



Designation: ~~D4856-99 (Reapproved 2004)~~ Designation: D4856 - 11

## ~~Standard Test Method for Determination of Sulfuric Acid Mist in the Workplace Atmosphere (Ion Chromatographic)~~ Determination of Sulfuric Acid Mist in Workplace Atmospheres Collected on Mixed Cellulose Ester Filters (Ion Chromatographic Analysis)<sup>1</sup>

This standard is issued under the fixed designation D4856; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

~~1.1 This ion chromatographic test method describes the determination of sulfuric acid mist in air samples collected from workplace atmospheres in a three-piece cassette filter.~~

1.1 This ion chromatographic test method describes the determination of sulfuric acid mist in air samples collected from workplace atmospheres on a mixed cellulose ester (MCE) filter.

NOTE 1—Other filter types such as quartz fiber, polytetrafluoroethylene (PTFE), and polyvinyl chloride (PVC) filters are also suitable.

1.2 The lower detection limit of this test method is 0.017 mg/m<sup>3</sup> of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) mist in 60 L of air sampled at 1 L/min.

~~1.3 This test method is relatively free from interference. The only known interference is soluble or partially soluble sulfate salts.~~

1.3 This test method is subject to interference from soluble and partially soluble sulfate salts. Other sulfur-containing compounds can be oxidized to sulfate and also interfere.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 No detailed instrument operating instructions are provided because of differences among various makes and models of ion chromatography (IC) systems. Instead, the analyst shall follow the instructions provided by the manufacturer of the particular instrument, analytical column, and suppressors used.

1.6 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. For specific precautionary statements, see Section 9.

### 2. Referenced Documents

~~2.1 ASTM Standards:~~ ASTM Standards:<sup>2</sup> [ards/sist/2e719681-1b25-4b8e-ab64-b358da9b6622/astm-d4856-11](https://www.astm.org/standards/sist/2e719681-1b25-4b8e-ab64-b358da9b6622/astm-d4856-11)

D882 Test Method for Tensile Properties of Thin Plastic Sheeting

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres ~~D1357 Practice for Planning the Sampling of the Ambient Atmosphere~~

~~D1605 Practices for Sampling Atmospheres for Analysis of Gases and Vapors~~

D1914 Practice for Conversion Units and Factors Relating to Sampling and Analysis of Atmospheres ~~D4327~~

D4327 Test Method for Anions in Water by Suppressed Ion Chromatography

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

2.2 ISO<sup>3</sup> and European Standards<sup>4</sup>:

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D22 on Sampling Analysis of Atmospheres and is the direct responsibility of Subcommittee on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Atmospheres.

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<sup>2</sup> 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.

<sup>3</sup> Withdrawn.

<sup>4</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

<sup>5</sup> Precision (CV=0.082) was obtained from NIOSH Contract No. CDC-99-74-45 on a range of 0.561–2.577 mg/M<sup>3</sup> using cellulose ester filters.

<sup>6</sup> Available from European Committee for Standardization (CEN), Avenue Marnix 17, B-1000, Brussels, Belgium, <http://www.cen.eu>.

ISO 648 Laboratory glassware—One Mark pipettes

ISO 7708 Air quality—Particle size fraction definitions for health-related sampling

ISO 1042 Laboratory glassware—One-mark volumetric flasks

EN 482 Workplace atmospheres—General requirements for the performance of procedures for the measurement of chemical agents.

EN 1540 Workplace atmospheres—Terminology

EN 13890 Workplace atmospheres—Procedures for measuring metals and metalloids in airborne particles—Requirements and test methods

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1356 and EN 1540.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *eluent*—ionic mobile phase used to transport the sample through the exchange columns.

3.2.2 *resolution*—ability of a column to separate constituents under specified test conditions.

### 4. Summary of Test Method

4.1 A known volume of air is drawn through a cassette filter holder containing a 37-mm mixed cellulose ester membrane filter of 0.8- $\mu\text{m}$  pore size. The filter is held in a 3-piece cassette supported by a cellulose back-up pad.

4.2 The filter is desorbed with Specification

4.1 A known volume of air is drawn through mixed cellulose ester membrane filter mounted in an appropriate air sampler. Examples are, but not limited to: inhalable samplers, 25-mm 3-piece, and 37-mm 3-piece cassettes supported by a back-up pad or screen.<sup>5</sup>

4.2 The sulfuric acid collected on the filter is desorbed with D1193 Type 1 water and an aliquot of the desorbed sample is injected into an ion chromatograph. The sample is pumped through two different ion exchange columns into a conductivity detector. Those columns are a precolumn and separator column which are packed with low-capacity anion exchanger. Ions are separated based on their affinity for the exchange sites of the resin. A method for anion suppression is used to reduce background conductivity of the eluent. Type 1 water. An aliquot of the desorbed sample solution is injected into an ion chromatograph to determine the sulfate ion concentration.

### 5. Significance and Use

5.1 Sulfuric acid is used in the manufacture of fertilizer, explosives, dyestuffs, other acids, parchment paper, glue, and the pickling of metal.

5.2 This test method has been found satisfactory for measuring sulfuric acid at levels required by federal occupational health regulations.

5.1 Sulfuric acid is used in the manufacture of fertilizer, explosives, dyestuffs, other acids, parchment paper, glue, lead acid batteries, textiles, etc., and in the pickling of metals.

5.2 This test method has been found to be satisfactory in the measurement of sulfuric acid for comparison with relevant occupational exposure limits.

### 6. Interferences

6.1 Soluble or partially soluble sulfate salts, for example, sodium or calcium sulfate, will constitute a positive bias.

6.1 Soluble or partially soluble sulfate salts, for example, sodium or calcium sulfate, will be measured as sulfuric acid. Other sulfur-containing compounds can be oxidized to sulfate and also interfere.

### 7. Apparatus

7.1 *Sampling Equipment:*

7.1.1 A 3-piece 37-mm filter cassette constructed of styrene acrylonitrile is used to hold the filter that is supported by a cellulose pad. The cassette has polyethylene end plugs that fit into an inlet and outlet sampling orifice.

7.1.2 The filter that is used in this procedure is a 37-mm diameter cellulose ester membrane filter of a 0.8- $\mu\text{m}$  pore size.

7.1.3 A portable sampling pump capable of a 1 L/min sampling rate with a filter cassette containing a backing pad and cellulose ester membrane filter previously described.

7.1.1 Air sampler designed to collect the appropriate aerosol size fraction needed. Examples are: inhalable sampler, 3-piece 37-mm or 25-mm filter cassette constructed of styrene acrylonitrile to hold the filter that is supported by a cellulose pad.

7.1.2 A MCE filter.

NOTE 2—This method may also be used with other appropriate filters such PTFE, Glass Fiber, PVC, and others.

<sup>5</sup> Consult Test Method D4327.

<sup>5</sup> Precision ( $CV_T = 0.082$ ) was obtained (NIOSH Contract No. CDC-99-74-45) over a range of 0.561 – 2.577 mg/M<sup>3</sup> using mixed cellulose ester filters.