Designation: E2409 – 13

Standard Test Method for Glycol Impurities in Mono-, Di-, Tri- and Tetraethylene Glycol and in Mono- and Dipropylene Glycol (Gas Chromatographic Method)[1]

This standard is issued under the fixed designation E2409; 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 gas chromatographic determination of glycol impurities in Mono-, Di- Tri- and Tetraethylene Glycol (MEG, DEG, TEG and TeEG) in the range of 5 to 3000 mg/kg, and in Mono- and Dipropylene Glycol (MPG and DPG) in the range 0.01 to 2.5 % (m/m).

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

1.3 Review the current Material Safety Data Sheets (MSDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

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]

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E300 Practice for Sampling Industrial Chemicals

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

2.2 *Other Document:*

Manufacturers' instruction manuals of gas chromatograph and digital integration system used.

3. Summary of Test Method

3.1 A portion of the test sample is analyzed by temperature-programmed, capillary gas chromatography over a polyethylene glycol column, using flame ionization detection. For quantification, the External Standard Technique or the Internal Standard (Marker) Technique are applied. When applying the Internal Standard Technique, the standard addition technique is used to eliminate the effect of other impurities present in the glycols. For this purpose, a blank glycol is used, as 100 % pure glycol samples are not available.

4. Significance and Use

4.1 Knowledge of the impurities is required to establish whether the product meets the requirements of its specifications.

5. Apparatus

5.1 *Gas Chromatograph(s),* provided with a sample splitter or on-column injection, flame ionization detector and temperature-programming facilities. Optional are pressure programming and auto sampler facilities. The instrument must be suitable for analysis according to the operating instructions given in Table 1 or Table 2.

5.1.1 *Columns*—The analytical column (chemically bonded cross-linked polyethylene glycol) used must completely separate

MEG, DEG, TEG, TeEG, PentaEG (Penta-ethylene Glycol) and 1,4-butanediol, or

MPG, DPG, TPG, and TePG

Figs. A1.1 through A1.5 show examples of chromatograms conforming to the requirements.

5.2 *Digital Integration Equipment.*

5.3 *Analytical Balance,* readability 0.1 mg, calibrated. Recalibrate or verify at regular intervals.

5.4 *Crimp Top Vials,* 1 mL and 5 mL.

5.5 *Crimper/De-capper,* for capping and de-capping the vials.

5.6 *Micro Syringes,* 10 µL.

5.7 *Bottles,* 50 mL, with screw cap.

[1] This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.02 on 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**

TABLE 1 Typical Operating Parameters for the GC Analysis of Glycol Impurities in MEG, DEG, TEG or TeEG

Column[A]	
Type	Capillary wide-bore
Material	Fused silica
Length × I.D.	15 m × 0.53 mm
Stationary Phase	Polyethylene glycol, for example, DB-Wax
Film Thickness	1 µm
Detector System	
Type	FID
Sensitivity	The ratio of the signal to the noise level must be at least 2:1 at a concentration of 5 mg/kg DEG in MEG
Temperatures	
Column Oven	0.05 min at 70°C Programmed from 70 to 230°C at 25°C/min 10 min at 230°C
Detector	250°C
Carrier Gas	Helium or Nitrogen
Calibration	see Section 9
Injected Volume	0.2 µL (on-column injection), or 0.5 µL up to 1 µL (using split injection technique)
Split Ratio	1:10 or appropriate split ratio to allow adequate sensitivity as defined under Detector System (only if split injection technique is used)

[A]The mentioned DB-Wax column is available from Agilent Technologies, 5301 Stevens Creek Blvd, Santa Clara, CA 95051, USA. Other column suppliers market equivalent stationary phases under trade names, therefore, it is permissible to use a different column from an alternative supplier. However, the chromatogram obtained must be identical, with regard to separation of the glycol components and 1,4-butanediol, to those illustrated in Figs. A1.1, A1.2, and A1.3, or A1.4 and A1.5.

TABLE 2 Typical Operating Parameters for the GC Analysis of Glycol Impurities in MPG or DPG

Column[A]	
Type	Capillary wide-bore
Material	Fused silica
Length × I.D.	30 m × 0.32 mm
Stationary Phase	Poly ethylene glycol, for example, DB-Wax
Film Thickness	0.5 µm
Detector System	
Type	FID
Sensitivity	The ratio of the signal to the noise level must be at least 2 to 1 at a concentration of 0.01 % (m/m) DPG in MPG
Temperatures	
Column Oven	5 min at 150°C Programmed from 150 to 180°C at 5°C/min 0 min at 180°C Programmed from 180 to 240°C at 30°C/min 10 min at 240°C
Detector	300°C
Carrier Gas	Helium
Calibration	see Section 9
Injected Volume	0.1 µL or 0.5 µL (using split injection technique)
Split Ratio	1 to 10 or appropriate split ratio to allow adequate sensitivity as defined under Detector System

6. Reagents and Materials

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.

6.2 *Calibration Standards:*

6.2.1 *Mono-ethylene Glycol* (MEG), minimum purity 99.5 % mass (m/m).

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

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

6.2.4 *Tetra-ethylene Glycol* (TeEG), of maximum purity available.

6.2.5 *Penta-ethylene Glycol* (PentaEG), of maximum purity available, or

6.2.6 *Mono-propylene Glycol* (MPG), minimum purity 99.5 % mass (m/m).

6.2.7 *Di-propylene Glycol* (DPG), minimum purity 99.5 % mass (m/m).

6.2.8 *Tri-propylene Glycol* (TPG), of maximum purity available.

6.2.9 *Tetra-propylene Glycol* (TePG), of maximum purity available.

6.3 *Internal Standard:*

6.3.1 *1,4-Butanediol,* minimum purity 97 % mass (m/m), for ethylene glycols, if necessary.

6.3.2 n-Octane, minimum purity 97 % mass (m/m), for propylene glycols, if necessary.

6.4 *Ethylene Glycol Quality Control Sample,* fiber grade MEG, DEG, TEG or TeEG *or Propylene Glycol Quality Control Sample, MPG or DPG* (only required if maintaining a control chart, see 10.5). Store nitrogen capped at a temperature between 0 and 5°C. Warm to ambient temperature before use.

6.5 *Water,* HPLC grade.

6.6 *Solutions:*

6.6.1 *Internal Standard Solution*—Weigh about 0.15 g 1,4-butanediol (m_1) to the nearest 0.1 mg into a 50 mL bottle. Add ultra-pure water up to a total mass of 50 g (m_2), weighing to the nearest 0.1 mg. Calculate the concentration of this solution to the nearest 0.1 mg/kg, or

6.6.2 *External Standard Solution,* of accurately known
MEG, DEG, TEG, and TeEG content, or
MPG, DPG, TPG, and TePG content,
to be used as external standard (see 9.4).

[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. (USPC), Rockville, MD.

7. Sampling, Test Specimens, and Test Units

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

8. Preparation of Apparatus

8.1 *Gas Chromatograph(s) and Column(s)*—Check the performance of the gas chromatograph and column as described in Section 9.

9. Calibration and Standardization

9.1 Two methods of quantification may be employed: the Internal Standard (Marker) Technique or the External Standard Technique.

9.2 *Internal Standard Technique for Ethylene Glycols:*

9.2.1 Prepare calibration solutions, containing 500, 1000 and 2000 mg/kg of each of the glycol components to be determined, by adding the relevant calibration standard (see 6.2) to a blank sample of the glycol being analyzed. Calculate the exact concentration of each glycol component (c_1) in the calibration solutions.

9.2.2 Weigh 0.5 g of each calibration solution (m_3) to the nearest 0.1 mg, into separate 5-mL vials. Add, also weighed to the nearest 0.1 mg, 0.5 g internal standard solution (see 6.6.1; m_4) and add HPLC grade water up to a total mass of approximately 5 g. Cap the vials and mix thoroughly.

9.2.3 Prepare a blank calibration solution by weighing 0.5 g blank sample of the glycol being analyzed (m_5), weighed to the nearest 0.1 mg, into a 5-mL vial. Add 0.5 g internal standard solution (see 6.6.1; m_6), also weighed to the nearest 0.1 mg, and add HPLC grade water up to a total mass of approximately 5 g. Cap the vial and mix thoroughly.

9.2.4 Calibrate separately for each impurity in MEG, DEG, TEG or TeEG by using the Internal Standard (Marker) Technique.

9.2.5 Fill a 1-mL sample vial with the calibration solution from the 5-mL vial (see 9.2.2 and 9.2.3). Close the vial by means of an aluminum crimp cap.

9.2.6 Analyze each calibration solution and the blank solution using the operating parameters given in Table 1. Inject each solution at least twice and calculate the average peak areas for each of the calibration solutions. Apply digital integration equipment for measuring the peak areas.

9.2.7 For each chromatogram, calculate the system response factor (f) of each of the components as described in 9.2.8 through 9.2.10.

9.2.8 Calculate the amount of internal standard (1,4-butanediol) added to the calibration solution:

$$\text{Mass of Internal Standard } (m_7),\ \text{g} = \frac{m_4 \times m_1}{m_2} \quad (1)$$

where:

m_1 = mass of 1,4-butanediol in internal standard solution (6.6.1), g,
m_2 = total mass of internal standard solution (6.6.1), g, and
m_4 = mass of internal standard solution added, g.

9.2.9 Calculate the amount of internal standard (1,4-butanediol) added to the blank solution:

$$\text{Mass of Internal Standard } (m_8),\ \text{g} = \frac{m_6 \times m_1}{m_2} \quad (2)$$

where:

m_6 = mass of internal standard solution added (9.2.3), g.

9.2.10 Calculate the response factor of each component of interest in the calibration solutions by means of the following equation:

$$f = \frac{c_1 \times 10^{-6}}{\left(\frac{m_7 \times A_1}{m_3 \times A_2}\right) - \left(\frac{m_8 \times A_3}{m_5 \times A_4}\right)} \quad (3)$$

where:

c_1 = added concentration of glycol compound in the calibration solution, (9.2.1), mg/kg,
A_1 = peak area of component in calibration solution, arbitrary units,
A_2 = peak area of internal standard in calibration solution, same arbitrary units,
A_3 = peak area of component in blank solution, same arbitrary units,
A_4 = peak area of internal standard in blank solution, same arbitrary units,
m_3 = mass of calibration solution (9.2.2), g,
m_5 = mass of blank solution (9.2.3), g,
m_7 = mass of internal standard in calibration solution, as obtained in 9.2.8, g, and
m_8 = mass of internal standard in blank solution, as obtained in 9.2.9, g.

9.2.11 Calculate the mean of the response factors. If the individual factors differ by more than 5 % from the mean response factor, repeat the measurement of the respective calibration solution.

9.3 *Internal Standard Technique for Propylene Glycols:* Calibrate by determining the calibration factor for each component of interest relative to the internal standard on the basis of peak area versus mass as follows:

9.3.1 Prepare a calibration solution by accurately weighing 0.5 g of each of the components of interest and of the internal standard, to the nearest 0.1 mg into a previously tarred, 50 mL bottle. Fill the bottle with a suitable solvent (for example, acetone/cyclohexane), close, and reweigh to the nearest 0.1 mg. Homogenize the calibration solution.

9.3.2 Analyze the calibration solution following the operating parameters given in Table 2. Introduce the calibration solution at least twice. Determine the areas of the components of interest and the reference component.

9.3.3 Calculate the mean peak areas of the components of interest for the calibration solution. If the two single peak areas differ by more than 3 % relative, repeat the analysis. If no satisfactory results can be obtained, stabilize the conditions and repeat 9.3.1 and 9.3.2.

9.3.4 Calculate the calibration factor (f_I) for all individual compounds, relative to the internal standard, by means of the following equation:

$$f_i = \frac{m_i \times A_m}{m_m \times A_i} \quad (4)$$

where:

m_i = mass of component i in calibration solution (9.3.1), g.
m_m = mass of internal standard in calibration solution (9.3.1), g.
A_i = peak area of component i (9.3.3), arbitrary units.
A_m = peak area of internal standard (9.3.3), same arbitrary units.

NOTE 1—An alternative for the empirical calibration factors as described in 9.2 and 9.3 is the use of theoretical factors, based on the molecular structure of the compounds of interest. Theoretical factors calculated are as follows: For MPG 3.045, for all DPG isomers 2.512, for all TPG isomers 2.244, all relative to octane. See Footnote 5.[5]

9.4 *External Standard Technique Ethylene Glycols, similar for Propylene Glycols:*

9.4.1 Prepare at least three calibration solutions, for example, containing 200, 500 and 1000 mg/kg of each of the glycol components to be determined, by adding the relevant calibration standard (see 6.2) to a blank sample of the glycol being analyzed and mix thoroughly. Weigh each glycol component to the nearest 0.1 mg and the blank glycol to the nearest 0.1 g. (See Table 3 for recommended weights.)

9.4.1.1 Calculate the exact concentration of each glycol component (C_i) in the calibration solutions. The calibration range can be adjusted if needed.

$$C_i = \frac{W_{(Comp;i)}}{W_{(Comp;i)} + W_{(Blank;i)}} \times \frac{10^6\ \mu g}{g} \quad (5)$$

where:

C_i = the concentration of each glycol component in the calibration standard of interest,
i = the calibration standard of interest,
$W_{(Comp;i)}$ = mass (g) of glycol component added to the calibration standard of interest, and
$W_{(Blank;i)}$ = mass (g) of blank glycol added to the calibration standard of interest.

9.4.2 Analyze each calibration solution and the blank solution using the operating parameters given in Table 1 or Table 2. Inject each solution at least twice.

9.4.2.1 Prepare a plot of area counts of the glycol component (*y*-axis) versus the concentration of the glycol component (mg/kg) added to the standard of interest (*x*-axis). Using a computer program, determine the best-fit line through the data using linear regression analysis. The relationship between concentration and peak area will be linear. Record the intercept value (concentration; mg/kg) where the resulting line crosses the *x*-axis (y = 0). Apply digital integration equipment for measuring the peak areas.

[5] Sternberg, J.C. *Gas Chromatography*, Academic Press, New York, 1962; pp. 231-267.

TABLE 3 External Standard Recommended Weights

Standard #	Target Weight of Glycol Component, ± 0.0001 g	Target Weight of High Purity Blank Glycol, ± 0.1 g
200 mg/kg	0.010	50
500 mg/kg	0.025	50
1000 mg/kg	0.050	50

9.4.2.2 Calculate the corrected concentration (mg/kg) of the glycol component in each calibration standard as follows:

$$\text{Corrected } C_i = C_i + Y \quad (6)$$

where:

Corrected C_i = the corrected concentration (mg/kg) of the glycol component in each calibration standard of interest,
C_i = the concentration (mg/kg) of the glycol component added to the calibration standard of interest, and
Y = absolute value of the concentration of blank glycol determined from the linear regression graph (intercept value) for each calibration standard.

9.4.3 For each chromatogram, calculate the system response factor *(f)* of each of the glycol components by means of the following equation:

$$f = \frac{\text{Corrected } C_i}{A_i} \quad (7)$$

where:

Corrected C_i = concentration of component in external standard solution, mg/kg, and
A_i = peak area of component, arbitrary units.

9.4.3.1 Calculate the mean of the response factors for each of the glycol components. If the individual factors differ by more than 5 % from the mean response factor, repeat the measurement of the respective calibration solution.

NOTE 2—Many gas chromatographs have the ability to calculate a calibration graph automatically after measuring the calibration solutions and subsequently to show the concentration of the component being measured directly on a display. In such cases, no calibration graphs need to be constructed. It is, however, recommended to verify the calibration procedure of the instrument and to establish the characteristics of the calibration graph according to suitable regression analysis software.

10. Procedure

10.1 *Internal Standard Technique for Ethylene Glycols:*

10.1.1 Weigh a test portion of 0.5 g (m_9), weighed to the nearest 0.1 mg, into a 5-mL vial.

NOTE 3—This method is for the determination of glycol impurities in the range of 5 to 3000 mg/kg. Higher levels of glycol impurities (< 5000 mg/kg) can be determined, if the intake is adjusted as follows:

$$\text{Mass Intake Sample, g} = \frac{2000}{c} \times 0.5 \quad (8)$$

where:

c = the expected maximum concentration of component in the sample, mg/kg.

10.1.2 Add 0.5 g internal standard solution (m_{10}), weighed to the nearest 0.1 mg, and add HPLC grade water up to a total mass of approximately 5 g and weigh. Close the vial and mix thoroughly.

10.1.3 Fill a 1-mL sample vial with the test solution. Close the vial by means of an aluminum crimp-cap.

10.1.4 Analyze the test solution using the operating parameters given in Table 1. Examples of the chromatograms are

shown in Figs. A1.1, A1.2 and A1.3. Apply digital integration equipment for measuring the area of the peaks.

10.2 *Internal Standard Technique for Propylene Glycols:*

10.2.1 Prepare a test solution by weighing 100 µL of internal standard to the nearest 0.1 mg into a previously tarred, 50 mL bottle. Fill the bottle with test sample, close and reweigh to the nearest 0.1 mg. Homogenize the test solution. Calculate the concentration of the internal standard in the test solution.

10.2.2 Analyze the test solution following the operating parameters given in Table 2. Apply the chromatography data system for measuring the areas of the peaks of interest. An example of a chromatogram is given in Fig A1.4.

10.3 *External Standard Technique*—When applying the external standard technique, analyze the test sample using the operating parameters given in Table 1 or Table 2. Sample dilution, with the sample glycol, may be necessary if the component of interest is beyond the range of the method. Apply digital integration equipment for measuring the area of the peaks.

10.4 *Determination of Water Content*—If it is required to calculate and report the purity of the sample, determine the water content in % mass (m/m) according to Test Method E1064.

10.5 *Quality Control*—It is recommended that a control chart for the concentration of the impurities in the glycol quality control sample be established and maintained according to generally accepted guidelines.[6] Measure the control sample each time a test sample(s) is tested, using the same calibration procedure as applied for the sample. If the measured value exceeds the action limit of the control chart, take appropriate action before proceeding with sample tests.

11. Calculation

11.1 *Internal Standard Technique (Ethylene Glycol):*

11.1.1 Calculate the amount (m_{11}) of internal standard (1,4-butanediol) added to the test sample by means of the following equation:

$$\text{Mass of Internal Standard } (m_{11}),\ \text{g} = \frac{m_{10} \times m_1}{m_2} \quad (9)$$

where:

m_{10} = mass of internal standard solution added, (10.1.2), g,
m_1 = mass of 1,4-butanediol in internal standard solution (6.6.1), g, and
m_2 = total mass of internal standard solution (6.6.1), g.

11.1.2 Calculate the concentration of each component of interest in the sample by means of the following equation:

$$\text{Glycol Component, mg/kg} = \frac{f \times m_{11} \times A_5}{m_9 \times A_6} \times 10^6 \quad (10)$$

where:

A_5 = peak area of the component, arbitrary units,
A_6 = peak area of internal standard, same arbitrary units,
f = relative response factor of component of interest, as obtained in 9.2.11,
m_9 = mass of sample, that is, without internal standard, (10.1.1), g, and
m_{11} = mass of internal standard in test solution, as obtained in 11.1.1, g.

NOTE 4—If the concentration of the calculated glycol component is required to be expressed in % mass (m/m), divide the result obtained above by a factor of 10^4.

11.2 *Internal Standard Technique (Propylene Glycol):*

11.2.1 Calculate the concentration of each component of interest by means of the following equation:

$$\text{Component, mg/kg or \% (m/m)} = f_i \times \frac{A_j}{A_{is}} \times C_{is} \quad (11)$$

where:

f_i = calibration factor for relevant component as obtained in 9.3.4, or theoretical factor (see Note 1).
A_j = peak area of relevant component peak in test solution (10.2.2), arbitrary units.
A_{is} = peak area of internal standard in test solution (10.2.2), same arbitrary units.
C_{is} = concentration of internal standard in test solution (10.2.1), mg/kg or % (m/m), whichever is relevant.

11.3 *External Standard Technique:*

11.3.1 Obtain the concentrations of the glycol impurities in the test sample in mg/kg, as presented by the software of the applied gas chromatograph (see Note 2). If an automated system is not being applied, read the concentration of the glycol impurities, in mg/kg, from the respective calibration graph, or

11.3.2 Calculate the concentration of the glycol components, in mg/kg, in the test sample by means of the following equation (see also Note 3):

$$\text{Glycol Component, mg/kg} = A_i \times f \quad (12)$$

where:

A_i = peak area of relevant glycol component, arbitrary units, and
f = the average response factor of component of interest, as obtained in 9.4.3.

11.4 *Purity:*

11.4.1 Calculate the purity of the sample by means of the following equation:

$$\text{Glycol of Interest Purity, \% mass (m/m)} = 100 - O - W \quad (13)$$

where:

O = other glycols, sum content as calculated in 11.1, 11.2, or 11.3, % mass (m/m) of each of the minor glycol components, and
W = water content of the sample, determined by Test Method E1064 (10.4), % mass (m/m).

12. Report

12.1 Report the concentrations of DEG in MEG and MEG in DEG to the nearest mg/kg and all other impurities to the nearest 10 mg/kg. Report the concentrations of DPG and TPG in MPG, and MPG, TPG, and TePG in DPG to the nearest

[6] *ASTM Manual on Presentation of Data and Control Chart Analysis: 7th edition*, ASTM Manual Series MNL 7A.

0.01 % mass (m/m). Report the purity of the sample to the nearest 0.01 % mass (m/m).

13. Precision and Bias

13.1 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 E2409, Standard Test Method for Glycol Impurities in Mono-, Di-, Tri- and Tetraethylene Glycol (Gas Chromatographic Method). Each of seventeen laboratories were asked to test four different materials. 14 laboratories tested MEG in DEG. 16 laboratories tested DEG in MEG. 9 laboratories tested DEG in TEG. 5 laboratories tested DEG in TeEG. 13 laboratories tested TEG in DEG. 5 laboratories tested TEG in TeEG. 10 laboratories tested TTEG in TEG. 4 laboratories tested PentaEG in TeEG. 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. E15-1063.[7]

13.1.1 *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.

13.1.2 *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.

13.1.3 *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.

13.1.3.1 The 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 E180, E1064 analysis.

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

13.2 *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.

13.3 The precision statement was determined through statistical examination of qualified results, from seventeen laboratories, on four materials. These four materials were described as the following: Fluid 1: Monoethylene Glycol. Fluid 2: Diethylene Glycol. Fluid 3: Triethylene Glycol. Fluid 4: Tetraethylene glycol. To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

13.4 Precision for MPG and DPG: to be evaluated.

14. Keywords

14.1 diethylene glycol; dipropylene glycol; gas chromatographic; monoethylene glycol; monopropylene glycol; tetraethylene glycol; tetrapropylene glycol; triethylene glycol; tripropylene glycol

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E15-1063.

TABLE 4 E2409 Glycol Impurities by GC

Test Result mg/kg	Sample	Average over all Laboratories	Repeatability Standard Deviation	Intermediate Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Intermediate Limit	Reproducibility Limit
DEG	MEG	374.59	7.3	7.3	34.0	20.6	20.6	95.3
MEG	DEG	1479.73	46.3	76.0	215.1	129.7	212.9	602.4
TEG	DEG	3499.69	92.8	143.2	306.5	260.0	401.0	858.3
DEG	TEG	489.32	56.8	70.9	201.7	159.1	198.5	564.9
TTEG	TEG	1020.00	96.3	96.3	244.1	269.8	269.8	683.5
DEG	TeEG	1646.25	55.4	55.4	95.4	155.1	155.1	267.1
TEG	TeEG	7908.35	221.9	221.9	1350.7	621.2	621.2	3782.0
PentaEG	TeEG	2084.93	58.7	72.9	156.3	164.5	204.1	437.5

ANNEX

(Mandatory Information)

A1. Example Chromatograms

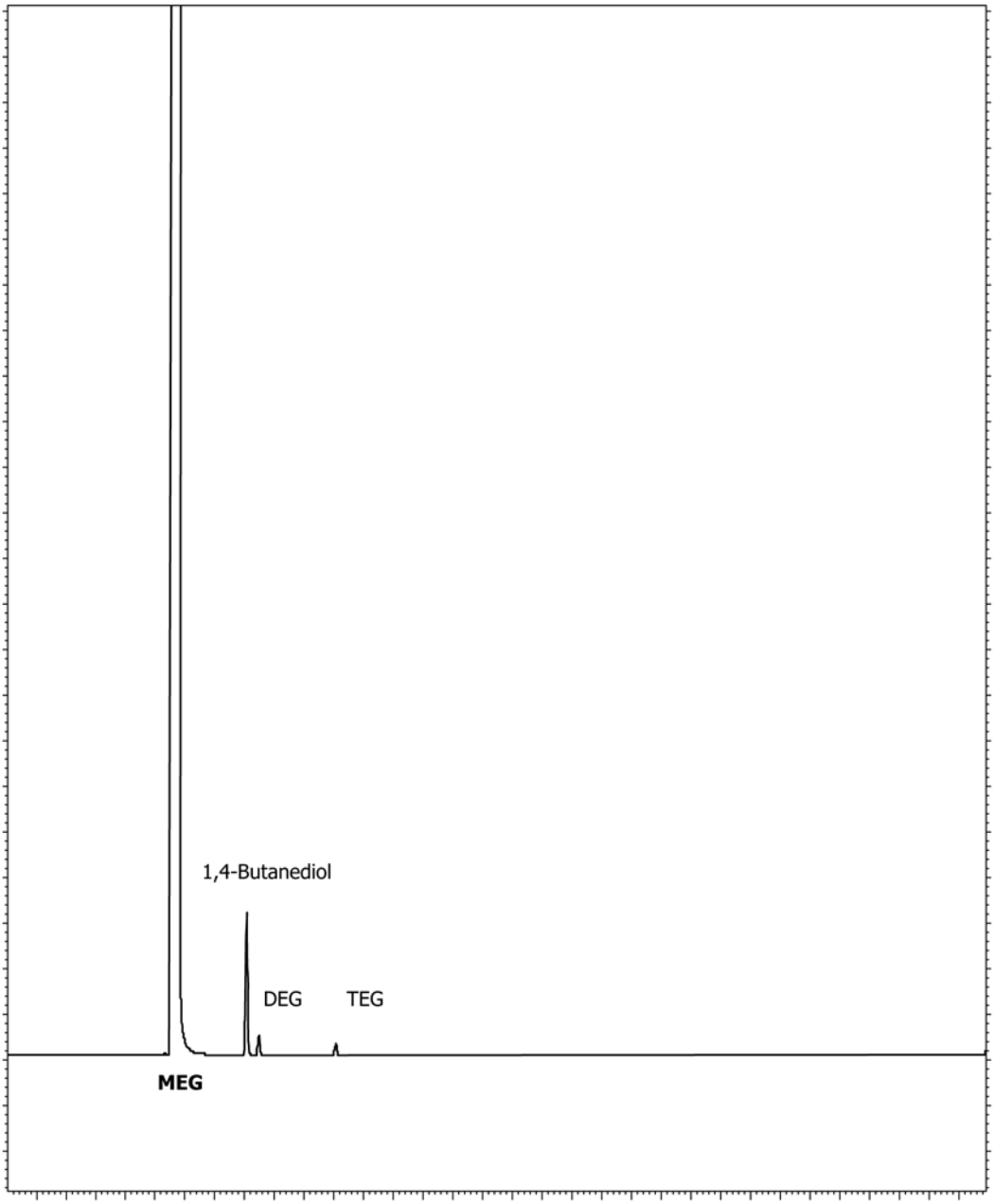

FIG. A1.1 Chromatogram of Glycol Impurities in MEG

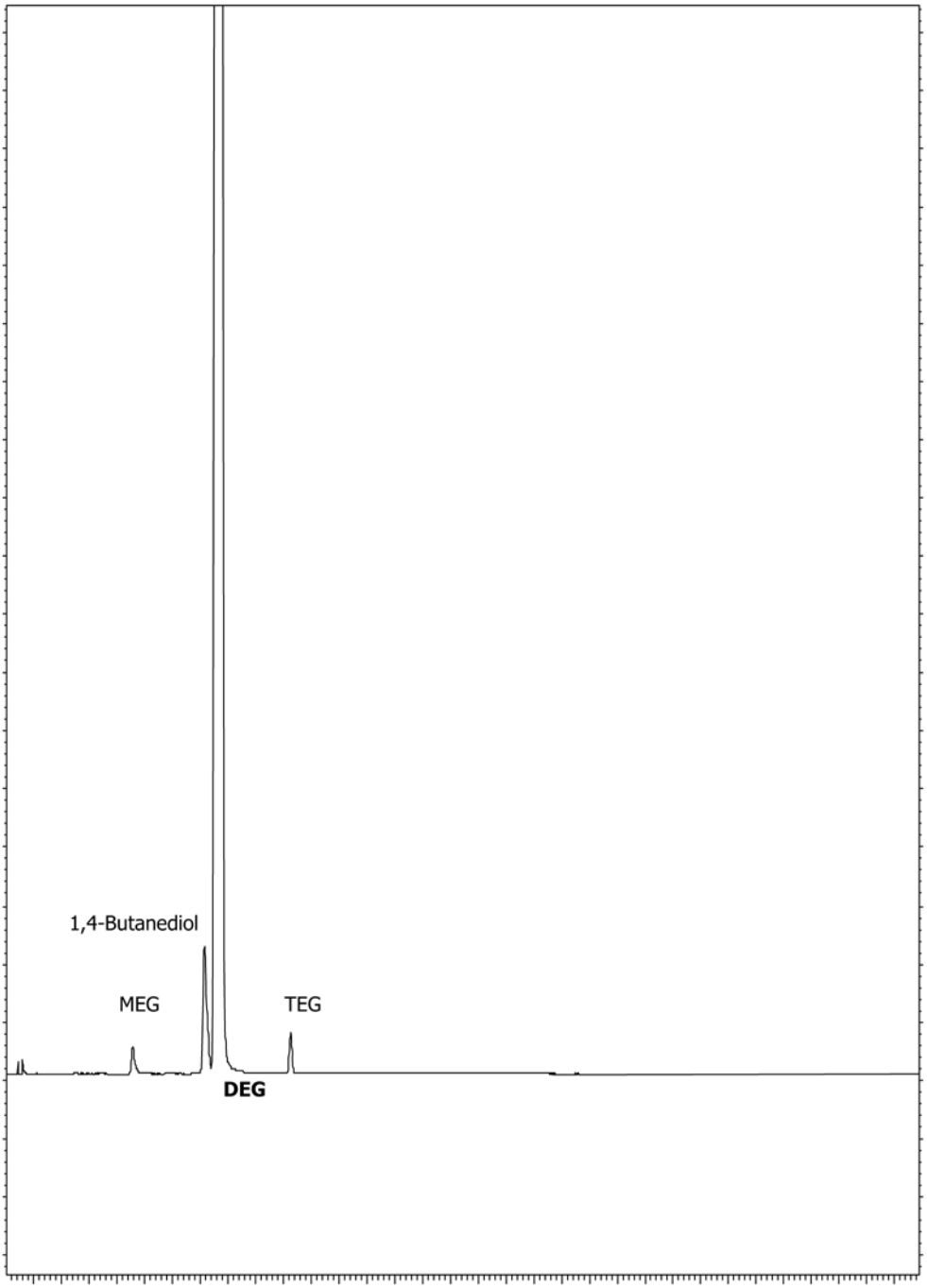

FIG. A1.2 Chromatogram of Glycol Impurities in DEG

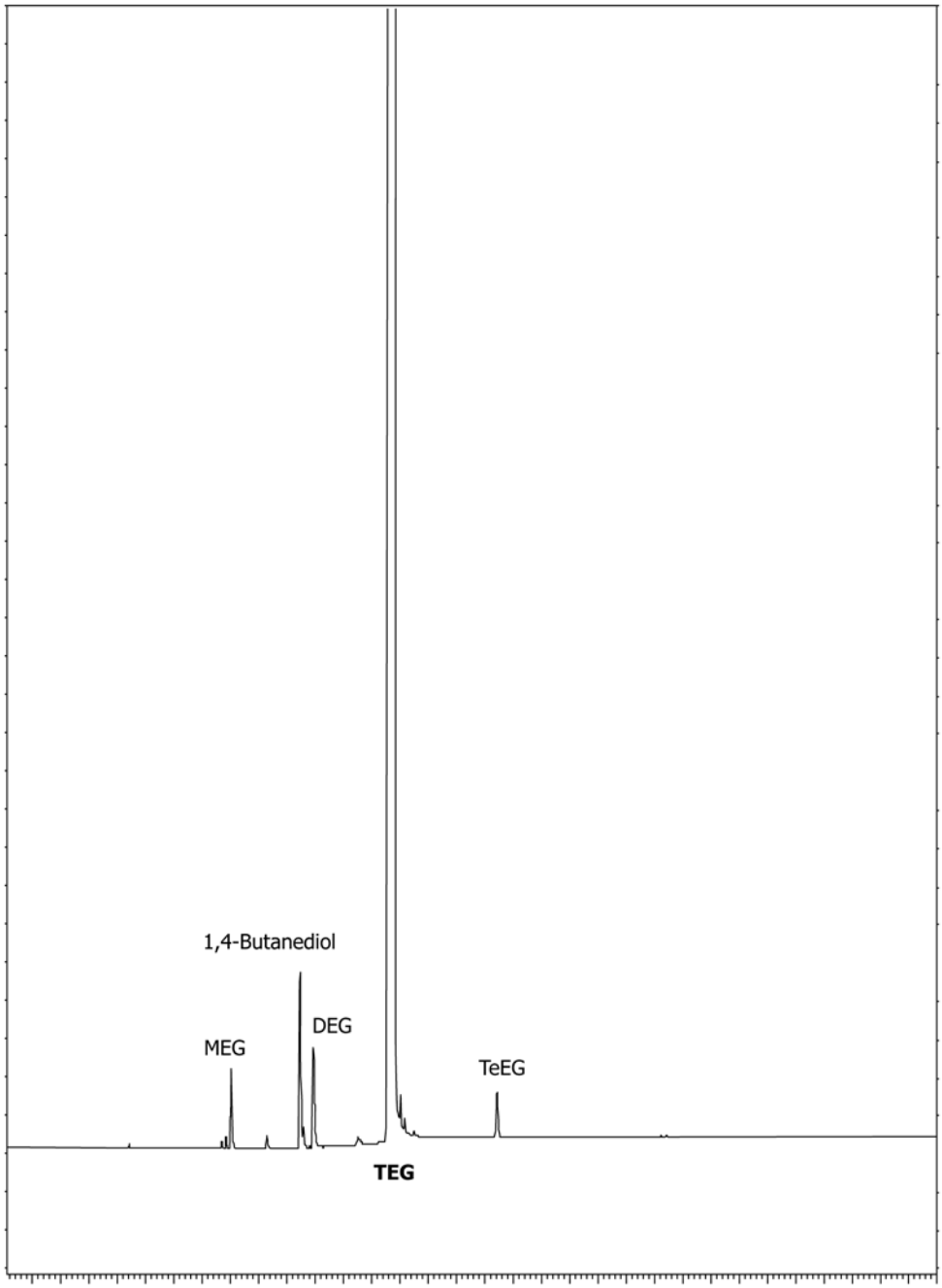

FIG. A1.3 Chromatogram of Glycol Impurities in TEG

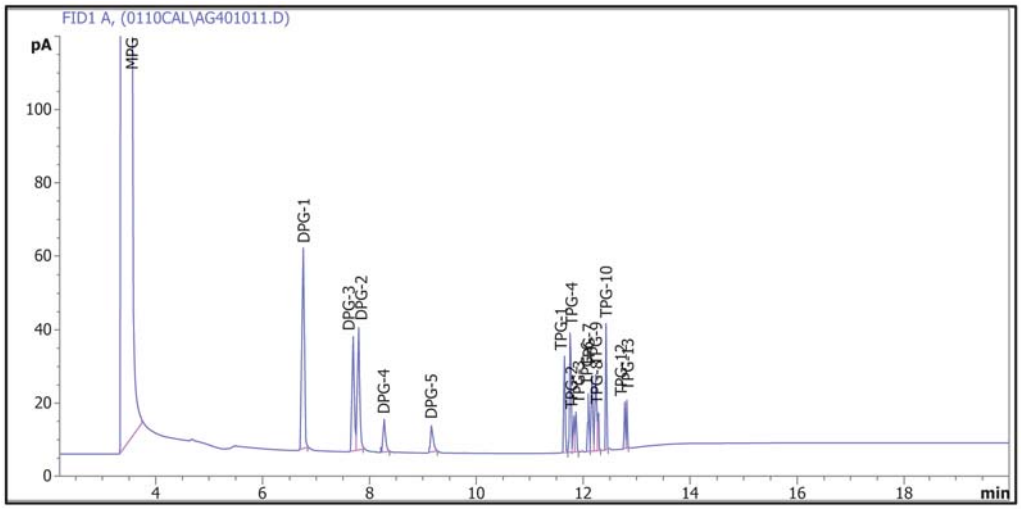

FIG. A1.4 Chromatogram of Glycol Impurities in MPG

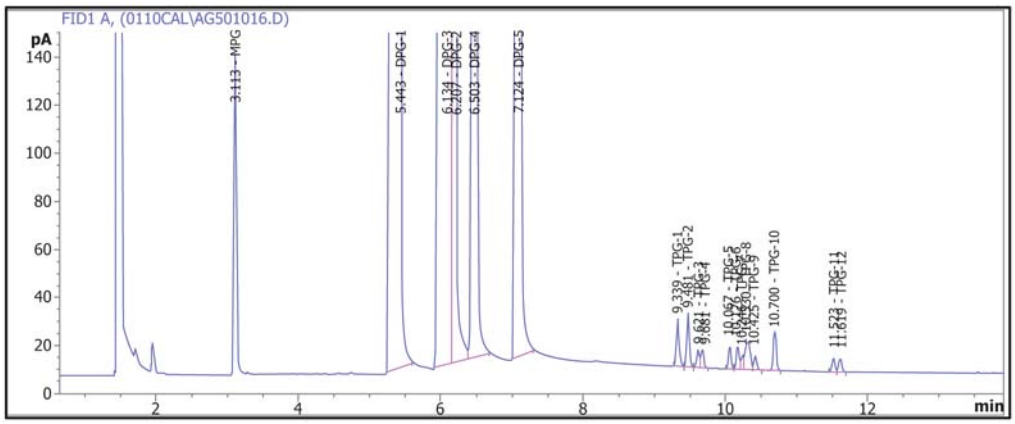

FIG. A1.5 Chromatogram of Glycol Impurities in DPG

SUMMARY OF CHANGES

Committee E15 has identified the location of selected changes to this standard since the last issue (E2409 – 08) that may impact the use of this standard. (Approved June 1, 2013.)

(1) Revised Precision and Bias statement.

(2) Introduction of MPG and DPG as sample types.