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Standard Guide for Selection of ASTM Analytical Methods for Implementation of International Cyanide Management Code Guidance[1]

This standard is issued under the fixed designation D7728; 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 selection of appropriate ASTM standard analytical methods for metallurgical processing sites to conform to International Cyanide Management Code guidance for the analysis of cyanide bearing solutions.

1.2 The analytical methods in this guide are recommended for the sampling preservation and analysis of total cyanide, available cyanide, weak acid dissociable cyanide, and free cyanide by Test Methods D2036, D4282, D4374, D6888, D6994, D7237, D7284, and D7511.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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
D1976 Test Method for Elements in Water by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy
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Test Methods for Cyanides in Water—Automated Methods for Total Cyanide, Weak Acid Dissociable Cyanide, and Thiocyanate (Withdrawn 2012)[3]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
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
D6696 Guide for Understanding Cyanide Species
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
D7511 Test Method for Total Cyanide by Segmented Flow Injection Analysis, In-Line Ultraviolet Digestion and Amperometric Detection
D7572 Guide for Recovery of Aqueous Cyanides by Extraction from Mine Rock and Soil

2.2 *ISO Standard:*[4]

ISO 17690:2015 Water Quality—Determination of Available Free Cyanide (pH 6) Using Flow Injection Analysis (FIA), Gas Diffusion, 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.

[4] Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

2.3 *Other References:*
International Cyanide Management Code[5]
National Water Quality Criteria for Cyanide[6]

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

3.2.2 *titratable cyanide, n*—cyanide measured by titration with silver nitrate to a rhodanine or silver electrode end point.

3.2.3 *weak and dissociable (WAD) cyanide, n*—available cyanide and weak acid dissociable cyanides, excluding determination of iron-, gold-, and cobalt-cyanide complexes.

4. Summary of Guide

4.1 Guidance is provided for selection of the appropriate analytical methods to determine cyanide for to apply to the International Cyanide Management Code guidance for analysis of cyanide in solution.

5. Significance and Use

5.1 This guide is intended as a means for selecting the proper methods for measuring cyanide to conform to the International Cyanide Management Code guidance related to the analysis of cyanide bearing solutions. Cyanide is analyzed in process solutions and in discharges in order to apply code guidance; however, improper sample collection and preservation can result in significant positive or negative bias, potentially resulting in over reporting or under reporting cyanide releases into the environment.

5.2 This guide contains comparative test methods that are intended for use in routine monitoring of cyanide. It is assumed that all who use methods listed in this guide will be trained analysts capable of performing them 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 Multiple interferences could affect the cyanide analytical results using methods enumerated in this guide. Refer to Practice D7365 for proper handling of the solutions during sampling, mitigation of interferences and preservation prior to cyanide analysis.

6.2 Unless otherwise specified, samples 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. Reagents and Materials

7.1 *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. 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.[7]

7.2 *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. Type III or IV water may be used if they do not cause measurable change in the blank or sample.

7.3 *Sample Bottles*—See 9.2 for further information about sample bottles.

8. Hazards

8.1 **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.

8.2 **Warning—**Many of the reagents used in this guide are highly toxic. These reagents and their solutions and extracted solids must be disposed of properly.

9. Sampling

9.1 Obtain a representative sample of the solution to be tested by using, where available, ASTM sampling methods developed for the cyanide processing industry (see Practice D7365).

9.2 Sampling methodology for materials of similar physical form shall be used where no specific methods are available.

9.3 Laboratory personnel and field samplers should follow the industry best practice or acceptable metallurgical methods for sampling and sample preparation of process solutions.

9.3.1 Refer to Guide D4840 for chain-of-custody procedures.

9.4 *Sample Containers:*

9.4.1 Sample containers shall be made of materials that will not contaminate the sample and bottles need to be cleaned thoroughly to remove all extraneous surface contamination

[5] Available from International Cyanide Management Institute, 1400 I Street, NW, Suite 550, Washington, DC 20005, https://www.cyanidecode.org.

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

[7] *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 *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.