Designation: E1944 – 98 (Reapproved 2021)

Standard Practice for
Describing and Measuring Performance of Laboratory Fourier Transform Near-Infrared (FT-NIR) Spectrometers: Level Zero and Level One Tests[1]

This standard is issued under the fixed designation E1944; 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 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 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.*

1.4 *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]

E131 Terminology Relating to Molecular Spectroscopy

E168 Practices for General Techniques of Infrared Quantitative Analysis

E932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrometers

E1252 Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis

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

3. Terminology

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

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

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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.

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 (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 Spectralon[3]-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.

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

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 comparison is made on-screen. In addition, if the interferogram was saved, it may be displayed or plotted and the center burst height recorded. Changes in the interferogram height are difficult to interpret since minor decreases in source temperature that only affect high frequencies can result in changes in interferogram height. These changes do not affect photometric accuracy.

7.1.1 *Reportage*—Report by making an overlay plot of Spectrum 1 energy ratioed against Reference 1 energy over the range of 95 to 105 % T, and by reporting the following energy ratios:

$$\text{For short} - \text{wave near infrared:} \tag{1}$$

$$\text{RATIO}^{800/1000(12\ 500/10\ 000)} = \text{E}^{800/1000(12\ 500/10\ 000)}$$

$$\text{For long} - \text{wave near infrared:}$$

$$\text{RATIO}^{1500/2000(6666.67/5000)} = \text{E}^{1500/2000(6666.67/5000)}$$

$$\text{RATIO}^{2000/2500(5000/4000)} = \text{E}^{2000/2500(5000/4000)}$$

Report the date and time of both spectra used, and the actual numbers of scans and measurement times, as well as details of the instrument set up conditions.

7.1.2 *Interpretation*—An overall drop in the energy level in which the largest percentage of change occurs at higher wavenumbers usually indicates interferometer misalignment or a reduction in source temperature. An overall drop in the energy level without wavelength (wavenumber) dependence suggests beam obstruction (vignetting) or misalignment of non-interferometer optical components. The appearance of bands or other features indicates purge gas contributions, beam obstruction by a partially transmitting object, oil or smoke deposition on mirrors or windows, or a forgotten sample within the beam. With cooled detectors (for example InSb), the appearance of a broad band around 1940 nm (5154.64 cm^{-1}) indicates ice deposition on the detector surface. Non-zero energy levels below the detector cut-off (more than 0.2 % of the maximum energy-level in the single beam spectrum) indicate system nonlinearities or detector saturation. On many instruments anomalous increases in the actual measurement time for a set number of scans indicate instrument problems (mis-triggering, white light misalignment, excessive purge rate, or interferometer drive-problems).

7.2 *One Hundred Percent Line Test*—Ratio Spectrum 2 to Spectrum 1. Note the noise level and any variations from 100 % transmittance (or reflectance) across the spectrum.

7.2.1 *Reportage*—Make an overlay plot of Spectra 1 and 2. Then ratio the two and plot the 100 % transmittance (or reflectance) line. The ordinate range should be 99 to 101 % T/R. If the noise or baseline drift exceeds these bounds, make a plot from 90 to 110 % T/R and consider performing Level One tests. Report the RMS (preferred) or peak-to-peak noise levels at over a ~8-18 nm (100 cm^{-1}) range centered at 800 nm (12 500 cm^{-1}), 1000 nm (10 000 cm^{-1}), 1500 nm (6666.67 cm^{-1}), 2000 nm (5000 cm^{-1}), 2500 nm (4000 cm^{-1}). If the instrument wavelength (wavenumber) range does not include some of these, substitute the nearest measurable wavelength (frequency).

7.2.2 *Interpretation*—Excessive noise may result from misalignment or source malfunction (refer to the energy spectrum test) or from a malfunction in the detector or the electronics. Repetitive noise patterns (for example, spikes or sinusoids) sometimes indicate digital problems. Isolated noise spikes may be digital malfunctions or they can indicate electromagnetic interference. Positive or negative bands often indicate a rapid change in purge quality. Simultaneously positive and negative sharp bands in the water region may indicate instrumental problems or excessive water vapor within the spectrometer. Deviations from the 100 % level (usually at lower wavelengths (higher wavenumbers) indicate interferometer, detector, or source instability (see Practice E1421).

7.3 *Check Sample Test*—Ratio Spectrum 3 to Spectrum 2 (or 1) to produce a check sample transmittance spectrum (or reflectance spectrum, when applicable). Convert all spectra to absorbance spectra. Subtract the stored absorbance check sample spectrum from this new absorbance check sample spectrum. Note any changes.

7.3.1 *Reportage*—Plot the check sample absorbance spectrum over the reported dynamic range of the instrument. Plot the subtraction result as a full scale spectrum.

7.3.2 *Interpretation*—Additional sharp features in the water vapor absorption regions indicate excessive water vapor in the sample compartment. Instrumental problems may include Jacquinot vignetting, source optics or laser misalignment, or interferometer scan problems. In the subtraction spectrum, first-derivative-like bandshapes that correspond to absorption band positions indicate these instrumental problems. Artifacts appearing only at the positions of the strongest (completely absorbing) bands may indicate phasing or other problems associated with detector non-linearity. Artifacts at both medium and strong band positions indicate analog electronic, ADC, or computer problems, or sampling jitter, (Zachor-Aaronsen distortion).

8. Level One Tests

8.1 *Nature of Test*—The tests described for Level One use only the check sample and are designed to more thoroughly test the instrument performance. The main purpose of Level One tests is to compare performance with previous results obtained on the same instrument. The tests can also be used to compare