Designation: D7572 – 13

StandardGuide for Recovery of Aqueous Cyanides by Extraction from Mine Rock and Soil[1]

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

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 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 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Test Methods for Cyanides in Water—Automated Methods for Total Cyanide, Weak Acid Dissociable Cyanide, and Thiocyanate (Withdrawn 2012)[3]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 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

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

[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.

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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.

*A Summary of Changes section appears at the end of this standard

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 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.

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