Designation: ~~E2193 – 16~~ E2193 – 23

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.~~ 220 nm to 350 nm. The results provide a measure of the purity of the sample with respect to ultraviolet (UV) 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, health, and environmental 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.

1.5 *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]

D1193 Specification for Reagent Water

~~D6299~~D6809 ~~Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance~~Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]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

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is 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**

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~~transmittance of the sample contained in a ~~50-mm or 10-mm~~ 10 mm cell is measured against water at a series of ~~wavelengths and the transmittance over a pathlength of 10 mm is calculated.~~wavelengths.

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~~UV transmittance of ~~monoethylene glycol~~ MEG 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~~UV range into the measurable UV range (near ~~190 to 250 nm). Monoethylene glycol~~ 190 nm to 250 nm). MEG 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,~~ 200 nm to 400 nm, having a spectral bandwidth of 2.0 nm or less at ~~220 nm,~~ 220 nm, wavelength accuracy ±0.5 nm or less at ~~220 nm,~~ 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~~ pathlength of 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.

5.1.2 Terminology E131, Practice E169, and Practice E275 have additional information on the operation of spectrophotometers.

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~~25 mL volumetric flask or bottle. Components should be clean and free of ~~ultraviolet~~UV 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,~~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),~~Commercial reference standards traceable to NIST SRM's are available. ~~is suitable.~~

6.3 *Standard Absorbance Filter,* with certified absorbance ~~values (if required; see~~ values.~~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~~ nm.~~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~~ accuracy.~~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).~~ immediately. 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).~~ immediately. 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~~ nm.~~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—*~~*Water*—~~HPLC grade or reagent water type I~~ Type I grade of reagent water (Specification D1193).

6.10 ~~*Monoethylene Glycol*~~ *MEG Quality Control Sample*—It is recommended to select a ~~Monoethylene Glycol~~MEG 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 Section ~~11.5~~14.

[4] ~~*Reagent Chemicals, American Chemical Society Specifications,*~~*ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials,* 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),~~(USPC), Rockville, MD.

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—*~~*MEG—*Although ~~monoethylene glycol,~~ MEG, in general, is not classified as dangerous or flammable and is 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~~UV 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~~ 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 ~~(~~(see ~~**Warning—**see 6.5~~ 6.5) in a ~~10-mm~~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 ~~(~~(see 6.2). Naphthalene is the preferred material for this purpose but holmium oxide is a sufficient alternative.

NOTE 2—Since the absorbance of ~~monoethylene glycol~~ MEG 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 (see 6.3) or solutions of suitable materials ~~(~~(see ~~**Warning—**see 6.7~~ 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~~6.8).

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, see 3.2.1~~)—~~)—Assemble the apparatus and flush thoroughly with nitrogen. Pass nitrogen through 20 mL of ~~monoethylene glycol~~ MEG contained in a ~~25-mL~~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. The recommended nitrogen stripping condition is as follows: nitrogen at 150 mL/min for 10 min.

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~~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~~ 10 mm matched cells with ~~HPLC grade~~ water (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.~~zero the system at 220 nm, 250 nm, 275 nm, and 350 nm, respectively.

NOTE 3—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~~ transmittance of the sample 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 ~~(~~(see 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.~~

~~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.~~

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