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Standard Test Method for the Screening of Cyanides in Waste[1]

This standard is issued under the fixed designation D 5049; 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 test method is applicable to the screening of cyanides in waste liquids, sludges, semisolids, and solids. The following test methods are included:

	Sections
Test Method A—Chloramine T	12 through 14
Test Method B—Prussian Blue	15 through 17
Test Method C—Cyantesmo Paper	18 through 20
Test Method D—Gas Detector Tube	21 through 25

1.2 This test method is designed and intended as a preliminary test to complement the more sophisticated quantitative analytical techniques that may be used to determine cyanide concentration. This test method offers, to 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 *This standard does not purport to address all of the safety problems 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.* Specific hazard information is given in Section 8 and 18.5.

2. Referenced Documents

2.1 *ASTM Standards:*
D 1193 Specification of Reagent Water[2]
D 2036 Test Methods for Cyanides in Water[3]
E 200 Practice for Preparation, Standardization, and Storage of Standard Solutions for Chemical Analysis[4]
2.2 *Code of Federal Regulations:*
21 CFR Part 1308[5]

3. Terminology

3.1 *Description of Term Specific to This Standard:*
3.1.1 *screening method*—a preliminary, qualitative, or semiquantitative 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 Test Methods

4.1 *Test 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 test method will indicate cyanides amenable to chlorination.

4.2 *Test 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 test method will indicate the presence of free cyanide and many of the complex cyanides.

4.3 *Test Method C, Cyantesmo Paper Method*—A portion of the sample is acidified in a flask or test tube releasing the simple 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 test method will indicate dissociable cyanide that could readily evolve hydrogen cyanide gas.

4.4 *Test 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 a positive presence of cyanide. This test method will indicate dissociable cyanide that could readily evolve hydrogen cyanide gas.

5. Significance and Use

5.1 This test method is intended for use by those in the waste management industries to determine the presence of potentially hazardous cyanides.

6. Interferences

6.1 Common interferences in the analysis for cyanide include oxidizing agents, sulfides, aldehydes, glucose, and other sugars, high concentration of carbonate, fatty acids, thiocyanate, and other sulfur containing compounds.

6.2 It is beyond the scope of these test methods to describe procedures for overcoming all of the possible interferences that may be encountered. Test Methods D 2036 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

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[1] This test method is under the jurisdiction of ASTM Committee D-34 on Waste Disposal and is the direct responsibility of Subcommitee D34.02 on Physical and Chemical Characterization.
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[2] *Annual Book of ASTM Standards*, Vol 11.01.
[3] *Annual Book of ASTM Standards*, Vol 11.02.
[4] *Annual Book of ASTM Standards*, Vol 15.05.
[5] Code of Federal Regulations available from the Superintendent of Documents, United States Government Printing Office, Washington, DC 20402.

Society where such specifications are available.[6] 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 E 200).

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III in Specification D 1193.

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: Warning—Test 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 safety gear must be worn to protect the analyst from corrosive materials and possible violent reactions.

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

9. Sample Collection, Preservation, and Handling

9.1 Samples should be analyzed as soon as possible or liquid samples preserved by using 2.5 *N* NaOH to adjust the pH of the sample to a range between 10 to 13, if the pH is >10.

9.2 Test Methods D 2036 can be referenced for additional preservation techniques.

10. Report

10.1 The report shall include the following information:

10.1.1 Sample identification,

10.1.2 Date of test,

10.1.3 Sample classification: positive or negative, and

10.1.4 Reference to the procedure used.

11. Quality Control

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

11.2 Suitability of this test method 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.

12. Precision and Bias

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

Test Method A—Chloramine T

13. Interferences

13.1 The presence of formaldehyde interferes with this test method.

13.2 The presence of high levels of reducing agents may interfere with this test 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*—Dissolve 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.

NOTE 2—Barbituric acid is now classified as a Schedule III drug by the Federal Drug Administration.[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 pipetes.

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 5 drops) of sample or extract from solid slurry in cavity of a spot plate or in a disposable beaker.

15.3 Add 1 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 4 drops of chloramine-T reagent and mix.

15.6 Add 4 drops of pyridine-barbituric acid reagent and mix again.

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

Test Method B—Prussian Blue

16. Interferences

16.1 Aqueous samples that are extremely alkaline (ph > 13) and contain cyanide may not show the Prussian blue color. Spike the sample with a 50 ppm standard. If the spiked sample does not show the presence of cyanides, a total cyanide distillation may be applied (Reference Test Methods D 2036).

16.2 Samples containing copper or nickel or having blue or green color may show positive interferences. These

[6] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc. New York, NY and the "United States Pharmacopeia."