Designation: D3268 – 91 (Reapproved 2011)

Standard Test Method for Separation and Collection of Particulate and Gaseous Fluorides in the Atmosphere (Sodium Bicarbonate-Coated Glass Tube and Particulate Filter Method)[1]

This standard is issued under the fixed designation D3268; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 The sodium bicarbonate-coated glass tube and membrane filter method provides a means for the separation and collection of gaseous atmospheric forms of fluoride reactive with sodium bicarbonate and particulate forms of fluoride which are collected by a filter. The test method is applicable to 12-h sampling periods, collecting 1 to 500 µg of gaseous fluoride at a 15 L/min (0.5 ft^3/min) sampling rate or about 0.1 to 50 µg/m^3. The length of the sampling period can therefore be adjusted so that the amount of fluoride collected will fall within this range. The actual lower limit of the test method will depend upon the sensitivity of the analytical method employed and the quality of reagents used in tube preparation and analysis. It is recommended that the lower limit of detection should be considered as two times the standard deviation of the monthly arithmetic mean blank value. Any values greater than the blank by less than this amount should be reported as "blank value."

1.2 The values stated in SI units are to be regarded as standard. The inch-pound units in parentheses may be approximate.

1.3 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

D3266 Test Method for Automated Separation and Collection of Particulate and Acidic Gaseous Fluoride in the Atmosphere (Double Paper Tape Sampler Method)

D3267 Test Method for Separation and Collection of Particulate and Water-Soluble Gaseous Fluorides in the Atmosphere (Filter and Impinger 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)

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 Gaseous fluorides are removed from the air stream by reaction with sodium bicarbonate coated on the inside wall of a borosilicate glass tube (Note 1). Particulate fluorides are collected on a filter following the tube. The fluoride collected by the tube is eluted with water or buffer and analyzed for fluoride. The particulate matter collected by the filter is eluted with acid and analyzed for fluoride (**1-4**).[4] The results are reported as µg/m^3 of gaseous or particulate in air at 25°C (77°F) and 101.3 kPa (29.92 in. Hg).

NOTE 1—Some particulate matter will collect on the wall of the sample tube. If this loss is to be evaluated, use 7test methods such as Test Method D3266 or Test Method D3267 for comparison since the filter for collecting particulate precedes the absorbers for gases Mandl and Weinstein (**2**)

[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.

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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.

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

[4] The boldface numbers in parentheses refer to references at the end of this test method.

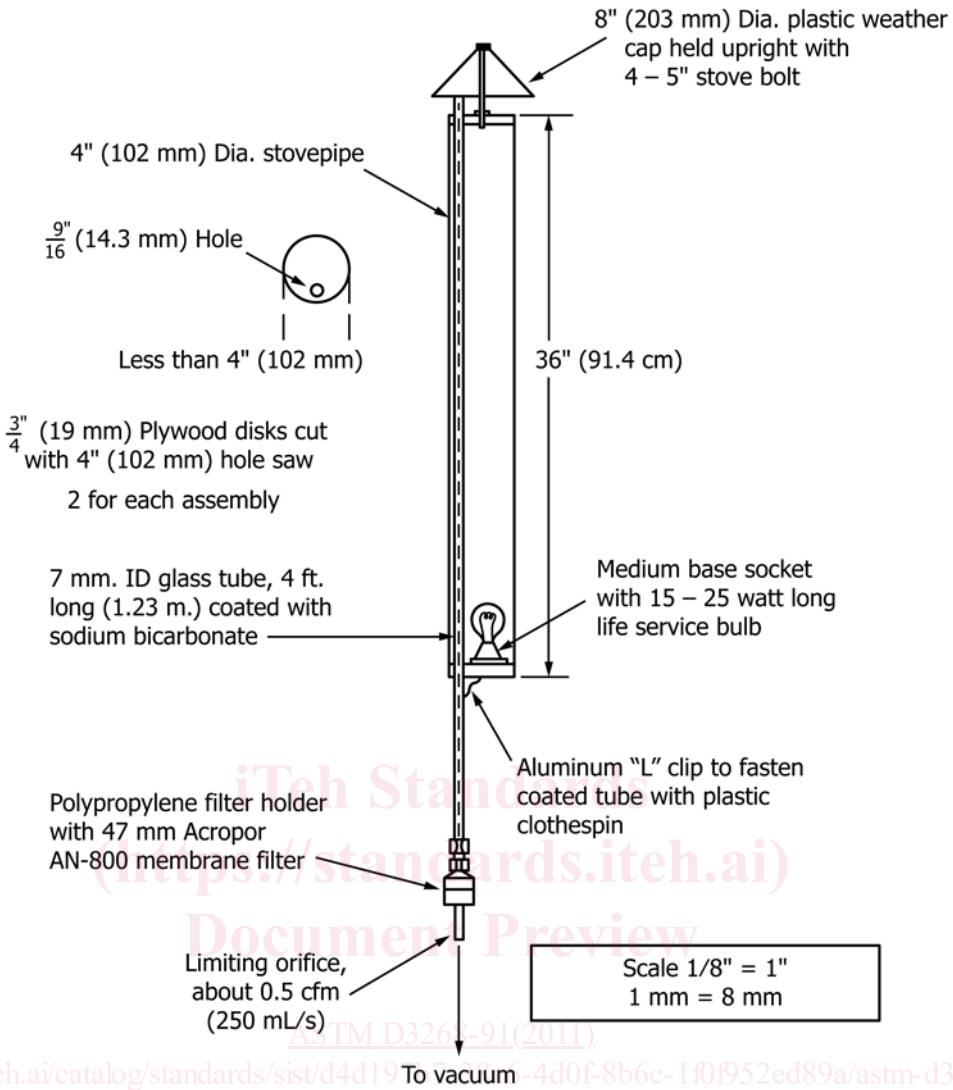

FIG. 1 Sodium Bicarbonate-Coated Glass Tube Illustrating Simple Heating Device

provide some information relative to potential loss of particulate matter.

5. Significance and Use

5.1 The sodium bicarbonate coated tube filter method provides a means of separating and collecting atmospheric gaseous fluoride and particulate fluoride samples.

5.2 Since the samples are collected on the dry tube and filter, the fluoride may be eluted with a small volume of eluant (see Section 10 for specific instructions on fluoride elution). Elution into a small volume and the sensitivity of the analytical methods employed allow the analysis of the collected fluoride to fractional parts of a microgram per cubic metre on samples taken for a 12-h period.

6. Interferences

6.1 Significant amounts of acid aerosols or gases might neutralize or acidify the bicarbonate coating and prevent quantitative uptake of gaseous fluoride from the atmosphere. If this potential interference needs to be evaluated, the alkalinity of the water extract may provide relevant information.

6.2 The presence of large amounts of aluminum or certain other metals or phosphates can interfere with subsequent analyses of the tubes or filters by calorimetric or electrometric methods. This is a problem inherent with any collection method for fluoride.

7. Apparatus

7.1 *Glass Tubing*—1200-mm (4-ft) lengths of 7-mm inside diameter borosilicate glass tubing, coated with sodium bicarbonate, in accordance with the requirements outlined in 7.6.

7.2 *Filter and Holder*—Connect the tubing directly to the filter holder and filter for the collection of particulate matter for particulate fluoride analysis (see Fig. 2).

NOTE 2—Use of material other than that recommended in footnote 6 or Fig. 2 will result in gaseous fluoride absorption on the material.

7.3 *Air Sampling System:*

7.3.1 The tube and filter are followed by an air sampling system which is capable of sampling at a rate of 15 L/min (0.5

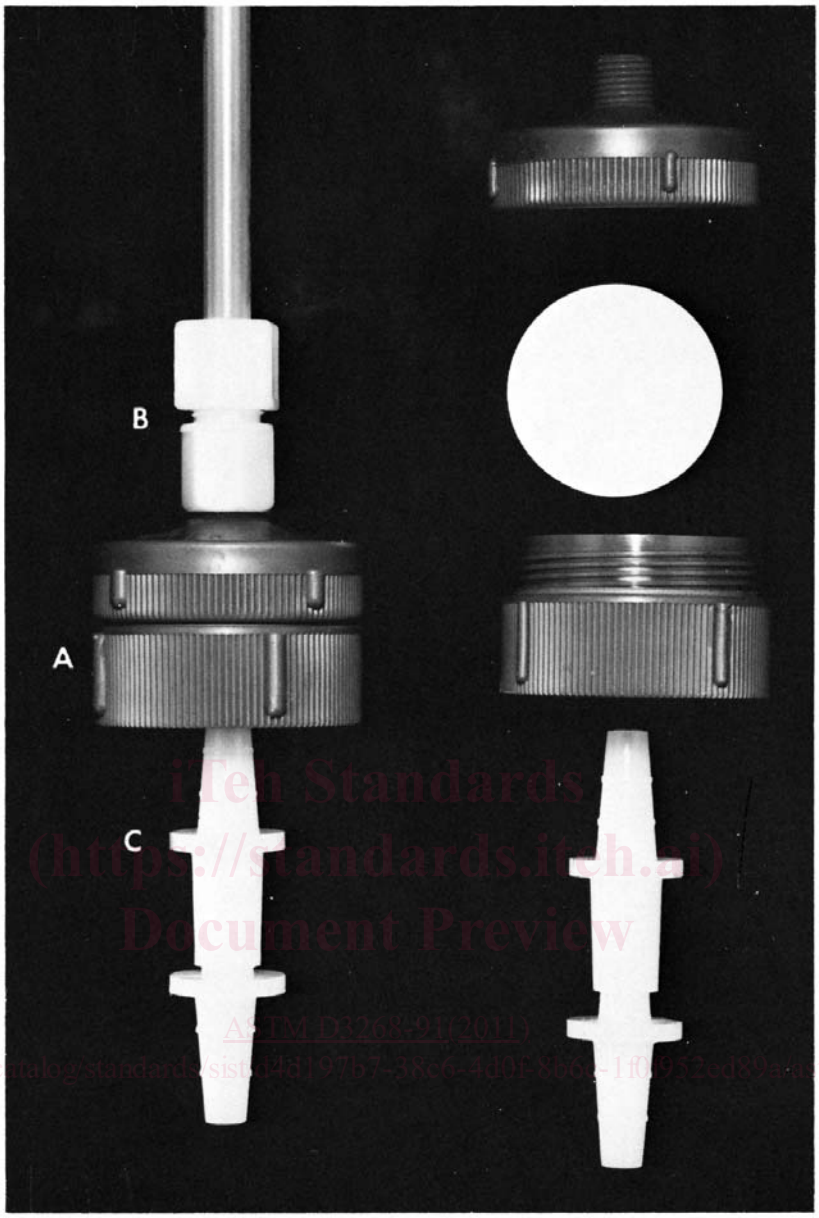

FIG. 2 Details of Attachment of the Filter Assembly and Limiting Orifice to a Bicarbonate-Coated Tube (7-mm Inside diameter). (A) Polypropylene Filter Holder. (B) Plastic Female Connector, (C) Limiting Orifice

ft^3/min) and measuring the total air sampled on a time rate basis or with a totalizing meter. See Test Method D3267 for sampling equipment, and the configuration and calibration.

7.3.2 The system shall be equipped so that pressure and temperature of the gas at the point of metering also are known for correcting sample volumes to standard conditions of 101.3 kPa (29.92 in. Hg) at 25°C (77°F).

7.3.3 Assemble the sampling system so that the inlet of the tube is 4 to 6 m above ground level (see Practice D1357) and protected from rain in such a manner as not to interfere with the free passage of aerosol fluorides.

7.4 *Light Bulb or Cone Heater,* 30-W, installed to heat the gases to a temperature where condensation will not occur.

7.5 *Configuration of Sampling Equipment*—Fig. 1 is a sketch of the sampling system. Other systems that meet the requirements outlined, are also satisfactory.

7.6 *Criteria for Coating of the Borosilicate Tubes:*

7.6.1 The coating shall be visible uniform coating on the full length of the tube.

7.6.2 The coating shall not contain any large crystals or heavy local deposits which could flake off and be collected with the aerosol fluorides.

7.6.3 The total coating shall contain less than 1 µg of fluoride when analyzed without exposure, including all the reagents used in the procedure. This is the reagent blank for the procedure.