Designation: D3266 – 91 (Reapproved 2005)

Standard Test Method for Automated Separation and Collection of Particulate and Acidic Gaseous Fluoride in the Atmosphere (Double Paper Tape Sampler Method)[1]

This standard is issued under the fixed designation D3266; 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 Department of Defense.

1. Scope

1.1 This test method describes the automatic separation and collection on chemically treated paper tapes of particulate and gaseous forms of acidic fluorides in the atmosphere by means of a double paper tape sampler. The sampler may be programmed to collect and store individual air samples obtained over time periods from several minutes to 3 h. A 30.5-m [100-ft] tape will allow unattended operation for the automatic collection of up to 600 samples.

1.2 The values stated in SI units are to be regarded as standard. The values given in brackets are for information only.

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]

D1071 Test Methods for Volumetric Measurement of Gaseous Fuel Samples

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

D3195 Practice for Rotameter Calibration

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)

D3270 Test Methods for Analysis for Fluoride Content of the Atmosphere and Plant Tissues (Semiautomated Method)

D3609 Practice for Calibration Techniques Using Permeation Tubes

D3614 Guide for Laboratories Engaged in Sampling and Analysis of Atmospheres and Emissions

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 paper tape to remove particulate matter which may contain fluoride and then through an alkali-treated paper tape to remove acidic fluoride gases.

4.2 The exhaust air is filtered through soda lime-glass wool, and the cleaned air is used to pressurize the front compartment to prevent fluoride contamination of the paper tapes from the ambient air.

4.3 Automatically, at the end of the preset sampling period, the vacuum pump is turned off, the tapes are indexed, and after indexing the vacuum pump is turned on. Indexing results in a "dead time" of several seconds.

4.4 The paper tapes are removed from the sampler after a selected period of operation and taken to an analytical work area where the individual sample spots are cut out, treated to dissolve the fluoride, and analyzed by potentiometric or photometric methods.[3,4,5]

[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] Mandl, R. H., Weinstein, L. H., Weiskopf, G. J., and Major, J. L. "The Separation and Collection of Gaseous and Particulate Fluorides." Paper CP-25A, 2D International Clean Air Congress, Washington, DC, 1970.

[4] Weinstein, L. H., and Mandl, R. H. "The Separation and Collection of Gaseous and Particulate Fluorides." VDI Berichte Nr. Vol 164, 1971, pp. 53 to 63.

[5] Lodge, James P. Jr., ed., "Methods of Air Sampling and Analysis," Intersociety Committee, 3rd ed., Lewis Publishers, Inc., 1988, pp. 352–356.

5. Significance and Use

5.1 This test method provides a means of automatically separating and collecting atmospheric particulate and acidic gaseous fluoride samples.

5.2 Since the samples are collected on dry tapes, the samples are in a form which allows elution of the fluoride content with a small volume of eluent. Consequently, the method allows analyses of air samples taken for a time period as short as several minutes.

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 acidic gaseous fluoride on the prefilter. If interfering quantities of such particulate metallic salts are present, the use of Test Methods D3268 is recommended because the acidic fluoride gases are collected prior to the filter.

6.2 Acid aerosols or gases might neutralize or acidify the alkali-treated tape and prevent quantitative uptake of the acidic fluoride gases from the atmosphere. If this potential interference is present the decreased alkalinity of the water extract (13.2.2.1) may provide relevant information.

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

6.4 There are several limitations of the test method that could possibly occur:

6.4.1 Although the acid-treated medium retentive prefilter has been shown to allow passage of hydrofluoric acid, it will restrict passage of particulate matter only as small as about 1 µm. Thus, smaller particulate matter may pass through the filter and impinge on or pass through the alkali-treated second tape.

6.4.2 The maximum sampling time recommended in the method is 3 h. This time is limited to minimize the possible effect of particulate matter sorbing the acidic fluoride gases or reducing the sampling rate.

7. Apparatus [6]

7.1 The double paper tape sampler is a modification of and utilizes the basic principles of the sequential paper tape sampler used for dust collection. The commercially available apparatus requires modification, as described in this test method, prior to use.[7] It consists of the following:

7.1.1 *Heated Inlet*—I_1, TFE-fluorocarbon, 1 m [3.3 ft] in length, 9.5 mm [⅜ in.] (outside diameter), encased in a 9.5 mm [⅜ in.] (inside diameter) aluminum tube. See Fig. 1. The aluminum jacket is wrapped in a constant wattage heating wire

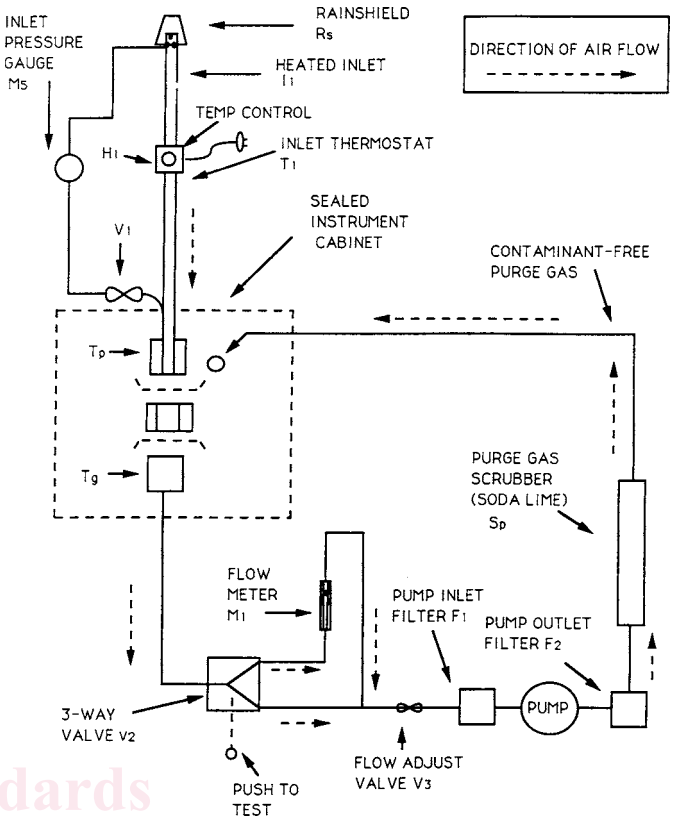


FIG. 1 Dual Tape Sampler Flow Schematic

of 25 W/m (8 W/ft). The tube is connected to the instrument with a TFE-fluorocarbon fitting.

7.1.1.1 *Rainshield*, R_s—Constructed of TFE-fluorocarbon.

7.1.1.2 *Proportional Temperature Controller*—H_1, with thermocouple reference point located at the bottom of the sample chamber.

7.1.1.3 *Inlet Thermostat*—T_1.

7.1.1.4 *Inlet Pressure Gauge*—M_5 with shutoff valve, V_1. One side of the gauge is connected to a TFE-fluorocarbon run tee placed between the intake tube and the sample block, and the other side is connected to a TFE-fluorocarbon run tee placed at the entrance to the intake tubing.

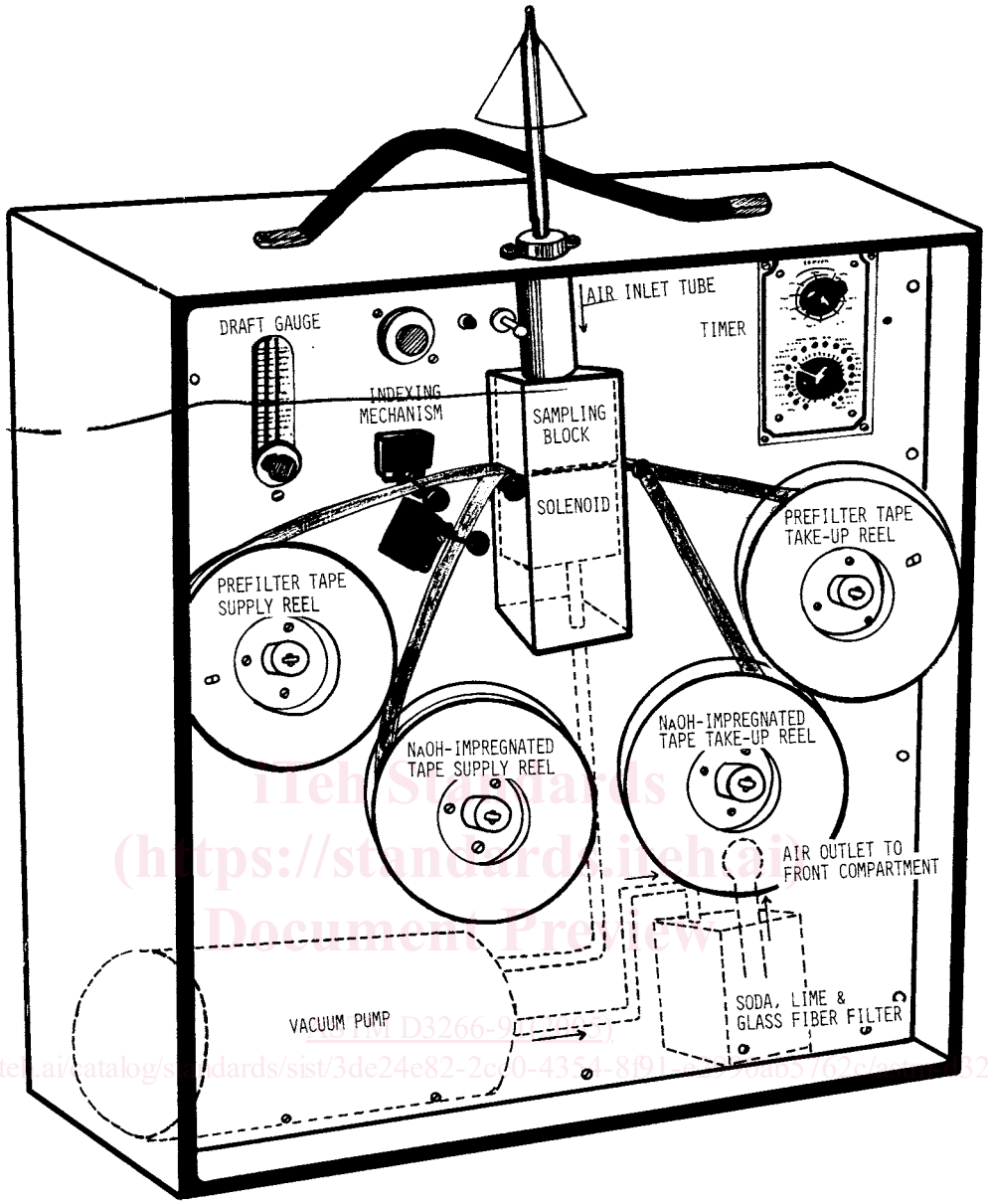
7.1.2 *Sampler*—See Figs. 1 and 2.

7.1.2.1 The upper part of the sampling block and sample inlet tube (Note 1) are constructed of polytetrafluoroethylene to minimize reactivity with acidic fluoride gases. The upper part of the sampling block (T_p) has a cylindrical cavity 25.4 mm [1 in.] in diameter with the inlet tube to the cavity perpendicular to the paper tapes. The lower part of the sampling block (T_g) shall be constructed of stainless steel with a 25.4 mm [1-in.] cylindrical cavity. The outlet tube from the cylindrical cavity passes at a right angle into the pump compartment. The lower block shall be spring-loaded with a total force of 1.36 kg [3 lb] against the lower surface of the upper block. The surfaces of the two blocks shall be machined flat to ensure a tight seal. The lower block shall be lowered by means of an electric solenoid which counteracts the spring pressure.

7.1.2.2 *Capstans*, positioned to guide the paper tapes through the sampling block and to the take-up reel.

[6] The sole source of supply of this apparatus known to the Committee is Anderson Samplers, Atlanta, GA. If you are aware of alternate suppliers, please provide this information to ASTM Headquarters, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428–2959. Your comments will recieve careful consideration at a meeting of the responsible technical committee, which you may attend.

[7] Zankel, K. L., McGirr, R., Romm, M. Campbell, Miller, R. "Measurement of Ambient Ground-Level Concentrations of Hydrogen Fluoride," *Journal of The Air Pollution Control Association*, Vol 37: 1191–1196 (1987).



Double Paper Tape Sampler

FIG. 2 Schematic Drawing of Double Paper Tape Sampler

7.1.2.3 The paper tapes shall be drawn through the sample block and wound on the take-up reels by 1⁄30 Hz (2 rpm) synchronous motors. Indexing is accomplished either by mechanical or photoelectric means to provide even spacing between samples. Provision is made by the use of tape perforated at regular intervals, or by some other means, to locate the collected sample spots for subsequent analysis. A relay is wired in series with the indexing mechanism to turn off the vacuum pump during tape transport.

7.1.2.4 *Interval Timer*, used to provide desired sampling times.

7.1.2.5 *Carbon-Vane Vacuum Pump*, to sample air, of nominal 30 L/min (1 ft^3/min) free-air capacity. This provides a sampling rate through two tapes of about 15 L/min (0.5 ft^3/min). Exhaust air from the pump is passed through a soda lime-glass wool filter (S_p) and the filtered air is used to pressurize the front compartment and prevent contamination by fluorides from the ambient air.

7.1.2.6 *Sample Flow Adjustment Valve*—An inline needle valve, V_3.

7.1.2.7 *Flow Indicator*—0–30 L/min (0–1 ft^3/min) M_1.

7.1.2.8 *Paper Tape*—38-mm [1.5-in.] wide, appropriately treated chemically (10.1).

7.1.2.9 Provision shall be made for manual override of the tape transport mechanism.

7.1.2.10 All fittings shall be constructed of TFE-fluorocarbon.

7.2 *Calibration Equipment*—See Fig. 3.

7.2.1 *Inlet Calibration Adapter*—To connect hose from flow calibration equipment to sampler inlet.