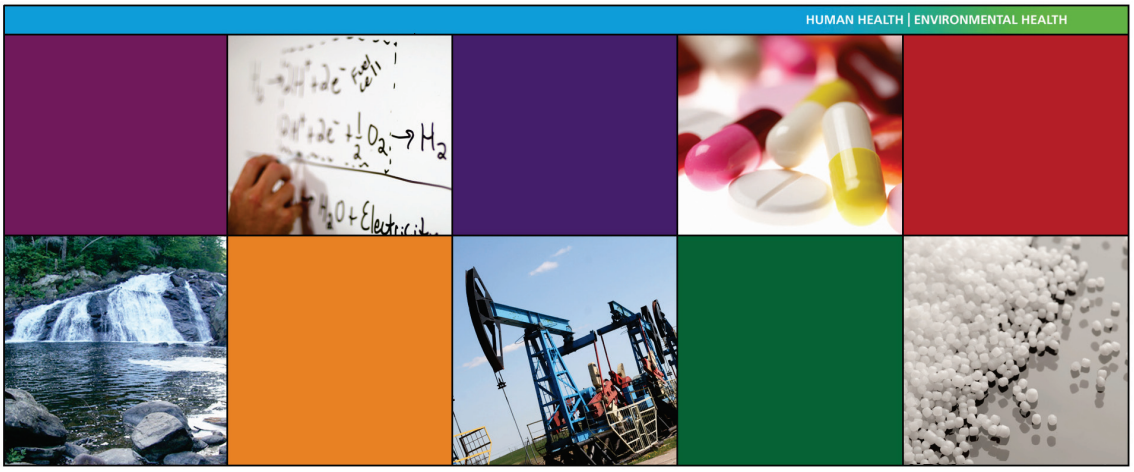
MOLECULAR SPECTROSCOPY

INSTRUMENT PERFORMANCE VALIDATION KIT



User's Guide

PerkinElmer®

Release History

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Performance Validation Kits

The Food and Drug Administration (FDA), the United Kingdom Accreditation Service (UKAS) and the Good Laboratory Practice (GLP) regulations all require that calibration of instrument accuracy can be traced to a certified reference material (CRM).

The MIR Performance Validation Kit (L1250404) contains a polystyrene Traceable Reference Material (TRM), and two types of reference material for routine use in the sample compartment of your spectrometer: three polystyrene cards for routine validation of wavenumber precision, and a glass transmission filter for recording ordinate repeatability.

The NIR Performance Validation Kit (L1250405) contains a polystyrene Traceable Reference Material (TRM).

The use of both kits is described here.

MIR Kit

The Instrument Performance Validation (IPV) kit enables you to generate a record of wavenumber calibration that is traceable to a standard reference material (SRM) calibrated by the National Institute of Standards and Technology (NIST). It also enables you to confirm that your instrument is performing consistently in day-to-day operation

In addition to the IPV kit, your instrument may have an internal APV (Automatic Precision Validator) kit. Two reference materials are installed in the optical module of the instrument and these internal reference materials can be used to provide automatic validation routines when used with Spectrum software.

NOTE: If you have a Spectrum 65 or Spectrum Two instrument that does not have internal reference materials, reference cards are provided that can be inserted in the sample slide.

Polystyrene film

The polystyrene cards in the IPV kit are used to test that the instrument is performing consistently, rather than for calibrating the accuracy of the wavenumber (abscissa) scale.

Calibration of wavenumber accuracy is performed using the TRM supplied in the IPV kit.

Traceability of the wavenumber axis comes from a certified polystyrene standard (SRM 1921b), measured by the National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899, USA. The NIST certificate of measurement provides wavenumber values for fourteen peaks, of which thirteen are recommended for certification using a center-of-gravity (also known as centroid) algorithm. Of these, we have selected five that show the least dependence on temperature and water vapor. These are nominally at 3082.22, 3060.14, 1601.38, 1583.04 and 1028.42 cm^{-1}. The calibration is valid for resolutions of 4 cm^{-1} or better.

NG11 Schott glass

The sample provided for testing the ordinate repeatability is an NG11 Schott glass filter with a nominal thickness of 1 mm. This is a very stable material, and has a series of broad spectral features with transmission between 70% and 0%. It can be used for testing that the instrument is performing consistently, but not for testing the accuracy of the transmittance scale. Accurate values for the transmittance of the glass sample are not available. Errors in transmittance caused by inter-reflections between this sample and other optical components vary from instrument to instrument, depending upon the precise location of the reference material.

Limitations of traceable wavenumber calibration

Wavenumber calibration using polystyrene is derived from the NIST SRM 1921b. Certified values for the most precisely known peak positions used have quoted uncertainties of about ±0.1 cm^{-1} for four of the peaks, and ±0.27 cm^{-1} for the peak at 1028.42 cm^{-1}.

These values were measured at 0.5 cm^{-1} resolution using an instrument at 23 ± 1 °C, but the expanded uncertainty values are quoted for 23 ± 5 °C.

We suggest that the uncertainty associated with routine measurement of the five recommended peaks in the spectrum of the traceable polystyrene sample, is realistically set at ±0.5 cm^{-1}. This level of precision is satisfactory for general analytical applications involving solid and liquid samples.

Measurement precision with a polystyrene film

Spectra collected using the reference materials in the IPV give peaks 10 cm^{-1} or more wide, and the peak positions can be consistently measured to ±0.1 cm^{-1}, provided that the noise level is low enough.

Peaks in the spectrum of polystyrene above 1800 cm^{-1} are broader than most of the peaks below 1800 cm^{-1} and may, therefore, show more variation for a given noise level. The peaks at 1943 and 1802 cm^{-1} are subject to interference by water vapor and should not be used.

NOTE: The recommended measurement conditions are 16 scans at 4 cm^{-1}. Under these conditions, the reported peak positions are unlikely to vary by more than ±0.1 cm^{-1} unless affected by water vapor.

NIR Kit

Instrument Performance Validation (IPV) enables you to routinely generate a record of wavenumber calibration that is traceable to a National Physical Laboratory (NPL) CRM. Using the NIR Performance Validation Macro, you can compare the result for a polystyrene Traceable Reference Material (TRM) and your internal polystyrene Automatic Precision Validator (APV) against a certified polystyrene standard (serial number: JR95), measured by the NPL, Teddington, TW1 0LW, UK.

The NPL certificate of measurement provides wavenumber values for two peaks, 5669.3 cm^{-1} and 4571.6 cm^{-1}, for resolutions of 4 cm^{-1} or better. The polystyrene TRM supplied with your instrument has been calibrated against this CRM, and a Certificate of Calibration printed at the factory is included with it. This certificate provides a record of calibration and traceability for the TRM to the NPL CRM.

Limitations of traceable wavenumber calibration

Certified values for the peak positions used have quoted uncertainties of ±1.5 cm^{-1} for the peak at 5669.3 cm^{-1} and ±1.0 cm^{-1} for the peak at 4571.6 cm^{-1}. We suggest that the uncertainty associated with routine measurement of the two recommended peaks, in the spectrum of a traceable polystyrene sample, is realistically set at these limits. This level of precision is satisfactory for general analytical applications involving solid and liquid samples.

Validating Instrument Calibration Using the TRM

To validate instrument calibration, tests must be performed under the same measurement conditions as those used to calibrate the TRM (Traceable Reference Material) at the factory. The Spectrum macros provided will use the appropriate instrument settings.

We also recommend that before starting the calibration you make sure that the instrument is optimally aligned.

Using Macros to Collect Reference Spectra

NOTE: To run the macro, you must be connected to your instrument. No sampling accessory other than the Slide Holder should be installed in the sample compartment.

1. Start Spectrum software and connect to your instrument.
2. Select **Macro** from the Setup menu.
   The Setup Macros tab is displayed.
3. Select **Add**, and then select **Import** from the drop-down list.
4. Browse to and select the macro appropriate for your instrument.
   The IPV macros are located in:
   C:\Program Files\PerkinElmer\Spectrum\IPV Kit\Macros\NIST1921b
   for Windows XP or Windows 7 (32-bit), or in
   C:\Program Files\PerkinElmer (x86)\Spectrum\IPV Kit\Macros\NIST1921b
   for Windows 7 (64-bit), where C: is the drive where Spectrum has been installed.
5. To run the macro, select **Macros** from the Process menu, and then select the appropriate macro from the sub-menu.

   OR

   Click  on the Process bar, and select the appropriate macro from the drop-down list.

   The macro runs. Follow the instructions on-screen. Spectra will be collected using the instrument settings contained in the macro.
   If you did not select the appropriate macro for your instrument, an error message will appear.

   You will be prompted during the macro to insert the TRM from the IPV kit into the sample slide in the sample compartment. If you do not have an internal filter wheel, during the macro run you will also be prompted to insert the calibrated polystyrene card (L1202057) supplied with your instrument into the sample slide.
   Spectra generated during the procedure will be addded to the Samples View in the Data Explorer and saved to C:\pel_data\Spectra.
6. Replace the TRM in its box and store it in a safe place.

After running the macro, we recommend that you use the Send To Word function to output your results to Word:

- Select **Send To Word** from the File menu, and then select **New Word Document** from the **Word** sub-menu.
  You can send a graph of your spectra and the contents of the Results Table.

Possible Reasons for Validation Failure

If the peaks were not found or lie outside the tolerance, the failure may be because:

- the wrong spectra were used;
- the wrong reference material was used to collect one of the spectra;
- the instrument was not aligned correctly;
- the noise level is too high;
- the beam was obstructed during validation, for example by an accessory in the beam path. The effect is shown most at high wavenumbers;

If you need more help, contact your PerkinElmer agent or office.

When Should I Recalibrate My Instrument?

You must check the calibration of your instrument if a new source, detector or power supply has been fitted to your instrument, and recalibrate if necessary.

You should also consider recalibrating your instrument if the peak positions in a spectrum collected from your TRM are not within ±0.3 cm^{-1} of those given in the column labeled TRM on the Certificate of Performance Validation.

NOTE: If you recalibrate your instrument, you must collect new ASTM reference spectra.

For recalibration purposes, we recommend that you use the peak at nominally 3060.14 cm^{-1} for the MIR. It is a symmetrical band, at high wavenumber and therefore sensitive to calibration errors. Any change in calibration will affect each peak by an amount proportional to its wavenumber. The change in position of peaks around 3000 cm^{-1} will be approximately double the change seen for peaks around 1600 cm^{-1}.

NOTE: The reference laser controls the wavenumber scale of the instrument and the scale is calibrated by measuring the effective wavenumber of the laser. This differs from the absolute value because the infrared radiation in the interferometer is not all exactly parallel to the laser beam. Slightly different calibrations are required for the different J-stops used for 2 cm^{-1} and 1 cm^{-1} resolution. Any calibration error will be proportional to the wavenumber value.

For recalibration purposes, we recommend that you use the peak at nominally 5669.3 cm^{-1}, for the NIR.

NOTE: The reference laser controls the wavenumber scale of the instrument and the scale is calibrated by measuring the effective wavenumber of the laser. This differs from the absolute value because the infrared radiation in the interferometer is not all exactly parallel to the laser beam. A different calibration is required for the Near Infrared Reflectance Accessory (NIRA) and remote accessories. Any calibration error will be proportional to the wavenumber value.

We recommend that, before starting the test, you make sure that the instrument is optimally aligned.

Routine Instrument Validation

For route instrument validation, we recommend that you use the Instrument Verification functionality in Spectrum software. This enables you to use both the Internal APV reference and an external sample card in the sample compartment (such as the TRM).

For more information see the Spectrum on-screen help which can be accessed by selecting **Contents** from the Help menu in Spectrum.

Routine Validation in MIR

What Peaks Should I Use?

Polystyrene film

There are five NIST certified peak positions for the polystyrene film. We recommend that you use the three peaks nominally at 3060.14, 1601.38 and 1028.42 cm^{-1} for testing wavenumber repeatability.

NG11 Schott glass filter

The most useful region for measurements with the glass filter is 4000 to 2000 cm^{-1}. Because the spectral features are very broad, there is no significant variation in the spectrum for resolutions of 8 cm^{-1} or worse. Those regions subject to atmospheric interference from water vapor around 3600 cm^{-1} and carbon dioxide around 2350 cm^{-1} should not be used. We recommend measuring the transmittance at 3990, 3030 and 2000 cm^{-1}.

For information, see the Spectrum on-screen Help.