



Designation: E2313 – 22

Standard Test Method for Aldehydes in Mono-, Di-, and Triethylene Glycol (using Spectrophotometry)¹

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1. Scope*

1.1 This test method describes the spectrophotometric determination of total aldehyde content in mono-, di-, and triethylene glycol (MEG, DEG, and TEG). Alkoxyalcohols (hemiacetals), if present, are co-determined, whereas dialkoxyalkanes (acetals), if present, are not. The results provide a measure of the purity of the sample with respect to total aldehyde content.

1.2 This test method is applicable to samples with total aldehyde concentration (as acetaldehyde) to 50 mg/kg or 35 mg/kg (as formaldehyde). The limit of detection (LOD) is 0.4 mg/kg and the limit of quantitation is 1.2 mg/kg.

NOTE 1—LOD and LOQ were calculated using the lowest level sample in the ILS.

1.3 The following applies for the purposes of determining the conformance of the test results using this test method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

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 *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 hazard statements, see Section 8 and 7.1.1 – 7.1.6.*

1.6 *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.*

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.

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2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

D2194 Test Method for Concentration of Formaldehyde Solutions

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

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)³

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

E288 Specification for Laboratory Glass Volumetric Flasks

E300 Practice for Sampling Industrial Chemicals

E969 Specification for Glass Volumetric (Transfer) Pipets

E1547 Terminology Relating to Industrial and Specialty Chemicals

2.2 Other Document:

Manufacturer's Instruction Manual of Spectrophotometer

3. Terminology

3.1 See Terminology E1547 for definitions of terms used in this test method.

4. Summary of Test Method

4.1 The total aldehyde content of the sample is determined spectrophotometrically. The aldehydes present are reacted with excess 3-methyl-2-benzothiazolinone hydrazone (MBTH) to form azines. An aqueous solution of iron (III) chloride is added, causing the oxidation of the excess MBTH to a reactive cation, which reacts with the previously formed azines producing bluish-green cations. Acetone or methanol is added to stop the oxidation reaction, to mask the excess MBTH and to obtain

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 The last approved version of this historical standard is referenced on www.astm.org.

*A Summary of Changes section appears at the end of this standard

a clear solution. The absorbance of the test solution is measured spectrophotometrically at 635 nm. The amount of aldehyde present in the test solution is derived from a previously prepared calibration graph. The aldehyde content of the sample, expressed as either acetaldehyde or formaldehyde, is calculated from the data obtained.

5. Significance and Use

5.1 Knowledge of the total aldehyde content of MEG is required to establish whether the product meets the requirements of its quality specifications.

6. Apparatus

6.1 *Spectrophotometer*, suitable for measurement at a wavelength of 635 nm, provided with cells with a pathlength of 10 mm.

NOTE 2—Further information on spectrophotometers can be obtained from Practice E275.

6.2 *Analytical Balance*, readable to 0.1 mg, calibrated. Recalibrate or verify at regular intervals.

6.3 *Stopwatch*, measuring accuracy ± 1 s or better. Verify at regular intervals.

6.4 *Buret*, calibrated, 100 mL, with a 50- or 75-mL reservoir on top of a lower portion calibrated in 0.1-mL divisions. A PTFE-fluorocarbon resin stopcock is suitable for this purpose. Recalibrate or verify at regular intervals.

6.5 *Adjustable Volume Dispensing Pipette(s)*, capable of dispensing volumes in the range 1.00 to 5.00 mL, with suitable disposable pipette tips. It is recommended to verify the capacity of the pipette(s) at regular intervals.

6.6 Glassware:

NOTE 3—In cases where volumetric glassware is to be used, it shall conform to Grade (or Class) A specifications as described in, for example, Specification E288 for volumetric flasks, or Specification E969 for one-mark pipettes.

7. Reagents

7.1 *Purity of Reagents*—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 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.

7.1.1 *Acetone (2-propanone)*, A.R. minimum purity 99.99 % (V/V). (**Warning**—Acetone is highly flammable. Keep container in a well-ventilated place. Keep away from sources of ignition. Do not breathe vapor. Take precautionary measures against static discharges.)

7.1.2 *Formaldehyde*, A.R., 36 to 40 % mass formaldehyde solution (Formalin). (**Warning**—Formaldehyde is classified as

a human carcinogen. Formaldehyde is toxic by inhalation, in contact with skin and if swallowed. Causes burns. Possible risks of irreversible effects. May cause sensitization by skin contact. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Wear suitable protective clothing, gloves and eye/face protection. In case of accident or if you feel unwell, seek medical advice immediately. Use only in well ventilated areas.)

7.1.3 *Hydrochloric Acid*, A.R., concentrated [36 % mass]. (**Warning**—Hydrochloric Acid is toxic by inhalation. Causes severe burns. Keep container in a well-ventilated place. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Wear suitable protective clothing, gloves and eye/face protection. In case of accident or if you feel unwell, seek medical advice immediately.)

7.1.4 *Iron (III) Chloride*, (FeCl₃·6H₂O), A.R. (**Warning**—Iron (III) Chloride causes burns. Avoid contact with skin and eyes. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Take off immediately all contaminated clothing. Wear eye/face protection. If swallowed, seek medical advice immediately and show the container or label.)

7.1.5 *Methanol*, A.R. minimum purity 99.99 % (V/V). (**Warning**—Methanol is highly flammable. Toxic by inhalation and ingestion. Keep container in a well-ventilated place. Keep container tightly closed when not in use. Keep away from sources of ignition. Avoid contact with skin. In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible.)

7.1.6 *3-Methyl-2-Benzothiazolinone Hydrazone Hydrochloride (MBTH)*. (**Warning**—MBTH is toxic if swallowed. Irritating to eyes. Keep locked up. Avoid contact with skin and eyes. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. This material and its container must be disposed of in a safe way. Wear suitable protective clothing. In case of accident or if you feel unwell, seek medical advice immediately. If swallowed, seek medical advice immediately and show the container or label.)

7.2 *Water*, reagent water conforming to Type I or II of Specification D1193.

7.3 Solutions:

7.3.1 *Standard Formaldehyde Solution*, 8.0 µg/mL, aqueous. Determine the exact concentration (c_1) of an A.R. 36 to 40 % mass formaldehyde solution (**Warning**—See 7.1.2.) according to Test Method D2194, expressing the result in g/100 mL. Weigh approximately 2.0 to 2.2 g (m_1) of the formaldehyde solution to the nearest 0.1 mg, into a 100-mL volumetric flask. Make up to the mark with water and mix thoroughly. Pipette 1.0 mL of this solution into a 1-L volumetric flask. Make up to the mark with water and mix thoroughly. Transfer this solution to a brown bottle with screw cap. Calculate the exact formaldehyde content of this solution (c_2), in µg/mL, by means of the following equation:

$$c_2, \mu\text{g/mL} = \frac{m_1 \times c_1}{10} \quad (1)$$

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.