Designation: D2914 – 15 (Reapproved 2022)

Standard Test Methods for Sulfur Dioxide Content of the Atmosphere (West-Gaeke Method)[1]

This standard is issued under the fixed designation D2914; 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 U.S. Department of Defense.

1. Scope

1.1 These test methods cover the bubbler collection and colorimetric determination of sulfur dioxide (SO_2) in the ambient or workplace atmosphere.

1.2 These test methods are applicable for determining SO_2 over the range from approximately 25 $\mu g/m^3$ (0.01 ppm(v)) to 1000 $\mu g/m^3$ (0.4 ppm(v)), corresponding to a solution concentration of 0.03 μg SO_2/mL to 1.3 μg SO_2/mL. Beer's law is followed through the working analytical range from 0.02 μg SO_2/mL to 1.4 μg SO_2/mL.

1.3 The lower limit of detection is 0.075 μg SO_2/mL (**1**),[2] representing an air concentration of 25 μg SO_2/m^3 (0.01 ppm(v)) in a 30-min sample, or 13 μg SO_2/m^3 (0.005 ppm(v)) in a 24-h sample.

1.4 These test methods incorporate sampling for periods between 30 min and 24 h.

1.5 These test methods describe the determination of the collected (impinged) samples. A Method A and a Method B are described.

1.6 Method A is preferred over Method B, as it gives the higher sensitivity, but it has a higher blank. Manual Method B is pH-dependent, but is more suitable with spectrometers having a spectral band width greater than 20 nm.

NOTE 1—These test methods are applicable at concentrations below 25 $\mu g/m^3$ by sampling larger volumes of air if the absorption efficiency of the particular system is first determined, as described in Annex A4.

NOTE 2—Concentrations higher than 1000 $\mu g/m^3$ can be determined by using smaller gas volumes, larger collection volumes, or by suitable dilution of the collected sample with absorbing solution prior to analysis.

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

1.8 **Warning—***Mercury has been designated by many regulatory agencies as a hazardous material that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Safety Data Sheet (SDS) for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.*

1.9 *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 8.3.1, Section 9, and A3.1.3.

1.10 *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:*[3]

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

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

D3195 Practice for Rotameter Calibration

D3609 Practice for Calibration Techniques Using Permeation Tubes

D3631 Test Methods for Measuring Surface Atmospheric Pressure

[1] These test methods are under the jurisdiction of ASTM Committee D22 on Air Quality and are the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[3] 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.

E1 Specification for ASTM Liquid-in-Glass Thermometers
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

2.2 *Other Standards:*

40 CFR Part 58 Probe and Monitoring Path Siting Criteria from Ambient Air Quality Monitoring, Appendix E[4]

3. Terminology

3.1 For definitions of terms used in this method, refer to Terminology D1356.

4. Summary of Test Methods

4.1 Sulfur dioxide (SO_2) is absorbed by aspirating a measured air sample through a tetrachloromercurate (TCM) solution, resulting in the formation of a dichlorosulfonatomercurate complex (**2, 3**). Ethylenediaminetetraacetic acid disodium salt (EDTA) is added to this solution to complex heavy metals that interfere with this method (**4**). Dichlorosulfonatomercurate, once formed, is stable to strong oxidants (for example, ozone and oxides of nitrogen) (**2**). After the absorption is completed, any ozone in the solution is allowed to decay (**5**). The liquid is treated first with a solution of sulfamic acid to destroy the nitrite anion formed from the absorption of oxides of nitrogen present in the atmosphere (**6**). It is treated next with solutions of formaldehyde and specially purified acid-bleached pararosaniline containing phosphoric acid (H_3PO_4) to control pH. Pararosaniline, formaldehyde, and the bisulfite anion react to form the intensely colored pararosaniline methyl sulfonic acid which behaves as a two-color pH indicator (**2**). The pH of the final solution is adjusted to the desired value by the addition of prescribed amounts of 3 *N* H_3PO_4 to the pararosaniline reagent (**5**).

5. Significance and Use

5.1 Sulfur dioxide is a major air pollutant, commonly formed by the combustion of sulfur-bearing fuels. The Environmental Protection Agency (EPA) has set primary and secondary air quality standards (**7**) that are designed to protect the public health and welfare.

5.2 The Occupational Safety and Health Administration (OSHA) has promulgated exposure limits for sulfur dioxide in workplace atmospheres (**8**).

5.3 These methods have been found satisfactory for measuring sulfur dioxide in ambient and workplace atmospheres over the ranges pertinent in 5.1 and 5.2.

5.4 Method A has been designed to correspond to the EPA-Designated Reference Method (**7**) for the determination of sulfur dioxide.

6. Interferences

6.1 The interferences of oxides of nitrogen are eliminated by sulfamic acid (**5, 6**), of ozone by time delay (**5**), and of heavy metals by EDTA and phosphoric acid (**4, 5**). At least 60 μg of Fe(III), 10 μg of Mn(II), and 10 μg of Cr(III), 10 μg of Cu(II) and 22 μg of V(V) in 10 mL of absorbing reagent can be tolerated in the procedure. No significant interference was found with 2.3 μg of NH_3 (**9**).

7. Apparatus

7.1 *For Sampling:*

7.1.1 *Absorber, Short–Term Sampling*—An all-glass midget impinger having a solution capacity of 30 mL and a stem clearance of 4 ± 1 mm from the bottom of the vessel is used for sampling periods of 30 min and 1 h (or any period considerably less than 24 h).

7.1.2 *Absorber, 24-h Sampling*—A glass or polypropylene tube 32 mm in diameter and 164 mm long with a polypropylene two-port cap (rubber stoppers are unacceptable because the absorbing reagent can react with the stopper to yield erroneously high SO_2 concentrations, and cause high and variable blank values). Insert a glass impinger stem, 6 mm inside diameter and 158 mm long, into one port of the absorber cap. Taper the tip of the stem to a small diameter orifice (0.4 ± 0.1 mm) such that a No. 79 jeweler's drill bit will pass through the opening but a No. 78 drill bit will not. Clearance from the bottom of the absorber to the tip of the stem shall be 6 ± 2 mm. Perform the orifice test before use to verify the orifice size. Permanently mark the 50 mL volume level on the absorber. See Fig. 1.

7.1.3 *Air Sample Probe*—A sample probe meeting the requirements of Section 7 of 40 CFR Part 58, Appendix E, (TFE-fluorocarbon, polypropylene, or glass with a residence time less than 20 sec), used to transport ambient air to the sampling train location. Design or orient the end of the probe to preclude the sampling of precipitation, large particles, etc.

7.1.4 *Moisture Trap*—Glass or polypropylene trap as shown in Fig. 1, placed between the absorber tube and flow control device to prevent entrained liquid from reaching the flow control device. Pack the tube with coconut charcoal and glass wool or with indicating silica gel. Charcoal is preferred when collecting long-term samples (1 h or more) if flow changes are routinely encountered.

7.1.5 *Cap Seals*—Seal the absorber and moisture trap caps securely to prevent leaks during use, by using heat-shrink material to prevent the caps coming loose during sampling, shipment, or storage.

7.1.6 *Filter,* membrane, of 0.8 to 2.0 μm porosity, with filter holder, to protect the flow controller from particles during long-term sampling. This item is optional for short-term sampling.

7.1.7 *Pump,* equipped with vacuum gauge, capable of maintaining a vacuum greater than 70 kPa (0.7 atm) at the specified flow rate across the flow control device.

7.1.8 *Flow Control and Measurement Devices:*

7.1.8.1 *Flow Control Device*—A calibrated rotameter and needle valve combination capable of maintaining and measuring air flow to within ±2 percent is suitable for short-term sampling but shall not be used for long-term sampling. A critical orifice can be used for regulating flow rate for both

[4] Available from U.S. Government Publishing Office (GPO), 732 N. Capitol St., NW, Washington, DC 20401, http://www.gpo.gov.