



Designation: E1866 – 97 (Reapproved 2021)

Standard Guide for Establishing Spectrophotometer Performance Tests¹

This standard is issued under the fixed designation E1866; 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 guide covers basic procedures that can be used to develop spectrophotometer performance tests. The guide is intended to be applicable to spectrophotometers operating in the ultraviolet, visible, near-infrared and mid-infrared regions.

1.2 This guide is not intended as a replacement for specific practices such as Practices E275, E925, E932, E958, E1421, or E1683 that exist for measuring performance of specific types of spectrophotometers. Instead, this guide is intended to provide guidelines in how similar practices should be developed when specific practices do not exist for a particular spectrophotometer type, or when specific practices are not applicable due to sampling or safety concerns. This guide can be used to develop performance tests for on-line process spectrophotometers.

1.3 This guide describes univariate level zero and level one tests, and multivariate level A and level B tests which can be implemented to measure spectrophotometer performance. These tests are designed to be used as rapid, routine checks of spectrophotometer performance. They are designed to uncover malfunctions or other changes in instrument operation, but do not specifically diagnose or quantitatively assess the malfunction or change. The tests are not intended for the comparison of spectrophotometers of different manufacture.

1.4 *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.*

1.5 *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.*

¹ This guide is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and Separation Science and is the direct responsibility of Subcommittee E13.03 on Infrared and Near Infrared Spectroscopy.

Current edition approved April 1, 2021. Published April 2021. Originally approved in 1997. Last previous edition approved in 2013 as E1866 – 97 (2013). DOI: 10.1520/E1866-97R21.

2. Referenced Documents

2.1 ASTM Standards:²

E131 Terminology Relating to Molecular Spectroscopy

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

E925 Practice for Monitoring the Calibration of Ultraviolet-Visible Spectrophotometers whose Spectral Bandwidth does not Exceed 2 nm

E932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrometers

E958 Practice for Estimation of the Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers

E1421 Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests

E1655 Practices for Infrared Multivariate Quantitative Analysis

E1683 Practice for Testing the Performance of Scanning Raman Spectrometers

3. Terminology

3.1 *Definitions*—For terminology relating to molecular spectroscopic methods, refer to Terminology E131.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *action limit, n*—the limiting value from an instrument performance test, beyond which the spectrophotometer is expected to produce potentially invalid results.

3.2.2 *check sample, n*—a single pure compound, or a known, reproducible mixture of compounds whose spectrum is constant over time such that it can be used in a performance test.

3.2.3 *level A test, n*—a pass/fail spectrophotometer performance test in which the spectrum of a check or test sample is compared against historical spectra of the same sample via a multivariate analysis.

3.2.4 *level B test, n*—a pass/fail spectrophotometer performance test in which the spectrum of a check or test sample is

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.

analyzed using a multivariate model, and the results of the analysis are compared to historical results for prior analyses of the same sample.

3.2.5 level one (1) test, *n*—a simple series of measurements designed to provide quantitative data on various aspects of spectrophotometer performance and information on which to base the diagnosis of problems.

3.2.6 level zero (0) test, *n*—a routine check of spectrophotometer performance, which can be done in a few minutes, designed to visually detect significant changes in instrument performance and provide a database to determine instrument performance over time.

3.2.7 optical reference filter, *n*—an optical filter or other device which can be inserted into the optical path in the spectrophotometer or probe producing an absorption spectrum which is known to be constant over time such that it can be used in place of a check or test sample in a performance test.

3.2.8 test sample, *n*—a process or product sample, or a mixture of process or product samples which has a constant spectrum for a finite time period and which can be used in a performance test. Test samples and their spectra are generally not reproducible in the long term.

4. Significance and Use

4.1 If ASTM Committee E13 has not specified an appropriate test procedure for a specific type of spectrophotometer, or if the sample specified by a Committee E13 procedure is incompatible with the intended spectrophotometer operation, then this guide can be used to develop practical performance tests.

4.1.1 For spectrophotometers which are equipped with permanent or semi-permanent sampling accessories, the test sample specified in a Committee E13 practice may not be compatible with the spectrophotometer configuration. For example, for FT-MIR instruments equipped with transmittance or IRS flow cells, tests based on polystyrene films are impractical. In such cases, these guidelines suggest means by which the recommended test procedures can be modified so as to be performed on a compatible test material.

4.1.2 For spectrophotometers used in process measurements, the choice of test materials may be limited due to process contamination and safety considerations. These guidelines suggest means of developing performance tests based on materials which are compatible with the intended use of the spectrophotometer.

4.2 Tests developed using these guidelines are intended to allow the user to compare the performance of a spectrophotometer on any given day with prior performance. The tests are intended to uncover malfunctions or other changes in instrument operation, but they are not designed to diagnose or quantitatively assess the malfunction or change. The tests are not intended for the comparison of spectrophotometers of different manufacture.

5. Test Conditions

5.1 When conducting the performance tests, the spectrophotometer should be operated under the same conditions as will

be in effect during its intended use. Sufficient warm-up time should be allowed before the commencement of any measurements.

5.1.1 If possible, the optical configuration used for measurements of test and check samples should be identical to that used for normal operations. If identical optical configurations are not possible, the user should recognize that the performance tests may not measure the performance of the entire instrument.

5.1.2 Data collection and computation conditions should generally be identical to those used in normal operation. Spectral data used in performance tests should be date and time stamped, and the results of the tests should be stored in a historical database.

6. Samples Used for Performance Testing

6.1 The sample used for performance testing is chosen to be compatible with the spectrophotometer configuration, and to provide spectral features which are adequate for the tests being performed.

6.1.1 The sample used for performance testing should generally be in the same physical state (gas, liquid, or solid) as the samples to be analyzed during normal operation of the spectrophotometer.

6.1.2 The sample used for performance testing should be physically and chemically compatible with the samples analyzed during normal operation.

6.1.3 The sample used for performance is chosen such that its spectrum is similar to the spectra which will be collected during normal operation.

6.1.4 The sample used for performance testing should have several significant absorbances ($0.3 < \text{absorbance} < 1.0$) across the spectral range used for normal operation of the spectrophotometer.

6.1.5 In order to adequately determine the photometric linearity of the instrument, the peak absorbance for at least one absorption band of the sample should be similar to and preferably slightly greater than the largest absorbance expected for samples measured during normal operation.

6.2 Check Samples—Check samples are generally used for conducting performance tests. Check samples are single pure compounds or mixtures of compounds of definite composition.

6.2.1 If mixtures are utilized as check samples, they must be prepared in a repeatable manner and, if stored, stored such that the mixture is stable over long periods of time. In preparing mixtures, components should be accurately pipetted or weighed at ambient temperature. It is recommended that mixtures be independently verified for composition prior to use.

6.2.2 While mixtures can be used as check samples, their spectra may be adversely affected by temperature sensitive interactions that may manifest themselves by wavelength (frequency) and absorbance changes.

6.3 Test Samples—A test sample is a process or product sample or a mixture of process or product samples whose spectrum is expected to be constant for the time period it is