



Standard Test Method for Fluorides in Workplace Atmospheres¹

This standard is issued under the fixed designation D 4765; 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 covers the simultaneous collection and separate measurements of gaseous and particulate fluoride found in certain industrial workplaces. The gaseous inorganic fluorides collected are reported in terms of fluoride; the procedure is not applicable to the collection or analysis of other fluoride-bearing gases (for example, fluorocarbon or fluorosulfur compounds). This test method covers sample collection, preparation, and fluoride measurement.

1.2 *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:

- D 1193 Specification for Reagent Water²
- D 1356 Terminology Relating to Sampling and Analysis of Atmospheres³
- D 1357 Practice for Planning the Sampling of the Ambient Atmosphere³
- D 5337 Practice for Flow Rate for Calibration of Personal Sampling Pumps³

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1356.

4. Summary of Test Method

4.1 Particulate material from a measured volume of air is collected by means of a membrane filter. Gaseous fluoride, from the sample of air, is absorbed by an alkali-impregnated cellulose pad placed immediately behind the membrane filter. The membrane filter and collected solids are made alkaline, ashed, and the residue fused with additional alkali. Finally, the fluoride is determined in a solution of the melt by use of a

fluoride ion-selective electrode. Gaseous fluoride is determined in an aqueous extract of the cellulose pad, also by means of the fluoride ion-selective electrode.

5. Significance and Use

5.1 The capability of this test method to collect and quantify both particulate and gaseous fluorides over the ranges normally encountered in industrial atmospheres makes it applicable for industrial hygiene evaluation and control purposes. The recommended range of this test method is from 0.005 to 5 mg F⁻/m³ air.

6. Interferences

6.1 Because an ion-selective electrode responds to ionic activity, insoluble and complex forms of fluoride must be released by appropriate combinations of fusion, adjustment of pH, and addition of complexing agents.

6.2 Acidity (pH) and ionic strengths of fluoride standard solutions must be matched to those of samples.

6.3 Temperature of sample and standard solutions must be controlled within $\pm 2^{\circ}\text{C}$.

7. Apparatus

7.1 *Personal Sampling Pump*, Equipped with a flow-monitoring device (rotameter, critical orifice) or a constant-flow device capable of drawing 2 L/min of air through the 0.8- μm membrane filter and pad for a period of 8 h.

7.2 *Filter Holder*—Plastic holders of the preloaded personal monitor type, that accept filters of 37-mm diameter, are preferred. The holder is to be numbered for identification.

7.3 *Membrane Filter*, of mixed-cellulose esters, 0.8- μm pore size, and of diameter to fit the filter holder (see 7.2).

7.4 *Cellulose Pad*, of size to fit the filter holder (see 7.2). The pad is commercially available as a plain, unregnated pad or as an alkali-impregnated pad.

7.5 *Crucibles*, 20-mL, nickel, Inconel,⁴ or platinum.

7.6 *Fluoride Ion-Selective Electrode*.

7.7 *Reference Electrode*, calomel type, preferably combined with the fluoride ion-selective electrode.

7.8 *Electrometer or Expanded Scale pH Meter*, with a millivolt scale for measurement of potentials.

¹ This test method is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.04 on Workplace Atmospheres.

Current edition approved April 10, 2003. Published June 2003. Originally approved in 1988. Last previous edition approved in 1998 as D 4765 – 98.

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

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

⁴ Inconel is a trademark for a group of corrosion-resistant alloys of nickel and chromium.