



Standard Test Method for Ultraviolet Transmittance of Monoethylene Glycol (Ultraviolet Spectrophotometric Method)¹

This standard is issued under the fixed designation E 2193; 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.*

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

2. Referenced Documents

2.1 ASTM Standards:²

E 131 Terminology Relating to Molecular Spectroscopy

E 169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near-Infrared 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.

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 $\pm 0.5\%$ *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 fused silica

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.

Current edition approved April 1, 2008. Published May 2008. Originally approved in 2002. Last previous edition approved in 2004 as E 2193 – 04.

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.

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

cells with pathlengths of 50 ± 0.1 mm or $10 \text{ mm} \pm 0.01$ 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. 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 9.)

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*, 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.³ 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 8.1.1).

NOTE 1—The standard reference material SRM 2034, available from the National Institute of Standards and Technology (NIST),⁴ is suitable.

6.3 *Standard Absorbance Filter*, with certified absorbance values (if required; see 8.1.2).

NOTE 2—The standard reference material SRM 2031, available from NIST,⁴ 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 8.1.3).

NOTE 3—The (potassium iodide) standard reference material SRM 2032, available from NIST,⁴ is suitable (see also 6.8).

6.5 *Naphthalene Solution*—(1 mg/L isooctane) Dissolve 1 mg naphthalene in 1000 mL of spectroscopic grade 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 8.1.2).

NOTE 4—The standard reference material, no. SRM 935a, available from NIST,⁴ 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 8.1.3).

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

6.9 *Water*, HPLC grade.

6.10 *Monoethylene Glycol Quality Control Sample*, (only required if maintaining a control chart, see 10.5). Store at a temperature between 0 and 5°C. Warm to ambient temperature before use.

7. Sampling

7.1 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, 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 9).

8. Preparation of Apparatus

8.1 *Spectrophotometer*—Check the performance of the spectrophotometer as described below. General information on

³ *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.

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

⁵ Available from Merck KGaA, Frankfurterstraße 250, D-64293 Darmstadt, Germany.

the measurement of performance of spectrophotometers is given in Practice E 275.

8.1.1 *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 10) at a wavelength setting of 0.6 nm below the value found for the naphthalene band maximum.

8.1.1.1 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.

8.1.2 *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).

8.1.3 *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).

8.2 *Glassware*—Thoroughly clean the cells, and other glassware, using the guidelines described in Practice E 275.

8.3 *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 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.

9. Sample Preparation (Option A only-skip to 10 if using Option B)

9.1 Strip the sample with nitrogen before measurement. Introduce 20 mL of test sample into a 25-mL volumetric flask 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.

10. Procedure

10.1 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.

10.2 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.

10.3 Empty the sample cell and dry with cell tissue. Carefully fill the sample cell with the test sample. 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 10.2 against the reference cell filled with water. Ensure that the direction of the cells in the holder is the same as noted in 10.2. Change the water in the reference cell for each set (10.2 and 10.3) of measurements made.

10.4 Empty the cells and rinse with water. Clean the cells at regular intervals, according to 8.2, and store filled with water.

NOTE 8—Although the determination in the procedure is described such that only one test result is obtained, it is required to perform a second (duplicate) determination to enable comparison of the duplicate results with the repeatability value given in Section 13.

10.5 *Quality Control*—It is recommended that a control chart for the UV transmittance in the Monoethylene 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 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 (10.3), and

A_c = cell correction at relevant wavelength λ (10.2).

11.2 Calculate the transmittance, T_{λ} , of the sample over a pathlength of 10 mm at each relevant wavelength by means of the following equation:

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

12. Report

12.1 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.

⁶ ASTM Manual on Presentation of Data and Control Chart Analysis: 7th Edition, ASTM Manual Series MNL 7A.

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 E 2193, Standard Test Method for Ultraviolet Transmittance of Monoethylene Glycol (Ultraviolet Spectrophotometric Method). Each of seventeen laboratories were asked to test two different materials. 16 laboratories tested MEG in U220nm. 15 laboratories tested MEG in U250nm. 17 laboratories tested MEG in U275nm. 15 laboratories tested MEG in U350nm. 12 laboratories tested MEG in S220nm. 12 laboratories tested MEG in S250nm. 12 laboratories tested MEG in S275nm. 12 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, 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. E15-1066.⁷

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 E 180. Practice E 180 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 E 180 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 E 180 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 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.

14. Keywords

14.1 monoethylene glycol; spectrophotometric; ultraviolet transmittance

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

TABLE 1 E 2193 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	unsparaged	80.65	0.293	0.828	1.445	0.819	2.319	4.047
250 nm	unsparaged	96.88	0.175	0.285	0.394	0.491	0.797	1.102
275 nm	unsparaged	98.76	0.180	0.373	0.752	0.505	1.045	2.105
350 nm	unsparaged	100.14	0.139	0.145	0.412	0.389	0.406	1.154

SUMMARY OF CHANGES

Committee E15 has identified the location of selected changes to this standard since the last issue (E 2193 – 04) that may impact the use of this standard. (Approved April 1, 2008.)

(I) Revised Precision and Bias statement.

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