

**Designation:** D7295 - 11 D7295 - 18

# Standard Practice for Sampling and Determination of Hydrogen Cyanide (HCN) in Combustion Effluents and Other Stationary Sources Combustion Effluents and Other Stationary Sources for the Subsequent Determination of Hydrogen Cyanide<sup>1</sup>

This standard is issued under the fixed designation D7295; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

- 1.1 This practice is used to <u>determine the concentration collect samples for the determination</u> 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<sup>-</sup>) 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.
- 1.5 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 safety, health, and health 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:<sup>2</sup>

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

<sup>&</sup>lt;sup>1</sup> 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.

Current edition approved March 1, 2011May 1, 2018. Published March 2011May 2018. Originally approved in 2006. Last previous edition approved in 20062011 as D7295-06:D7295-11. DOI: 10.1520/D7295-11.10.1520/D7295-18.

<sup>&</sup>lt;sup>2</sup> 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.



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

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<sup>-</sup>) in the sodium hydroxide solution. The CN<sup>-</sup> 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.

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  - 4.3 The concentration of HCN in the atmosphere or stack is calculated as described in 9.6.003 dda c/astm-d7295-18

# 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 midget 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 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  $120 \pm 5^{\circ}$ C when sampling stack emissions in moist air in order to prevent condensate from forming in the sample train.



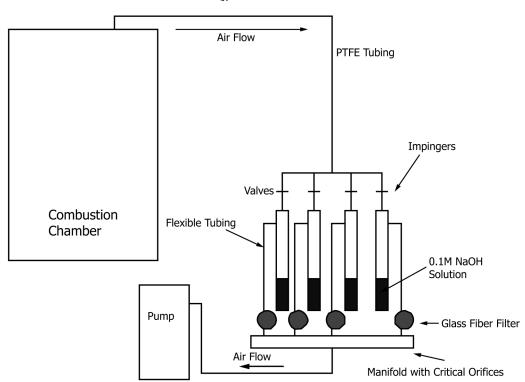


FIG. 1 Example of Sample Train (Closed Chamber Sampling, Non-Isokinetic)

- 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.

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- 6.1.6 *Thermometer*—Measure the temperature of the stack or atmosphere with a mercury thermometer or an equivalent digital thermometer as described in Guide E2877 capable of accurately reading within  $\pm 0.1$ °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 Chemical Society, where such specifications are available<sup>3</sup>. 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.

<sup>&</sup>lt;sup>3</sup> 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.