



Standard Practice for Describing and Measuring Performance of Laboratory Fourier Transform Near-Infrared (FT-NIR) Spectrometers: Level Zero and Level One Tests¹

This standard is issued under the fixed designation E 1944; 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 practice covers two levels of tests to measure the performance of laboratory Fourier transform near infrared (FT-NIR) spectrometers. This practice applies to the short-wave near infrared region, approximately 800 nm ($12\,500\text{ cm}^{-1}$) to 1100 nm (9090.91 cm^{-1}); and the long-wavelength near infrared region, approximately 1100 nm (9090.91 cm^{-1}) to 2500 nm (4000 cm^{-1}). This practice is intended mainly for transmittance measurements of gases and liquids, although it is broadly applicable for reflectance measurements.

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.*

2. Referenced Documents

2.1 ASTM Standards:

E 131 Terminology Relating to Molecular Spectroscopy²

E 168 Practices for General Techniques of Infrared Quantitative Analysis²

E 932 Practices for Describing and Measuring Performance of Dispersive Infrared Spectrometers²

E 1252 Practice for General Techniques for Qualitative Analysis²

E 1421 Practice for Describing and Measuring Performance of Fourier Transform Infrared (FT-IR) Spectrometers: Level Zero and Level One Tests²

3. Terminology

3.1 For definitions of terms used in this practice, refer to Terminology E 131. All identifications of spectral regions and absorbance band positions are given in nanometers (nm), and

wavenumbers (cm^{-1}); and spectral energy, transmittance, reflectance, and absorbance are signified by the letters E, T, R and A respectively. A subscripted number signifies a spectral position in nanometers, with wavenumbers in parenthesis (for example,

$A^{1940(5154.64)}$, denotes the absorbance at 1940 nm or 5154.64 cm^{-1}).

4. Significance and Use

4.1 This practice permits an analyst to compare the general performance of a laboratory instrument on any given day with the prior performance of that instrument. This practice is not intended for comparison of different instruments with each other, nor is it directly applicable to dedicated process FT-NIR analyzers. This practice requires the use of a check sample compatible with the instrument under test as described in 5.3.

5. Test Conditions

5.1 *Operating Conditions*—In obtaining spectrophotometric data for the check sample, the analyst must select the proper instrumental operating conditions in order to realize satisfactory instrument performance. Operating conditions for individual instruments are best obtained from the manufacturer's instructional literature due to the variations with instrument design. It should be noted that many FT-NIR instruments are designed to work best if left in standby mode when they are not in use. A record should be kept to document the operating conditions selected during a test so that they can be duplicated for future tests. Note that spectrometers are to be tested only within their respective recommended measurement wavelength (wavenumber) ranges.

5.2 Instrumental characteristics can influence these measurements in several ways. Vignetting of the beam (that is, the aperture of the sample cell is smaller than the diameter of the near infrared beam at the focus) reduces the transmittance value measured in nonabsorbing regions, and on most instruments can change the apparent wavelength (or wavenumber) scale by a small amount, usually less than 0.01 nm (0.1 cm^{-1}). Focus changes can also change transmittance values, so the sample should be positioned in the same location in the sample compartment for each measurement. The angle of acceptance

¹ This practice is under the jurisdiction of ASTM Committee E-13 on Molecular Spectroscopy and is the direct responsibility of Subcommittee E 13.03 on Infrared Spectroscopy.

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² *Annual Book of ASTM Standards*, Vol 03.06.

(established by the f number) of the optics between the sample and detector significantly affects apparent transmittance. Heating of the sample by the beam or by the higher temperatures which exist inside most spectrometers changes absorbances somewhat, and even changes band ratios and locations slightly. Allow the sample to come to thermal equilibrium prior to measurement.

5.3 The recommended check sample should meet the following requirements: the check sample should be fully compatible with the requirements for repeatable sample presentation to the measuring spectrophotometer. The check sample should consist of a single pure compound or precisely known mixture of compounds which is spectroscopically stable over months or years. The spectra obtained from such a check sample should be known to indicate changes in the spectrophotometer, not the check sample itself. It is recommended that independent verification of the integrity of the check sample be used prior to test measurement. The check sample should be measured under precisely the sample measurement conditions of temperature, humidity, and instrument set up configuration. Suggested check samples may include, but are not limited to the following: for gases, water vapor at 5.89 Torr and 1 atmosphere in a 2 m gas cell, or methane at 18 psig pressure in a 10 cm gas cell; for liquids, pure spectroscopic grade hydrocarbon compounds (for example, toluene, decane, isooctane, etc.), or precise mixtures of these pure compounds; for reflectance measurements of solids, rare earth oxides mixed with white halon powder, or Spectralon³-based rare earth oxide reflectance standards. Reference reflectance standards yielding a featureless, near 100 % reflectance spectrum are pure powdered sulfur, halon, or Spectralon.

6. Level Zero Tests

6.1 *Nature of Tests*—Routine checks of instrument performance can be performed within a few minutes. They are designed to uncover malfunctions or other changes in instrument operation but not to specifically diagnose or quantitatively assess any malfunction. For Level Zero tests, a resolution of 4 cm^{-1} and a nominal measurement time of 30 s is recommended. Resolution and measurement times can be specified to match conditions used for routine measurement applications. The exact measurement time, along with the date, time, sample identification, number of scans, and operator's name, should always be recorded.

6.2 *Philosophy*—The philosophy of the tests is to use previously stored test results as bases for comparison and the visual display screen or plotter to overlay the current test results with the reference results (known to be good). If the old and new results agree, they are simply reported as no change. Level Zero consists of three tests. Run the tests under the same conditions that you would normally use to run a sample (that is, sample temperature, purge time, warm-up time, beam splitter type, detector configuration, etc.)

6.3 *Variations in Operating Procedure for Different Instruments*—Most of the existing FT-NIR instruments should

be able to use the tests in this procedure without modification. However, a few instruments may not be able to perform the tests exactly as they were written. In these cases, it should be possible to obtain the same final data using a slightly different procedure. The FT-NIR manufacturer should be consulted for appropriate alternative procedures.

6.4 *Sample*—The check sample used for performance tests is described in 5.3. The same sample should be used for all test comparisons (note serial number, or other identifying information, of sample) as well as orientation of the sample within the sample compartment during test measurements.

6.5 *Reference Spectra*—Two spectra acquired and stored during the last major instrument calibration are used as references. These spectra will be identified as Reference 1 and Reference 2.

6.5.1 Reference 1 is a Fourier-transformed single-beam energy spectrum of an empty beam. (in this and all later usage, empty beam means that nothing is in the sample path except dry air or the purge gas normally present within the spectrometer sample compartment). For reflectance measurements this spectrum is a spectrum of a flat, pure reflectance standard approximating 100 % R.

6.5.2 Reference 2 is a transmittance spectrum of the check sample. For reflectance measurements this spectrum is a reflectance spectrum of the check sample.

6.6 *Repeatability of Procedures*—Care should be taken that each of the spectral measurements is made in a consistent and repeatable manner, including sample orientation (although, different spectral measurements do not necessarily use the identical procedure). In particular, for those instruments having more than one sample beam or path in the main sample compartment, all of the test spectra always should be measured using the same optical path.

6.7 *Measurements*—Three test spectra will be acquired and stored. The test spectra will be identified hereafter as Spectrum 1, Spectrum 2, and Spectrum 3.

6.7.1 *Spectrum 1*—An empty-beam spectrum stored as a Fourier-transformed single beam energy spectrum (or as an interferogram). If stored as an interferogram, it must be transformed before use in the ensuing tests.

6.7.2 *Spectrum 2*—An empty-beam spectrum taken immediately after Spectrum 1. This spectrum should be stored as either a Fourier-transformed single-beam energy spectrum or as a transmittance spectrum ratioed against Spectrum 1.

6.7.3 *Spectrum 3*—A spectrum of the check sample obtained reasonably soon after Spectrum 2. This spectrum should be stored as a transmittance spectrum (or reflectance spectrum, when applicable) ratioed against either Spectrum 1 or Spectrum 2, or as a single-beam energy spectrum. To reproducibly insert the sample, the serial number (or other identifying information) should be right side up facing the instrument detector (or aligned in a manner that allows repeatable measurements each time the check sample is measured).

7. Level Zero Test Procedures

7.1 *Energy Spectrum Test*—Overlay Spectrum 1 and Reference 1. Note any changes in energy level across the spectrum. Ratio Spectrum 1 to Reference 1. Video display resolution may limit the accuracy to which this test can be interpreted if the

³ Spectralon, available from Labsphere, Inc., P.O. Box 70, Shaker St., North Sutton, NH 03260-0070, has been found satisfactory for this purpose.