



# Polystyrene Reflection Standard for ATR Accessory Qualification

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# Polystyrene Reflection Standard for ATR Smart Accessory Qualification

This document describes the traceable polystyrene standard for the attenuated total reflection (ATR) Smart accessories from Thermo Fisher Scientific. The standard can be used to verify the performance of a Thermo Scientific FT-IR spectrometer with the installed accessory.

The ATR standard is available for Thermo Scientific ATR Smart accessories purchased with the ValPro™ System Qualification package. For convenience, the standard is mounted in a holder designed for ATR sampling using the accessory's built-in pressure device.

The ATR standard is traceable to a certified standard stored at our factory. This document explains the company's internal process for measuring the ATR standard to establish traceability, including all associated data and reports.

## About the traceable ATR standard

The ATR standard contains a thick (~0.038 inch) polystyrene reference sample. Polystyrene has defined absorption bands which can be used to verify wavelength accuracy in the mid-infrared spectral range (4,000 to 400  $\text{cm}^{-1}$ ). The sample 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. For the purposes of form and function, we use thick polystyrene for ATR measurements and take steps to establish traceability to the certified 1.5 mil polystyrene standard. See "Measuring the traceable standard."

The ATR sample is serialized. After the sample has been measured and the measured values verified to be within the tolerance ranges of the certified standard, the sample is considered to be traceable to that standard and is therefore referred to as a "traceable standard."

**Note** It is technically correct to say that the sample is not traceable until the verification is completed and the report is approved but, to improve clarity, the standard is referred to as traceable throughout this document. ▲

The traceable ATR standard is used to demonstrate the wavelength accuracy and reproducibility of the accessory. Based on European Pharmacopoeia (EP) recommendations, we selected five peaks in the mid-infrared spectrum of polystyrene to demonstrate wavelength accuracy and reproducibility. The peaks are located at approximately 2849.5  $\text{cm}^{-1}$ , 1601.2  $\text{cm}^{-1}$ , 1583.0  $\text{cm}^{-1}$ , 1154.5  $\text{cm}^{-1}$ , and 1028.3  $\text{cm}^{-1}$ . These peaks were chosen because of their uniform shape and the relative absence of spectral interference in the selected regions and because they cover the mid-infrared spectral range.

Note The EP recommends using seven peaks to measure instrument wavelength accuracy and reproducibility. However, due to inherent differences between the transmission and ATR sampling techniques, we select only the five peaks described above to establish traceability for ATR sampling. ▲

## Measuring the traceable ATR standard

For the purposes of form and function, we use thick polystyrene for ATR reflection measurements. This section describes the process used to establish that each thick polystyrene ATR standard can be traced to a secondary thick polystyrene ATR standard, which is traceable to a certified 1.5 mil polystyrene transmission standard. All measurements are made using a Thermo Scientific spectrometer that meets factory test specifications and has passed the ValPro spectrometer operational qualification (OQ) test and the Quantification Algorithms OQ test (see the operational qualification manual for the spectrometer for details) and a Thermo Scientific ATR Smart Accessory that has passed the OQ test for the accessory (see *Qualifying the Smart Orbit, Smart Performer and Smart iTR* for details).

Background measurements are taken through the ATR crystal without a sample in place. We use the same crystal material for background measurements that is used to measure the samples and standards.

## Establishing traceability to the certified 1.5 mil standard

To establish the validity of our traceability approach, we first tested a 1.5 mil polystyrene sample against a certified NIST 1.5 mil polystyrene standard using the transmission sampling technique and the wavenumber accuracy values and uncertainties recommended by the European Pharmacopoeia to verify that this secondary standard is traceable to the certified standard. Then we measured the 1.5 mil secondary standard using the single-bounce attenuated total reflection (ATR) technique, which requires that the material be compressed against the ATR crystal (this was a destructive test of the material). We then selected a thick polystyrene material that is better suited for repetitive

testing using the ATR technique and measured the thick polystyrene using the same single-bounce ATR technique and crystal that was used to measure the secondary 1.5 mil standard. We compared the results from the two materials and techniques and determined that the peak locations and uncertainties for the EP recommended peaks measured with the thick polystyrene sample using the ATR technique could be related directly to the location and uncertainty values measured with the certified 1.5 mil standard using the transmission technique. However, due to optical effects related to the ATR accessory, two of the bands specified in the EP method ( $3060\text{ cm}^{-1}$  and  $1942.5\text{ cm}^{-1}$ ) have been omitted. In summary, we consider the secondary 1.5 mil polystyrene transmission standard and the secondary thick polystyrene ATR reflection standard to be equally valid for the purposes of establishing traceability to the certified standard.

## Measuring the thick polystyrene samples

We take the following steps to measure each thick polystyrene sample to establish traceability.

1. Install the secondary thick polystyrene ATR standard on the ATR crystal. The standard must be mounted on the Thermo Scientific sample holder for ATR sampling.
2. Measure the secondary ATR standard two times.
3. Remove the secondary ATR standard from the instrument.
4. Install the thick polystyrene sample on the ATR crystal. The sample must consist of the same material as the secondary standard and it must be mounted on the same type of sample holder.
5. Measure the thick polystyrene sample two times.
6. Remove the polystyrene sample from the instrument.
7. Print the traceability report. The reports show the results of the measurements.
8. Verify that the values measured from the sample are within the specified tolerance ranges of the secondary ATR standard.
9. If the measured values are within the tolerance ranges of the secondary standard, sign the report and acquire the required approval signatures. The signatures demonstrate that the sample can be traced to the certified standard.

## Uncertainty in the measurements

The polystyrene peak measurements are reported in wavenumbers ( $\text{cm}^{-1}$ ). 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 five peak location measurements are:  $\pm 1.0 \text{ cm}^{-1}$  for the nominal peaks at  $2850 \text{ cm}^{-1}$ ,  $1601 \text{ cm}^{-1}$ ,  $1583 \text{ cm}^{-1}$ ,  $1154 \text{ cm}^{-1}$ , and  $1028 \text{ cm}^{-1}$ .

## Storage and expiration of the traceable ATR standard

If the thick polystyrene sample is properly handled and stored, the sample measurements are accurate, within the specified measurement uncertainties, for two years from the date of issue shown on the sample holder.

We store the traceable standards under controlled conditions of registration and access and do not release them, even temporarily, from this controlled storage prior to issue. After a standard has been issued, it 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 accessory and the standard (see the qualification manual for the accessory for instructions). When not in use, store the standard in its original UV-protective case and store the case in a dark place at normal room temperature.

Because the 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 report

The measurement process to establish traceability of the ATR standard produces a report titled “Wavelength Accuracy Verification of the ATR Polystyrene Standard.” The report provides the results from the polystyrene measurements and shows that the polystyrene is traceable to the secondary thick polystyrene ATR standard.



The report includes the following information:

- Serial number of the traceable ATR standard
- Date the report was created
- Serial number of the instrument used in the traceability process
- Serial number of the secondary thick polystyrene ATR standard used to establish traceability
- Signature and date the report was verified
- Approval signature
- Issue date (the date the traceable standard was approved for shipment from our factory)

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

A copy of the Wavelength Accuracy Verification report is provided with this document. The report is traceable to the serial number of the traceable ATR standard and the serial number of the secondary thick polystyrene ATR standard. We archive the Wavelength Accuracy Verification report and the associated data.

The report shows the location values for the five peaks (absorptions) that were measured for the polystyrene standard (approximately 2849.5  $\text{cm}^{-1}$ , 1601.2  $\text{cm}^{-1}$ , 1583.0  $\text{cm}^{-1}$ , 1154.5  $\text{cm}^{-1}$ , and 1028.3  $\text{cm}^{-1}$ ). Background measurements are taken through the ATR crystal without a sample in place. We use the same crystal material for background measurements that is used to measure the standards and samples.

The Wavelength Accuracy Verification report is divided into four sections. The first section shows the measurements of the secondary thick polystyrene ATR standard used to verify the traceable ATR 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 secondary polystyrene ATR standard

The section of the report titled “Measurement of the ATR Polystyrene Standard” shows the reference values for the secondary thick polystyrene ATR standard and the values measured at our 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 “Measured Average,” “Meas1,” and “Meas2” show the individual peak location values of the secondary thick polystyrene ATR standard measured at our factory and the average of the two measurements.

## Results from the traceable ATR standard

The section of the report titled “Measurement of the Traceable ATR Polystyrene Standard” shows the measurements of the traceable ATR standard compared to the measurements made on the secondary thick polystyrene ATR 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 secondary standard. (These are the same values report in the Measured Average column in section one of the Wavelength Accuracy Verification report.)

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

## Summary data from the ATR standard

The section of the report titled “Traceable ATR Polystyrene Standard Summary” shows the peak location values used to describe the traceable ATR 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 ATR standard.

## Verifying the traceable standard

We assign each standard a unique serial number and includes the serial number information in the traceability report. To verify the traceable standard, check that the serial number printed on the standard itself matches the serial number of the traceable standard printed on the traceability report.

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