Designation: E 2193 – 02

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 the 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:*

E 131 Terminology Relating To Molecular Spectroscopy[2]

E 169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis[2]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[3]

E 275 Practice for Describing and Measuring Performance of Ultra-violet, Visible and Near Infrared Spectrophotometers[2]

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. After sparging with nitrogen, the absorbance of the sample contained in a 50-mm cell is measured against water at a series of wavelengths and the transmittance over a pathlength of 10 mm is calculated.

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.

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 fused silica cells with pathlengths of 50 ± 0.1 mm and 10 mm.

5.2 *Nitrogen Stripping Apparatus*, 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.[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.

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[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] *Annual Book of ASTM Standards*, Vol 03.06.

[3] *Annual Book of ASTM Standards*, Vol 15.05.

[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. (USPC), Rockville, MD.

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),[5] 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,[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 8.1.3).

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) 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,[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 8.1.3).

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*, HPLC grade.

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 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*—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

9.1 Strip the sample with nitrogen before measurement.

[5] NIST, 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460, U.S.A.

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

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 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. Fill the sample cell with the nitrogen-sparged test sample and 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.

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:

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

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 %.

13. Precision and Bias

13.1 *Precision*—The following criteria should be used to judge the acceptability of results (see Note 9):

13.1.1 *Repeatability* (Single Analyst)—The standard deviation for a single determination has been estimated to be the value shown in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such runs is also shown in Table 1.

NOTE 9—The precision estimates are based on the analysis of 15 samples analyzed in duplicate in April 1982 by one analyst in one laboratory. The range of percent transmittance values for the samples are shown in Table 1. The equations in Practice E 180 were used in developing these precision estimates. Because the data are from only one laboratory, no estimates of Laboratory Precision or Reproducibility are possible. An interlaboratory study is planned for 2002.

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

14. Keywords

14.1 monoethylene glycol; spectrophotometric; ultraviolet transmittance

TABLE 1 Transmission Precision-Repeatability

Wavelength, nm	Range of average % *T* for 15 samples	Standard deviation, % *T*	Degrees of freedom	95 % Limit, % *T* absolute
220	80.8 – 95.1	0.5273	15	1.5
250	87.0 – 99.4	0.3469	15	0.97
275	90.9 – 99.5	0.3924	15	1.1
350	98.3 – 100.0	0.2530	15	0.71