



Standard Test Methods for Analysis for Fluoride Content of the Atmosphere and Plant Tissues (Semiautomated Method)¹

This standard is issued under the fixed designation D 3270; 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 These test methods describe the semiautomated procedure for the analyses of various types of samples for the purpose of determining total fluoride. Since the test methods incorporate microdistillation of the sample, they may be applied to any fluoride-containing solution where standards of identical composition have been carried through the same sample preparation procedures and have proven to provide quantitative recovery when analyzed by the semiautomated system. Conversely, the methods shall not be applied for analyses until the applicability has been demonstrated.

1.2 In normal use, the procedure can detect 0.1 µg/mL of F. The normal range of analysis is from 0.1 to 1.6 µg/mL of F. Higher concentrations can be analyzed by careful dilution of samples with reagent water. If digested samples routinely exceed 1.6 µg/mL of F, the analytical portion of the pump manifold can be modified to reduce sensitivity. However, the best procedure is to analyze a smaller aliquot of the sample. Most accurate results are obtained when the fluoride concentration falls in the middle or upper part of the calibration curve.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 and health practices and determine the applicability of regulatory limitations prior to use. See 8.3, 10.2.4, and 10.2.5 for additional precautions.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres³

¹ These test methods are under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and are the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

Current edition approved March 10, 2000. Published May 2000. Originally published as D 3270 - 91. Last previous edition D 3270 - 91 (1995)^{ε1}.

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 11.03.

D 3266 Test Method for Automated Separation and Collection of Particulate and Acidic Gaseous Fluoride in the Atmosphere (Double Paper Tape Sampler Method)³

D 3267 Test Method for Separation and Collection of Particulate and Water-Soluble Gaseous Fluorides in the Atmosphere (Filter and Impinger Method)³

D 3268 Test Method for Separation and Collection of Particulate and Gaseous Fluorides in the Atmosphere (Sodium Bicarbonate-Coated Glass Tube and Particulate Filter Method)³

D 3269 Test Methods for Analysis for Fluoride Content of the Atmosphere and Plant Tissues (Manual Procedures)³

D 3614 Guide for Laboratories Engaged in Sampling and Analysis of Atmospheres and Emissions³

3. Terminology

3.1 *Definitions*—For definitions of terms used in these methods, see Terminology D 1356.

4. Summary of Test Methods

4.1 These semiautomated methods are based on the distillation of hydrogen fluoride (HF) from the sample and subsequent reaction of the distillate with alizarin fluorine blue-lanthanum nitrate reagent, to form a blue complex which is measured colorimetrically at 624 nm (**1**)⁴, or the subsequent measurement with a specific ion probe.

4.2 General, Plant Material:

4.2.1 The plant material including leaf samples, washed or unwashed, is dried, and ground, then dissolved with perchloric acid and diluted to 50 mL with water. In the case of leaf samples, an appreciable amount of fluoride may be deposited on the external leaf surfaces. This fluoride behaves differently physiologically from fluoride absorbed into the leaf and it is often desirable to wash it from the surface as a preliminary step in the analysis. Details of a leaf-washing process are described in 9.1.

4.3 General, Atmospheric Samples:

4.3.1 Test Methods D 3269 contains acceptable procedures and also techniques for the proper preparation of atmospheric samples. Test Methods D 3266, D 3267, and D 3268 are

⁴ The boldface numbers in parenthesis refer to the references at the end of this method.

