Designation: E2313 – 15

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

This standard is issued under the fixed designation E2313; 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 describes the spectrophotometric determination of total aldehyde content in the range of 0.5 to 50 mg/kg (as acetaldehyde) or 0.3 to 35 mg/kg (as formaldehyde) 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 Review the current Safety Data Sheets (SDS) for detailed information concerning toxicity, first aid procedures, and safety precautions for all materials used in this test method.

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

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water
D2194 Test Method for Concentration of Formaldehyde Solutions
D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]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

2.2 *Other Document:*

Manufacturer's Instruction Manual of Spectrophotometer

3. Summary of Test Method

3.1 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 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.

4. Significance and Use

4.1 Knowledge of the total aldehyde content of monoethylene glycol is required to establish whether the product meets the requirements of its quality specifications.

5. Apparatus

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

NOTE 1—Further information on spectrophotometers can be obtained from Practice E275.

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

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[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.16 on Industrial and Specialty Product Standards.

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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] 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**

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

5.4 *Apparatus,* as described in Test Method D2194.

5.4.1 *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.

5.4.2 *Erlenmeyer Flask,* approximately 5-mL capacity.

5.4.3 *Vials,* specimen, short, style, approximately 5-mL capacity.

5.4.4 *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.

5.5 *Glassware:*

NOTE 2—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.

5.5.1 *Graduated Pipettes,* capable of quantitatively transferring between 0.5 and 5 mL, or

5.5.2 *Volumetric Pipettes,* 1.0, 2.0, 3.0, 4.0, 5.0 mL (if no adjustable volume dispensing pipettes are used).

5.5.3 *Volumetric Pipette,* 25 mL.

5.5.4 *Volumetric Flasks,* 100 mL, 1 L.

5.5.5 *Brown Bottle,* 1 L, with screw cap, for storage.

6. Reagents

6.1 *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.

6.1.1 *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.)

6.1.2 *Formaldehyde,* A.R., 36 to 40 % mass formaldehyde solution (Formalin). (**Warning—**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.)

6.1.3 *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.)

6.1.4 *Iron (lll) Chloride,* ($FeCl_3 \cdot 6H_2O$), 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.)

6.1.5 *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.)

6.1.6 *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.)

6.2 *Water,* reagent water conforming to Type IV of Specification D1193.

6.3 *Solutions:*

6.3.1 *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 6.1.2) 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:

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

where:

c_1 = concentration of concentrated formaldehyde solution used, g/100 mL, and

m_1 = mass of concentrated formaldehyde solution taken, g.

The concentrated and diluted formaldehyde solutions are stable in excess of six months if stored at room temperature. Do not store in a refrigerator. Prepare fresh solutions every six months.

6.3.2 *Hydrochloric Acid,* 50 %(V⁄V). Dilute one volume of A.R. concentrated hydrochloric acid (**Warning—**See 6.1.3) with one volume of water.

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[4] *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 *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USP), Rockville, MD.

6.3.3 *Iron (III) Chloride Solution,* 0.3 % mass, aqueous. Dissolve 5 g of A.R. iron (III) chloride. $6H_2O$ (**Warning**—See 6.1.4) in water, adding 1 or 2 drops of A.R. concentrated hydrochloric acid to stabilize solution (**Warning—**See 6.1.3) and dilute to 1 L with water. Prepare a fresh solution daily.

6.3.4 *Methyl-2-benzothiazolone Hydrazone Solution* (MBTH solution), 0.4 % mass, aqueous. Dissolve 400 ± 1 mg of MBTH hydrochloride (**Warning—**See 6.1.6) in water in a 100-mL volumetric flask. Make up to the mark and mix thoroughly. Prepare a fresh solution daily.

6.4 *Reagents,* as described in Test Method D2194, for the standardization of formaldehyde solution (see 6.3.1).

6.4.1 *Sodium Sulfite Solution,* 12.5 % mass, aqueous. Dissolve 125 g of anhydrous sodium sulfite (Na_2SO_3) in water and dilute to 1 L.

NOTE 3—Sodium sulfite gradually oxidizes to sodium sulfate on exposure to air and therefore should be kept in a tightly closed container. For best results freshly prepared reagent should be used.

6.4.2 *Sulfuric Acid,* 0.5 mol/L (1.0 N). Prepare and standardize sulfuric acid (H_2SO_4) against 0.5 mol/L (0.5 N) sodium hydroxide (NaOH) solution which has been standardized against potassium acid phthalate to a thymolphthalein end point.

6.4.3 *Thymolphthalein Indicator,* 0.1 % mass, alcohol solution. Dissolve 1.0 g of thymolphthalein in 100 mL of methanol, ethanol, or isopropanol and dilute to 1 L with additional alcohol.

6.5 *Ethylene Glycol Quality Control Sample*—It is recommended to select an Ethylene Glycol sample (fiber grade MEG, DEG or TEG), similar to the product being analyzed and to use it as a quality control sample (**Warning—**See 7.2). To this end, ensure to obtain a sufficient amount and store it in such a way that it is stable for a known period of time and use it as such during this period of time only. For more details, see 11.8.

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 *Ethylene Glycol*—Although (mono-, di- and tri-) ethylene 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 case of contact with eyes rinse with plenty of water and seek medical advice.

8. Sampling, Test Specimens, and Test Units

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

9. Preparation of Apparatus

9.1 *Spectrophotometer*—Check the performance of the spectrophotometer at regular intervals according to the guidelines given in Practice E275 and the manufacturer's instruction manual.

9.2 *Glassware*—Thoroughly clean the cells and other glassware using 50 % (V⁄V) hydrochloric acid (**Warning—See** 6.1.3) according to the guidelines in Practice E275. If the glassware is persistently stained by iron (III) chloride, clean the affected glassware in a concentrated hydrochloric acid bath. Do not use acetone or methanol for cleaning, since this reacts with MBTH.

10. Calibration

10.1 Introduce the amounts of reagents as listed in Table 1 into separate 100-mL volumetric flasks (see 9.2) in the following way: First introduce the standard formaldehyde solution and water by means of a volumetric pipette (5.5.2), followed by MBTH solution.

10.2 Proceed as described in 11.5 through 11.7, measuring the absorbances of the calibration solutions, including the blank solutions, against water at 635 nm in a 10-mm cell.

10.3 If the mean blank absorbance exceeds its upper control limit, clean the glassware and repeat the calibration (see 11.8.3).

10.4 Calculate the exact amount, in µg, of formaldehyde present in each of the calibration solutions, which will be in the order of 0.0, 8.0, 16.0, 24.0, 32.0 and 40.0 µg.

10.5 Construct a calibration graph by plotting the absorbances against the corresponding amount of formaldehyde in each calibration solution. Apply simple linear regression to obtain the best straight line (calibration function).

10.6 Evaluate the linearity of the calibration function obtained by calculating the correlation coefficient and/or by plotting the y-residuals versus the known amounts and visually evaluate the results.

NOTE 4—Many spectrophotometers have the ability to calculate a calibration graph automatically after measuring the calibration solutions and subsequently to show the amount/concentration of the component being measured directly on a display. In such cases, no calibration graphs need to be constructed and the calculation can be started with 12.2. It is, however, recommended to verify the calculation procedure of the instrument and to establish the characteristics of the calibration graph according to suitable regression analysis software.

10.7 *Verification*—The entire calibration graph is based on only one starting solution. In such a case, a weighing error, for example, could introduce significant errors when reading concentrations in test solutions. It is, therefore, recommended to either:

TABLE 1 Amounts of Reagents to be Added to the Calibration Solutions

Calibration Solution, No.	Standard Formaldehyde Solution, mL	Water, mL	MBTH Solution, mL
Blank 1	0.0	5.0	5.0
Blank 2	0.0	5.0	5.0
Blank 3	0.0	5.0	5.0
1	1.0	4.0	5.0
2	2.0	3.0	5.0
3	3.0	2.0	5.0
4	4.0	1.0	5.0
5	5.0	0.0	5.0

10.7.1 Prepare a separately weighed control solution with an accurately known amount of approximately 20 µg of formaldehyde and perform 10.2, measuring the absorbance of the control solution. Obtain the amount of formaldehyde of the control solution from the calibration graph/function. If this value and the calculated value of the control solution differ by more than the confidence limits for the control solution, as derived from the confidence limits for intercept and slope of the calibration function, repeat the calibration.

10.7.2 Maintain a record or control chart of the slope of the calibration graph. If a significant difference with earlier values is obtained, repeat the calibration.

11. Procedure

11.1 Place a 100-mL volumetric flask onto the balance and tare or record the tare weight to the nearest 0.1 mg. Pipet 1 mL of sample into the flask, reweigh the flask and record the increase in weight as the mass of the test portion (m_2).

11.2 Using a pipette, add 4 mL water to the sample flask to make a total volume of 5 mL in each flask.

11.3 Prepare a blank solution in the same way by taking 5 mL of water in place of the prescribed volumes of test sample and water.

11.4 Using a pipette, add 5 mL of MBTH solution (see 6.3.4) into each 100-mL flask.

11.5 Thoroughly swirl the flasks and allow to stand for 30 min at room temperature. Pipette 25 mL of iron (III) chloride solution (see 6.3.3) into each of the flasks. Again mix well by thorough swirling and allow to stand for exactly 5 min at room temperature. Make up to the mark with acetone or methanol (**Warning—**See 6.1.1 and 6.1.5) and homogenize.

11.6 Measure the absorbances of the test solutions relative to water, at 635 nm in a 10-mm cell.

11.7 If the blank absorbance exceeds its upper control limit (see 11.8.3), clean the glassware and repeat the procedure.

NOTE 5—Repeat the procedure when a test solution reads an absorbance less than 0.200 by increasing the sample size in 11.1 to 4 mL and reducing the amount of water to 1 mL. Do not use more than 5 mL of sample. Repeat the procedure when a test solution reads an absorbance greater than 0.800. Decrease the test portion to give an approximate reading of 0.5, and follow 11.1 through 11.7. Do not simply dilute the test solution with acetone (or methanol) and re-measure the absorbance; absorbance does not correlate linearly with serial dilution for this technique.

11.8 *Quality Control*—Although the procedure above is described such that only one test result is obtained, it is recommended to either:

11.8.1 Perform a second (duplicate) determination, to enable comparison of the duplicate results with the listed repeatability limit in Table 2. Choose this option if this test method is performed on an infrequent basis.

11.8.2 Use statistical quality control (SQC) principles in order to monitor its state of in-control, of which a summary is given below. For more detailed guidance, refer to Practice D6299. Choose this option if this test method is performed on a regular basis.

11.8.2.1 Analyze the QC sample under intermediate precision conditions and construct a control chart for aldehydes.

11.8.2.2 While testing regular samples, gather new SQC data. Maintain the control chart and evaluate the data according to the rules supplied. In short, if the measured value is within the control chart action limits and part of a random data pattern, the system can be considered in statistical control. If the measured value exceeds an action limit or belongs to a non-random data pattern, this is an indication of the system being out of statistical control. In that case, investigate for the root cause and take remedial action(s) to eliminate this. Next, reanalyze the QC sample to verify the system is in statistical control again, before proceeding with sample tests.

11.8.2.3 Continue to analyze the QC sample on a regular basis. The frequency depends on the criticality of the test.

11.8.2.4 From SQC data obtained under statistical control, calculate the intermediate precision. Compare this value with the intermediate precision limit as included in Table 2.

11.8.3 It is recommended to maintain a record or control chart of the blank solutions, measured at the same time as the control sample. Use the control chart to monitor the mean and standard deviation of the blank solutions. If an individual blank measurement falls outside the action limits of the control chart (3 standard deviations of the mean), but in no case by more than 0.07 absorbance units, acid-clean all the glassware and reanalyze the batch of samples.

12. Calculation

12.1 From the calibration graph or calculated calibration function (10.5), obtain the amount of formaldehyde (m_3), in µg, present in the test solution.

12.2 Calculate the total aldehyde content of the test sample by means of one of the following equations:

$$\text{Aldehyde (as formaldehyde), mg/kg} = \frac{m_3}{m_2} \quad (2)$$

$$\text{Aldehyde (as acetaldehyde), mg/kg} = \frac{44.05}{30.03} \times \frac{m_3}{m_2} \quad (3)$$

where:

m_3 = amount of formaldehyde as obtained in 12.1, µg, and
m_2 = mass of test portion from 11.1, g.

TABLE 2 E2313 Aldehydes in MEG, DEG, and TEG

Test Result mg/kg	Sample	Average over all Laboratories	Repeatability Standard Deviation	Intermediate Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Intermediate Limit	Reproducibility Limit
Aldehydes	MEG	4.22	0.12	0.23	1.30	0.35	0.64	3.63
Aldehydes	DEG	29.69	0.79	1.06	11.42	2.20	2.96	31.98
Aldehydes	TEG	169.29	4.01	12.86	63.66	11.22	36.01	178.24

13. Report

13.1 Report the total aldehyde content of the sample, expressed as either acetaldehyde or formaldehyde, to the nearest 0.1 mg/kg.

14. Precision and Bias[5]

14.1 In 2007, Committee E15 on Industrial and Specialty Chemicals conducted and completed Interlaboratory Study #52 to determine precision data for six test methods used in the analysis of glycols. The precision of this test method is based on the interlaboratory study of E2313, Standard Test Method for Aldehydes in Mono-, Di-, and Triethylene Glycol (Spectrophotometric Method). Each of seventeen laboratories were asked to test three different materials. 17 laboratories tested MEG, 13 laboratories tested DEG and 12 laboratories tested TEG. Every test result represents an individual determination. Two test results were conducted on each of two days for a total of four test results per assay. Note that in the combined study, 8 labs used a single analyst, 7 labs used two analysts (on different days) and 2 labs did not record this information. In the event that there were missing values for one or more labs, this information was noted in the results. The details of this study are given in ASTM Research Report No. RR:E15-1065.

14.1.1 *Repeatability*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the r value for that material; r is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

14.1.2 *Reproducibility*—Two test results shall be judged not equivalent if they differ by more than the R value for that material; R is the interval representing the difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

14.1.3 *Intermediate Precision*—The day-to-day standard deviation within a laboratory for results produced by the same operator, determined through statistical analysis following Practice E180. Practice E180 was used to conform to this particular study design which required an estimate of intermediate precision. The statistical analysis was conducted using the SAS statistical analysis software, Version 8.0.

14.1.3.1 The Practice E180 analysis considers the two test results from each day as being run under repeatability conditions and estimates the repeatability, intermediate, and reproducibility precision for each assay. The repeatability precision would be estimated from the two sets of duplicate test results within each day, and the intermediate precision would be estimated from the agreement between the two days, all pooled over laboratories. Caveat: Since two days is a short time period, the intermediate precision would probably be underestimated by the Practice E180 analysis.

14.1.4 Any judgment in accordance with these two statements would have an approximate 95 % probability of being correct.

14.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

14.3 The precision statement was determined through statistical examination of qualified results, from seventeen laboratories, on three materials. These three materials were described as the following: Fluid 1: Monoethylene glycol. Fluid 2: Diethylene glycol. Fluid 3: Triethylene glycol. To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

15. Keywords

15.1 aldehydes; diethylene glycol; MBTH; monoethylene glycol; spectrophotometric; triethylene glycol

SUMMARY OF CHANGES

Committee E15 has identified the location of selected changes to this standard since the last issue (E2313-08) that may impact the use of this standard. (Approved Nov. 1, 2015.)

(1) The title was revised.
(2) Revisions were made in the following sections: 1 – 5, 6.1.5, 6.4.2, 6.5, 9.2, 10 – 13. Calibration, Procedure, and Calculation updated.
(3) Section 7 Hazards was added.
(4) Tables 1 and 2 were revised.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E15-1065.

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