



Designation: D7572 – 11

Standard Guide for Recovery of Aqueous Cyanides by Extraction from Mine Rock and Soil After Remediation of Process Releases¹

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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 practice 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 practice can also be applied to other analytical methods for cyanide, for example, US EPA Method 335.4.

1.3 The values stated in SI units are to be regarded as standard.

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.*

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 Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water
- D4282 Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion

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.

D4374 Test Methods for Cyanides in Water—Automated Methods for Total Cyanide, Weak Acid Dissociable Cyanide, and Thiocyanate

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

D6888 Test Method for Available Cyanide 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

D6696 Guide for Understanding Cyanide Species

D7237 Test Method for 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

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

2.2 U.S. EPA Methods:³

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 practice, refer to Terminology D1129 and Guide D6696.

³ Available from United States Environmental Protection Agency (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

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*—*n*—*n*—in sampling, for a the opening of the 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, refrigerated and transported to the laboratory where they are weighed, sub-sampled, the moisture is determined 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 concentration of cyanides dissolved in water present in the solids as the result of the process release.

5. Significance and Use

5.1 This practice is intended as a means for obtaining an extract from mine rock and soil samples measure cyanide concentrations. 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, potentially resulting in unnecessary permit violations or undetected cyanide releases into the environment.

5.2 This practice 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 remediated mine rock or soil in the field.

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

5.4 This practice produces extracts that are amenable to the determination of minor (trace) constituents. When minor constituents 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 practice uses a comparative test method and is intended for use as a routine method for monitoring remediated mine rock and soils after process releases and remediation. It is assumed that all who use this practice 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.

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 practice. 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 within 72 hours 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 practice 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 $\pm 2^\circ\text{C}$ in a range of 100 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 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.

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

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this practice. 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.

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 *Annual 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.