

EXTERNAL NEAR INFRARED REFLECTANCE ACCESSORY



User's Guide



Release History

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Warnings and Safety **Information**

General Safety

The External Near Infrared Reflectance Accessory (External NIRA) forms part of the Spectrum 100 Series and Spectrum 400 Series FT-IR Spectrometers, which have been designed and tested in accordance with PerkinElmer specifications and in accordance with the safety requirements of the International Electrotechnical Commission (IEC). The Accessory conforms to IEC 61010-1 ("Safety Requirements for electrical equipment for measurement, control and laboratory use") as it applies to IEC Class 1 (earthed) appliances and therefore meets the requirements of EC low voltage directive 2006/95/EC.

Electrical Safety

The accessory power supply is separate from the instrument power supply.

- Connect the accessory to a power supply line that includes a switch or other means of disconnection from the electricity supply.
- Only plug the accessory into an electricity-supply socket that is provided with a protective earth connection.



WARNING

Any interruption of the protective earth conductor inside or outside the accessory or disconnection of the protective earth terminal can make the accessory dangerous.

The accessory has an IEC Insulation class I rating for external circuits – only connect other equipment that meets the requirements of IEC 61010-1, IEC 60950 or equivalent standards.

Radiation Emitted by the Spectrum 100/400 NIR Source

The Spectrum 100/400 NIR source is a quartz halogen bulb that emits ultraviolet, visible and infrared radiation. The majority of this energy is in the infrared region. Do not stare into the beam produced by this bulb. For further safety and warning information see the *Spectrum 100 Series User's Guide* (L1050021) or the *Spectrum 400 Series User's Guide* (L1050056), as applicable. These are distributed as .pdf files on the *Spectrum Manuals CD* (L1050002).

Measurements of the infrared radiation emitted from the spectrometer's external beam port show that exposure limits recommended by the American Conference of Governmental Industrial Hygienists (ACGIH) and the International Commission on Non-Ionizing Radiation Protection (ICNIRP) will not be exceeded during normal operation.



WARNING

*Do not look directly into the beam emitted from the external beam port.
Prolonged exposure (> 10 seconds) may cause permanent eye damage.*

Introduction

Overview

The External NIRA is used for gathering diffuse reflectance spectra. It is an external accessory for a Spectrum 100N FT-NIR or Spectrum 400 FT-IR/FT-NIR spectrometer that incorporates the NIR wavelength range.



Figure 1 Spectrum 400 FT-IR/FT-NIR Spectrometer with External Near Infrared Reflectance Accessory (External NIRA) Fitted

The External NIRA is not sited inside the instrument's sample compartment. The sample compartment can be used for other purposes, such as another accessory, and/or, if applicable, a technique that requires the spectrometer's alternate wavelength range (typically MIR).

Diffuse reflectance measurements using the NIRA

The External NIRA is used for collecting diffuse reflectance spectra from solids, liquids and powders. The technique is non-destructive and requires minimal sample preparation. It is particularly useful for undiluted powders and granular materials. You can make both qualitative and quantitative measurements.

How it Works

The sample is placed on a sapphire window located on the top of the accessory. The circles and lines on the plate help you center the sample over the window.

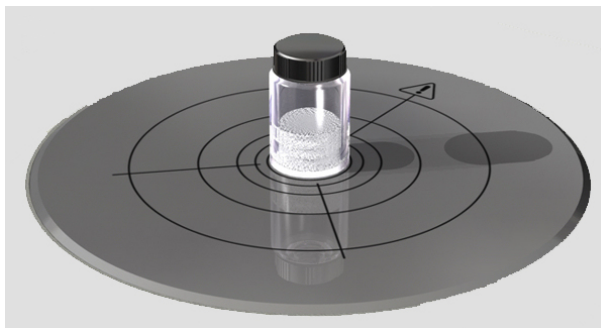


Figure 2 Vial Placed on the NIRA Top Plate

The infrared beam is switched to the external beam port on the right of the spectrometer, enters the accessory through the port on its left side, and is focused and directed onto the sample.

NIR light hitting the sample is absorbed, reflected and scattered over a wide range of angles.

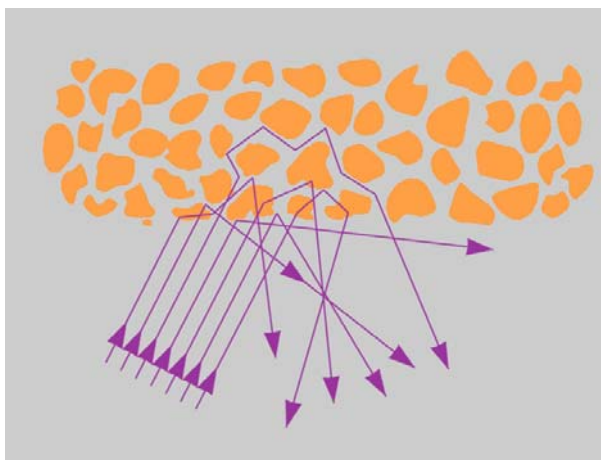


Figure 3 Diffuse Reflectance

The NIRA is designed to direct the diffusely reflected beam from the sample onto an internal detector so that the components absorbed by the sample can be measured.

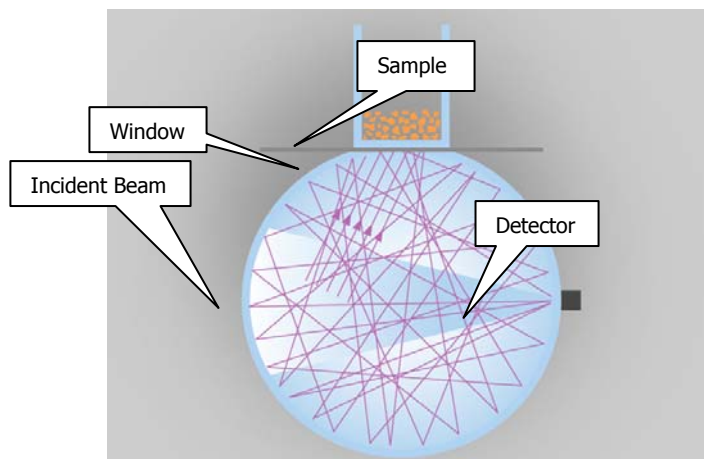


Figure 4 Diffuse Reflectance within a NIRA

Sample Options

Because it is a non-destructive technique, reflectance is useful for a wide range of samples from emulsions and powders to tablets. For many types of sample, no preparation is required. For example, a piece of textile to be analyzed can be placed directly on the sapphire window to obtain an NIR spectrum.

It is also useful for samples inside containers. Samples can be scanned through a beaker or plastic bag, and tablets can be scanned while still in the blister pack. For example, a glass sample vial containing a powder to be analyzed can be placed directly on the sapphire window to obtain an NIR spectrum.

A loose powder cup is provided with the NIRA Sampling Kit (see page 19) that can be used as a container for loose powders. You are also supplied with a tablet holder with three different sized inserts.

Installing the Accessory

Electrical Requirements

A suitable mains power socket must be available for the accessory power supply. The power supply can operate on electricity supplies of 50 or 60 Hz and in the range 100–230 V without adjustment.

For further information, see the *Spectrum 100 Series Getting Started Guide* (L1050001) or the *Spectrum 400 Series Getting Started Guide* (L1050055), as appropriate.

Fitting the Accessory

A new External NIRA is installed by a PerkinElmer Service Engineer who will install an external accessory bracket in the handhold on the right of the instrument (if not already fitted) and any internal parts required. The accessory should not be removed unless:

- You want to use the external beam port on the right of the instrument with a different external accessory.

See "Appendix 3: Removing and Refitting the External NIRA" on page 53.

- The spectrometer must be moved to a new location.

For information about moving the spectrometer, refer to see the *Spectrum 100 Series User's Guide* (L1050021) or the *Spectrum 400 Series User's Guide* (L1050056), as applicable. These are distributed on the *Spectrum Manuals CD* (L1050002).



WARNING

The spectrometer is a heavy precision instrument, so two people are required for safe handling.

CAUTION

Do not attempt to lift the instrument, or to move it to another location, when an external accessory is attached. The optical alignment of the accessory may be disturbed.

The Instrument Install Wizard

A new External NIRA is installed on a Spectrum 100N FT-NIR or Spectrum 400 FT-IR/FT-NIR spectrometer by a PerkinElmer Service Engineer who will, if necessary, install the software on a PC that is connected to the instrument, and then install the accessory.

The External NIRA is supported by Spectrum version 6.3.2 (or later) for the Spectrum 400 Series Spectrometers or version 6.3.3 (or later) for the Spectrum 100 Series Spectrometers.

If you have a Spectrum 100 Series Spectrometer and would like to use the External NIRA with Spectrum Express software, you will need Spectrum Express version 1.2 (or later).

NOTE: If you have Spectrum Express software and do not have an instrument installed, see the *Spectrum Express Installation Leaflet* (L1050054) that shipped with your software for details of the Instrument Install Wizard. The accessory will be detected by the Spectrum Express software when fitted in your spectrometer.

Before using the External NIRA with Spectrum or Spectrum Express software:

1. If you have removed and refitted the External NIRA (see "Appendix 3: Removing and Refitting the External NIRA" on page 53), make sure that the accessory data cable is fitted correctly.
2. Switch the accessory power on, wait for a few seconds, and then switch the instrument on.

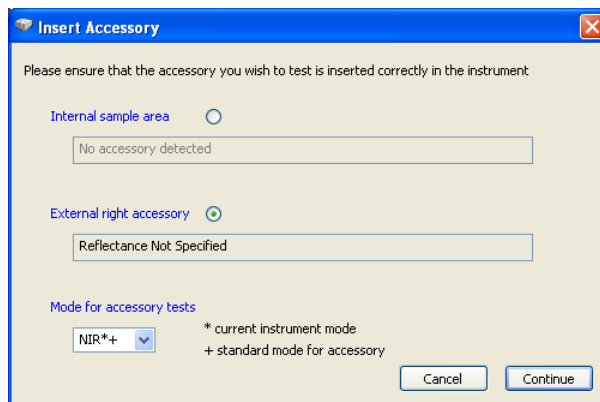
OR

If the accessory power supply has been interrupted for any reason, switch the instrument off, switch the accessory power on, wait for a few seconds, and then switch the instrument on.

When the instrument is switched on, it performs self-checks that detect the presence of the external accessory.

To use the Instrument Install Wizard to install the External NIRA:

1. Log in to the Spectrum software as an Administrator.
2. From the **Administration** menu, select **Instrument and Accessory Configuration**, and then select **Add Accessories**.
The Instrument Install Wizard **Welcome** page is displayed.
3. Select the instrument on which the accessory is installed and then click **Next**.
The **Install Accessories** page is displayed.
The External NIRA is NOT shipped with a configuration disk.
4. Click **Next**.
The **Insert Accessory** dialog is displayed.



NOTE: If the external accessory has not been detected by the instrument, the dialog does not include the External right accessory.

(i) Click **Cancel** to close the Insert Accessory dialog and display the list of tests, and then click **Back** to return to step 3.

(ii) Check whether:

- The accessory data cable is connected correctly.
If necessary, reconnect the data cable.
- The accessory power supply has been interrupted.
Switch the instrument off, switch the accessory power on, wait for a few seconds, and then switch the instrument on.
The instrument performs self-checks that detect the presence of the external accessory.

(iii) When the checks have been completed, click **Next**.
The updated Insert Accessory dialog is displayed.

5. Select **External right accessory** and then, in the **Mode for accessory tests** drop-down list, select **NIR*+** (or **NIR+**).

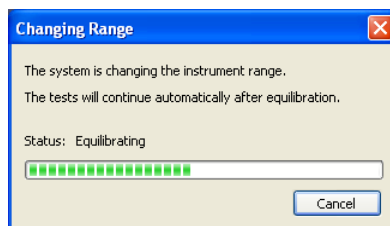
+ indicates that NIR is the standard wavelength range for testing the accessory.

* indicates that NIR is the current wavelength range. If * is not present, a wavelength range change will be performed before testing begins.

6. Click **Continue**.

If the current wavelength range is NIR, the wizard begins testing the accessory. Go to step 7.

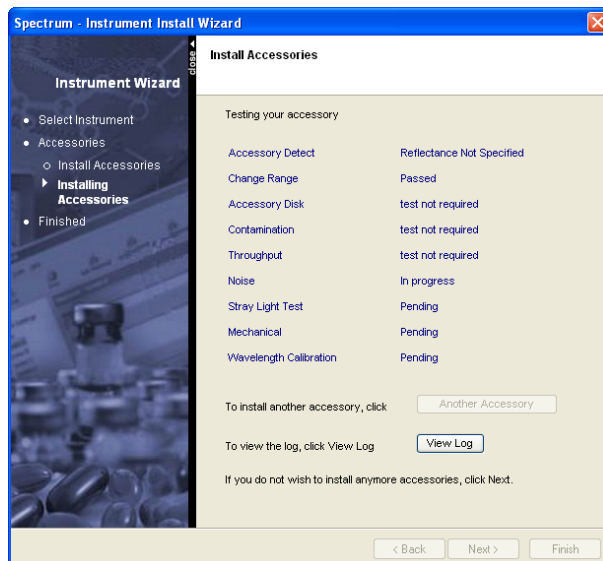
If you have a Spectrum 400 dual-range instrument, and the current wavelength range is not NIR, the Changing Range dialog is displayed.



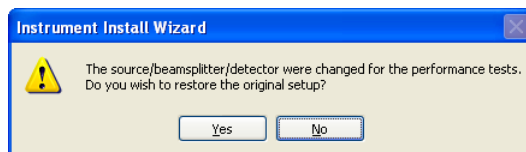
NOTE: Depending on the beamsplitters and detectors fitted, the instrument requires some time to equilibrate after changing ranges.

If you do not want to continue, click **Cancel**.

After the change has been completed, the wizard displays a list of tests and begins testing the accessory.



7. When the accessory tests and calibrations tests are complete you can install another accessory, if required, by clicking **Another Accessory**. You are returned to the start of the installation wizard.
8. Click **View Log** if you want to see the test results in more detail.
If you want to view the results of the accessory tests later, the log is stored at C:\Program Files\PerkinElmer\ServiceIR\<Instrument Serial Number>\Performance Tests xxx\PerformanceTests.log (where xxx is an incremental number).
9. Click **Next**.
The **Finish** page is displayed.
10. Click **Finish** to complete the installation and close the Instrument Install Wizard.
If the source, beamsplitter, or detector was changed to facilitate testing, you are given an opportunity to return the instrument to its original setup



11. Click **Yes** or **No** as appropriate.

The Sampling Kit

The following items are provided with the External NIRA:

Part Number	Description	Quantity
L1240413	Loose Powder Sample Holder	1
L1241418	Powder Sampling Plate	1
L1245010	Sample Alignment Tool	1
L9001026	Cleaning Brush	1
04967843	Spatula	1
04974047	Tweezers	1
L1241632	Tablet Holder (Locating ring)	1
L1241628	Tablet Holder Insert (5 mm aperture)	1
L1241629	Tablet Holder Insert (7 mm aperture)	1
L1241630	Tablet Holder Insert (9 mm aperture)	1
L1241634	Spectralon Reference	1
L1245009	Tutorial Sample	1
L9001029	4 ml Glass Vial and Cap	10

Loose Powder Sample Holder

The Loose Powder Sample Holder is a deep cup with an optically flat base that enables you to sample loose powders. This is a useful alternative if sampling in conventional vials causes interferences due to absorption by the vial base.

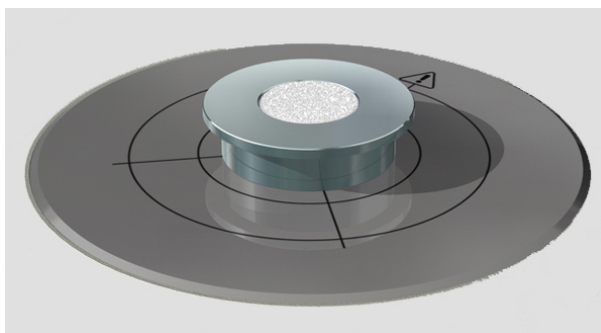


Figure 5 Loose Powder Sample Holder

Powder Sampling Plate

This is a metal plate used when filling sample cups.

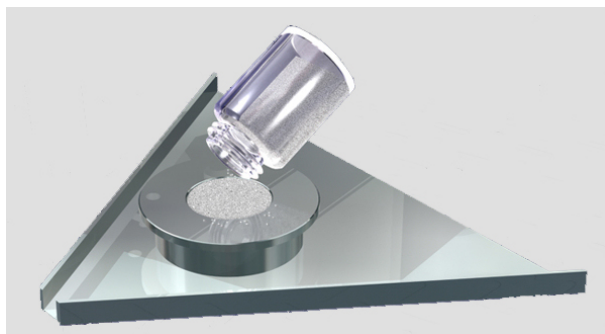


Figure 6 Powder Sampling Plate

1. Place a Sample Cup in the center of the Powder Sampling Plate.
2. Overfill the Sample Cup with your sample and push a straight edge across the top of the Sample Cup so that the powder is level across the cup.
3. Remove the Sample Cup from the Powder Sampling Plate.
You can pour the spilt powder away using the narrow edge of the Sampling Plate.

Sample Alignment Tool

The Sample Alignment Tool is a magnetic device that helps you position sample containers of the same type (such as vials or beakers) in the same place on the NIRA.

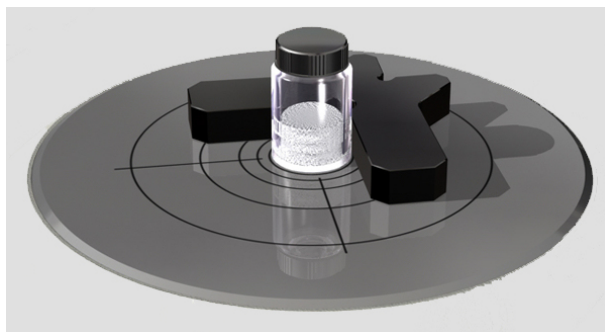


Figure 7 Sample Alignment Tool

Cleaning Brush

This is used for cleaning out the powder cups and brushing powder spillages off the surface of the NIRA.

Spatula

The spatula is used for transferring powder samples to a powder cup.

Tablet Holders

The Tablet Holder has a set of different-sized insert apertures for use with tablets of different shapes and sizes.

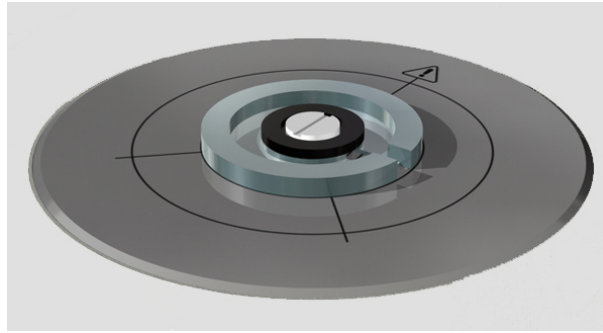


Figure 8 Tablet Holder

Spectralon Reference

The Spectralon Reference is used for collecting background spectra if you are using the NIRA in manual operating mode and performing certain system checks. It is placed on top of the Sample Window.

Tutorial Sample

The sample of Calcium Ascorbate is used in the Spectrum software tutorial named *Using the Near Infrared Reflectance Accessory (NIRA)*.

- To run the tutorial, select the **Help** menu, click **Learning Spectrum**, and then click **Using the NIRA**.



Using the Accessory with
Spectrum Software

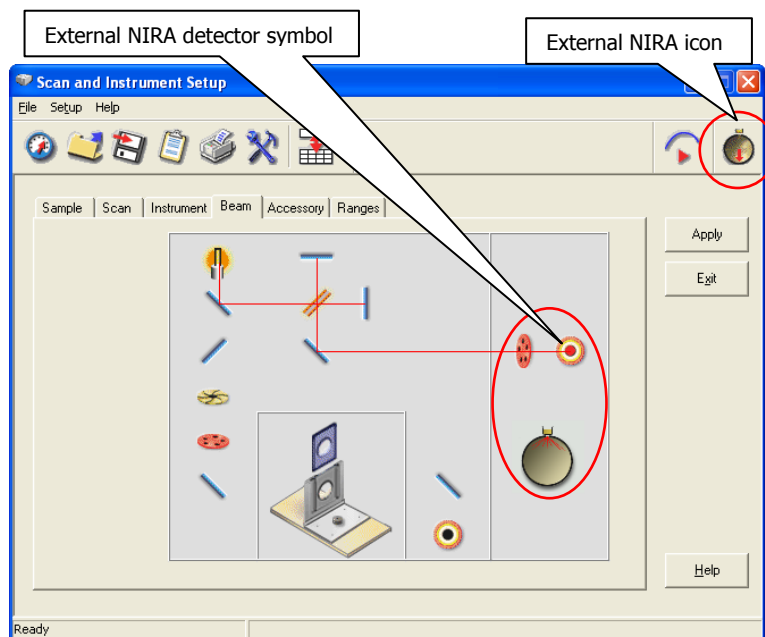
Selecting the Accessory

To select the External NIRA using the Spectrum software:

1. Select the **Setup** menu and then click **Instrument**.

The Scan and Instrument Setup dialog is displayed, showing the Instrument tab.

2. Select the **Beam** tab.



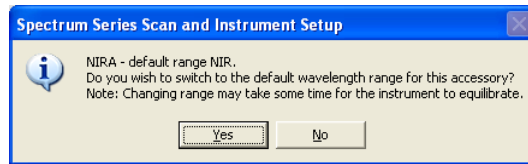
NOTE: If the External NIRA elements are not displayed in the Beam tab, check whether:

- The accessory data cable is connected correctly.
If necessary, reconnect the data cable. The Beam tab is updated.
- The accessory power supply has been interrupted.
Switch the instrument off, switch the accessory power on, wait for a few seconds, and then switch the instrument on. The instrument performs self-checks that detect the presence of the external accessory. The Beam tab is updated.

3. Click the External NIRA detector symbol.

The accessory is selected, as indicated by the icon at the top right of the Scan and Instrument Setup dialog.

4. If you have a Spectrum 400 dual-range instrument and NIR is not the current wavelength range, you are offered an opportunity to change range.



Select either **Yes** or **No**.

5. If you want this to be the default instrument set up, click **Apply** in the Scan and Instrument Setup dialog.

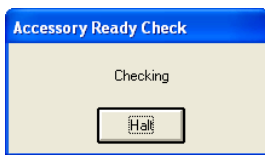
Accessory Ready Check

The software automatically performs a short Accessory Ready Check procedure after you start Spectrum and connect to the instrument:

- If the applied instrument setup includes the External NIRA. That is, the Beam tab would have shown the External NIRA selected when you last exited Spectrum.
- When you select the External NIRA for the first time in a Spectrum session.

If, for any reason, you want to stop the Accessory Ready Check:

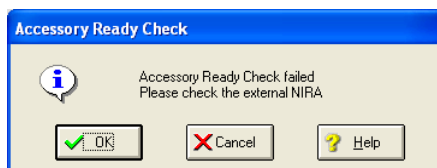
1. Click **Accessory Ready Check** in the Windows Taskbar.
The Accessory Ready Check dialog is displayed.



2. Click **Halt**.

If the Accessory Ready Check fails to complete:

1. Click **Accessory Ready Check** in the Windows Taskbar.
The Accessory Ready Check failed dialog is displayed.



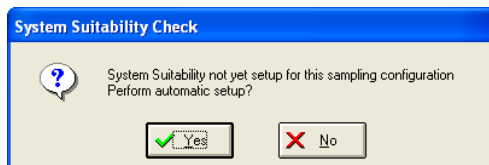
2. Make sure that the accessory data cable is connected correctly and then click **OK**.
If the check still fails, contact your local PerkinElmer Service Representative.

Setting up System Suitability Checks

System Suitability is the procedure of demonstrating that your complete system, including the External NIRA, is performing fit-for-purpose. This is a common requirement for regulatory compliance. The System Suitability checks provide a test of wavenumber precision and a check on noise using a traceable polystyrene sample. In the checks described below, the internal polystyrene sample in the External NIRA is used.

1. Select the **Setup** menu, click **Validation**, and then click **System Suitability** in the sub-menu.

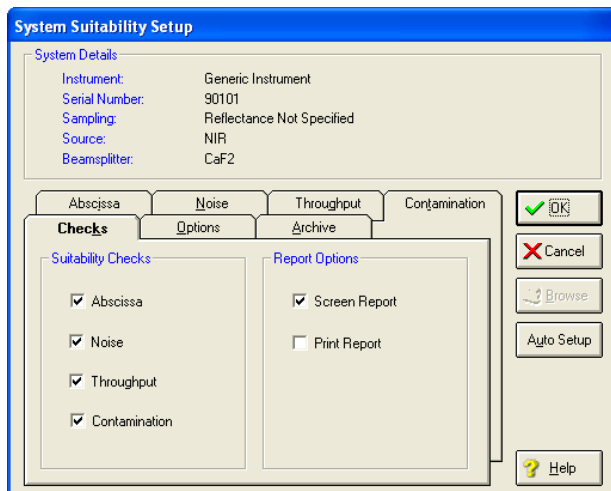
The following message is displayed:



2. Click **Yes**.

The system collects reference spectra for the Noise and Contamination checks automatically.

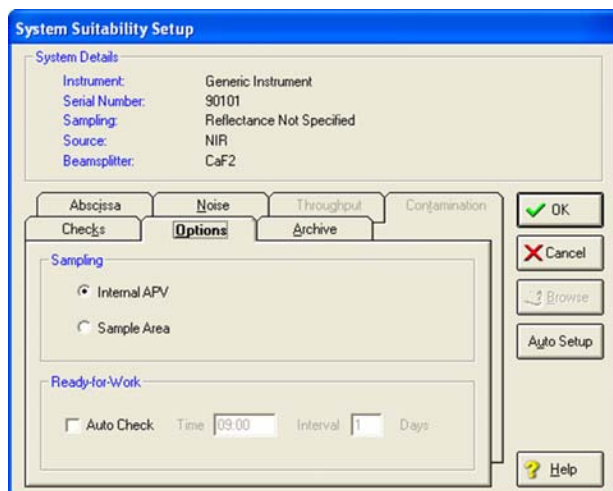
The System Suitability Setup dialog is displayed.



3. Select **Screen Report** and/or **Print Report**.

If there are errors reported by the test, the report is displayed on the screen whether this option was selected or not.

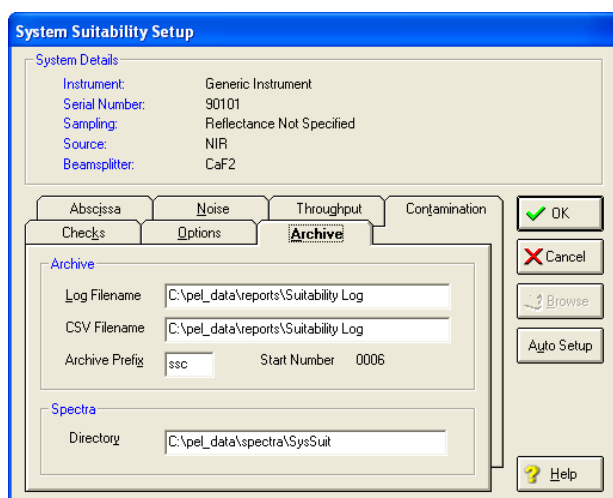
4. Select the **Options** tab.



5. Make sure that **Internal APV** is selected.

6. Select the **Archive** tab.

The default **Log Filename** path, **Archive Prefix** and **Directory** where the spectra are stored are displayed.



7. If required, enter an alternative **Log Filename**, **Archive Prefix** or **Directory**.

We suggest that you use the defaults.

8. Click **OK**.

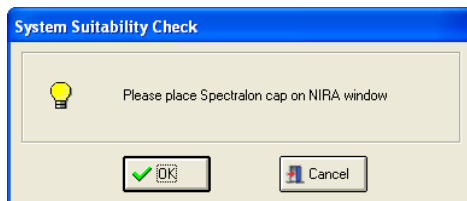
System Suitability checks for are now set up for this specific instrument configuration.

The Abscissa and Noise tabs contain the default values for the System Suitability checks. For further information about these checks and also for information about **Throughput** and **Contamination** checks see the on-screen Help.

Performing System Suitability Checks

1. Select the **Instrument** menu, click **Validate**, and then click **System Suitability** in the sub-menu.

The System Suitability Check dialog is displayed:

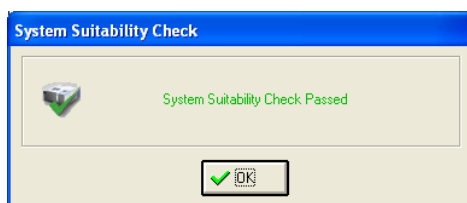


2. Place the Spectralon Reference (L1241634) provided on the window on the External NIRA top plate, and then click **OK**.

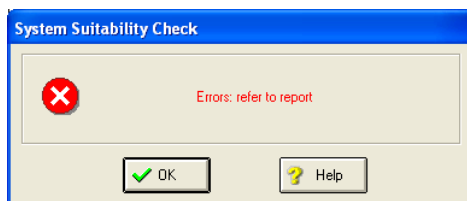
The validation is performed using the current System Suitability setup.

When the validation is complete the System Suitability report is printed and/or displayed (depending on the setup option selected).

If the System Suitability checks pass, the following message is displayed.



If the System Suitability checks fail, the following message is displayed:



and the report is displayed on the screen.

The errors described below are errors that may be expected, caused, and potentially remedied by the user. Report any other system errors to your PerkinElmer Service Representative.

Abscissa Test

Nominal position not within the abscissa range of the abscissa reference spectrum - This means that an abscissa position specified for the test was outside the range of the generated spectrum. This can occur in a System Suitability check if the default scan range for the sampling configuration has been modified since the System Suitability for that sampling configuration was setup.

Peak not found - This means that no peak (or at least no peak greater than the abscissa test threshold) was found near to the nominal position specified. To correct this, check that the material used produces the peak expected at the required threshold, re-specify the position, or switch off that element of the test.

Noise test

Specified abscissa range is not encompassed by the noise spectrum - This means that the abscissa range over which the noise is to be measured is not covered by the scan range of the collected spectrum. This can occur in a System Suitability check if the scan range for the sampling configuration has been modified since the System Suitability for that sampling configuration was setup.

3. If necessary, close the window containing on-screen report, and click **OK** to close the System Suitability Check dialog.

Your External NIRA is now ready for use.

Performing a Scan in Spectrum

The External NIRA has two modes of operation: manual and interleaved. In manual mode you collect sample and background spectra separately. In interleaved mode the instrument determines when to collect background scans.

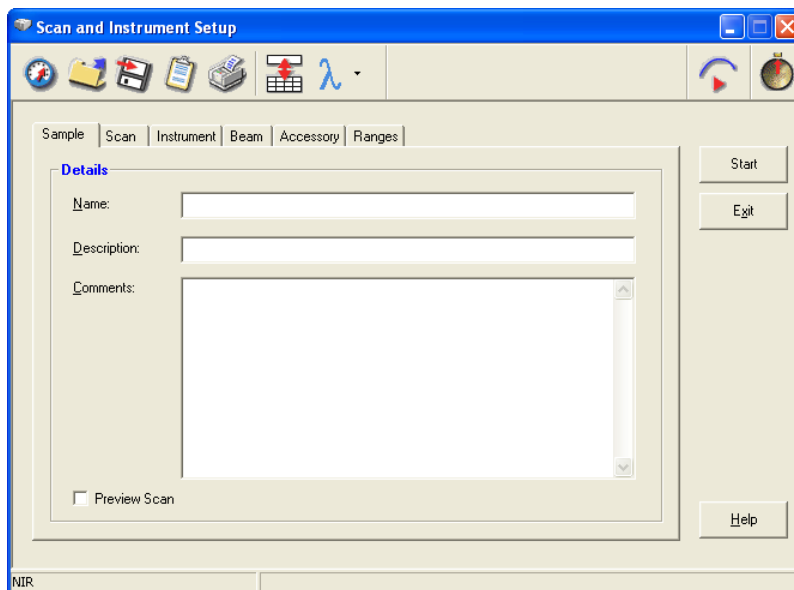
For optimum results it is often important to maintain consistency when using the NIRA for all samples within an application. For example, if the NIRA was used in interleaved mode to collect reference spectra for a library, the NIRA must be used in interleaved mode when collecting spectra that you wish to compare against the library spectra.

Interleaved Scanning

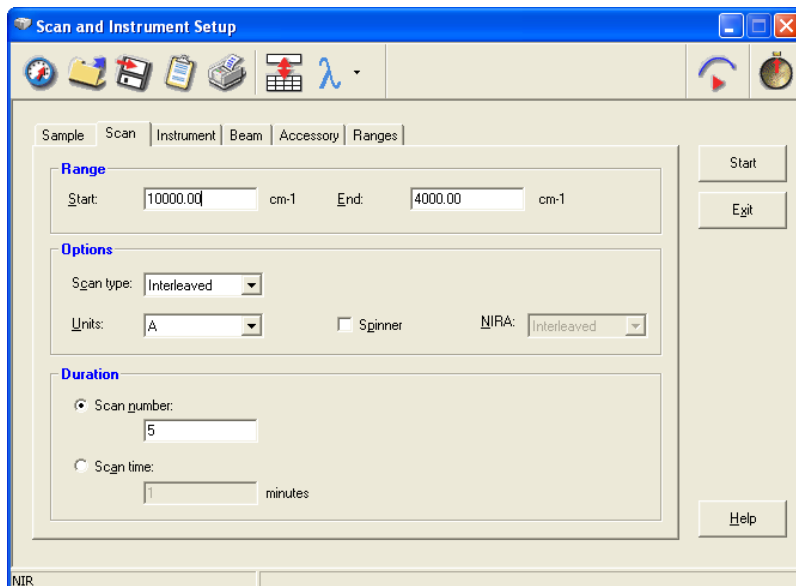
The simplest scan configuration is known as interleaved mode. Interleaved mode enables sample and background scans to be collected together. If you specify a relatively small number of sample scans, the instrument collects that number of background scans, followed by sample scans. For a larger number of scans, groups of background scans alternate with groups of sample scans.

In Interleaved mode the background spectrum will be taken in the lower position and the sample spectrum will be taken in the upper position.

1. Place your sample on the External NIRA.
 2. Select **Scan** from the Instrument menu.
- The Scan and Instrument Setup dialog is displayed.



3. Enter the name of the sample and any description and comments on the Sample tab.
 4. Select the **Scan** tab.
 5. Select **Interleaved** as the **Scan type**.
- The **NIRA** field is grayed.



6. Enter scan parameters and instrument settings as required.

7. Click **Start** to begin collecting data.

The Display tab appears as scans are collected and then the spectrum appears in the graph window in the main application.

Manual Scanning

There are two options available for collecting sample and background scans: Upper and Lower. The default position for sample and background scans is set in the Accessory Configuration dialog, which can be accessed by Advanced users.

- **Upper** – the scan is taken in the upper position. A background scan in the upper position is useful if, for example, you want to take a background from a reference material (such as the Spectralon sample supplied) or from of a vial containing a background reference material to ratio against a vial containing your sample.
- **Lower** – the scan is taken in the lower position, from inside the NIRA sphere.

Performing a Background Scan

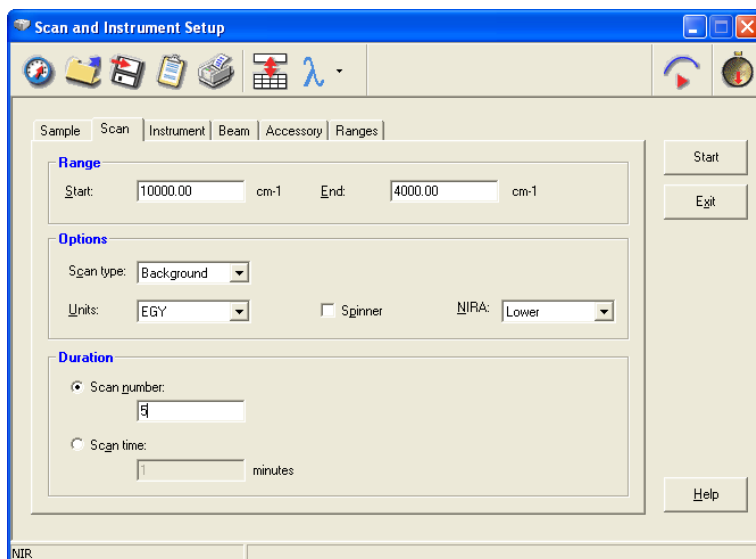
Before you collect a sample spectrum you must collect an appropriate background spectrum.

1. Select **Scan** from the Instrument menu.

The Scan and Instrument Setup dialog is displayed. The amount of information displayed will depend on your user level. For further information on user levels see the on-screen Help.


2. Enter a **Name** for the background spectrum and any **Description** and **Comments** that you wish to save with the background spectrum.

3. Select the **Scan** tab.



4. Select **Background** as the **Scan type**.
The default background position is displayed in the **NIRA** field.
5. If required, select a new **NIRA** position from the drop-down list.
6. Enter scan parameters and instrument settings as required.
7. Click **Start** to begin collecting data.
The background scan is collected and you are now ready to collect a sample scan.



Alternatively, click  to collect a background scan.

Performing a Sample Scan

1. Place the sample to be analyzed on the External NIRA.
2. Select **Scan** from the Instrument menu.
3. Enter the name of the sample and any description and comments on the Sample tab.
4. Select the **Scan** tab.
5. Select **Sample** as the **Scan type**.
The default sample position is displayed in the **NIRA** field.
6. Enter scan parameters and instrument settings as required.
7. Click **Start** to begin collecting data.
The Display tab appears as scans are collected and then the spectrum appears in the graph window in the main application.

NOTE: Spectrum automatically alerts you when you need a new background, and can be configured to request a new background at set intervals (using Data Collection Setup). For more information, see the Spectrum on-screen Help.

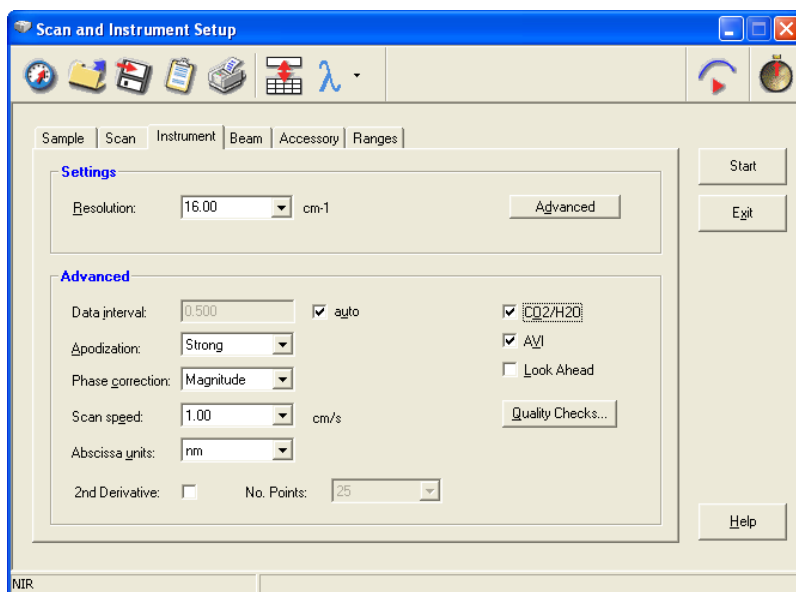
Scanning in Nanometers

The default Abscissa units are wavenumbers (cm^{-1}). To change the Abscissa units you must have Advanced User status.

To collect a scan in nanometers (nm):

1. Select the **Instrument** tab.
2. Click **Advanced**.
3. Select **nm** from the **Abcissa units** drop down list.

The **Start** and **End** Range values on the Scan tab are now displayed in nm.



**Using the Accessory with
Spectrum Express Software**

Selecting the Accessory

When the External NIRA is installed in your instrument, the Setup Instrument BeamPath tab is updated to show that you now have the External NIRA in position.

- To direct the beam to the External NIRA, select **Right external** as the **Beam Location** from the drop-down list.

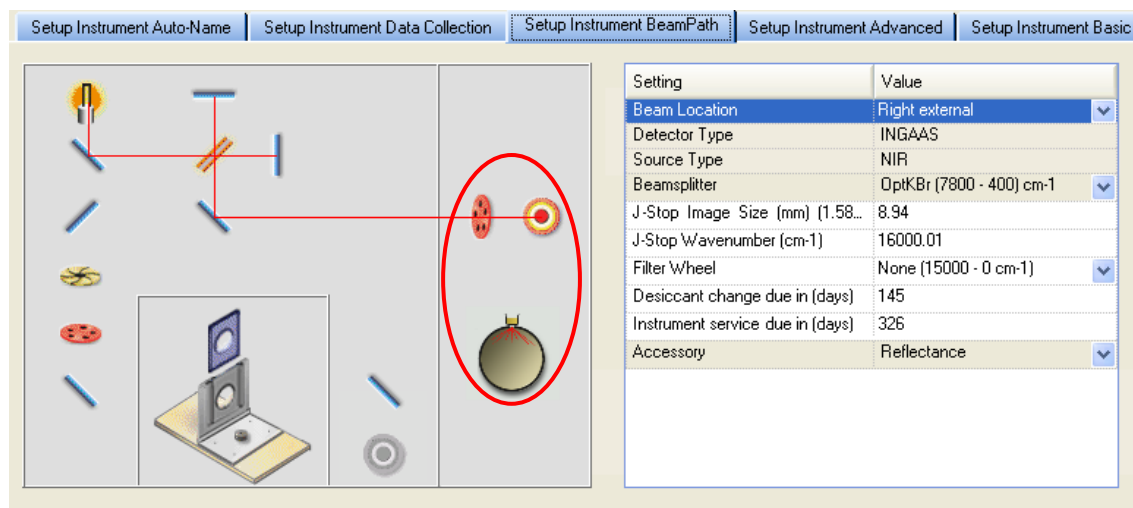


Figure 9 Setup Instrument BeamPath tab with the beam path directed to the External NIRA (circled)

Contamination Ready Check in Spectrum Express Software

NOTE: For information of how to set up Ready Checks see the Spectrum Express on-screen Help. The following description assumes that it is already set up and enabled.

To perform a Ready Check:

1. From the Measurement menu, select the Instrument Checks sub-menu and then **Contamination** from the Ready Checks available.
The Ready Checks dialog is displayed.
2. Place the Spectralon Reference (L1241634) provided on the window on the NIRA top plate, and then click **Scan**.
A new background spectrum is collected, compared to the reference background spectrum and the result of the test is displayed.
3. If required, click the link that enables you to see a print preview of the Instrument Ready Checks Report.





Performing a Scan in Spectrum Express

1. Check the scan and instrument parameters on the Instrument Settings toolbar and, if required, enter a new, unique **Sample ID** and **Description** for your sample.
When your accessory is installed in the instrument, Spectrum Express will default to the instrument settings that were last used to perform a successful scan with that accessory.

Start (cm-1)	End (cm-1)	Accumulations	Sample ID	Description
4000	650	4 Scans	Administrator 37	Sample 0

Figure 10 The Instrument Settings toolbar

NOTE: The **Sample ID** and **Description** are automatically supplied by the *AutoName* function. See *AutoName* in the *Setup and Administration* book in the Spectrum Express Help file for more information.

2. Click  on the Measurement toolbar to collect a spectrum.
If Spectrum Express detects that a Background spectrum is required, the Scan icon will display a flag at the bottom right-hand corner . Click on the icon and a background scan will be collected before the sample spectrum.
If at any time you wish to collect a background scan but do not wish to save it, click .
If you wish to collect a background and save the spectrum, select **Background** as the **Scan Type** on the Setup Instrument Basic tab and then click .

The NIRA starts in **Automatic** mode, and so for most applications you will not need to change the accessory settings. However, if you do wish to change the accessory settings, for example, to collect a **Background** scan in the **Upper** position, see *Manual Scanning*.

Manual Scanning

1. Select **Instrument** from the Setup menu.
The Setup Instrument tabs are displayed.
2. Select the **Setup Instrument Basic** tab.

The screenshot shows the 'Setup Instrument Basic' tab with the following settings:

- Settings:**
 - Abscissa units: Wavenumber
 - Start (cm⁻¹): 7800
 - Ordinate Units: ArbY
 - End (cm⁻¹): 4000
- Scan Settings:**
 - Resolution (cm⁻¹): 2
 - Scan Type: Interleaved
 - ☐ Data Interval (cm⁻¹): 0.5
 - Accumulations: 30 Scans
- Accessory:**

NIRA	
Item	Value
Sampling Details	Not Specified
Spinner	NO
NIRA	Automatic

Figure 11 Setup Instrument Basic tab with Interleaved as the Scan Type

3. Select the **Scan Type** for your spectrum.
4. Select the **NIRA** setting from the drop-down list.

There are three options for the **NIRA** setting:

- **Automatic** – a background scan is taken in the Lower position and a sample scan is taken in the Upper position for all scan types.

NOTE: These settings are always used if the Scan Type is **Interleaved**. (If Interleaved is selected the NIRA option is not available.)

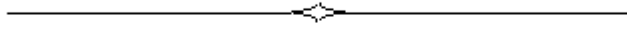
- **Lower** – the scan is taken in the Lower position, from inside the NIRA sphere.
- **Upper** – the scan is taken in the Upper position.

A Background scan in the Upper position is useful if, for example, you want to take a background of a vial containing a background reference material to ratio against a vial containing your sample. If you do not have a blank sample in place, and you wish to collect a **Background** spectrum in the Upper position, ensure that the Spectralon Reference (L1241634) provided is placed on the window on the NIRA top plate.

For optimum results it is often important to maintain consistency when using the NIRA for all samples within an application. For example, if the NIRA was used in Interleaved mode to collect reference spectra for a library, the NIRA must be used in Interleaved mode when collecting spectra that you wish to compare against the library spectra.

NOTE: You can change the abscissa units on the Setup Instrument Basic tab. The **Start** and **End** values will automatically update. The units available are wavenumbers, nanometers and microns.

NOTE: The Spectrum Express Help file describes how to format, process and report your results. To open the Help file, select **Contents** from the Help menu.



Appendices

Appendix 1: NIRA Liquids Transflectance Accessory

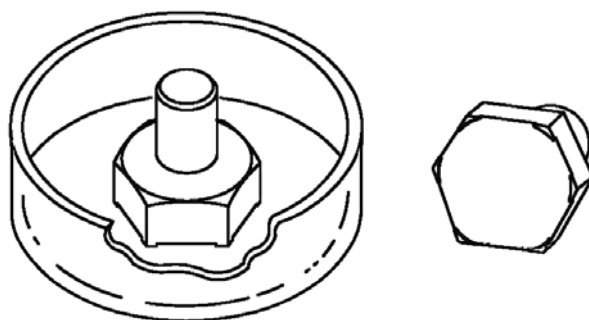


Figure 12 The NIRA Liquids Transflectance Accessory

The Liquids Transflectance Accessory extends the range of sampling using the External NIRA. This optional accessory facilitates the sampling of liquids and gels, providing users with a near-universal sampling system for the QC screening of a wide range of solids, pastes, liquids and gels.

The Liquids Transflectance Accessory comprises a flat, clear glass sample dish into which the sample is placed, and a machined aluminum diffuse reflector which is pressed into the sample.

The accessory enables simple, convenient qualitative analysis of liquids and gels at the normal NIRA sampling position, significantly enhancing the versatility of the NIRA sampling system.

The low cost means that it should be treated as disposable, although it can be re-used for some samples if thoroughly cleaned in between successive samples.

It provides the most convenient method for sampling viscous and transparent gels and resins, and - for certain resins which cannot be cleaned from sampling accessories - the only practical method.

The machined aluminum hexagonal reflector features integral precision-turned spacers which raise the reflector off the base of the sample dish, allowing two passes of the beam through the sample, to provide a total path length of approximately 0.5 mm.

The Transflectance Accessory is intended to be used by operators of the NIRA, without any additional training, other than following the procedures and recommendations given in this *User's Guide*.

This low-cost accessory is intended for simple qualitative screening of liquids and gels. Careful adherence to the procedures given in this User's Guide will produce the required results. For more demanding quantitative measurements, we recommend the standard transmission configuration with the basic sample slide.

NOTE: To deal with unavoidable and unwanted reflections which slightly affect the cosmetic appearance of the spectra, a simple Spectrum Procedure is available which allows users to automatically correct for this effect. This is provided on a disk included with the Liquids Transflectance Accessory Kit.

A typical application of the Liquids Transflectance Accessory is shown on page 45.

List of Parts

As soon as you receive the Liquids Transflectance Accessory kit (L1185153), make sure that it includes the parts listed below. If anything is missing or damaged, contact the carrier and PerkinElmer.

Part Number	Description	Quantity
L1181106	Sample Dish	5
L1181105	Machined Diffuse Reflector	5
L1100609	NIR Liquid Sample Procedure disc	1

Interleaved Scanning using the Liquids Transflectance Accessory

1. Place the sample material into the sample dish, ensuring a sufficient depth in the dish to fill the space under the diffuse reflector once inserted.
2. Handling the diffuse reflector only by its upper section, as shown in Figure 13, place it into the center of the sample dish.

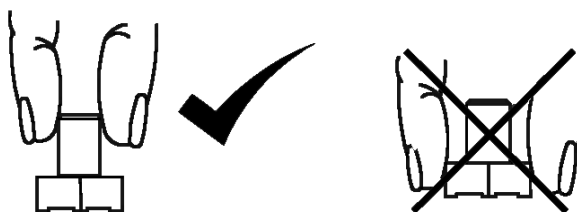


Figure 13 Handling the Diffuse Reflector

3. Apply sufficient downward pressure to ensure that the integral spacers of the reflector are in contact with the base of the dish, and that there are no air bubbles trapped between the reflector and the sample.
4. Position the sample dish and reflector centrally over the sample window, as shown in Figure 14.

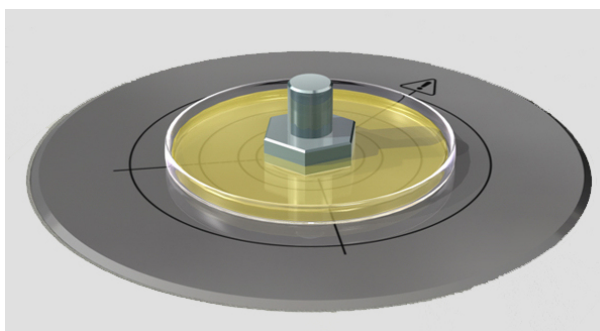


Figure 14 Liquids Transflectance Accessory in Position

5. To ensure consistency between results, always take care to position the reflector in the centre of the dish and the dish centrally over the sample window.
You are now ready to collect your spectrum.

6. Enter the name of the sample and any description.
7. Select **Interleaved** as the **Scan type**.
The **NIRA** field is grayed. The background spectrum will be taken in the lower position and the sample spectrum will be taken in the upper position.
8. Enter scan parameters and instrument settings as required.
9. Click **Start** (Spectrum software) or **Scan** (Spectrum Express software) to begin collecting data.

Manual Scanning using the Liquids Transflectance Accessory

The Background Scan

1. Handling the diffuse reflector only by its upper section, as shown in Figure 13, place it into the center of the sample dish.
2. Position the sample dish and reflector centrally over the upper sample window, using the sample locator, as shown in Figure 14.
You are now ready to collect your background spectrum.
3. Enter the name of the Background and any description.
4. Select **Background** as the **Scan type**.
The default background position is displayed in the **NIRA** field.
5. Enter scan parameters and instrument settings as required.
6. Click **Start** (Spectrum software) or **Scan** (Spectrum Express software).
The background scan is collected and you are now ready to collect a sample scan.

The Sample Scan

1. Place the sample material into the sample dish, ensuring a sufficient depth in the dish to fill the space under the diffuse reflector once inserted.
2. Handling the diffuse reflector only by its upper section, as shown in Figure 13, place it into the center of the sample dish.
3. Apply sufficient downward pressure to ensure that the integral spacers of the reflector are in contact with the base of the dish, and that there are no air bubbles trapped between the reflector and the sample.
4. Position the sample dish and reflector centrally over the sample window, as shown in Figure 14.
5. To ensure consistency between results, always take care to position the reflector in the center of the dish and the dish centrally over the sample window, using the sample locator if required.
You are now ready to collect your spectrum.
6. Place the sample to be analyzed on the NIRA
7. Enter the name of the sample and any description and comments on the Sample tab.

8. Select **Sample** as the **Scan type**.
The default sample position is displayed in the **NIRA** field.
9. Enter scan parameters and instrument settings as required.
10. Click **Start** (Spectrum software) or **Scan** (Spectrum Express software).

Discrimination of Sorbitol, Glycerol and Polyethylene Glycol using the NIRA Liquids Sampling Accessory

Sorbitol, glycerol and polyethylene glycol are all commonly used in the healthcare industry. It is a simple analysis to discriminate between them by FT-NIR using the NIRA Liquids Sampling Accessory. Transflectance measurements are fast and convenient: each analysis takes only a few seconds. A three component library was created as a demonstration, the spectra are shown below. The sorbitol spectrum is dominated by water although there are sorbitol bands at 5880 and 5600 cm^{-1} .

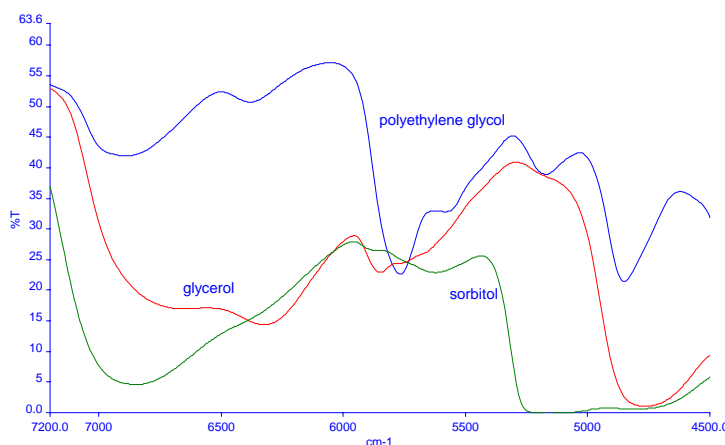


Figure 15 FT-NIR Spectra of Polyethylene Glycol, Glycerol and Sorbitol

An unknown sample was challenged against the library using the COMPARE algorithm. A correlation of 1 indicates an exact match whereas a correlation of 0 indicates no similarity between the spectra.

The unknown was identified as polyethylene glycol with a correlation of 0.96.

Compare - GLY200.SP		
File:	Correlation:	Factor:
polygly.sp	0.9608	1.0632
glycerol.sp	0.7068	0.4931
sorbitol.sp	0.0101	0.0008

Appendix 2: External NIRA Sample Spinner



Figure 16 External NIRA Sample Spinner

What is the Sample Spinner Used For?

This accessory is designed to provide faster and more representative sampling of non-homogenous samples such as wheat, blended feeds and polymer chips, when used in conjunction with the External NIRA.

Particularly suited for samples with particle sizes exceeding 1 mm, the sample spinner obviates the need for collecting spectra of representative batches and then calculating a mean spectrum from the series of results obtained.

Using the sample spinner enables faster, easier analysis and reduces effects such as sample orientation, inhomogeneity and re-pack.

The sample spinner also offers the benefit of non-destructive sampling, as no grinding is required.

<p>NOTE: A comparison between two methods of collecting spectra is shown on page 47.</p>

Typical Sample Spectra

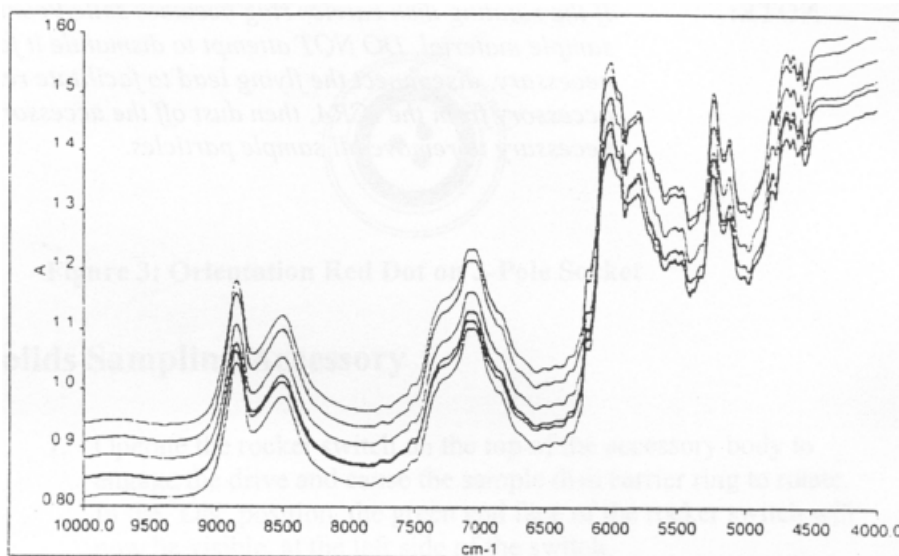


Figure 17 Representative Spectra Collected on a NIRA (traditionally used to calculate a mean)

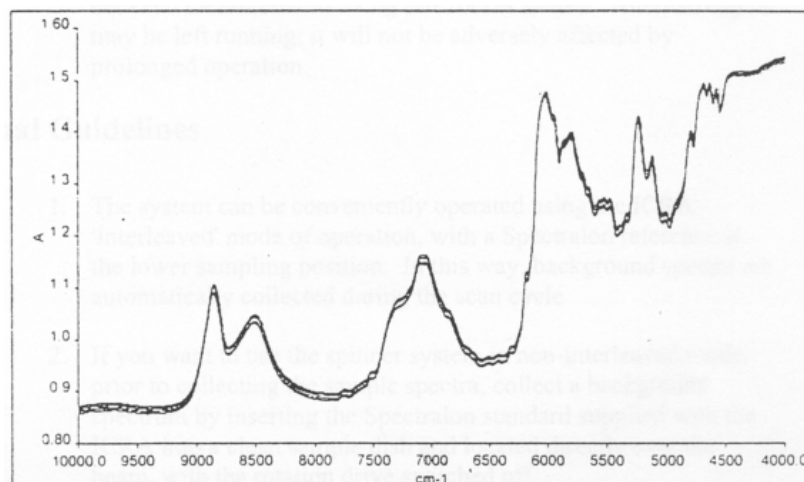


Figure 18 Replicate Spectra (collected using the sample spinner)

Using the Sample Spinner

The sample spinner consists of a housing with a built-in drive unit for a rotating carrier ring.

Material to be sampled is placed in a sampling dish, which is positioned on the carrier ring. The carrier ring, together with the sampling dish, rotates at a fixed speed when activated by the Spectrum software.

The sample spinner locates on top of the External NIRA and receives electrical drive power via a connector that is plugged into a connector on the accessory (See "Installing the Sample Spinner" on page 49).

When the sample spinner is properly positioned, sampling dishes placed into the carrier ring will be located directly above the sample window.

The accessory is intended to be used without any additional training, other than following the procedures and recommendations given in this User's Guide.

List of Parts

As soon as you receive the External NIRA Sample Spinner, make sure that it includes the parts listed below. If anything is missing or damaged, contact the carrier and PerkinElmer.

Part Number	Description	Quantity
L1250042	External NIRA Sample Spinner	1
L1181257	Sampling Dish (90 mm diameter)	5

Optional Items

Part Number	Description	Quantity
L1185305	Quartz Base Sample Dish	1

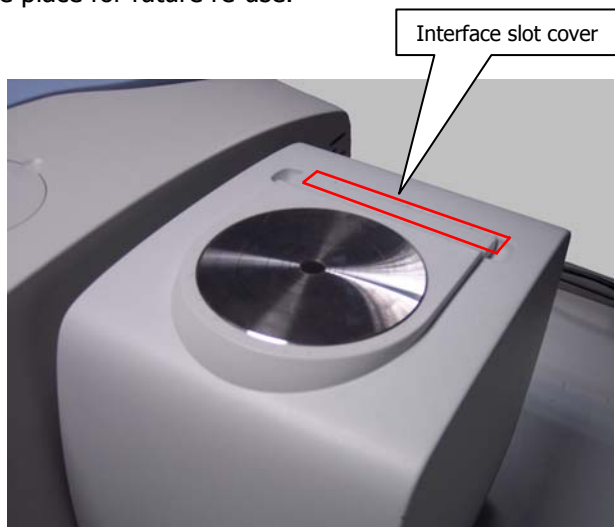
NOTE: For more information about the Quartz Base Sample Dish, see page 51.

Installing the Sample Spinner

CAUTION

Take care when installing the sample spinner that the electrical connection does not get tangled or damaged as this may prevent the sample spinner from operating correctly.

1. Remove the interface slot cover on the External NIRA.
Store it in a safe place for future re-use.



2. Plug the electrical connection on the sample spinner into the interface slot connector on the External NIRA.
The connector is a 4-pin push fit that must be aligned correctly. Align the red dot on the electrical connection with the red dot on the interface slot connector.

3. Making sure that the electrical connection does not get tangled, slide the locating feet of the sample spinner into the interface slot.



4. Gently push the locating feet fully into the interface slot.



The spinner is now correctly positioned over the sampling window.

Using the NIRA Sample Spinner with Spectrum Software

1. Select **Instrument** from the Setup menu.
The Scan and Instrument Setup dialog is displayed.
2. Select the **Scan** tab.
3. Select the **Spinner** check box.
The accessory will rotate when you start a scan and will stop when the scan is completed.

NOTE: If the NIRA Sample Spinner is fitted but the **Spinner** check box is not selected the NIRA Sample Spinner will not rotate during scanning.

Using the NIRA Sample Spinner with Spectrum Express Software

1. Select **Instrument** from the Setup menu.
The Setup Instrument tabs are displayed.
2. Select the **Setup Instrument Basic** tab.
3. Ensure that **Yes** is selected in the **Spinner** drop-down list in the Accessory section.
The accessory will rotate when you start a scan and will stop when the scan is completed.

NOTE: If the Spinner is connected, the software will default to **Yes**, but if you do not wish the Spinner to rotate during scanning, select **No** from the drop-down list.

Preparing your Sample

It is good practice to use a consistent quantity of sample. Ensure that the entire sampling dish is filled to a depth of at least 5-10 mm. Sampling dishes may be used with or without a lid as required.

Cleaning

If the rotating dish carrier ring becomes soiled or contaminated by sample material, DO NOT attempt to dismantle it for cleaning. Disconnect the spinner from the NIRA and then dust off the spinner. If necessary, invert the spinner to remove all sample particles.

The Quartz Base Sample Dish

The optional Quartz Base Sample Dish L1185305 (Figure 19), enables quantitative and qualitative measurements of large granular materials.

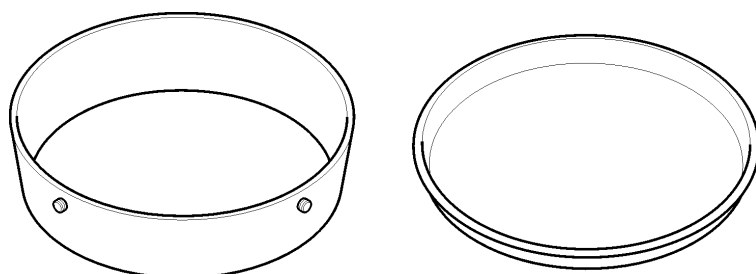


Figure 19 The Quartz Base Sample Dish

The quartz (Suprasil TM) base has a low OH content and is especially suited to more demanding applications. Highly reproducible results are ensured by minimizing the variations due to borosilicate glass that can occur with standard sample dishes.

Care of the Quartz Base Sample Dish

- Do not place the dish on a rough surface as this will scratch the quartz base.
- Do not overfill the dish. Always leave a gap of 1–4 mm between the sample and the rim of the dish to allow the lid to be fitted. If the dish is overfilled the quartz base may crack when attempting to fit the lid.
- Avoid touching the quartz base with your fingers as the marks will affect the results obtained.

Using the Quartz Base Sample Dish

1. Place the dish on a flat surface; preferably on a paper towel placed on a level tray. Pour the sample into the dish leaving at least a 1–4 mm gap from the top of the dish.
At least 1 cm depth of sample is needed for analysis.
2. Level off the sample ensuring a uniform layer of sample adjacent to the quartz base.
3. Screw on the lid.
4. Check once more that the sample is distributed evenly on the quartz base, especially around the edge.
5. Place the dish centrally on the NIRA Sample Spinner.

Cleaning the Quartz Base Sample Dish

When cleaning the dish avoid touching the quartz base with your fingers. Use the soft brush supplied with the NIRA to remove any particles.

If airborne particles are sticking to the glass use an antistatic gun to remove the static.

To remove greasy deposits, wipe the quartz base with a swab that has been dipped in isopropyl alcohol. Make sure that the base is thoroughly dry before reusing the dish.

Appendix 3: Removing and Refitting the External NIRA

In the first instance, the External NIRA is fitted by a PerkinElmer Service Engineer. The accessory should not be removed unless:

- You want to use the external beam port on the right of the instrument with a different external accessory.
- The spectrometer must be moved to a new location.

For information about moving the spectrometer, refer to see the *Spectrum 100 Series User's Guide* (L1050021) or the *Spectrum 400 Series User's Guide* (L1050056), as applicable. This is distributed on the *Spectrum Manuals CD* (L1050002).



WARNING

The spectrometer is a heavy precision instrument, so two people are required for safe handling.

CAUTION

Do not attempt to lift the instrument, or to move it to another location, when an external accessory is attached. The optical alignment of the accessory may be disturbed.

Removing the External NIRA

The External NIRA is attached using two bolts to an external accessory bracket fitted in the handhold on the right of the instrument.

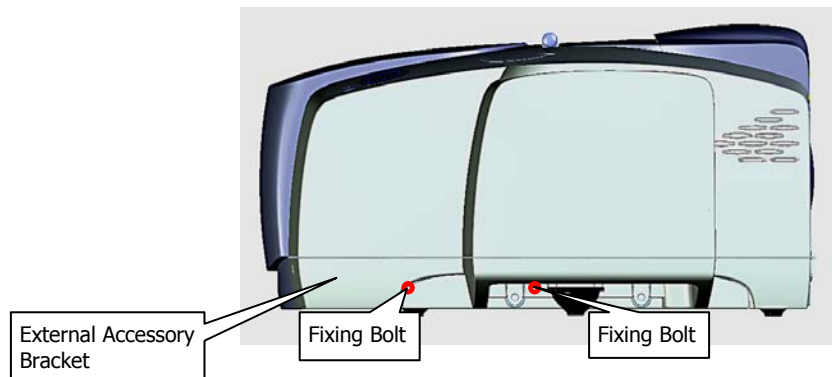


Figure 20 Location of External Accessory Fixing Bolts

1. Switch off the power supply to the accessory, wait for a few seconds, and then disconnect the accessory power cable from the back of the accessory.
The accessory is powered using a separate external power supply.
2. Remove the data cable from the accessory to the instrument.
The data cable connects to the back of the accessory and to the EXT.R port on the back of the instrument.

3. Use the 6 mm hex key supplied to carefully undo the two 100 mm M8 socket-headed bolts that attach the accessory to the external accessory bracket. These bolts are accessible from under the baseplate of the accessory, looking from the right of accessory (Figure 20). Each bolt is fitted with a spacer (between the accessory baseplate and the accessory bracket), a plain washer and a spring washer.

CAUTION

DO NOT attempt to remove the external accessory bracket or to transfer the bracket to another spectrometer.

The external accessory bracket is fitted by a PerkinElmer Service Engineer, along with any other internal components that may be required.

4. Lift the External NIRA carefully, and store it upright in a cool, dry, safe place. Make sure the fixing bolts, spacers and washers are kept with the accessory for future use.
5. When you have completed removing an external accessory, if the instrument is off, turn it on.

OR

If the instrument is on, turn it off, wait for a few seconds, and then turn it on. The instrument performs self-checks that detect the removal of the accessory.

Refitting the External NIRA

To fit the External NIRA:

- The instrument's external beam port must be fitted with a suitable window (typically, KBr).
- There must be at least 300 mm (12 inches) of free bench space to the right of the instrument.
- A suitable power socket must be available for the accessory power supply.
- An external accessory bracket must be fitted in the handhold on the right of the instrument.

The accessory is attached to the external accessory bracket using two 100 mm M8 socket head bolts, two spacers, two plain washers and two spring washers.

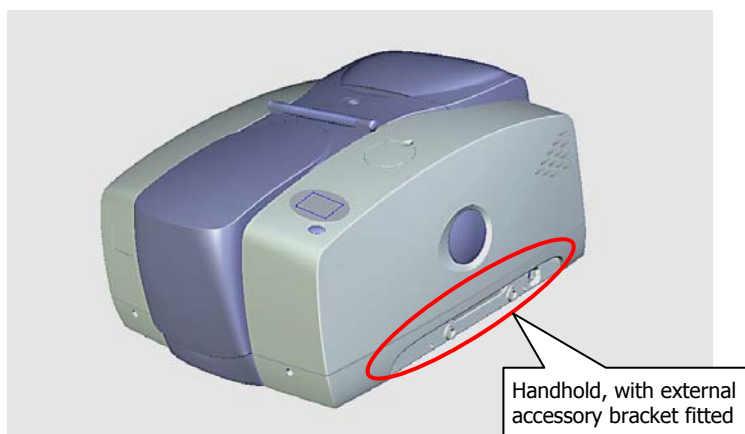


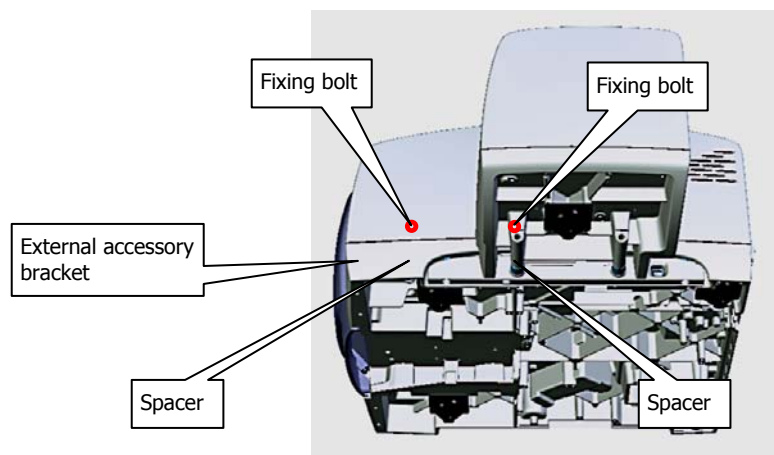
Figure 21 External Accessory Bracket

CAUTION

If an external accessory bracket is not fitted, DO NOT attempt to transfer the bracket from another spectrometer.

The external accessory bracket is fitted by a PerkinElmer Service Engineer, along with any other internal components that may be required.

1. Place a spring washer and then a plain washer on each bolt, and then push the bolts through the fixing holes under the External NIRA baseplate.



WARNING

DO NOT lift the spectrometer when fitting the External NIRA.

The underside of the spectrometer is shown for illustration only.

Figure 22 Location of External Accessory Spacers

2. Add a spacer to each bolt and then carefully align the bolts with the threaded holes in the external accessory bracket.

3. Using the 6 mm hex key supplied, turn each bolt until it engages in the external accessory bracket, make sure the bolts are perpendicular to the bracket, and then carefully and evenly tighten the bolts.

If these bolts engage incorrectly, the accessory may be misaligned or its fixings damaged.

Do not over-tighten the bolts.

4. Connect the data cable to the back of the accessory and to the EXT.R port on the back of the instrument.
5. Connect the accessory power cable to the back of the accessory, and power the accessory.
The accessory utilizes a separate external power supply.
6. If necessary, connect the instrument power supply.
7. If the instrument is off, turn it on.

OR

If the instrument is on, turn it off, wait for a few seconds, and then turn it on.

The instrument performs self-checks that detect the presence of the accessory.