Designation: E2193 – 16

Standard Test Method for Ultraviolet Transmittance of Monoethylene Glycol (using Ultraviolet Spectrophotometry)[1]

This standard is issued under the fixed designation E2193; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers a procedure for the determination of the transmittance of monoethylene glycol (1,2-ethanediol; MEG) at wavelengths in the region 220 to 350 nm. The results provide a measure of the purity of the sample with respect to ultraviolet absorbing compounds.

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 *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.* For specific hazard statements, see Section 7.

1.4 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.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

E131 Terminology Relating to Molecular Spectroscopy

E169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]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

2.2 *Other Document:*

Manufacturer's Instruction Manual of Spectrophotometer

3. Summary of Test Method

3.1 The product is sampled in such a way as to avoid extraneous contamination and air contact. The absorbance of the sample contained in a 50-mm or 10-mm cell is measured against water at a series of wavelengths and the transmittance over a pathlength of 10 mm is calculated.

3.2 This test method can be performed with two options as to sample preparation prior to UV measurement.

3.2.1 Option A: Nitrogen sparging of the sample (see 4.2).

3.2.2 Option B: No nitrogen sparging.

4. Significance and Use

4.1 Knowledge of the ultraviolet transmittance of monoethylene glycol is required to establish whether the product meets the requirements of its quality specifications.

4.2 Dissolved oxygen in organic solvents, such as MEG, forms complexes that shift the solvent absorption from the vacuum ultraviolet range into the measurable UV range (near 190 to 250 nm). Monoethylene glycol has a UV absorption peak at 180 nm. For MEG-oxygen complexes, this peak is shifted to a longer wavelength, thus increasing the absorbability at 220 nm.

4.2.1 However, this effect is not observed in water. There is no significant measurable effect due to dissolved oxygen in water that would require nitrogen sparging prior to using for collection of the reference spectrum.

4.2.2 Nitrogen sparging and re-measurement of suspect or borderline glycol samples at 220 nm can be used as a tool to rule out or confirm the presence of UV affecting contaminants other than oxygen.

[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. Apparatus

5.1 *Ultraviolet Spectrophotometer,* double beam, suitable for measurement at wavelengths in the region 200 to 400 nm, having a spectral bandwidth of 2.0 nm or less at 220 nm, wavelength accuracy ±0.5 nm or less at 220 nm, wavelength repeatability 0.3 nm or less at 220 nm and a photometric accuracy of ±0.5 % *T* or less, in the transmittance region above 50 % *T*. Stray light shall be less than 0.1 % at 220 nm. The instrument shall be provided with matched quartz (fused silica) cells with pathlengths of 50 ± 0.1 mm or 10 mm ± 0.01 mm. Use of 50-mm cell should provide better precision.

5.1.1 *Optional Single Beam Spectrophotometer*—Use the same cell for measurement of the blank and the sample.

5.2 *Nitrogen Stripping Apparatus (Option A, 3.2.1),* consisting of an oil-free pressure reducing valve to fit the nitrogen cylinder, a control valve, vinyl tubing and a disposable glass pipette to be inserted in a 25-mL volumetric flask or bottle. Components should be clean and free of ultraviolet contaminants. Avoid contacting the sample with any plastic material containing plasticizers. Plasticizers can leach out of the material and cause erroneous results. Replace the disposable pipette by a clean, new one after each sample handling. (See Section 10.)

5.3 *Bottles,* capacity at least 0.5 L, with lined, well-fitting cap. Use a fresh bottle for each determination.

5.4 *Glassware:*

5.4.1 *Volumetric Flask or Bottle,* 25 mL.

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 *Holmium Oxide Wavelength Calibration Filter,* calibrated (if required; see 9.1.1).

NOTE 1—The standard reference material SRM 2034, available from the National Institute of Standards and Technology (NIST),[5] is suitable.

6.3 *Standard Absorbance Filter,* with certified absorbance values (if required; see 9.1.2).

NOTE 2—The standard reference material SRM 2031, available from NIST,[5] is suitable. In addition, SRM 935a may be used (see 6.4).

6.4 *Stray Light Filter,* for measuring stray light at 220 nm (if required; see 9.1.3).

NOTE 3—The (potassium iodide) standard reference material SRM 2032, available from NIST,[5] is suitable (see also 6.8).

6.5 *Naphthalene Solution*—(1 mg/L isooctane) (**Warning—** Isooctane is highly flammable and irritating to the respiratory system. Avoid contact with skin. Naphthalene is irritating to the skin, eyes, and respiratory system. It may cause sensitization by skin contact. Avoid contact with eyes. Wear suitable protective clothing.)

6.6 *Nitrogen,* minimum purity 99.99 % (V/V), oil-free.

6.7 *Potassium Dichromate or Potassium Chromate,* for checking photometric accuracy (if required; see 9.1.2).

NOTE 4—The standard reference material, no. SRM 935a, available from NIST,[5] is suitable.

(**Warning—**Potassium dichromate is harmful in contact with skin, toxic if swallowed, and very toxic by inhalation. It is irritating to the respiratory system and skin. Risk of serious damage to eyes. It may cause sensitization by skin contact. It may cause heritable genetic damage. It may cause cancer by inhalation. In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible). Avoid exposure—obtain special instructions before use. This material or its container, or both, must be disposed of as hazardous waste. Avoid release to the environment. Refer to special instructions/safety data sheets.) (**Warning—**Potassium chromate is irritating to eyes, respiratory system, and skin. It may cause sensitization by skin contact. It may cause heritable genetic damage. It may cause cancer by inhalation. In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible). Avoid exposure—obtain special instructions before use. This material or its container, or both, must be disposed of as hazardous waste. Avoid release to the environment. Refer to special instructions/safety data sheets.)

6.8 *Potassium or Sodium Iodide Solution,* for measuring stray light at 220 nm (if required; see 9.1.3).

NOTE 5—The "UV-VIS Standard" sodium iodide solution,[6] is suitable. Potassium iodide solutions can be prepared from NIST standard reference material SRM 2032 (see 6.4 and Note 3).

6.9 *Water, Low UV Absorbance*—HPLC grade or reagent water type I (Specification D1193).

6.10 *Monoethylene Glycol Quality Control Sample*—It is recommended to select a *Monoethylene Glycol* sample 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 11.5.

7. Hazards

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

7.2 *Monoethylene Glycol*—Although monoethylene glycol, in general, is not classified as dangerous or flammable and is

[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 *Analar 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.

[5] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

[6] Available from Merck KGaA, Frankfurterstraße 250, D-64293 Darmstadt, Germany.

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

8. Sampling

8.1 The following precautions must be observed carefully since the ultraviolet transmittance is very sensitive to small amounts of extraneous material contaminating the sample and to oxygen dissolved in the sample through air contact. The sample connection must be protected against accidental contamination and designed so that it will permit convenient positioning of the sample bottle to the sample outlet in order to minimize air contact, that is, the descending stream of sample should be as short as possible. Purge the sampling line thoroughly with sample. Fill the bottle partly with sample (**Warning—**see 7.2), shake and discard. Repeat the rinsing procedure. Then take the sample in a "gentle stream," thus filling the bottle to within 10 mm of the top. A "gentle stream" is a rate of flow that avoids spattering, splashing, or other aggressive manifestations on the part of the sample flow. Cap and avoid excessive shaking during transport (see also Section 10).

9. Preparation of Apparatus

9.1 *Spectrophotometer*—Check the performance of the spectrophotometer as described below. General information on the measurement of performance of spectrophotometers is given in Practice E275.

9.1.1 *Wavelength Accuracy*—Check the wavelength accuracy of the spectrophotometer at 220 nm, in accordance with the manufacturer's instructions, for example, by means of a naphthalene solution (**Warning—**see 6.5) in a 10-mm cell. If the scale reading at the observed band maximum differs by more than 0.3 nm from 220.6 nm (wavelength of naphthalene band maximum), measure the absorbance in the actual procedure (Section 11) at a wavelength setting of 0.6 nm below the value found for the naphthalene band maximum.

9.1.1.1 Alternatively, wavelength accuracy may be checked using the calibrated holmium oxide filter (6.2). Naphthalene is the preferred material for this purpose but holmium oxide is a sufficient alternative.

NOTE 6—Since the absorbance of monoethylene glycol rises considerably at wavelengths shorter than 220 nm, it is essential that the wavelength position in this region is accurately set.

9.1.2 *Photometric Accuracy*—Check that the photometric accuracy of the spectrophotometer is in accordance with the instrumental specification (see 5.1), for example, by means of standard absorbance filters or solutions of suitable materials (**Warning—**see 6.7).

9.1.3 *Stray Light*—Check that any stray light emanating from the spectrophotometer at 220 nm does not exceed the instrumental specification (see 5.1), for example, by means of a stray light filter or a solution of a suitable material (see 6.4 and 6.5).

9.2 *Glassware*—Thoroughly clean the cells, and other glassware, using the guidelines described in Practice E275, that is, using water, methanol, or a mild sulfonic detergent. Do not use acetone or ultrasonic baths to clean cells.

9.3 *Nitrogen Stripping Apparatus (Option A, 3.2.1)*—Assemble the apparatus and flush thoroughly with nitrogen. Pass nitrogen through 20 mL of monoethylene glycol contained in a 25-mL volumetric flask or bottle and check the quality of the nitrogen by measuring the absorbance at 220 nm, in order to see whether it remains constant after a possible initial decrease due to the removal of dissolved oxygen.

10. Sample Preparation (Option A only. Skip to Section 11 if using Option B)

10.1 Strip the sample with nitrogen before measurement. Introduce 20 mL of test sample into a 25-mL volumetric flask or bottle and pass a brisk stream of nitrogen (see 5.2, 5.4 and 6.6) through the sample for 15 min, using a clean disposable pipette. Stopper the flask.

11. Procedure

11.1 Adjust the spectrophotometer to the optimum instrument settings, selecting the slit width to give a spectral bandwidth of 2.0 nm or less. Spectral bandwidth of 2.0 nm is preferred as lower bandwidths increase the noise level of the spectral data.

11.2 Fill two 50-mm or 10-mm matched cells with HPLC grade (see 6.9) water. Make sure the cell windows are clear and the water is free of bubbles. Place the cells in the cell compartment of the spectrophotometer, noting the direction of the cells inside the cell holder, and measure the absorbances at 220, 250, 275, and 350 nm, or any other wavelengths required by the relevant product specification. Use the cell with the higher absorbance as the sample cell, the other as the reference cell, and record the absorbances observed as the cell corrections at the various wavelengths.

NOTE 7—With properly matched cells, the cell correction is less than 0.01 absorbance units.

11.3 Empty the sample cell and dry with cell tissue. Carefully fill the sample cell with the test sample (at room temperature (**Warning—**see 7.2)). Avoid producing bubbles in the sample. Without changing the adjustments of the spectrophotometer, measure and record the absorbances at the same set of wavelengths as measured in 11.2 against the reference cell filled with water. Ensure that the direction of the cells in the holder is the same as noted in 11.2. Change the water in the reference cell for each set (11.2 and 11.3) of measurements made.

11.4 Empty the cells and rinse with water. Clean the cells at regular intervals, according to 9.2, and store filled with water.

11.5 *Quality Control*—Although the procedure in Section 9 is described such that only one test result is obtained, it is recommended to either:

11.5.1 Perform a second (duplicate) determination, to enable comparison of the duplicate results with the listed repeatability limit in Table 1. Choose this option if this test method is performed on an infrequent basis.

TABLE 1 E2193 UV Transmittance of MEG

Test Result %T at Wavelength	Sample	Average Over All Laboratories	Repeatability Standard Deviation	Intermediate Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Intermediate Limit	Reproducibility Limit
220 nm	sparged	92.01	0.394	1.905	3.458	1.103	5.334	9.682
250 nm	sparged	98.49	0.232	0.462	0.737	0.650	1.293	2.063
275 nm	sparged	98.97	0.162	0.372	0.392	0.453	1.042	1.098
350 nm	sparged	100.18	0.155	0.220	0.334	0.433	0.615	0.936
220 nm	unsparged	80.65	0.293	0.828	1.445	0.819	2.319	4.047
250 nm	unsparged	96.88	0.175	0.285	0.394	0.491	0.797	1.102
275 nm	unsparged	98.76	0.180	0.373	0.752	0.505	1.045	2.105
350 nm	unsparged	100.14	0.139	0.145	0.412	0.389	0.406	1.154

11.5.2 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.

11.5.2.1 Analyze the QC sample under intermediate precision conditions and construct a control chart for data obtained for one of the wavelengths of interest.

11.5.2.2 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.

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

11.5.2.4 From SQC data obtained under statistical control, calculate the intermediate precision. Compare this value with the intermediate precision limit as included in Table 1.

12. Calculation

12.1 Calculate the net absorbance, A_λ, of the sample over a pathlength of 10 mm at each relevant wavelength, by means of the following equation. If 10 mm cells were used in the measurement, do not divide by 5.

$$A_\lambda, = \frac{A_s - A_c}{5} \quad (1)$$

where:

A_s = absorbance found in sample measurement at relevant wavelength λ (11.3), and

A_c = cell correction at relevant wavelength λ (11.2).

12.2 Calculate the transmittance, T_λ, of the sample over a pathlength of 10 mm at each relevant wavelength by means of the following equation:

$$T_\lambda, \% = 10^{(2-A_\lambda)} \quad (2)$$

13. Report

13.1 Report the following information:

13.1.1 Report the transmittance of the sample at each relevant wavelength to the nearest 0.1 %. The use of Option A (Nitrogen sparged) or Option B (non-sparged) and the cell size should be included in the report.

14. Precision and Bias[7]

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 E2193, Standard Test Method for Ultraviolet Transmittance of Monoethylene Glycol (using Ultraviolet Spectrophotometry). Each of 17 laboratories were asked to test two different materials. Sixteen laboratories tested MEG in U220nm. Fifteen laboratories tested MEG in U250nm. Seventeen laboratories tested MEG in U275nm. Fifteen laboratories tested MEG in U350nm. Twelve laboratories tested MEG in S220nm. Twelve laboratories tested MEG in S250nm. Twelve laboratories tested S275nm. Twelve laboratories tested MEG in S350nm. 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 labs used a single analyst, seven labs used two analysts (on different days) and two 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 RR:E15-1066.

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

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E15-1066.

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 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 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 two materials. These two materials were described as the following: Fluid 1: Monoethylene glycol-Sparged; Fluid 2: Monoethylene glycol- Unsparged. 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 monoethylene glycol; spectrophotometry; ultraviolet transmittance

SUMMARY OF CHANGES

Committee E15 has identified the location of selected changes to this standard since the last issue (E2193 – 08) that may impact the use of this standard. (Approved Jan. 1, 2016.)

(1) Title of test method was revised.
(2) Specification D1193 and Practice D6299 were added to Section 2 Referenced Documents.
(3) Revisions were made in the following sections: 1.4, 5.1, 5.2, 5.4.1, 6.5, 6.9, 6.10, 8.1, 9.2, 9.3, 10.1, 11.3, and 11.5.
(4) The following sections were added: 7, 11.5.1, 11.5.2, and 11.5.2.1 – 11.5.2.4.
(5) Note 8 was deleted.