampler fed from a single point comprising equally-sized or adjustable proportional collection pans. For use with this guide, there must be a minimum opening width of at least 3 times the nominal size.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this guide. 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.⁵ Other grades

⁵ *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. (<http://uk.vwr.com>)

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 that meets the purity specifications of Type I or Type II water, presented in Specification D1193.

8.3 *Concentrated Sodium Hydroxide Solution (0.4 M)*—In a 1 L volumetric flask, dissolve 16 g NaOH in reagent water and dilute to volume.

8.4 *Dilute Sodium Hydroxide Solution Extraction Fluid (0.04 M)*—In a 1 L volumetric flask, add 100 mL of 0.4 M NaOH in reagent water and dilute to volume.

8.5 *Sample Bottles*—See Section 10.4.1 for further information about sample bottles.

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). Adequate ventilation is necessary when handling cyanide solutions and a fume hood should be utilized whenever possible.

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

10. Sampling

10.1 Obtain a representative sample of the mine rock or soil to be tested by using, where available, ASTM sampling