



Designation: D 5578 – 94 (Reapproved 1999)^{e1}

Standard Test Method for Determination of Ethylene Oxide in Workplace Atmospheres (HBr Derivatization Method)¹

This standard is issued under the fixed designation D 5578; 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.

^{e1} NOTE—Editorial corrections were made throughout in December 1999.

1. Scope

1.1 This test method covers a method of collecting and analyzing samples to determine the amount of ethylene oxide (ETO) present in workplace atmospheres.

1.2 This test method can be used to provide a time-weighted average (TWA) over the concentration range from 0.2 to 30 ppm (v).

1.3 This test method can be used to determine 15-min excursions (STEL) ranging from 10 to 500 ppm (v).

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *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 Section 9 for specific safety hazards.*

2. Referenced Documents

2.1 ASTM Standards:

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres²

D 3686 Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)²

D 3687 Practice for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption Method²

E 355 Practice for Gas Chromatography Terms and Relationships³

2.2 Other Standard:

Occupational Safety and Health Administration, U.S. Department of Labor, Title 29, *Code of Federal Regulations*, Part 1910, Subpart Z, Section 1910.1047.⁴

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology D 1356, and Practice E 355.

4. Summary of Test Method

4.1 A known volume of air is pumped through a glass tube packed with carbon molecular sieve, surface area 400 m²/g impregnated with hydrogen bromide (HBr) where ETO is adsorbed and converted to 2-bromoethanol.

4.2 The tube contains two reactive sections for sample collection. The backup section collects vapors that pass through the front section and is used to determine if the collection capacity of the front section has been exceeded.

4.3 The resultant derivative, 2-bromoethanol, is desorbed with a mixture of acetonitrile/toluene and analyzed using a gas chromatograph equipped with an electron capture detector.

4.4 Desorption efficiency is determined by spiking tubes with known amounts of 2-bromoethanol and analyzing with the same procedure used for air samples.

4.5 Quantitation is achieved by comparing peak areas from sample solutions with areas from standard solutions.

5. Significance and Use

5.1 Ethylene oxide is a major industrial chemical with production volume ranked in the top 25 chemicals produced in the United States. It is used in the manufacture of a great variety of products as well as being a sterilizing agent and fumigant.

5.2 This test method provides a means of determining exposure levels of ETO in the working environment at the presently recommended exposure guidelines.

¹ This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.04 on Workplace Atmospheres.

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² *Annual Book of ASTM Standards*, Vol 11.03.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

5.2.1 OSHA Permissible Exposure Limit (PEL) 1 ppm, 15-min excursion limit 5 ppm (CFR, Part 1910, Subpart Z, Section 1910.1047).⁴

5.2.2 ACGIH Threshold Limit Value (TLV) 1 ppm (1.8 mg/m³), suspected human carcinogen.⁵

6. Interferences

6.1 Derivatives and other compounds that have identical or nearly the same retention time as 2-bromoethanol during the gas chromatographic analysis will interfere.

6.2 Interferences can sometimes be resolved by altering gas chromatographic operating conditions. The presence of 2-bromoethanol under a chromatographic peak can be verified using a mass spectrometer.

7. Apparatus

7.1 *Carbon Molecular Sieve*, surface area 400 m²/g, HBr sampling tube.

7.1.1 *Preparation of Collection Medium*—Add 20 mL of HBr (24 %) to 70 g of carbon molecular sieve, surface area 400 m²/g, in a glass jar. Cap the jar and mix the contents thoroughly for 5 min by rotating. Allow to equilibrate and dry overnight or for 12 h.

7.1.2 *Tube Preparation*—Insert a plug of silanized glass wool into a 10-cm by 6-mm outside diameter (4-mm inside diameter) glass tube. Pack the front section of the tube with 400 mg of the reactive adsorbent (7.1.1), using gentle tapping or vibration to promote uniform packing. Insert another plug of silanized wool and pack 200 mg of the adsorbent in the backup section. Hold the backup section in place by firmly inserting an additional glass wool plug. The tubes may be flame-sealed or sealed with polyethylene caps. Provide a numerical identification for each lot of tubes.

7.1.3 *Tube Holder*, capable of preventing breakage and protecting worker during sampling.

7.1.4 *High-Density Polyethylene or Polypropylene Caps*, tight-fitting, for resealing used tubes.

7.2 Pump and Tubing:

7.2.1 *Sampling Pumps*, having stable low flow rates ($\pm 10\%$ of set flow rate) within the range from 20 to 100 mL/min for up to 8 h.

7.2.2 *Rubber or Plastic Tubing*, 6-mm inside diameter, for connecting collection tube to pump. All tubing must be downstream (between tube and pump) of collection tube to prevent contamination or loss of sample.

7.3 *Vials*, glass with TFE-fluorocarbon lined caps, 10 mL, for desorbing samples and storing standards.

7.4 *Pipettes*, 5 mL, for adding desorbing solution to samples.

7.5 *Syringes*, 10, 50, and 100- μ L syringes, for preparing standards.

7.6 *Gas-Tight Syringe*, 2 μ L, with low dead-volume needle.

7.7 *Gas Chromatograph (GC)*:

7.7.1 *Gas Chromatograph*, with an electron capture detector and a suitable readout device.

7.7.2 *Chromatographic Column*, packed or capillary columns in accordance with 7.7.2.1 and 7.7.2.2 have been found suitable for this analysis.

7.7.2.1 *Packed*, 3.7 m by 3 mm (12 ft by 1/8 in.), stainless steel, packed with 10 % diethylene glycol succinate on diatomaceous earth, flux-calcined, silanized, 80/100 mesh.

7.7.2.2 *Capillary*, 30-m by 0.53-mm inside diameter fused silica capillary column with polyethylene glycol phase.

8. Reagents

8.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Acetonitrile*, pesticide grade.

8.3 *2-Bromoethanol*, commercially available at 98 % purity or better.

8.4 *Desorbing Solution*, 1+1 (v/v) mixture of acetonitrile and toluene.

8.5 *Sodium Carbonate* (Na₂CO₃).

8.6 *Toluene*, pesticide grade.

9. Hazards

9.1 Minimize exposure to all reagents and solvents by performing all sample and standard preparations as well as tube desorption in a well-ventilated hood.

9.2 Avoid inhalation and skin contact with all reagents and solvents.

9.3 Use suitable protective holders when collecting samples and handle used tubes carefully to prevent injury.

10. Calibration

10.1 Sample Pump Calibration:

10.1.1 Calibrate the sample pump flow in accordance with Practice D 3686, with the ETO sampling tube positioned vertically and in line during calibration of the pump.

10.1.2 Calibrate the flow rate of the pump at 20 mL/min for TWA sampling and 100 mL/min for STEL sampling depending on the duration of the sample and the volume of sample needed.

10.2 Gas Chromatograph Calibration:

10.2.1 Prepare a 2-bromoethanol stock solution (1 μ g/ μ L) by adding 57 μ L of 2-bromoethanol to 100 mL of toluene. If refrigerated, this solution is stable for at least one month.

10.2.2 To a series of four 10-mL vials containing 5.0 mL of desorbing solution, add 0.0, 50, 100, and 200 μ L of stock solution, thus, providing calibration standards equivalent to

⁵ *Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices*, American Conference of Governmental Industrial Hygienists, 6500 Glenway Avenue, Building D-7, Cincinnati, OH 45211-4438.

⁶ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.