Designation: E202 – 18

Standard Test Methods for Analysis of Ethylene Glycols and Propylene Glycols[1]

This standard is issued under the fixed designation E202; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 These test methods cover the chemical and physical analysis of the commonly available grades of ethylene glycol, diethylene glycol, triethylene glycol, propylene glycol, and dipropylene glycol. The key sections appear in the following order:

	Sections
Purity of Reagents	4
Specific Gravity	6 – 8
Distillation Range	10 – 12
Acidity	14 – 16
Water	18 – 20
Iron	22 – 24
Color	26 – 28
Gas Chromatographic Analysis	30 – 32
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1.2 Review the current appropriate Safety Data Sheets (SDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

1.3 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.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard with the exception of foot-pound for apparatus descriptions.

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.*

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

D1078 Test Method for Distillation Range of Volatile Organic Liquids

D1193 Specification for Reagent Water

D1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)

D1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D5386 Test Method for Color of Liquids Using Tristimulus Colorimetry

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]

E203 Test Method for Water Using Volumetric Karl Fischer Titration

E394 Test Method for Iron in Trace Quantities Using the 1,10-Phenanthroline Method

E611 Test Methods for Low Concentrations of Diethlyene Glycol in Ethylene Glycol by Gas Chromatography

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

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

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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**

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs
E1615 Test Method for Iron in Trace Quantities Using the FerroZine Method
E2409 Test Method for Glycol Impurities in Mono-, Di-, Tri- and Tetraethylene Glycol and in Mono- and Dipropylene Glycol(Gas Chromatographic Method)
E2679 Test Method for Acidity in Mono-, Di-, Tri- and Tetraethylene Glycol byNon-Aqueous Potentiometric Titration

2.2 *ASTM Adjuncts:*

Adjunct ADJD6300 Determination of Precision and Bias for Use in Test Methods for Petroleum Products and Lubricants[4]

2.3 *Other Document:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200[5]

3. Significance and Use

3.1 These test methods measure certain chemical and physical properties of ethylene glycols and propylene glycols and may be used to determine compliance with specification in which limits are established for these properties. For those tests that use the procedure of another ASTM test method, that test method should be consulted for additional information on the significance and use of that test.

3.2 Alternative test methods and technology for several of the methods can be found in the Appendix. Use of these methods is optional and individuals using the alternative methods should assure themselves that the method is sufficient and appropriate for the application. Precision data presented in this standard is only for the original test methods listed.

4. Purity of Reagents

4.1 *Purity of Reagents*—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.[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.

4.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type II or III.

5. Quality Control

5.1 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 common guidelines.[7] Measure the control sample each time a test sample(s) is tested. If the measured value exceeds the action limit of the control chart, take appropriate action before proceeding with sample tests.

SPECIFIC GRAVITY

6. Procedure

6.1 Determine the relative density of the sample at 20/20°C using the pycnometer test method in accordance with Test Methods D891, except determine the water and sample weights of the pycnometer at 20.0 ± 0.1°C.

7. Report

7.1 Report the relative density at 20/20°C (in air) to the nearest 0.0001 unit.

8. Precision and Bias

8.1 The following criteria should be used for judging the acceptability of results (see Note 1):

8.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0000651 unit at 96 dF. The 95 % limit for the difference between two such runs is 0.0002 unit.

8.1.2 *Laboratory Precision (Within-Laboratory, Between-Days)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.0000598 units at 48 df. The 95 % limit for the difference between two such averages is 0.0002 unit.

8.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.000191 unit at 5 dF. The 95 % limit for the difference between two such averages is 0.0005 unit.

NOTE 1—These precision estimates are based on interlaboratory studies performed in 1962 and 1963 on six samples of the five glycols whose specific gravity values range from approximately 1.0233 to 1.1255. A total of ten laboratories cooperated in the studies in which each analyst performed duplicate determinations on each sample on each of two days.[8] Practice E180 was used in developing these precision estimates.

8.2 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

9. Quality Guidelines

9.1 Laboratories shall have a quality control system in place.

9.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

9.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

[4] Available from ASTM International Headquarters.

[5] Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov.

[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.

[7] ASTM Manual on Presentation of Data and Control Chart Analysis, 7th Edition, ASTM Manual Series MNL 7A (revision of Special Technical Publication STP 15D.

[8] Supporting data have been filed at ASTM Headquarters and may be obtained by requesting Research Report RR:E15-0013. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Guide E2409 Glycol Impurities by Gas Chromatography (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

9.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

9.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical quality control practices.

DISTILLATION RANGE

10. Procedure

10.1 Determine the distillation range of the sample in accordance with Test Method D1078. Use the conditions as specified in Test Method D1078, and the ASTM Solvents Distillation Thermometer shown in Table 1 of Test Method D1078. (See Note 2 for certain allowable exceptions in applying this test method to triethylene glycol.)

NOTE 2—In the distillation of triethylene glycol, it may not be possible to collect the first drop of liquid within 15 min or to maintain the prescribed distillation rate of 4 to 5 mL/min with some sources of gas. In this case, up to 30 min can be allowed to collect the first drop, and a distillation rate of 2 to 3 mL/min is satisfactory. Alternatively, the flask chamber may be covered with a suitable shield so that only the upper neck and thermometer are exposed to room air to achieve the specified rates.

10.2 Use the following values of *K* in the equation for barometric correction (Test Method D1078):

Chemical	*K*
Ethylene glycol	0.045
Diethylene glycol	0.050
Triethylene glycol	0.055
Propylene glycol	0.043
Dipropylene glycol	0.051

11. Report

11.1 Report the corrected temperatures to the nearest 0.1°C at each volume required by the specification for the glycol being analyzed.

12. Precision and Bias

12.1 *Interlaboratory Study*: [9, 10]

12.2 The precision of this test method was obtained from an interlaboratory study conducted in 2000 involving manual and automatic distillation procedures. The study involved six samples of different boiling point ranges, done in duplicate. Ten laboratories performed automatic Test Method D1078 distillation, and five laboratories performed manual Test Method D1078 distillation. It was found that the precision is dependent on the boiling point temperature. The data were statistically evaluated using ASTM D2PP software (ASTM Adjunct ADJD6300).[4]

12.3 *Repeatability*—Two results, each the mean of two runs, obtained by the same operator should be considered suspect if they differ by more than the repeatability values shown in Table 1 at a 95 % confidence level.

12.4 *Reproducibility*—Two results, each the mean of two runs, obtained by operators in different laboratories should be considered suspect if they differ by more than the reproducibility values shown in Table 1 at a 95 % confidence level.

12.5 *Bias:*

12.5.1 *Absolute Bias*—Since the temperature measuring devices specified by this test method are calibrated against the normal boiling point of toluene (99.9+ % purity), this test method has no bias with respect to pure toluene as a reference material.

12.5.2 *Relative Bias Between Manual and Automatic D1078 Distillation*—Statistical comparison between the variances of automatic and manual D1078 distillation results did not indicate any statistically significant difference. Statistical comparison of the averages of the six samples used in the study indicated that the paired-sample, two-tailed, t-test for the initial boiling point (IBP) and 50 % distillation point showed a small relative bias that is not statistically significant. A small but statistically significant bias was indicated for the automatic and manual D1078 dry point (DP). The observed bias (if any) are only for the samples studied and may not be necessarily applicable to other samples.

NOTE 3—In cases of dispute, the parties involved may agree to designate either the manual or the automatic method to be the referee test method. If an agreement on which method to designate cannot be made, the referee test method will be the manual method.

13. Quality Guidelines

13.1 Laboratories shall have a quality control system in place.

[9] Supporting data have been filed at ASTM Headquarters and may be obtained by requesting Research Report RR:E15-1114. Contact ASTM Customer Service at service@astm.org.

[10] Supporting data have been filed at ASTM Headquarters and may be obtained by requesting Research Report RR:E15-1123. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Precision for Acidity in Glycols

Glycol ID	Grand Avg (mg/kg)	Standard Deviation (mg/kg)	Degrees of Freedom	95 % Range mg/kg absolute
MEG	1.66	0.100	5	0.280
DEG	1.75	0.114	5	0.319
TEG		1.370	5	3.836
TTEG	4.71	0.277	5	0.777

TABLE 3 Accuracy for Acidity in Glycols Acidity as Acetic Acid in MEG

Actual Concentration (mg/kg)	Found Concentration (mg/kg)	Average Recovery (%)
6.62	6.04	91.2
11.91	10.90	91.5
27.30	25.67	94.0
51.51	48.72	94.6

13.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

13.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

13.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

13.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical quality control practices.

ACIDITY

14. Procedure

14.1 Determine the acidity of the sample in accordance with Test Method E2679.

15. Report

15.1 Report the acidity as acetic acid to the nearest 0.1 mg/kg for the sample.

16. Precision and Bias

16.1 *Precision*—The following criteria should be used to judge the acceptability of the results (see Note 4):

16.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be the value given in Table 2 at the indicated degrees of freedom. The 95 % limit of difference between two such runs is also given in Table 2.

16.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—The precision of the procedure for measuring acidity is being determined.

16.1.3 *Reproducibility (Multilaboratory)*—The precision of the procedure for measuring acidity is being determined.

NOTE 4—The precision statements are preliminary based on 5 analyses by one analyst on two days for samples of MEG, DEG, TEG and TTEG containing approximately 1.7 mg/kg, 1.8 mg/kg, 33.0 mg/kg and 4.7 mg/kg acidity as acetic acid respectively. An interlaboratory study is planned for 2009/2010. Practice E180 was used in developing these precision estimates.

16.2 *Bias*—The bias of this test method was determined by spiking samples of MEG with acetic acid in the 5 to 50 mg/kg range and analyzing the spiked and unspiked samples. The accuracy (recovery) was estimated to be the values given in Table 3 based on the titration curves. The bias depends upon the accuracy of the titration, weighing of the spike and the extent of any interferences.

17. Quality Guidelines

17.1 Laboratories shall have a quality control system in place.

17.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

17.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

17.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

17.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical quality control practices.

WATER

18. Procedure

18.1 Determine the water content of the sample using any suitable Karl Fischer reagent titration method. Test Method E1064 is recommended.

19. Report

19.1 Report the water content to the nearest 0.001 weight %.

20. Precision and Bias

20.1 In 2007, ASTM International Committee E15 on Industrial and Specialty Chemicals conducted and completed Interlaboratory Study No. 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 Test Method E1064, conducted in 2007. Each of 17 laboratories were asked to test three different materials. Fourteen laboratories tested MEG, 13 laboratories tested DEG and 13 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, eight laboratories used a single analyst, seven laboratories used two analysts (on different days), and two laboratories did not record this information. In the event that there were missing values for one or more laboratories, this information was noted in the results. See Table 4.

20.1.1 *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

TABLE 4 Test Method E1064 Water in Organic Liquids by Coulometric Karl Fischer Titration

Test Result % weight	Sample	Average over all Laboratories	Repeatability Standard Deviation	Intermediate Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Intermediate Unit	Reproducibility Limit
Water	MEG	0.0086	0.0009	0.0014	0.0025	0.0026	0.0038	0.0071
Water	DEG	0.0649	0.0012	0.0014	0.0049	0.0032	0.0039	0.0137
Water	TEG	0.0498	0.0019	0.0129	0.0157	0.0054	0.0361	0.0439

the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

20.1.2 *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.

20.1.3 *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.

20.1.3.1 The E180 analysis considers the two test results from each day as being run under 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 analysis.

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

20.2 *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.

20.3 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

20.3.1 To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

21. Quality Guidelines

21.1 Laboratories shall have a quality control system in place.

21.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

21.1.2 A quality control sample is stable material isolated from the production process and representative of the sample being analyzed.

21.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

21.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical control practices.

IRON

22. Procedure

22.1 Determine the iron content of the sample in accordance with Test Method E1615.

23. Report

23.1 Report the iron content to the nearest 0.001 µg/g.

24. Precision and Bias

24.1 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 E1615. Each of 15 laboratories were asked to test three different materials. Thirteen laboratories tested MEG, 11 laboratories tested DEG, and 10 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 laboratories used a single analyst, 7 laboratories used 2 analysts (on different days) and 2 laboratories did not record this information. In the event that there were missing values for one or more laboratories, this information was noted in the results. The details of this study are given in an ASTM Research Report.[11]

24.1.1 *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.

24.1.2 *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.

[11] Supporting data have been filed at ASTM Headquarters and may be obtained by requesting Research Report RR:E15-1064. Contact ASTM Customer Service at service@astm.org.

24.1.3 *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.

24.1.3.1 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 PracticeE180 analysis.

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

24.2 *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.

24.3 The precision statement was determined through statistical examination of qualified results, from fifteen laboratories, on three materials. These three materials were described as the following:

Fluid 1: Monoethylene Glycol
Fluid 2: Diethylene Glycol
Fluid 3: Triethylene Glycol

24.3.1 To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

25. Quality Guidelines

25.1 Laboratories shall have a quality control system in place.

25.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

25.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

25.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

25.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical control practices.

COLOR

26. Procedure

26.1 Determine the color of the sample in accordance with Test Method D1209.

27. Report

27.1 Estimate and report the color to the nearest one platinum-cobalt unit.

28. Precision and Bias

28.1 The following criteria should be used for judging the acceptability of results (see Note 5):

28.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0 unit at 40 dF. The 95 % limit for the difference between two such runs is two units.

28.1.2 *Laboratory Precision (Within-Laboratory, Between-Days)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.64 unit at 46 dF. The 95 % limit for the difference between two such averages is two units.

28.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 2.47 units at 9 df. The 95 % limit for the difference between two such averages is seven units.

NOTE 5—These precision estimates are based on interlaboratory studies performed in 1962 and 1963 on a total of six samples of the five glycols whose color ranged from 2 to 21 platinum-cobalt units. Because the test results are based on visual comparison of the untreated sample with standards, duplicate determinations at low levels of color are almost always in perfect agreement. This was confirmed in the 1962 study of two samples of ethylene glycol with average colors of 2 and 21 platinum-cobalt units. The standard deviation for duplicate determinations was estimated to be 0.0 units at 40 dF. Therefore, the stated 95 % limit in the repeatability statement is based on the reporting of results to the nearest one unit. The 1963 study omitted the duplicate determinations. A total of ten laboratories cooperated in the studies in which each analyst performed duplicate determinations on each sample on each of two days.[8] Practice E180 was used in developing these precision estimates.

28.1.4 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

29. Quality Guidelines

29.1 Laboratories shall have a quality control system in place.

29.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

29.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

29.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

29.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical control practices.

GAS CHROMATOGRAPHIC ANALYSIS

30. Procedure

30.1 Determine the purity of Ethylene Glycol samples in accordance with E2409. For Propylene Glycol purity analysis, refer to the Alternative Test Methods in the Appendixes.

31. Report

31.1 Report the concentrations of DEG in MEG and MEG in DEG to the nearest mg/kg and all other impurities to the