

Transmission Standards for FT-IR Spectrometer Qualification

Using Traceable Polystyrene and Traceable
NG11 Glass

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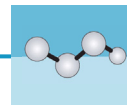
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Nicolet Series FT-IR Validation Wheel

This document describes the traceable polystyrene and traceable NG11 glass transmission standards for the Fourier transform infrared (FT-IR) spectrometers from Thermo Fisher Scientific. The standards can be used to verify spectrometer performance.

The transmission standards are required for Thermo Scientific FT-IR systems purchased with the ValPro™ System Qualification package. The standards are offered in two configurations, as a standards wheel (both standards are mounted on a wheel that is installed inside the spectrometer) and as a set of individual standards (each standard is mounted on a card that fits the transmission sample holder in the spectrometer sample compartment).

The transmission standards are traceable to certified standards stored at Thermo Fisher Scientific. This document explains the company's internal process for measuring the transmission standards to establish traceability, including all associated data and reports.

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About the Transmission Standards

The transmission set contains one each of the following samples:

- A Schott NG11 absorbing optical glass reference. NG11 glass produces a spectrum with absorption bands that have different relative intensities over a broad spectral range. Selected bands can be used to verify the instrument linearity.
- A 1.5 mil polystyrene reference. Polystyrene has defined absorption bands which can be used to verify wavelength accuracy in the mid-infrared spectral range (4,000 to 400 cm^{-1}).

The transmission samples are serialized. After the samples have been calibrated and the measured values verified to be within the tolerance ranges of the certified standards, the samples are considered to be traceable to those standards and are therefore referred to as “traceable standards.”

Note It is technically correct to say that the samples are not traceable until the calibration and verification are completed and the report is approved but, to improve clarity, the standards are referred to as traceable throughout this document.

Traceable Glass Standard

The traceable glass standard is used to demonstrate that the instrument detector produces a linear response. It is traceable to a NG11 glass standard with a standard thickness of ~1 millimeter that was tested and certified to samples measured at the National Institute of Standards and Technology (NIST). The certified standard is reported in the NIST Report of Calibration (Reference: 6000167599).

We selected seven locations in the mid-infrared spectrum of the NG11 glass to demonstrate detector linearity. The zero-gradient calibration points chosen for calibration produce transmittance values of approximately 76% at 3990 cm^{-1} , 15% at 3512 cm^{-1} , 37% at 3031 cm^{-1} , 6% at 2739 cm^{-1} , 17% at 2598 cm^{-1} , 10% at 2473 cm^{-1} , and 0.01% at 2010 cm^{-1} .

Traceable Polystyrene Standard

The traceable polystyrene standard is used to demonstrate the wavelength accuracy and reproducibility of the instrument. It is traceable to a polystyrene standard with a standard thickness of ~1.5 mil (~0.038 mm) that was measured and certified by the National Institute of Standards and Technology (NIST) according to the NIST Certificate for Standard Reference Material 1921b.

Based on European Pharmacopoeia (EP) recommendations, we selected seven peaks in the mid-infrared spectrum of polystyrene to demonstrate wavelength accuracy and reproducibility. The peaks are located at approximately 3060.0 cm^{-1} , 2849.5 cm^{-1} , 1942.9 cm^{-1} , 1601.2 cm^{-1} , 1583.0 cm^{-1} , 1154.5 cm^{-1} , and 1028.3 cm^{-1} . These peaks are recommended because of their uniform shape and the relative absence of spectral interference in the region and because they cover the mid-infrared spectral range.

Measuring the Traceable Standards

We use the following process to establish traceability of the transmission standards. All measurements are made using an instrument that meets factory test specifications and has passed ValPro spectrometer operational qualification (OQ) test and the Quantification Algorithms OQ test (see the Operational Qualification manual for the spectrometer for details).

The background material used for the measurements is air. This is accomplished by removing the sample from the instrument beam path during the background measurement or collecting the background through the empty position of the standards wheel.

Note In order to preserve the certified standards, we generally use the process described below to create a secondary standard that is traceable to the certified standard, and then use the secondary standard to create the traceable standards that ship with our instruments. We consider the certified standard and the secondary standard to be equally valid for the purposes of establishing traceability to the certified standard.

Instructions used to measure transmission samples to establish traceability:

1. Install the certified NG11 glass standard (or a secondary standard) in the instrument.

The standard can be mounted on a wheel that is installed inside the spectrometer or on a card that fits the transmission sample holder in the spectrometer sample compartment.

2. Measure the certified (or secondary) glass standard three times.

Note If using card-mounted standards, remove the card-mounted glass standard before continuing.

3. Install the certified (or secondary) polystyrene standard in the instrument.
4. Measure the certified (or secondary) polystyrene standard two times.
5. Remove the certified (or secondary) standard or wheel from the instrument.
6. Install the NG11 glass sample (or the sample wheel) in the instrument.
7. Measure the glass sample three times.

Note If using card-mounted standards, remove the card-mounted glass sample before continuing.

8. Install the polystyrene sample in the instrument.
9. Measure the polystyrene sample two times.
10. Remove the polystyrene sample (or the sample wheel) from the instrument.
11. Print the traceability reports (one for each sample type).
The reports show the results of the measurements.
12. Verify that the values measured from the samples are within the specified tolerance ranges of the certified standards.
13. If the measured values are within the tolerance ranges of the certified standards, sign the reports and acquire the required approval signatures.

The signatures demonstrate that the samples can be traced to the certified standards.

Uncertainty in the Measurements

Measurements of the NG11 glass are reported in % transmittance. The uncertainties of the NG11 glass measurements are based on historical calibration data for certified NG11 glass standards.¹ The uncertainties for the seven transmission measurements are: $\pm 0.18\%$ at 3990 cm^{-1} , $\pm 0.08\%$ at 3512 cm^{-1} , $\pm 0.12\%$ at 3031 cm^{-1} , $\pm 0.06\%$ at 2739 cm^{-1} , $\pm 0.09\%$ at 2598 cm^{-1} , $\pm 0.08\%$ at 2473 cm^{-1} , and $\pm 0.01\%$ at 2010 cm^{-1} .

The polystyrene peak measurements are reported in wavenumbers (cm^{-1}). The uncertainties of the polystyrene peak measurements are taken from the European Pharmacopoeia (EP), Fifth Edition, General Chapter 2.2.24, Verification of the Wavenumber Scale. The uncertainties for the seven peak location measurements are: $\pm 1.0\text{ cm}^{-1}$ for the nominal peaks at 3060 cm^{-1} , 2850 cm^{-1} , 1943 cm^{-1} , 1601 cm^{-1} , 1583 cm^{-1} , 1154 cm^{-1} , and 1028 cm^{-1} .

Storage and Expiration of the Traceable Standards

If the samples are properly handled and stored, the sample measurements are accurate, within the specified measurement uncertainties, for the following period:

- Samples mounted on a wheel are accurate for five years from the date of issue shown on the wheel.
- Card-mounted samples are accurate for two years from the date of issue shown on the card.

Thermo Fisher Scientific stores the glass and polystyrene samples under controlled conditions of registration and access and does not release them, even temporarily, from this controlled storage prior to issue. After the samples have been issued, they must be handled and stored under controlled conditions to maintain accuracy.

To prevent contamination of the sample, do not allow it to contact chemicals or your skin. Degradation or contamination of the sample could affect the qualification results. To verify accuracy, we recommend annual re-qualification of the instrument and the standard (see the Installation Re-Qualification manual for the spectrometer for instructions).

When not in use, card-mounted samples must be stored in their original UV-protective case and the case stored in a dark place at room temperature. Samples mounted on a wheel must be stored in a serviced and functioning Thermo Scientific FT-IR spectrometer.

NOTICE Improper use or care of the instrument, or improper maintenance of the instrument, may cause permanent damage to the standards wheel.

¹Historical National Physics Laboratory (NPL) calibration data supported by the technical report showing equivalency between NIST calibrated and NPL certified NG11 glass. Chunnillal, C.J., Clarke, F. J. J., Smart, M.P., Hanssen, L. M., Kaplan, S.G., 2003 NIST-NPL comparison of mid-infrared regular transmittance and reflectance, *Metrologia* 40 S55

Samples mounted on a wheel must be replaced after five years from the date of issue. Because card-mounted samples are handled regularly and, therefore, exposed to potential contamination, damage and degradation, they must be replaced after two years from the date of issue.

About the Traceability Reports

The measurement process to establish traceability of the transmission standards produces two reports. The first report, titled “Linearity Calibration of the Validation Wheel Standard,” provides the calibration data for the photometric linearity of the serialized, traceable NG11 glass and demonstrates that the glass is traceable to the NIST-certified NG11 glass standard. The second report, titled “Wavelength Accuracy Verification of the Validation Wheel Polystyrene Standard,” provides the results from the polystyrene measurements and shows that the polystyrene is traceable to the certified NIST standard.

The reports include the following information:

- Serial number(s) of the traceable standards or wheel
- Date the report was created
- Serial number of the instrument used in the traceability process
- Serial number(s) of the certified (or secondary) standards or wheel used to establish traceability
- Signature and date the report was verified
- Approval signature
- Issue date (the date the traceable standards were approved for shipment from the Thermo Fisher Scientific factory)

The signatures at the end of each report confirm traceability to the NIST certificates.

Linearity Calibration Report

The Linearity Calibration report provides the calibration data for the photometric linearity of the serialized, traceable NG11 glass standard and demonstrates that the glass is traceable to the certified NIST standard. A copy of the report is provided with this document. The report is traceable to the serial number of the traceable standard or wheel and the serial number of the certified (or secondary) standard or wheel. The Linearity Calibration report and the associated calibration data are archived at Thermo Fisher Scientific.

The report shows the % transmittance values for the seven locations that were measured for the glass standard (approximately 3990 cm^{-1} , 3512 cm^{-1} , 3031 cm^{-1} , 2739 cm^{-1} , 2598 cm^{-1} , 2473 cm^{-1} and 2010 cm^{-1}). The background material used for the measurements is air.

The Linearity Calibration report is divided into four sections. The first section shows the measurements of the NIST-certified (or secondary) NG11 glass standard used to verify the traceable glass standard. The second section shows the measurements from the traceable glass standard compared with the measurements made on the certified (or secondary) glass standard. The third section shows the calibration data for the traceable glass standard. The last section bears the signatures that confirm traceability.

Results From the Certified Glass Standard

The section of the report titled “Measurement of the Schott NG11 Glass Standard” shows the reference values for the NG11 glass standard and the values measured at the Thermo Fisher Scientific factory.

The column labeled “Actual” shows the measured value and the uncertainty value for each wavelength as reported on the NIST Certificate of Calibration.

The columns labeled “Meas1,” “Meas2,” “Meas3,” and “Average” show the individual signal intensity values of the certified (or secondary) glass standard measured at the Thermo Fisher Scientific factory and the average of the three measurements.

The Slope value results from a linear regression analysis of the Actual measurements compared with the Average measurements referred to above. The measured slope must fall between 0.985 and 1.015 inclusive. The regression equation is used to calibrate the traceable glass standard.

Results From the Traceable Glass Standard

The section of the report titled “Measurement of the Traceable Validation Wheel Glass Standard” shows the measurements of the traceable glass standard compared to the measurements made on the certified (or secondary) standard.

The columns labeled “Meas1,” “Meas2,” “Meas3,” and “Average” show the individual signal intensity values of the traceable glass standard and the average of the three measurements. The column labeled “+-” shows the calculated range of uncertainty of those measurements.

The column labeled “Interp” shows the same values that are found in the “Average” column.

Summary Data From the Glass Standard

The section of the report titled “Traceable Validation Wheel Glass Standard Summary” shows the % transmittance values used to describe (calibrate) the traceable glass standard. The values are taken directly from the Interp column in section 2 of the Linearity Calibration report. These values are defined to be the true measurement values for the traceable glass standard.

Wavelength Accuracy Verification Report

The Wavelength Accuracy Verification report provides the results from the polystyrene measurements and shows that the polystyrene is traceable to the certified (or secondary) NIST standard. A copy of the report is provided with this document. The report is traceable to the serial number of the traceable standard or wheel and the serial number of the certified (or secondary) standard or wheel. The Wavelength Accuracy Verification report and the associated data are archived at Thermo Fisher Scientific.

The report shows the location values for the seven peaks (absorptions) that were measured for the polystyrene standard (approximately 3060.0 cm^{-1} , 2849.5 cm^{-1} , 1942.9 cm^{-1} , 1601.2 cm^{-1} , 1583.0 cm^{-1} , 1154.5 cm^{-1} , and 1028.3 cm^{-1}). The background material used for the measurements is air.

The Wavelength Accuracy Verification report is divided into four sections. The first section shows the measurements of the NIST-certified (or secondary) polystyrene standard used to verify the traceable polystyrene standard. The second section shows the values measured from the traceable polystyrene standard. The third section shows the verification data for the traceable standard. The last section bears the signatures that confirm traceability.

Results From the Certified Polystyrene Standard

The section of the report titled “Measurement of the Polystyrene Standard” shows the reference values for the NIST-certified polystyrene standard and the values measured at the Thermo Fisher Scientific factory.

The column labeled “Expected Location” shows the nominal value for each wavelength as described in chapter 2.2.4 of the European Pharmacopoeia. The column labeled “Uncert” shows the EP-defined uncertainty values for those measurements.

The columns labeled “Meas1,” “Meas2,” and “Measured Average” show the individual peak location values of the certified (or secondary) polystyrene standard measured at the Thermo Fisher Scientific factory and the average of the two measurements.

Results From the Traceable Polystyrene Standard

The section of the report titled “Measurement of the Traceable Polystyrene Standard” shows the measurements of the traceable polystyrene standard compared to the measurements made on the certified (or secondary) polystyrene standard.

The column labeled “Expected Location” shows the nominal value for each wavelength as described in chapter 2.2.4 of the European Pharmacopoeia.

The column labeled “Reference Location” shows the calculated average of the two measurements taken from the certified (or secondary) polystyrene standard. (These are the same values report in the Measured Average column in section one of the Wavelength Accuracy Verification report.)

The columns labeled “Meas1,” “Meas2,” and “Measured Average” show the individual peak location values of the traceable polystyrene standard and the average of the two measurements.

Summary Data From the Polystyrene Standard

The section of the report titled “Traceable Validation Wheel Polystyrene Standard Summary” shows the peak location values used to describe the traceable polystyrene standard. The values are taken directly from the Measured Average column in section 2 of the Wavelength Accuracy Verification report. These values are defined to be the true measurement values for the traceable polystyrene standard.

Verifying the Traceable Standards

Thermo Fisher Scientific assigns each standard or standards wheel a unique serial number and includes the serial number information in the traceability reports. To verify the traceable standards, check that the serial number printed on the standards wheel installed in the instrument (or the serial numbers printed on the card-mounted standards) matches the serial number of the traceable standards printed on the traceability reports.

We position the serial number of the standards wheel so it is visible at the top of the wheel when it is in the home position. If the instrument has a detector hatch, open the hatch to view the serial number for the standards wheel. Otherwise, open the instrument cover to view the serial number.