Designation: D8311 – 20

Standard Test Method for Impurities in Monoethylene Glycol by Gas Chromatography with Normalization[1]

This standard is issued under the fixed designation D8311; 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 gas chromatographic determination of impurities in monoethylene glycol including 1,3-dioxolane-2-methanol, diethylene glycol (DEG) and triethylene glycol (TEG). The purity of monoethylene glycol (MEG) is also calculated. A similar test method, using the internal standard calibration technique and the external standard calibration technique, is Test Method E2409.

1.2 This test method is applicable for monoethylene glycol purities of 98.0 mass % or higher.

1.3 The limit of detection (LOD) for 1,3-dioxolane-2-methanol, DEG and TEG is 0.0002 mass %.

1.4 In determining the conformance of the test results using this method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

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

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.*

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]

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E300 Practice for Sampling Industrial Chemicals

E355 Practice for Gas Chromatography Terms and Relationships

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1064 Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

E2409 Test Method for Glycol Impurities in Mono-, Di-, Tri- and Tetraethylene Glycol and in Mono- and Dipropylene Glycol(Gas Chromatographic Method)

2.2 *Other Document:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200[3]

3. Summary of Test Method

3.1 The specimen to be analyzed is injected into a gas chromatograph equipped with a flame ionization detector (FID) and a capillary column.

3.2 The peak area of each component is measured and adjusted using relative calibration factors. The concentration of each component is calculated based on its relative percentages of total adjusted peak area and normalized to 100.0000 %.

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.14 on Alcohols & Glycols.

Current edition approved April 1, 2020. Published May 2020. DOI: 10.1520/D8311-20.

[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 Occupational Safety and Health Administration (OSHA), 200 Constitution Ave., NW, Washington, DC 20210, http://www.osha.gov.

4. Significance and Use

4.1 This test method is suitable for setting specifications and for use as an internal quality control tool where these products are produced or are used. Typical impurities are: 1,3-dioxolane-2-methanol, diethylene glycol, and triethylene glycol.

4.2 This method may not detect all components and there may be unknown components that would be assigned inappropriate relative calibration factors and thus, the results may not be absolute.

5. Apparatus

5.1 *Gas Chromatograph*—Any instrument having a flame ionization detector and a splitter injector suitable for use with a fused silica capillary column may be used, provided the system has sufficient sensitivity, linearity, and range to determine 0.0001 mass %, while not exceeding the full scale of either the detector or the electronic integration for the major component. It shall have a split injection system that will not discriminate over the boiling range of the samples analyzed. The system should be capable of operating at conditions given in Table 1.

5.2 *Columns*—The choice of column is based on resolution requirements. Any column may be used that is capable of resolving all significant impurities from the major component. The column and conditions described in Table 1 have been used successfully and shall be used as a referee in cases of dispute.

5.3 *Injector*—The specimen must be precisely and repeatedly injected into the gas chromatograph. An autoinjector is required.

5.4 *Syringe*—Chromatographic, capable of delivering appropriate µL volumes.

5.5 *Electronic Integrator* chromatography data system is required.

TABLE 1 Recommended Method Parameters

Inlet	Split
Temperature, °C	300
Column	
Material	fused silica
Stationary Phase	6 % Cyanopropyl-phenyl - 94 % dimethyl polysiloxane
Length, m	30
Internal diameter, mm	0.32
Film thickness, µm	1.8
Column temperature program	
Initial temperature, °C	80
Initial time, min	0.1
Programming rate, °C/min	25
Final, °C	240
Time 2, min	10
Carrier gas	Helium, Nitrogen or Hydrogen
Flow velocity, mL/min	1.5 (Helium), 1 (Nitrogen) or 1.5 (Hydrogen)
Split ratio	30:1
Sample size, µL	1.0
Detector	FID
Temperature, °C	300

6. Reagents and Materials

6.1 *Purity of Reagent*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Reagents with an establish purity greater than ACS reagent grade may be used.

6.2 *Calibration Standards:*

6.2.1 Mono-ethylene Glycol (MEG), minimum GC purity 99.95 mass %. As a blank MEG sample, the concentrations of each component of interest should not be higher than 0.005 mass %.

6.2.2 Di-ethylene Glycol (DEG), minimum purity 99.5 mass %.

6.2.3 Tri-ethylene Glycol (TEG), minimum purity 99.5 mass %.

6.2.4 1,3-Dioxolane-2-methanol, minimum purity 95 mass %.

6.3 Carrier gas (helium, nitrogen, or hydrogen), makeup gas and detector gases 99.999 % (v/v) pure or better. Oxygen in carrier gas less than 1 ppm, less than 0.5 ppm is preferred. Purify carrier, makeup and detector gases to remove oxygen, water, and hydrocarbons.

6.4 Air for the FID should contain less than 0.1 ppm total hydrocarbon.

7. Hazards

7.1 Consult current OSHA regulations and supplier's Safety Data Sheets and local regulations for all materials used in this test method.

7.2 *Monoethylene Glycol*—Although monoethylene glycol, in general, is not classified as dangerous or flammable and is not expected to impose a health hazard when used under normal conditions, it is recommended to avoid inhalation and contact with skin and eyes. Wear suitable protective clothing and gloves. Do not breathe gas, fumes, vapor, or spray. Use only in well-ventilated areas. In cases of contact with eyes, rinse with plenty of water and seek medical advice.

8. Sampling

8.1 Follow the relevant instructions for sampling as given in Practice E300.

9. Preparation of Apparatus

9.1 Follow manufacturer's instructions for mounting and conditioning the column into the chromatograph and adjusting the instrument to the conditions described in Table 1, allowing sufficient time for the equipment to reach equilibrium. See Practice E355 and Practice E1510 for additional information on gas chromatography practices and terminology.

[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.