



Designation: D7295 – 18

Standard Practice for Sampling Combustion Effluents and Other Stationary Sources for the Subsequent Determination of Hydrogen Cyanide¹

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1. Scope

1.1 This practice is used to collect samples for the determination of gaseous hydrogen cyanide (HCN) from any combustion device or atmosphere where cyanide may be present. While primarily designed for the measurement of gas phase HCN, the sample collection described in this practice also includes cyanide ion (CN⁻) absorbed particles that may be present in the sampling atmosphere.

1.1.1 Samples can be collected from a closed chamber such as the NBS smoke box described in Test Method E662 provided it is equipped with sampling ports.

1.1.2 Open chambers such as industrial work areas or large scale fires can be monitored for HCN with this practice.

1.1.3 The HCN emissions of a flow through system can be determined by sampling from its discharge stack. Examples of such systems include large scale manufacturing applications and the cone calorimeter described in Test Method E1354.

1.2 This practice can be used to monitor HCN levels in lab scale fire smoke effluents in order to estimate toxicity of gases produced from burning materials. See Guide E800.

1.3 The concentration range of hydrogen cyanide will be dependent on the volume of gas sampled, the volume of sodium hydroxide solution placed in the impinger during sampling, and the analytical method used to measure cyanide. For example, the lower limit of detection would be 0.002-mg/m³ when 0.1-m³ of combustion effluent is collected into 100-mL sodium hydroxide solution based on a detection limit of 0.002 mg/L cyanide in the impinger solution when using the flow injection analysis (FIA) system described in Test Method D6888.

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 This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.6 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
- D1356 Terminology Relating to Sampling and Analysis of Atmospheres
- D2036 Test Methods for Cyanides in Water
- D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
- D3154 Test Method for Average Velocity in a Duct (Pitot Tube Method)
- D3614 Guide for Laboratories Engaged in Sampling and Analysis of Atmospheres and Emissions
- D3685/D3685M Test Methods for Sampling and Determination of Particulate Matter in Stack Gases
- D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
- D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps
- D6696 Guide for Understanding Cyanide Species
- D6888 Test Method for Available Cyanides with Ligand Displacement and Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection
- D7365 Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide

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.

[E337 Test Method for Measuring Humidity with a Psychrometer \(the Measurement of Wet- and Dry-Bulb Temperatures\)](#)

[E662 Test Method for Specific Optical Density of Smoke Generated by Solid Materials](#)

[E800 Guide for Measurement of Gases Present or Generated During Fires](#)

[E1354 Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter](#)

[E2877 Guide for Digital Contact Thermometers](#)

3. Terminology

3.1 Refer to Terminology [D1356](#) for terminology related to sampling and analysis of atmospheres.

3.2 For definitions of terms related to cyanide testing, refer to Guide [D6696](#) and Test Method [D6888](#).

3.3 For definitions of terms related to the measurement of gases present or generated during fires, refer to Guide [E800](#).

3.4 For additional information related to sampling and analysis of atmospheres, see Guide [D3614](#).

4. Summary of Practice

4.1 A known volume of gaseous sample is bubbled through an impinger containing 0.1-M sodium hydroxide solution utilizing a calibrated pumping system. Particulates that may pass through the impinger are captured on a glass fiber filter then recombined back into the impinger solution prior to analysis. Because HCN is soluble in aqueous solutions, it may be present in water droplets formed from either a wet scrubber system or condensation in the stack. Because of this, samples that contain moist air should be sampled near isokinetic conditions.

4.2 During the sampling process, hydrogen cyanide (HCN) is converted to cyanide ion (CN⁻) in the sodium hydroxide solution. The CN⁻ is analyzed with a flow injection analysis (FIA) system described in Test Method [D6888](#) or with a suitable analytical method such as ion chromatography described in Test Methods [D2036](#).

4.2.1 Colorimetric and ion selective electrode methods, also described in Test Methods [D2036](#) can be used for screening purposes.

4.3 The concentration of HCN in the atmosphere or stack is calculated as described in [9.6](#).

5. Significance and Use

5.1 Hydrogen cyanide is highly toxic. In relatively low quantities, hydrogen cyanide can cause asphyxia and death.

5.2 The National Fire Protection Association has assigned a flammability rating of 4 (severe fire hazard) to hydrogen cyanide.

6. Apparatus

6.1 Sample Collection Train

6.1.1 *Constant Flow Pumping System*—Pumps used to collect samples should be able to accurately pump from 0.1-L/min

to 15-L/min or at the desired flow rate. Personal industrial hygiene sampling pumps or high volume sampling pumps may be utilized. More than one sample can be collected with a single pump using a manifold with flow control valves. The system should be calibrated as described in Practice [D5337](#) or using a suitable commercially available calibrator.

6.1.2 *Impinger Sample Collection Vessels*—Standard mini or midjet impingers with standard tip. Greenberg-Smith impingers equipped with a standard tip with 500-mL capacity may be used when larger flow rates are desired. Impinger connections should consist of leak free ground glass joints to avoid the loss of HCN during sampling and should be free of any silicone grease. The use of a single impinger has shown to yield an average of 97 % efficiency in combustion effluents; however, higher efficiencies are possible using additional impingers in series. Without prior knowledge of the atmosphere, it is recommended to use a backup impinger to determine if breakthrough occurs. Test each impinger for cyanide as a separate sample in order to demonstrate capture efficiency; mathematically combine the results by adding the observed concentrations.

6.1.3 *Sample Collection Tubing and Probes*—Collection tubing and probes should be constructed of inert material such as PTFE or PTFE-lined stainless steel. Vinyl tubing can be used in the sample train where flexible tubing is necessary, but the aforementioned are preferred. The sample lines should be heated at $120 \pm 5^\circ\text{C}$ when sampling stack emissions in moist air in order to prevent condensate from forming in the sample train.

6.1.4 *Glass Fiber Filters*—Type A/E, 1 μm binder free borosilicate glass fiber filter and cassette assembly cartridge for air sampling applications. The filters are used between the impingers and pumping system to capture fine particulates that may pass through the impingers and to protect the pumping system. It is recommended to use 13-mm filters for mini-impingers and 47-mm filters for Greenberg-Smith impingers.

6.1.5 *Duplicate and Matrix Spike Sample Trains*—It is recommended to setup duplicate impingers to evaluate precision. In addition, a second set of impingers can be fortified with a known amount of cyanide (see [8.11](#)) to evaluate recovery. [Fig. 1](#) shows an example of such a system.

6.1.6 *Thermometer*—Measure the temperature of the stack or atmosphere with a thermometer as described in Guide [E2877](#) capable of accurately reading within $\pm 0.1^\circ\text{C}$.

7. Interferences

7.1 Sulfide, aldehydes, and oxidizing agents in the atmosphere are possible interferences.

7.2 Prior to conducting sampling and analysis, review [9.4](#) in order to avoid cyanide degradation or inaccurate results.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the American

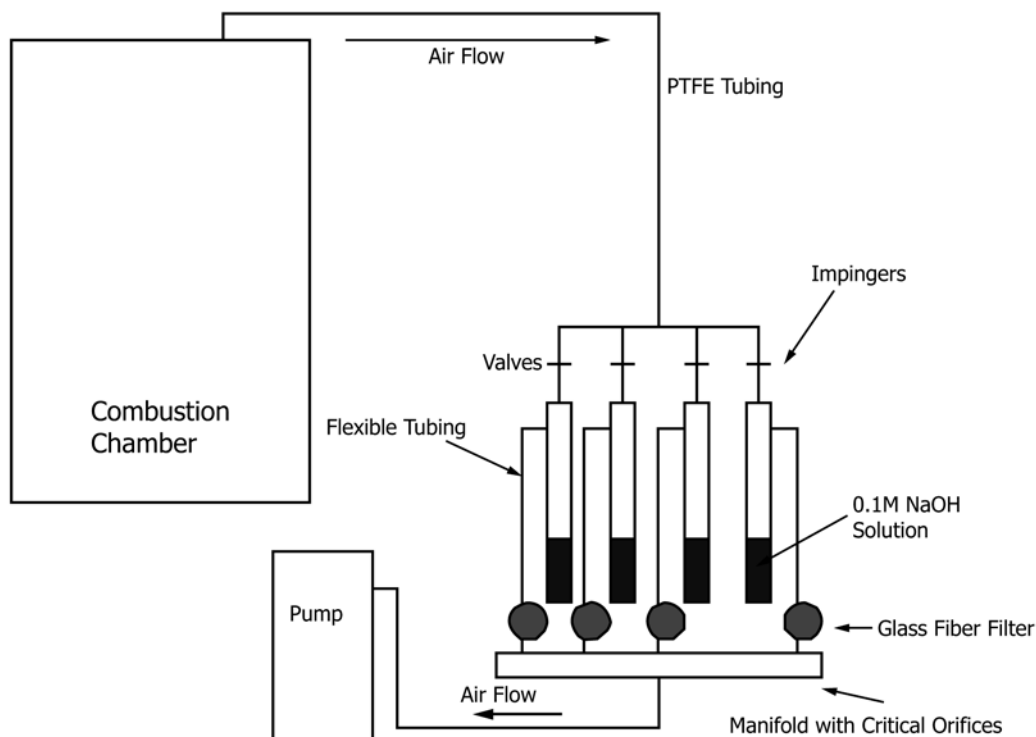


FIG. 1 Example of Sample Train (Closed Chamber Sampling, Non-Isokinetic)

Chemical Society, where such specifications are available³. 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I, II, or III of Specification D1193.

8.3 *Impinger Solution (0.10 M NaOH)*—In a 1-L volumetric flask, dissolve 4.0 g NaOH in approximately 800-mL laboratory water and dilute to 1L.

8.4 *Ethylenediamine (EDA) Solution*—Weigh 3.5 g EDA into a 100-mL volumetric flask and dilute to volume with water.

8.5 *Cadmium Chloride*, granular, powdered.

8.6 *Lead Acetate Test Strips*, used to test for the presence of sulfide in the impinger solutions.

8.7 *Leur Lock Syringe Filter*—0.45 μm filter attached to syringe

8.8 *KI Starch Paper Test Strips*, used to indicate the presence of oxidizing agents

8.9 *Sodium Arsenite*— NaAsO_2 is used to neutralize oxidizing agents

8.10 *pH Indicator Strips*, wide range pH paper.

8.11 *Cyanide Matrix Spike Solution (1000 $\mu\text{g}/\text{mL}$ CN)*—Dissolve 2.51 g of KCN and 2.0 g of NaOH in 1 L of water. Standardize with silver nitrate solution as described in Test Methods D2036, paragraph 16.2. Store the solution under refrigeration and check concentration approximately every six months and correct if necessary. Commercially prepared solutions are also available and can be used for spiking purposes. (**Warning**—Because of the toxicity of cyanide, great care must be exercised in its handling. Acidification of cyanide solutions produces toxic hydrocyanic acid (HCN). All manipulations must be done in the hood so that any HCN gas that might escape is safely vented.)

9. Procedure

9.1 *Stack Evaluation (Flow Through Systems)*:

9.1.1 Determine the velocity of the stack as described in Test Method D3154 or U.S. EPA Method 2.⁴

9.1.2 Because HCN is soluble in aqueous solutions, it may be present in water droplets formed from either a wet scrubber system or condensation in the stack. Because of this, samples that contain moist air should be sampled near isokinetic conditions. To determine if the moisture in the stack is saturated, test with the wet bulb/dry bulb procedure described in Test Method E337 or estimate the moisture content by U.S. EPA Method 4.⁵

9.1.3 If isokinetic sampling is required, see Test Methods D3685/D3685M or U.S. EPA Method 5⁶ to determine the probe

³ *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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia*.

⁴ Method 2, Velocity—S-Type Pitot, U.S. Environmental Protection Agency.

⁵ Method 4, Moisture Content, U.S. Environmental Protection Agency.

⁶ Method 5, Particulate Matter, U.S. Environmental Protection Agency.