



Designation: D5049 – 23

Standard Practice for Screening of Cyanides in Waste¹

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1. Scope

1.1 This practice is applicable to the screening of cyanides in waste liquids, sludges, semisolids, and solids. The following screening methods are included:

	Sections
Method A—Chloramine T	13 – 15
Method B—Prussian Blue	16 – 18
Method C—Cyanide Test Paper	19 – 21
Method D—Gas Detector Tube	22 – 26
Method E—Ion Selective Electrode	27 – 31

1.2 This practice is designed and intended as a compilation of screening tests to complement more sophisticated quantitative analytical techniques that may be used to determine cyanide concentration. This practice provides the user the option and the ability to “screen” waste for potentially hazardous levels of cyanide when the more sophisticated techniques are not available and the total waste composition is unknown.

1.3 The values stated in SI units are to be regarded as the 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.* Specific hazard information is given in Section 8 and 18.5.

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

D1193 Specification for Reagent Water

¹ This practice is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of D34.01 on Monitoring and Characterization.

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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.

D2036 Test Methods for Cyanides in Water
D5681 Terminology for Waste and Waste Management
D6696 Guide for Understanding Cyanide Species
D7365 Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide
D8273 Practice for Determination of Total and Available Cyanide in Solid Waste and Soil after Alkaline Extraction
E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

2.2 *Code of Federal Regulations:*³

21 CFR Part 1308 Schedules of Controlled Substances

3. Terminology

3.1 See Terminology **D5681** for definitions of terms related to waste and waste management.

3.2 See Guide **D6696** for terminology specific to the various forms of cyanide.

3.3 Definitions of Terms Specific to This Standard:

3.3.1 *screening method*—a preliminary, qualitative, or semi-quantitative test, developed from classical qualitative and quantitative techniques, that is designed to efficiently give the user specific information about a waste that will aid in determining waste identification, process compatibility, and safety in handling.

4. Summary of Screening Methods

4.1 *Method A, Chloramine-T Method*—The presence of cyanides can be observed when cyanides react with chloramine-T reagent at pH 8 to form cyanogen chloride. The addition of barbituric acid to cyanogen chloride gives intensive red color. This method indicates cyanides amenable to chlorination.

4.2 *Method B, Prussian Blue Method*—The pH of a sample is adjusted to pH 12 with NaOH and subsequently, solutions of ferrous sulfate and ferric chloride are added. By the addition of concentrated sulfuric acid in the presence of cyanides, a deep blue color (Prussian blue) is produced. This method indicates the presence of free cyanide and many complex cyanides.

³ Code of Federal Regulations available from the Superintendent of Documents, United States Government Printing Office, Washington, DC 20402.

4.3 Method C, Cyanide Test Paper Method—A portion of the sample is acidified in a flask or test tube releasing hydrogen cyanide gas from cyanides. The presence of hydrogen cyanide gas is indicated by a color change in the cyanide screening paper that is held just above the solution. This method indicates dissociable cyanide that could readily evolve hydrogen cyanide gas.

4.4 Method D, Gas Detector Tube Method—A portion of the sample is acidified in a beaker to release cyanide as hydrogen cyanide gas. The gas is funneled through a detector tube, creating a color stain in the tube in proportion to the concentration of cyanide gas in the vapor. A definite color change in the detector tube indicates the presence of cyanide. This method indicates dissociable cyanide that could readily evolve hydrogen cyanide gas.

4.5 Method E, Ion Selective Electrode—The pH of a portion of the sample is adjusted to pH >11 with NaOH. When the sensing element of the ion selective electrode is in contact with a solution containing cyanide ions, an electrode potential develops which corresponds to the cyanide concentration. This method will indicate the presence of free cyanide and weak cyanide complexes with metals which could readily evolve hydrogen cyanide gas. The method is suitable for waste liquids, sludges, semisolids, and solids and is the safest among these listed screening methods because the high pH virtually eliminates the potential of operator exposure to HCN.

5. Significance and Use

5.1 This compilation of methods is intended for use by those in the waste management and related industries to determine the presence of cyanides.

6. Interferences

6.1 Common interferences include oxidizing agents, sulfides, aldehydes, glucose and other sugars, high concentration of carbonate, fatty acids, thiocyanate, and other sulfur containing compounds. Each of these interferences and the extent to which they interfere is dependent upon the technique used. Sulfide and other reduced sulfur containing compounds, aldehydes, and oxidizing agents interfere with all methods by reacting with CN in the sample reducing its concentration. An exception is thiocyanate, which may form in the presence of cyanide and sulfur compounds present in the sample, resulting in a false positive by Methods A and C.

6.2 It is beyond the scope of these methods to describe procedures for overcoming all of the possible interferences that may be encountered. Practice D7365 can be referenced for interferences and their possible elimination.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents 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 (see Practice E200).

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I, II, or III in Specification D1193.

7.3 *Sodium Hydroxide (2.5 N)*—Dissolve 100 g sodium hydroxide (NaOH) in water and dilute to 1 L in a volumetric flask.

7.4 *Cyanide Solution, Standard (50 mg/L)*—Dilute 0.125 g potassium cyanide (KCN) with NaOH solution to 1 L in a volumetric flask.

8. Hazards

NOTE 1—Methods B, C, and D are designed to release highly toxic hydrogen cyanide vapors by means of acidification of alkaline materials. All tests must be performed under a laboratory fume hood. In addition, sufficient personal protective equipment (PPE) must be worn to protect the analyst from inhalation, corrosive materials, and violent reactions.

8.1 Avoid inhalation and skin or eye contact with all hazardous wastes.

9. Sample Collection, Preservation, and Handling

9.1 Samples should be analyzed as soon as possible. Liquid samples can be preserved for up to 14 days by using 2.5 N NaOH to adjust the pH of the sample to a range between 10 to 12.

9.2 Test Methods D2036 and Practice D7365 provide information on additional preservation techniques and elimination of potential interferences.

10. Report

10.1 The report shall include the following information:

- 10.1.1 Sample identification,
- 10.1.2 Sample source,
- 10.1.3 Sampling procedure,
- 10.1.4 Date of test,
- 10.1.5 Sample classification: positive or negative, and
- 10.1.6 Reference to the analytic procedure used.

11. Quality Control

11.1 Method/reagent blanks, duplicates, fortification (spikes) samples (where applicable), and quality control check samples of appropriate matrices should be performed at an action level specified and documented by the laboratory at an appropriate frequency.

11.2 Suitability of this compilation of tests should be determined by each laboratory using appropriate standards for the action level and the matrix of concern.

11.3 A standard shall be run with each batch of samples.

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 *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.

12. Precision and Bias

12.1 No statement is made about either the precision or bias of this practice since the result merely states whether there is conformance to the criteria for success specified in the procedure.

Screening Method A—Chloramine T

13. Interferences

13.1 The presence of formaldehyde interferes with this screening method.

13.2 The presence of high levels of reducing agents interferes with this screening method.

13.3 Thiocyanate (SCN^-) reacts with chloramine-T, creating a positive interference.

14. Reagents and Materials

14.1 *Chloramine-T* (100 g/L)—Dissolve 1 g chloramine-T in 10 mL water.

14.2 *Sodium Hydroxide, Standard Solution* (0.1 M)—Dissolve 4.0 g sodium hydroxide (NaOH) in water and dilute to 1 L in a volumetric flask.

14.3 *Potassium Dihydrogen Phosphate* (0.1 M)—Dissolve 13.6 g potassium dihydrogen phosphate (KH_2PO_4) in water and dilute to 1 L in a volumetric flask.

14.4 *Phosphate Buffer pH 8*—Mix 46.1 mL 0.1 M NaOH and 50 mL of 0.1 M KH_2PO_4 .

14.5 *Pyridine-Barbituric Acid Solution*—Mix 1.5 g barbituric acid with 5 mL of water. Add 7.5 mL of pyridine. Add 1.5 mL of concentrated hydrochloric acid (HCl) and dilute to 25 mL with water. The solution will completely dissolve upon dilution with water.

NOTE 2—Barbituric acid is classified as a Schedule III drug by the U.S. Department of Justice, Drug Enforcement Administration, Diversion Control Division.⁵ This reagent requires locked storage and record keeping.

14.6 White ceramic spot plate or disposable beaker.

14.7 Stirring rod.

14.8 Disposable transfer pipettes.

14.9 pH indicator strip, with a range of 0 to 14.

15. Procedure

15.1 Slurry solid samples (1 + 10) with water.

15.2 Place 0.25 mL (approximately five drops) of sample or extract from solid slurry in cavity of a spot plate or in a disposable beaker.

15.3 Add one drop of phosphate buffer and mix.

15.4 Check the pH with indicator paper and continue drop by drop the addition of buffer solution until pH 8 is obtained.

15.5 Add four drops of chloramine-T reagent and mix.

15.6 Add four drops of pyridine-barbituric acid reagent and mix again.

15.7 Observe color change. Presence of cyanides is indicated by red color.

Screening Method B—Prussian Blue

16. Interferences

16.1 See Practice [D7365](#) for listings of cyanide test methods.

16.2 Samples containing copper or nickel or having blue or green color may show positive interferences. These samples should be analyzed for available or total cyanide using one of the methods listed in Practice [D7365](#).

16.3 Solid samples suspected of containing high concentrations of cyanide, Prussian Blue, or other metal-metal cyanide complexes should be extracted using Practice [D8273](#).

17. Reagents and Materials

17.1 *Sodium Hydroxide* (2.5 N)—Dissolve 100 g sodium hydroxide (NaOH) in water and dilute to 1 L in a volumetric flask.

17.2 *Ferrous Sulfate Heptahydrate* (100 g/L)—Dissolve 100 g of ferrous sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) in water and dilute to 1 L in a volumetric flask. Preserve the solution by adjusting the pH to 2 with concentrated sulfuric acid (H_2SO_4).

17.3 *Ferric Chloride Hexahydrate* (50 g/L)—Dissolve 50 g of ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) in water and dilute to 1 L in a volumetric flask.

17.4 Sulfuric acid (concentrated H_2SO_4).

17.5 Glass bottle or jar.

17.6 pH indicator strip with a range of 0 to 14.

18. Procedure

18.1 Place approximately 1 to 5 g of sample into beaker.

18.2 Adjust pH to 12 to 13 by the addition of 2.5 N NaOH (5 to 10 mL may be required). Slurry solid samples by adding approximately 1 + 10 water solution before checking pH. Check with pH paper.

18.3 Add 5 to 10 mL FeSO_4 (100 g/L) solution and mix.

18.4 Add 5 to 10 mL of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (50 g/L) and mix.

18.5 Add the concentrated sulfuric acid slowly to adjust the pH below 1.0 (check with pH indicator paper).

NOTE 3—Mixing concentrated sulfuric acid with an alkaline solution can result in a violent reaction with the generation of heat. In addition, hydrogen cyanide vapors are generated upon acidification of alkaline materials containing cyanides. All tests must be performed under a laboratory fume hood, and personal protective equipment (PPE) must be worn to protect the analyst from inhalation, corrosive materials, and violent reactions.

18.6 A light green-blue color indicates the presence of cyanide and shall be recorded as positive.

⁵ Title 21 United States Code (USC) Controlled Substances Act, Subchapter I—Control and Enforcement, Part B—Authority to Control; Standards and Schedules, 812. Schedules of controlled substances.