Designation: E925 – 09 (Reapproved 2022)

Standard Practice for Monitoring the Calibration of Ultraviolet-Visible Spectrophotometers whose Spectral Bandwidth does not Exceed 2 nm[1]

This standard is issued under the fixed designation E925; 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.

INTRODUCTION

In the application of spectrophotometric methods of analysis it is the responsibility of the analyst to verify and validate that the instrument is functioning properly and is capable of providing acceptable analytical results. It is preferable that the verification of instrument performance be accomplished through the use of reference materials whose properties have been accurately determined. Such materials are readily available, and their use in the tests and measurements described in this practice is satisfactory for evaluating the performance of spectrophotometers whose spectral bandwidth does not exceed the value for which the intrinsic or certified properties are valid. A compromise maximum permissible spectral bandwidth of 2 nm is recommended for the reference materials and error tolerances recommended here.

This practice covers some of the essential instrumental parameters that should be evaluated to ensure the acceptability of the analytical data routinely obtained on the instrument. These parameters include the accuracy of the wavelength and absorbance scales and stray radiant power levels.

The accuracy of the wavelength scale in both the ultraviolet (UV) and visible regions is determined using the sharp absorption bands of a holmium oxide glass or solution filter. The absorbance scale accuracy in the UV region (235 nm to 350 nm) is determined using acidic solutions of potassium dichromate. In the visible region (440 nm to 635 nm) the absorbance accuracy is determined using individually certified neutral density glass filters. The use of these reference materials provides a valid and relatively simple means to test the errors in the wavelength and absorbance scales of small spectral bandwidth spectrophotometers in the spectral ranges indicated. A simplified version of the opaque filter method is provided as a test for excessive stray radiant energy.

1. Scope

1.1 This practice covers the parameters of spectrophotometric performance that are critical for testing the adequacy of instrumentation for most routine tests and methods[2] within the wavelength range of 200 nm to 700 nm and the absorbance range of 0 to 2. The recommended tests provide a measurement of the important parameters controlling results in spectrophotometric methods, but it is specifically not to be inferred that all factors in instrument performance are measured.

1.2 This practice may be used as a significant test of the performance of instruments for which the spectral bandwidth does not exceed 2 nm and for which the manufacturer's specifications for wavelength and absorbance accuracy do not exceed the performance tolerances employed here. This practice employs an illustrative tolerance of ±1 % relative for the error of the absorbance scale over the range of 0.2 to 2.0, and of ±1.0 nm for the error of the wavelength scale. A suggested maximum stray radiant power ratio of 4×10^{-4} yields <1 % absorbance bias at an absorbance of 2. These tolerances are chosen to be compatible with many chemical applications while comfortably exceeding the uncertainty of the certified values for the reference materials and typical manufacturer's specifications for error in the wavelength and absorbance scales of the instrument under test. The user is encouraged to develop and use tolerance values more appropriate to the requirements of the end use application. This procedure is designed to verify quantitative performance on an ongoing

[1] This practice is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and Separation Science and is the direct responsibility of Subcommittee E13.01 on Ultra-Violet, Visible, and Luminescence Spectroscopy.

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[2] Routine tests are defined as those in which absorbance data obtained on a sample are compared to those of a standard sample preparation.

basis and to compare one instrument's performance with that of other similar units. Refer to Practice E275 to extensively evaluate the performance of an instrument.

1.3 This practice should be performed on a periodic basis, the frequency of which depends on the physical environment within which the instrumentation is used. Thus, units handled roughly or used under adverse conditions (exposed to dust, chemical vapors, vibrations, or combinations thereof) should be tested more frequently than those not exposed to such conditions. This practice should also be performed after any significant repairs are made on a unit, such as those involving the optics, detector, or radiant energy source.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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:*[3]

E131 Terminology Relating to Molecular Spectroscopy

E169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

E387 Test Method for Estimating Stray Radiant Power Ratio of Dispersive Spectrophotometers by the Opaque Filter Method

E1866 Guide for Establishing Spectrophotometer Performance Tests

2.2 *NIST Publications:*[4]

NIST Special Publication 260-54 Certification and Use of Acidic Potassium Dichromate Solutions As An Ultraviolet Absorbance Standard

NIST Special Publication 260-102 Holmium Oxide Solution Wavelength Standard from 240 to 640 nm—SRM 2034

NIST Special Publication 260-116 Glass Filters as a Standard Reference Material for Spectrophotometry—Selection, Preparation, Certification, and Use of SRM 930 and SRM 1930

NIST Special Publication 260-140 Technical Specifications for Certification of Spectrophotometric NTRMs

2.3 *ISO Publications:*[5]

ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

ISO Guide 34 General Requirements for the Competence of Reference Material Producers

3. Terminology

3.1 *Definitions:*

3.1.1 For the definitions of terms used in this practice, refer to Terminology E131.

3.1.2 For a description of the instrumental parameters evaluated in this practice, refer to Practice E275.

3.1.3 For a description of quantitative ultraviolet spectrophotometric techniques, refer to Practices E169.

4. Significance and Use

4.1 This practice permits an analyst to compare the performance of an instrument to the manufacturer's supplied performance specifications and to verify its suitability for continued routine use. It also provides generation of calibration monitoring data on a periodic basis, forming a base from which any changes in the performance of the instrument will be evident.

5. Reference to this Calibration-Monitoring Procedure

5.1 Reference to this practice in any spectrophotometric calibration-monitoring scheme shall constitute due notification that the adequacy of the spectrophotometer performance has been evaluated by means of this practice. Performance is considered to be adequate when the data obtained are within the stated tolerances from the true values.

6. Instrument Operation

6.1 In obtaining spectrophotometric calibration data the analyst must select the proper instrumental operating conditions to realize satisfactory instrument performance. Operating conditions for individual instruments are best obtained from the manufacturer's literature because of variations in instrument design.

6.2 When using reference materials, all the components of the spectrophotometer must be functioning properly. In addition, the temperature of the specimen compartment should be between 20 and 25°C. Matched solution cells should be used for calibration purposes.

6.3 Each of the above factors in instrument operation is important in the determination of wavelength and absorbance accuracy.

7. Determination of Wavelength Accuracy in the Ultraviolet and Visible Spectral Regions

7.1 *Discussion*—The holmium oxide glass filter **(1, 2)**[6] or solution standard (NIST Special Publication 260-102) may be

[3] 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.

[4] Available from National Technical Information Service (NTIS), 5301 Shawnee Rd., Alexandria, VA 22312, http://www.ntis.gov.

[5] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

[6] The boldface numbers in parentheses refer to a list of references at the end of this standard.