Designation: D4856 – 23

Standard Test Method for Determination of Sulfuric Acid Mist in Workplace Atmospheres Collected on Mixed Cellulose Ester Filters (Ion Chromatographic Analysis)[1]

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 (ε) 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 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 0.001 mg/sample or 0.017 mg/m^3 of sulfuric acid (H_2SO_4) mist in 60 L of air sampled at 1 L/min.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see Section 9.

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

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

D4327 Test Method for Anions in Water by Suppressed Ion Chromatography

D4840 Guide for Sample Chain-of-Custody Procedures

D5337 Practice for Setting and Verifying the Flow Rate of Personal Sampling Pumps

D8358 Guide for Assessment and Inclusion of Wall Deposits in the Analysis of Single-Stage Samplers for Airborne Particulate Matter

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

E288 Specification for Laboratory Glass Volumetric Flasks

E1154 Specification for Piston or Plunger Operated Volumetric Apparatus and Operator Qualification

2.2 *ISO and European Standards:*

ISO 648 Laboratory glassware — Single-volume pipettes[3]

ISO 7708 Air quality — Particle size fraction definitions for health-related sampling[3]

ISO 18158 Workplace air — Terminology[3]

ISO 20581 Workplace air — General requirements for the performance of procedures for the measurement of chemical agents[3]

ISO 21438-1 Workplace atmospheres — Determination of inorganic acids by ion chromatography — Part 1: Non-volatile acids (sulfuric acid and phosphoric acid)[3]

[1] This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

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

EN 13205 Workplace exposure — Assessment of sampler performance for measurement of airborne particle concentrations[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1356 and ISO 18518.

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.2 *resolution, n*—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 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, with filters supported by a back-up pad or screen.[5]

4.2 The sulfuric acid collected on the filter is desorbed with Specification D1193 Type 1 water or eluent. 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, 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.

NOTE 2—In some countries the occupational exposure limit value (OELV) for sulfuric acid is related to the thoracic aerosol fraction; in such cases it is recommended to use a sampler for the thoracic aerosol fraction (ISO 20581).[6]

6. Interferences

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 Air sampler designed to collect the appropriate aerosol size fraction needed (ISO 7708). 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.

NOTE 3—Some size-selective samplers are designed to collect the targeted fraction of airborne particles on the filter, and any particulate matter deposited on the internal surfaces of the sampler (separate from the filter) is not considered part of the sampled air. Other size-selective samplers are designed such that all airborne particles which pass through the entry orifice(s) are of interest, hence particulate matter deposited on the inner walls of the sampler does form part of the sample. In such cases it will be necessary to account for particulate material collected on the inner walls of the sampler (in addition to that collected on the filter). Refer to Guide D8358 for additional information.

7.1.2 Mixed cellulose ester (MCE) filters.

NOTE 4—Other filter types such as quartz fiber, polytetrafluoroethylene (PTFE), and polyvinyl chloride (PVC) filters are also suitable.

7.1.3 A personal sampling pump capable of maintaining the required sampling rate through the sampler, throughout the sampling period.

7.2 *Ion Chromatograph:*

7.2.1 *Pump,* capable of delivering a constant flow of 0.1 mL to 5 mL of eluent per minute at a pressure of from 3.4 MPa to 34 MPa (500 psi to 5000 psi).

7.2.2 *Injection Valve,* a low dead volume, non-metallic valve fitted with a sample loop having a volume of up to 500 µL, for injecting the samples into the eluent stream. An autosampler can be attached for automation.

7.2.3 *Guard Column,* a column placed before the separator column to protect it from being fouled by particulate matter.

7.2.4 *Separator Column,* a column packed with anion exchange resin that is suitable for resolving the sulfate anion.

7.2.5 *Suppressor Module,* a module to reduce the total conductivity of the eluent, suitable for use with the separator column and capable of converting the eluent and separated anions to their respective acid forms.

7.2.6 *Conductivity Detector,* a low-volume, flow-through, temperature-compensated, electrical conductivity cell.

7.2.7 *Recorder, Integrator, or Computer,* a device for the purpose of measuring peak height or area, compatible with the detector output, and capable of recording detector response as a function of time.

7.2.8 *Eluent Reservoir,* a container suitable for storing a prepared eluent solution.

7.2.9 *Eluent Generation System,* for continuous production of eluent, suitable for use with the selected separator column, as an alternative to using a manually-prepared eluent.

7.2.10 *Ultrasonic Bath,* preferably with a timer, suitable for use in the ultrasonic extraction.

7.3 *Laboratory Supplies:*

7.3.1 *One-mark Volumetric Flasks,* capacities between 10 mL and 2000 mL.

7.3.2 *One-mark Pipets,* complying with the requirements of ISO 648; or *Piston-Operated Volumetric Pipettors and Dispensers*, complying with the requirements of Specification E1154.

7.3.3 *Disposable Screw-cap Polyethylene Vessels,* of 15 mL capacity.

7.3.4 *Disposable Filters,* polytetrafluorethylene (PTFE), pore size 0.45 µm, for use in ion chromatography.

7.3.5 *Disposable 2 mL or 5 mL Syringes,* with Luer lock connector, for use with disposable filters, or for sample injections without an autosampler.

[4] Available from European Committee for Standardization (CEN), Avenue Marnix 17, B-1000, Brussels, Belgium, http://www.cen.eu.

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

[6] Breuer, D.; Heckmann, P.; Gusbeth, K.; Schwab, G.; Blaskowitz, M.; Moritz, A."Sulfuric acid at workplaces - Applicability of the new Indicative Occupational Exposure Limit Value (IOELV) to thoracic particles." *Journal of Environmental Monitoring*, Vol. 14 (2012), pp. 440-445.