



Designation: ~~D3267~~—~~12~~ D3267 – 20

Standard Test Method for Separation and Collection of Particulate and Water-Soluble Gaseous Fluorides in the Atmosphere (Filter and Impinger Method)¹

This standard is issued under the fixed designation D3267; 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 provides a procedure for separation and collection of particulate and water-soluble gaseous fluorides from the atmosphere on a chemically treated filter and in an impinger of ~~particulate and water-soluble gaseous fluoride in the atmosphere.~~ impinger. The sampling rate may vary from 30 L/min ~~(1.0 ft³/min)~~ (1.0 ft³/min) to 1515 L/min ~~(0.5 ft³/min)~~ (0.5 ft³/min) for longer sampling periods depending on the atmospheric fluoride concentration. This test method is not intended to be applied to gaseous fluorine compounds that are not water-soluble ~~water-soluble~~.

1.2 There are several limitations of the test method:

1.2.1 Although the acid-treated, medium retentive, prefilter has been shown to allow passage of HF, it ~~will restrict~~ restricts passage of particulate matter only as small as about 1 μm . Thus, smaller particulate matter may pass through the filter and be collected in or pass through the impingers.

1.2.2 The maximum sample volume to be taken using this test method prior to changing the acid-treated prefilter is recommended as 12 m³. This recommendation is made to minimize any effects due to particulate matter build-up.

1.3 The values stated in SI units are to be regarded as ~~the standard.~~ the standard. The ~~inch-pound units values given in parentheses are for information only.~~ inch-pound units values given in parentheses are for information only after SI units are provided for information only and are not considered 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 ~~safety~~, health, and environmental practices ~~health~~ and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see 7.27.4.3 and 7.4.6.

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

D1071 Test Methods for Volumetric Measurement of Gaseous Fuel Samples

¹ This test method 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 ~~April 1, 2012~~ Dec. 1, 2020. Published ~~May 2012~~ January 2021. Originally approved in 1973. Last previous edition approved in ~~2005~~ 2012 as ~~D3267-91 (2005)~~ D3267-12. DOI: ~~10.1520/D3267-12~~ 10.1520/D3267-20.

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~~ standard's Document Summary page on the ASTM website.

- D1193 Specification for Reagent Water
- D1356 Terminology Relating to Sampling and Analysis of Atmospheres
- D1357 Practice for Planning the Sampling of the Ambient Atmosphere
- D2009 Practice for Collection by Filtration and Determination of Mass, Number, and Optical Sizing of Atmospheric Particulates (Withdrawn 1995)³
- D3268 Test Method for Separation and Collection of Particulate and Gaseous Fluorides in the Atmosphere (Sodium Bicarbonate-Coated Glass Tube and Particulate Filter Method)
- D3269 Test Methods for Analysis for Fluoride Content of the Atmosphere and Plant Tissues (Manual Procedures) (Withdrawn 2010)³
- D3270 Test Methods for Analysis for Fluoride Content of the Atmosphere and Plant Tissues (Semiautomated Method)
- E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)

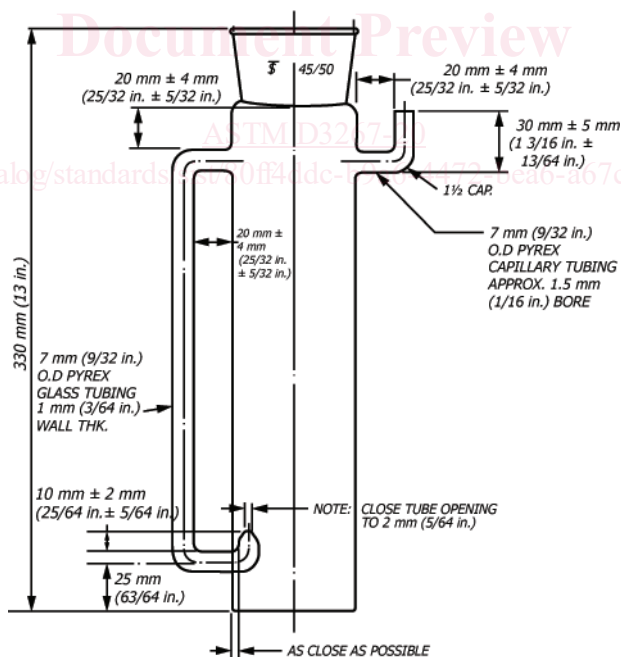
3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1356.

4. Summary of Test Method

4.1 Air is drawn through an air-inlet tube (see Practice D1357) and is first passed through an acid-treated prefilter to remove particulate matter that may contain fluoride and then through an impinger to remove water-soluble fluorides.

4.2 The prefilter and impinger solution are removed from the sampling system at the end of the selected sampling period and taken to an analytical work area and analyzed by either a potentiometric or photometric method (1-4).⁴ See Test Methods D3269 and/or D3270.

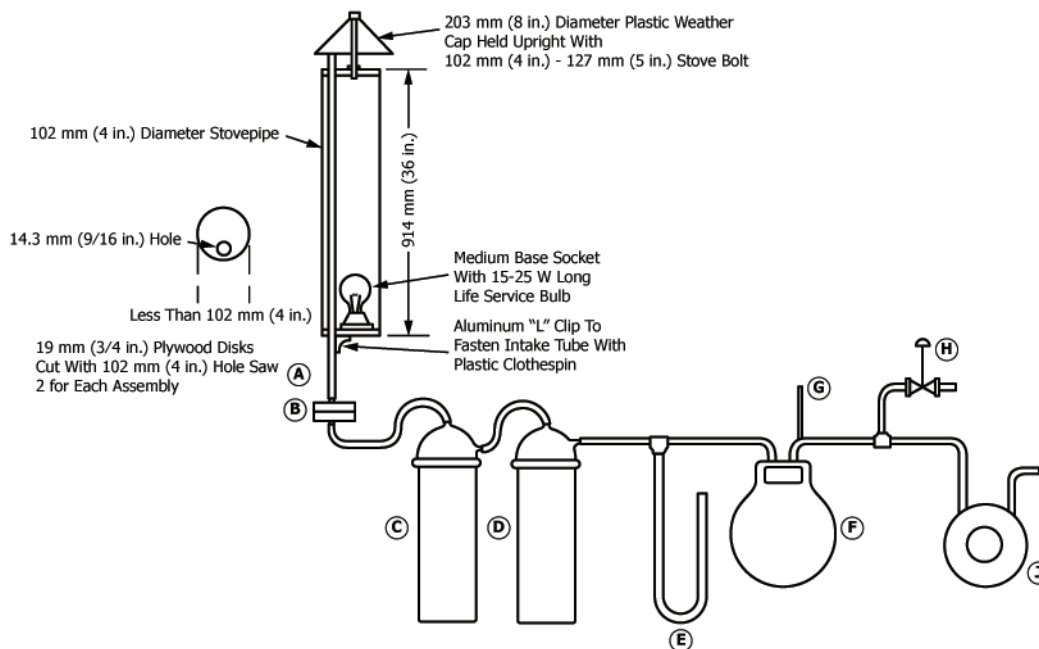


SI Equivalents											
in.	1/16	3/64	5/64	5/32	13/64	9/32	25/64	25/32	63/64	1 1/16	13
mm	1.5	1	2	4	5	7	10	20	25	30	330

FIG. 1 Modified Standard Impinger

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ The boldface numbers in parentheses refer to the references at the end of this standard.



- A Sampling tube
- B Filter holder
- C Standard impinger
- D Deminster (dry-impinger)
- E Manometer
- F Meter
- G Thermometer
- H Metering valve
- J Vacuum pump

SI Equivalents						
in.	9/16	3/4	4	5	8	36
mm	14	19	102	127	203	914

FIG. 2 Diagram of a Sample Train for Short-Term Sampling of Atmospheric Fluorides

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5. Significance and Use

5.1 Sources of particulate and water-soluble gaseous fluorides in the atmosphere include the fertilizers, aluminum reduction plants, phosphate processors, steel mills, coal burning operations, brick and tile manufacturers, and various less significant sources (5). Gaseous fluorides can cause adverse effects when ingested or inhaled by animals or humans, or absorbed by plants. The procedures documented in this test method provide a means of determining of particulate and water soluble gaseous fluoride in atmospheres.

5.2 This test method provides a means of separation and collection of particulate and water soluble gaseous fluoride and provides samples that are convenient to analyze.

6. Interferences

6.1 Particulate metallic salts, such as those of aluminum, iron, calcium, magnesium or rare-earth elements, may react with and remove some or all of the water-soluble gaseous fluoride on the prefilter. If interfering quantities of such particulate metallic salts are present, the use of Test Method D3268 is recommended because the acidic fluoride gases are collected prior to the filter.

6.2 Aluminum or certain other metals or phosphates can interfere with subsequent analyses by photometric or electrometric methods. These potential interferences are discussed in Test Methods D3269 and D3270.

7. Apparatus

7.1 *Standard Impinger*; (see Fig. 2E)-(C) for sampling covering periods up to 3 h. When sampling is initiated, the collecting impinger shall contain 75 to 150 mL of reagent water (see 8.2).

7.2 Impinger, Modified, Standard, Smith-Greenburg (Fig. 1). For sampling periods from 3 to 72 h, see Fig. 1 and Fig. 3C. (C). **Warning**—If a different size impinger is used, the recovery of the sample should be determined.)

NOTE 1—If a different size impinger is used, the recovery of the sample should be determined.

7.3 Water Level Control Device, for use with long-term sampling train (see Fig. 3).

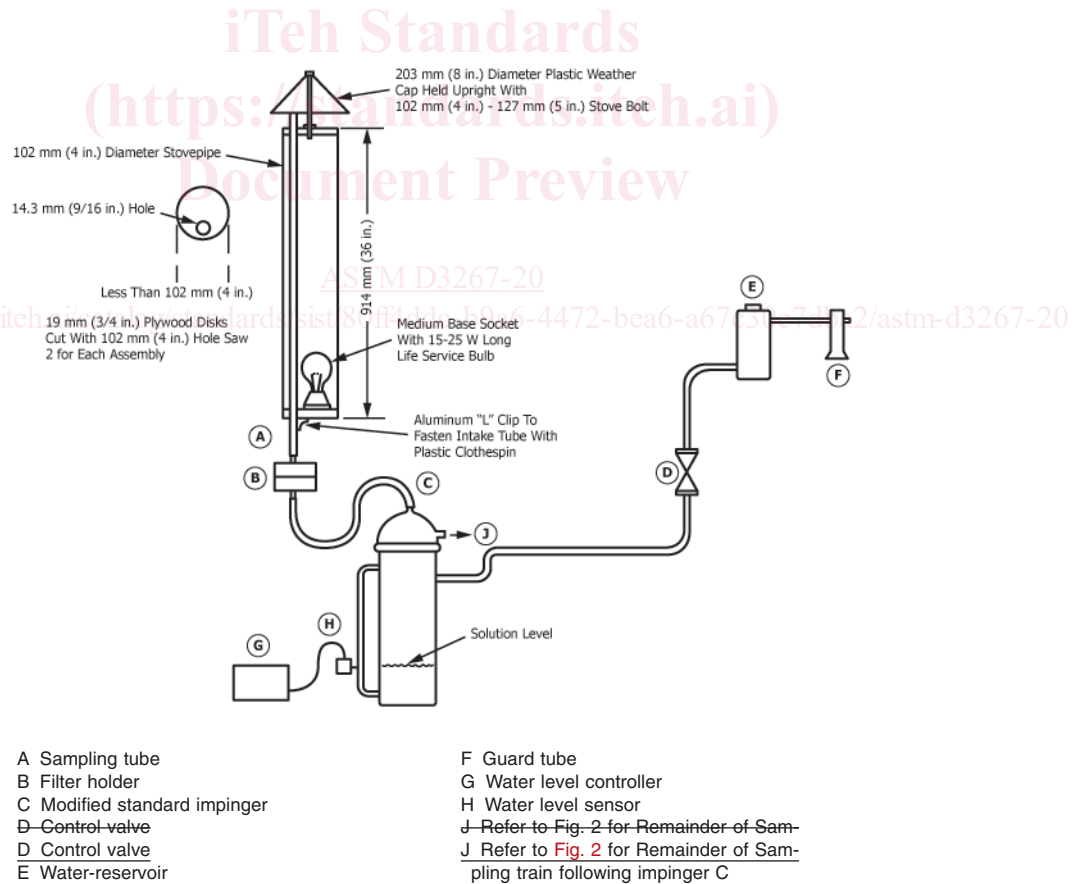
7.3.1 Liquid Level Controller. (See Fig. 3G (G and H).)

7.3.2 Control Valve, solenoid-activated. (See Fig. 3D (D).)

7.3.3 Water Reservoir, equipped with a guard tube containing an alkaline reagent to prevent fluoride contamination. (See Fig. 3E (E and F).)

7.4 Sampling Equipment (see Fig. 1, Fig. 2, and Fig. 3 for dimensions and details).

7.4.1 Sampling Tube (see Fig. 2A (A)), constructed of a ~~1225 mm (4 ft)~~ 1225 mm (4 ft) length of a material that is inert to the atmosphere being sampled. (See Note 4.2.)



SI Equivalents

in.	9/16	3/4	4	5	8	36
mm	14	19	102	127	203	914

FIG. 3 Diagram of a Modified Standard Impinger for Long-Term Sampling of Atmospheric Fluorides

NOTE 2—Stainless steel coated with plastic material is preferred because of its rigidity and permanence. Plastic materials which have been shown to have demonstrated a low absorbance for gaseous fluorides ~~are include~~ polytetrafluoroethylene, polyethylene, polypropylene, and polychlorotrifluoroethylene.

~~However, The user is cautioned that high air velocities through some types of plastic tubing may create a static charge and cause deposition of particulate matter. Consequently, if plastic is selected for use, it should be tested to demonstrate that this effect does not occur to a significant degree. is insignificant with respect to the measurement's intended use.~~

7.4.2 *Filter Holder*, stainless steel or polypropylene, ~~(polypropylene (see Fig. 2B), (B)),~~ used to hold the acid-treated prefilter. (See Note 23.)

NOTE 3—~~Note that filter~~ Filter changes are recommended after 12 m³ are sampled.

7.4.3 *Mercury Manometer* ~~((see Fig. 2E), (E)),~~ for the determination of pressure in the metered sampling line. A calibrated vacuum gage/gauge is also satisfactory. **(Warning—**Mercury has been designated by many regulatory agencies as a hazardous substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.)

7.4.4 *Dry Test Meter* ~~((see Fig. 2F), (F)),~~ for measuring the total volume sampled.

7.4.5 *Thermometer*—Temperature measuring devices such as RTDs (~~Resistance Temperature Devices~~), thermistors (~~resistance temperature devices~~), thermistors, and organic liquid-in-glass thermometers meeting the requirements of specific applications may be used.

7.4.6 *Control Valve* ~~((see Fig. 2H), (H)),~~ used to adjust the sample rate through a dry test meter. (See 7.4.4.) **(Exercise Warning—**Exercise care to see that all sampling apparatus is clean and free of contamination. Clean with a detergent low in fluoride and phosphate, followed by thorough rinsing with water (see 8.2.) care to ensure that all sampling apparatus components are clean and free of contamination. Clean sampling equipment with a detergent low in fluoride and phosphate, followed by thorough rinsing with water (see 8.2).)

7.4.7 *Vacuum Pump* (see Fig. 2J), (J)), capable of a 30 L/min (1 ft³/min) sampling rate and capable of continuous operation over the maximum sample period selected under the environmental conditions imposed by the sampling location.

<https://standards.iteh.ai/catalog/standards/sist/80ff4ddc-b9a6-4472-bea6-a67c30e7dba2/astm-d3267-20>

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵

8.2 *Purity of Water*—Water shall be Type II reagent water conforming to Specification D1193. Additionally, test the water with a specific ion electrode or electrode, by concentration and photometric analysis—analysis, or other suitable analytic method to demonstrate that it contains less than 0.005 µg/mL of fluoride.

8.3 *Alcoholic Citric Acid Solution* (0.1 N)—Dissolve 4.203 g 4.203 g of citric acid monohydrate (C₆H₈O₇·H₂O) in 200 mL of 95 % ethyl alcohol:alcohol (C₂H₅OH).

8.4 Chemically treated filters are used as the prefilter. (See 10.1.)

8.5 *Sodium Hydroxide Solution* (5N)—(5.0 N)—Dissolve 200 g of sodium hydroxide (NaOH) in 250 mL of water in a 1 L volumetric flask, swirl to mix, cool, and dilute to 1000 mL with water. Mix thoroughly.

⁵ *Reagent Chemicals, American Chemical Society Specifications, ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

8.6 *Sodium Hydroxide Solution (1.0 N)*—Dissolve 40.0 g of NaOH in 250 mL of reagent water in a ~~1000-mL~~ 1000 mL volumetric flask. Swirl to mix, cool, and dilute to 1000 mL with reagent water. Mix thoroughly.

8.7 *Sulfuric Acid (1.0 N)*—Add 28.0 mL of concentrated H₂SO₄ (sp gr 1.84) to 250 mL of reagent water in a ~~1000-mL~~ 1000 mL volumetric flask. Swirl to mix, cool, and dilute to 1000 mL with reagent water. Mix thoroughly.

8.8 *Total Ionic Strength Adjustment Buffer (TISAB)*—Add 57 mL of glacial acetic acid, ~~acid (CH₃COOH)~~, 58 g of sodium chloride (~~NaCl~~)(NaCl), and 4.0 g of CDTA ((1,2-cyclohexylene-dinitrilo)tetraacetic acid) to 500 mL of distilled water. Stir and add 5 N NaOH solution (see 8.5) slowly until the pH is between 5.0 and 5.5. Cool and dilute to 1 L.

8.9 *TISAB (1:1)*—Dilute the full strength TISAB (see 8.8) 1:1 with ~~an equal amount of~~ water.

9. Calibration

9.1 Calibrate the dry test meter (see 7.4.4) in accordance with Test Methods **D1071**.

10. Procedure

10.1 *Treatment of Prefilter:*

10.1.1 Clean sampler components with a detergent low in fluoride and phosphate, followed by rinsing with water (see 8.2).

10.1.2 Immerse a medium retentive prefilter (~~cleaned~~ **Warning**—Exercise care to see that all sampling apparatus is clean and free of contamination. Clean with a detergent low in fluoride and phosphate, followed by thorough rinsing with water (see 8.2.)) ~~in as in accordance with below in~~ the alcoholic citric acid solution (~~0.1~~ (0.1 N)) (see 8.3) and place under an infrared lamp to dry. After drying and prior to use, store the treated filter in a sealed container to prevent contamination.

NOTE 4—~~If the person using this test method demonstrates a requirement for there is a need for a prefilter of higher retentivity, retentivity than described in this test method, other filters may be used.~~ used as appropriate. Membrane filters, if used, require reduction of the alcohol content of the alcoholic citric acid solution to 65 % to prevent deformation of the ~~membrane~~ pores.

10.2 *Sampling:*

10.2.1 *Test the System for Leaks:*

10.2.1.1 Fill the impinger with the ~~required~~ volume of reagent water (see 8.2) that will be ~~utilized~~ used during sampling.

10.2.1.2 Turn on the vacuum pump (see 7.4.7) and develop a vacuum slightly greater than that required for the desired sampling rate. For short-term sampling (3 h or less), utilize a flow rate of 30 L/min (1 ft³/min). For long-term sampling (24 to 72 h), a flow rate of 15 L/min (0.5 ft³/min) is used with the modified standard impinger (see 7.2) and water level controller (see 7.3) to compensate for solution evaporation.

NOTE 4—For short term sampling (3 h or less), utilize a flow rate of 30 L/min (1 ft³/min). For long-term sampling (24 to 72 h), a flow rate of 15 L/min (0.5 ft³/min) shall be used with the modified standard impinger (see 7.2) and the water level controller (see 7.3) to compensate for solution evaporation.

10.2.2 During the sampling period, record the time, vacuum, and meter reading at the beginning and end of ~~the sampling period.~~ sampling. Also record barometric pressure in accordance with Test ~~Method~~ Methods **D3270** and the ambient temperature at intervals during the sampling ~~period.~~ period as stated in Test Methods **D3270**.

10.2.3 Use field blanks with each sample. (~~See~~ **Note 4.**) ~~two filters to the field and use one for sampling. Use the second filter as a field blank. Transport an impinger containing 150 mL of deionized water to the sampling site and treat in the same manner as the sample impingers except do not connect it to a sample train. Use this impinger as a field blank.~~

NOTE 5—~~Take two filters to the field and use one for sampling. Use the second filter as a field blank. Carry an impinger containing 150 mL of deionized water to the sampling site and treat in the same manner as the sample impingers except do not connect into a sample train. Use this impinger as a field blank.~~

10.2.4 At the end of ~~the sampling period, sampling,~~ transfer the filter and impinger contents to suitable containers for transporting to the work area where analyses are to be performed.

10.3 See Practice **D1357** for guidance on sampling.

11. Preparation and Analysis of Samples for Fluoride Analysis

11.1 Carry out the analysis in a work area with an atmosphere free of ~~contamination by fluorides,~~ fluoride contamination.

11.2 *Preparation of Impinger Solutions for Fluoride Analysis:*

11.2.1 Measure the volume of liquid in the impinger by weight if a tare weight is available on the impinger or by transferring to a tared polyethylene bottle and weighing the total contents after transfer. Record the weight of the liquid contents. Mix the contents thoroughly.

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