NIRA II
NEAR INFRARED REFLECTANCE ACCESSORY

User's Guide

PerkinElmer®

Release History

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Contents

Warnings and Safety Information

General Safety

The NIRA II Near Infrared Reflectance Accessory is intended for use with a PerkinElmer Frontier FT-NIR or Frontier FT-IR/FT-NIR spectrometer.

These instruments 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.

Radiation Emitted by the Instrument NIR Source

The NIR source in PerkinElmer Frontier FT-NIR and Frontier FT-IR/FT-NIR spectrometers uses 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.

Measurements of the infrared radiation emitted from the spectrometer's sample window 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 sample window. Prolonged exposure (>10 seconds) may cause permanent eye damage.

For further safety and warning information, refer to the appropriate User's Guide for your instrument. These are distributed, as .pdf files, on the *IR & Raman Manuals CD* (L1050002).

Introduction

The NIRA II Near Infrared Reflectance Accessory is used for collecting diffuse reflectance spectra of solids, granules, pastes, powders and turbid liquids. Clear liquids can also be analyzed using the liquids transflectance accessory (see page 34). The technique is non-destructive. It is particularly useful for undiluted powders and granular materials. You can make both qualitative and quantitative measurements.

The NIRA II has been specifically designed to give excellent reproducibility when the same analysis is performed using a number of different spectrometer and accessory combinations. This has been achieved by:

- Designing an optical system of high uniformity and consistency, so that the positioning of the sample has a negligible effect on the spectrum.
- Rapidly collecting alternate measurements of sample and background throughout the scanning of the spectrum to eliminate the effect of drift (see page 17).
- Minimizing the amount of stray light entering the detector, and correcting for the effect of any stray light that is measured (see page 26).
- Introducing a reference correction to standardize the internal reference material of the NIRA II against a chosen reference standard (see page 28).

The use of the optional Sample Spinner (see page 13) allows results of optimum precision to be obtained.

Figure 1 The NIRA II Near Infrared Reflectance Accessory with optional Sample Spinner

How it Works

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

Figure 2 NIRA II top plate

The infrared beam from the instrument enters the NIRA II through the port on the left-hand side of the accessory and is directed upward through the sample window in the top plate.

Near infrared light hitting the sample is absorbed, reflected and scattered over a wide range of angles, as shown schematically in Figure 3.

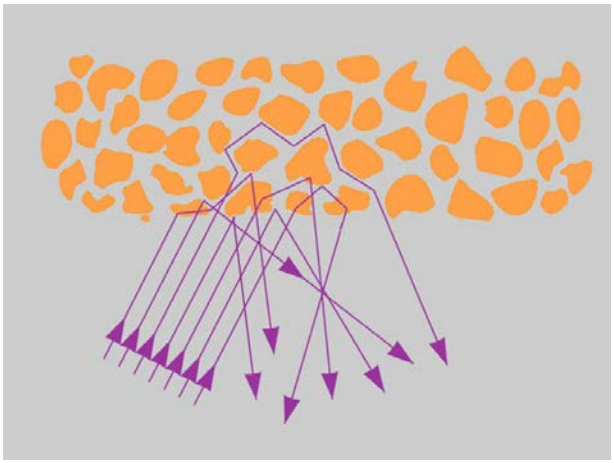


Figure 3 Diffuse Reflectance

The NIRA II is designed to collect the diffusely reflected light from the sample and direct it onto the detector so that the diffuse reflectance spectrum of the sample can be measured.

Diffuse reflectance is measured relative to a suitable reference material. The NIRA II contains an internal reference material that you can use to take a background spectrum, or you can place a suitable external reference material over the sample window. See *Reference Correction* on page 28 for more information.

Sample Options

Because it is a non-destructive technique, diffuse 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, to analyze the coating on a textile, a piece of the fabric can be placed directly on the sample window to obtain an NIR spectrum.

Near infrared diffuse reflectance 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 sample window to obtain an NIR spectrum.

A range of Petri dishes and vials (see Figure 4) is available from PerkinElmer that can be either placed directly on the NIRA II top plate or (in the case of the Petri dishes) used with the optional Sample Spinner. See *Additional Parts* on page 24 for more information.

Figure 4 Sample containers available from PerkinElmer

Packing List

The following items are provided with the NIRA II:

Part Number	Description	Quantity
L1100711	NIRA II Accessory Shipping Disk	1
L1281032	NIRA II Cover	1
L1245010	Sample Alignment Tool	1
L1285013	Standard Sample	1

NIRA II Accessory Shipping Disk

This disk contains an electronic copy of the test certificate for your NIRA II accessory, and the Window and Reference Correction spectra that will be installed by a PerkinElmer Service Representative when your accessory is commissioned (see *Appendix A: NIRA II Correction Routines* on page 25 for further information).

NIRA II Cover

The NIRA II Cover helps protect the top plate and sample window of the NIRA II from dust and contamination when not in use (see Figure 5). It can be used when the Sample Spinner is in place on the top plate. It should also be placed over the sample window when the Stray Light Correction spectrum is being collected. See *Stray Light Correction* on page 26 for further information.

Figure 5 NIRA II Cover in position

Sample Alignment Tool

The Sample Alignment Tool is a device that helps you to reproducibly position vials, beakers and other containers on the NIRA II sample window. It is magnetically held in position on the top plate (see Figure 6). You can use the tool with either side facing upward; a stud just below the sample window in the top plate fits into slots on both sides of the tool, which allows its position to be adjusted to accommodate containers of varying sizes.

Figure 6 Sample Alignment Tool in use

Standard Sample

The Standard Sample is used by the PerkinElmer Service Representative during the installation of your accessory. It is also required if your Spectrum software ever needs to be re-installed. It is not used in any other operation involving the NIRA II.

Keep the Standard Sample in a safe place with the lid on to ensure that it remains clean.

Figure 7 NIRA II Standard Sample

Installing the Accessory

Fitting the Accessory

The NIRA II can be simply installed into the sample compartment of the spectrometer, after first removing any other sampling accessory fitted.

To remove the current accessory and install the NIRA II:

1. Raise the sample cover to the vertical position, press the release clip and lift the cover upwards, clear of the spectrometer.
   Store it in a safe place for future use.

NOTE: Some versions of the Frontier spectrometer do not have a sample cover.

2. Reach in under the base of the current accessory and pull the release handle towards you to release the accessory.

3. Slide the accessory out of the sample area (see Figure 8).
   Store it in a safe place for future use.

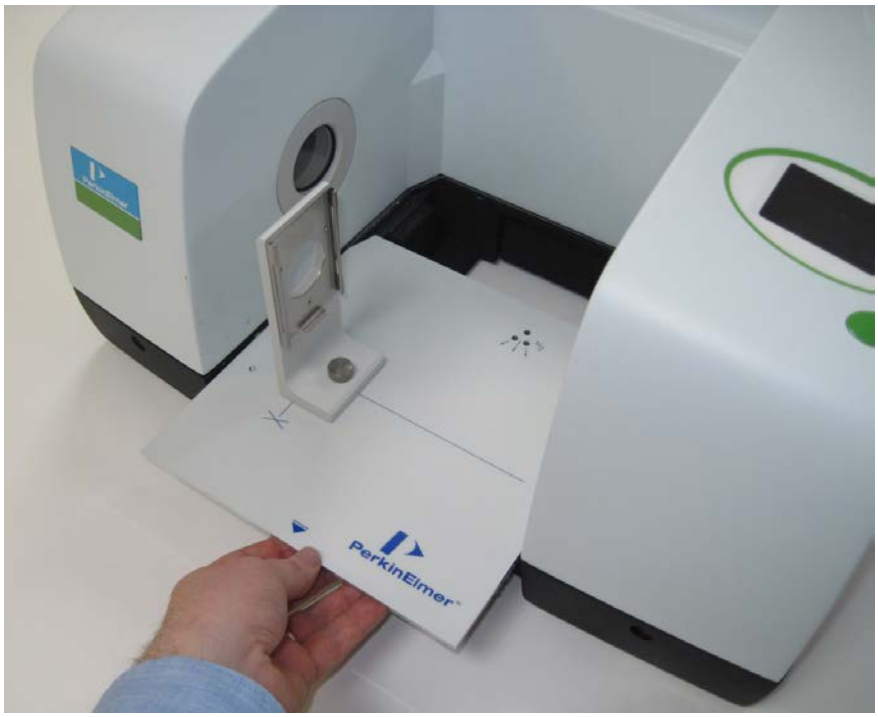


Figure 8 Removing the basic sample slide

4. Slide in the NIRA II, rest the back of it on the ledge in the sample area and slide it into position. Push it firmly home to ensure that the multi-way connector on the rear of the accessory engages properly with the spectrometer connector.
   When correctly installed, a seal around the inside of the NIRA II cover protects the optics in the sample area from contamination if samples are spilt.

Figure 9 NIRA II installed in a Frontier FT-NIR/dual range Spectrometer

NOTE: If you have Spectrum software and do not have an instrument installed, see the *Spectrum Administrator's Guide* (L1050095) on the *IR & Raman Manuals CD* (L1050002) for details of the Instrument Install Wizard.

Accessory Detection by Spectrum 10 Software

NOTE: The NIRA II will only be detected and operate correctly when used with Spectrum software version 10.4.1 or later. If you are using a spectrometer purchased previously, you may also need to update the firmware. See *Appendix C: Upgrading a Spectrometer to Control the NIRA II* on page 36 for further details.

The Spectrum 10 software detects the presence of the accessory, and the NIRA II icon is added to the toolbar. The Setup Instrument Basic (see Figure 10) and Setup Instrument BeamPath (see Figure 11) tabs are updated to show that the NIRA II is in position.

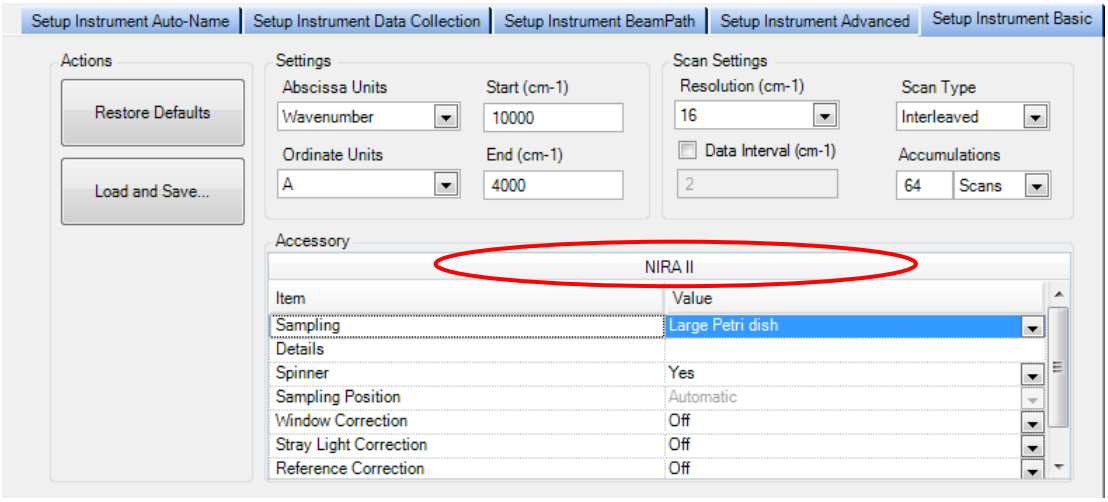


Figure 10 Setup Instrument Basic tab with NIRA II (circled)

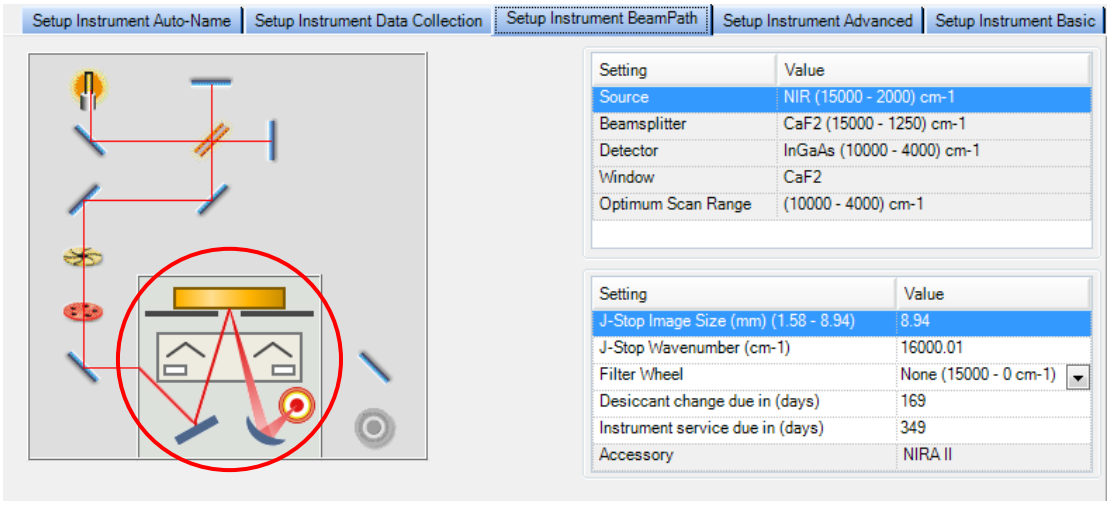


Figure 11 Setup Instrument BeamPath tab with NIRA II (circled)

NIRA II Sample Spinner

The NIRA II Sample Spinner is an optional accessory that is designed to provide more representative sampling of non-homogenous samples such as wheat, blended feeds and polymer chips, when used in conjunction with a NIRA II. It also optimizes the precision of the results obtained for apparently homogeneous samples.

Particularly suited for samples with particle sizes exceeding 1 mm, the NIRA II Sample Spinner obviates the need for collecting spectra of representative batches and then calculating a mean spectrum from the series of results obtained (see Figure 13).

Using the NIRA II Sample Spinner enables easier analysis and reduces the effect of sample orientation, inhomogeneity and re-pack. It offers the benefit of non-destructive sampling, as no grinding is required.

Two versions of the NIRA II Sample Spinner are available for Petri dishes with approximate diameters of 100 mm (L1280417) and 60 mm (L1280410) (see Figure 12).

Figure 12 NIRA II Sample Spinners for 100 mm (left) and 60 mm (right) Petri dishes

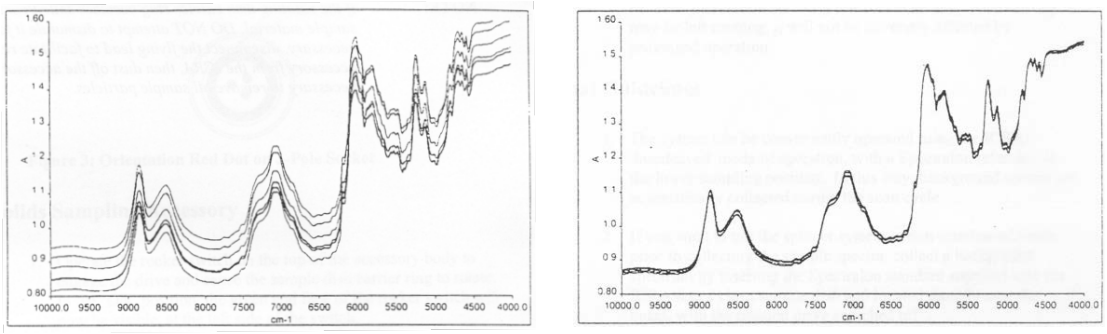


Figure 13 Replicate spectra taken with (right) and without (left) using a Sample Spinner

Using the NIRA II Sample Spinner

The NIRA II Sample Spinner is composed of a housing with built-in drive unit, incorporating a rotating turntable.

Material to be sampled is placed in a Petri dish, which is positioned on the turntable. The turntable, together with the Petri dish, rotates at a speed of approximately 20 rpm when activated by the Spectrum 10 software.

The NIRA II Sample Spinner locates on three studs in the top plate of the NIRA II and receives electrical drive power wirelessly via a transmitter coil located in the top plate of the NIRA II. When the Sample Spinner is properly positioned, Petri dishes placed onto the turntable will be located above the sample window (see Figure 14).

CAUTION	*To avoid the possibility of losses in the wireless power transmission and intermittent operation of the Sample Spinner, keep the Spinner and top plate free of metal objects.*

A red LED on the motor housing will flash if the Sample Spinner detects a high load on the drive motor.

CAUTION	*Do not operate the Sample Spinner for more than a few minutes with the LED flashing. Check that the sample weight is below 300 g. If the flashing LED persists, clean the Sample Spinner (refer to* Cleaning the Sample Spinner *on page 25) to remove any material clogging the drive mechanism.*

The accessory is intended to be used by operators of the NIRA II 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 NIRA II Sample Spinner kit, make sure that it includes the parts listed below. If anything is missing or damaged, contact the carrier and PerkinElmer.

Description	Quantity
NIRA II Sample Spinner	1
Spinner Cover Hex Key and Screws Kit (L1280427)	1

Installing the NIRA II Sample Spinner

The NIRA II Sample Spinner should be fitted after the NIRA II is installed in the spectrometer (see page 10).

1. Check that the top plate of the NIRA II is clean and dry (see *Cleaning the NIRA* II on page 19 for further information).

2. Holding the drive unit, locate the NIRA II Sample Spinner in position on the top plate, using the thick green line as a guide (see Figure 14).

   The Sample Spinner should "click" into place when correctly positioned. The Spinner is designed to sit in exactly the same position each time it is placed on the top plate.

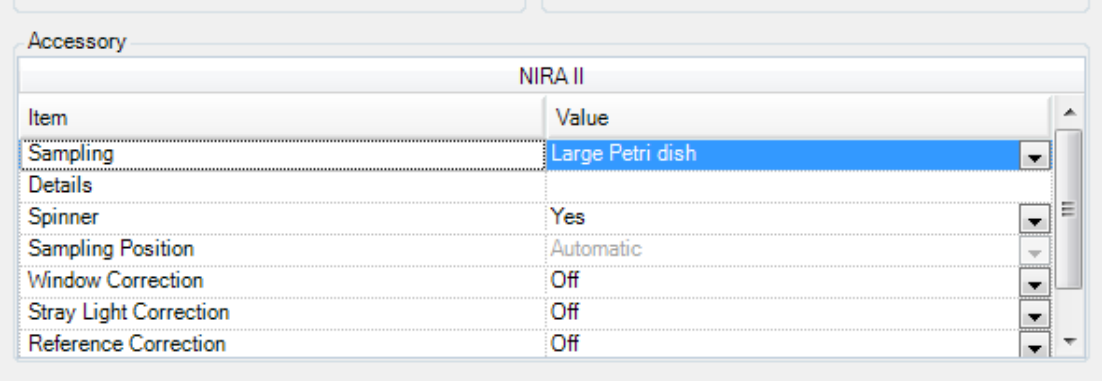
Figure 14 NIRA II Sample Spinner in position over sample window

Using the NIRA II Sample Spinner with Spectrum 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 Sample Spinner is connected, the software will default to **Yes**, but if you do not want 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 mm. Sampling dishes may be used with or without a lid as required. A maximum sample weight of 300 g is recommended.

To reduce the risk of spilling sample on to the Spinner, fill the sampling dish on a surface away from the NIRA II and check that the outside of the dish is clean before placing it on to the turntable.

Performing a Scan in Spectrum 10

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 10 will default to the instrument settings that were last used to perform a successful scan with that accessory.

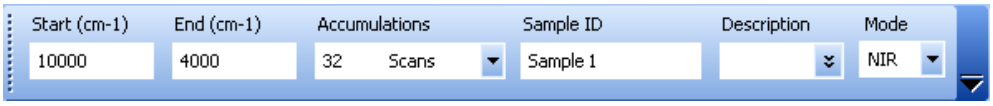


Figure 15 The Instrument Settings toolbar

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

2. Place your sample over the sample window.

3. Click  on the Measurement toolbar to collect a spectrum.

   The NIRA II starts in **Interleaved** mode. In this mode, background scans are automatically collected from the internal reference material as part of the sample scan; you do not need to collect a separate background.

For most applications, we recommend that the default instrument settings are used. However, if you want to change the settings (for example, to collect a **Background** scan in the **External** 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.

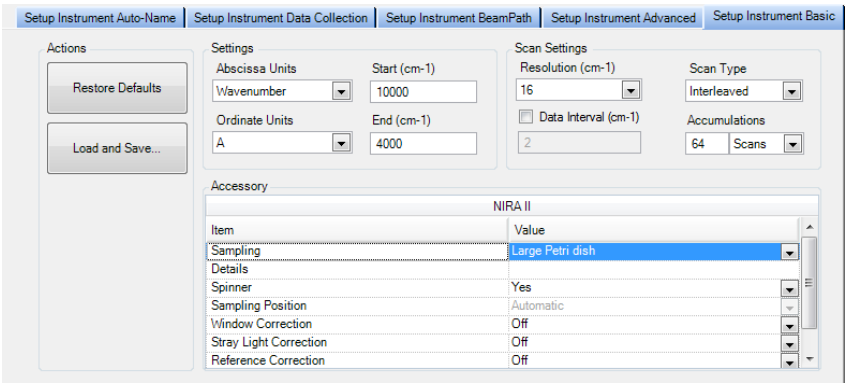


Figure 16 Setup Instrument Basic tab with Interleaved as the Scan Type

3. Select the **Scan Type** for your spectrum.

   The recommended setting for this option is **Interleaved**.

   The other options available are: Background, Sample, Single Beam and Interferogram. See the Spectrum on-screen Help for more information on these settings.

4. Select the **Sampling Position** setting from the drop-down list.

   There are three options for the **Sampling Position** setting:

   - **Automatic** – a background scan is always taken in the Internal position (from the internal reference material) and a sample scan is always taken in the External position (at the sample window) for all scan types.

> **NOTE**: The **Automatic** setting is always used if the Scan Type is **Interleaved**. (If Interleaved is selected, the Sampling Position option is not available.)

   - **Internal** – the scan is taken from the internal reference material inside the NIRA II.

   - **External** – the scan is taken at the sample window.

     A Background scan in the External 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 External position, ensure that a suitable white reflector such as the 99% reflectance polymer (L1281920/1) is placed on the sample window in the NIRA II top plate.

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

5. If Spectrum 10 detects that a Background spectrum is required, the Scan icon will display a flag at the bottom right-hand corner  . Click 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 (see Figure 16) and then click  .

   If you switch on one or more of the NIRA II correction routines (see page 25) without the corresponding reference spectra being available, then the Scan icon will display a warning  , and you will not be able to collect a spectrum. Position the mouse pointer over the icon to view the corrections that are affected.

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

Maintenance

Cleaning the NIRA II

The outer casing of the NIRA II is designed to be easily cleaned to prevent spurious peaks occurring in the spectrum due to cross-contamination.

- Use a damp cloth to wipe off any spilt sample material.
  For water-insoluble residue, use a mild solvent such as ethanol or propan-2-ol (iso-propyl alcohol, IPA).

CAUTION	*Do not use acetone to clean the NIRA II.*

Care should be taken when cleaning the window in the top plate to prevent scratching. We recommend the following procedure:

1. Moisten an unused lens cleaning tissue with a little deionised water or ethanol.
2. Wipe gently over the window in a straight line.
   Do not use any area of the tissue more than once; any residue picked up previously could scratch the window when wiped over it a second time.
3. Allow the top plate to dry thoroughly before continuing with the next analysis.

CAUTION	*Do not submerge the NIRA II or place it under running water when cleaning it.*

When the accessory is not in use, the NIRA II cover can be used to reduce the need for frequent cleaning of the top plate and window (see page 8).

Replacing Scratched Windows

Over time, some scratching of the window may occur. The light-scattering effect of these scratches can be corrected (see *Stray Light Correction* on page 26). However, correcting for high levels of stray light may adversely affect the quality of your sample data. The Stray Light Correction Wizard will inform you if the level of stray light is too high for the correction to be applied, in which case you should clean the sample window thoroughly. If this does not reduce the stray light sufficiently, then you should replace the sample window.

To replace the window, the entire top plate of the NIRA II must be replaced. A new Window Correction spectrum is supplied with each top plate which must be installed on your system. Contact your PerkinElmer Service Representative for further information.

Cleaning the Sample Spinner

To prevent cross-contamination between samples, regularly wipe down the outer surface of the NIRA II Sample Spinner with a damp cloth.

If the rotating turntable becomes heavily soiled or contaminated by sample material, the drive mechanism may experience a high load, as indicated by a red LED flashing on the motor housing. If this should happen, the Sample Spinner can be partially dismantled for more thorough cleaning. This level of cleaning should not be required frequently unless the Sample Spinner is used extensively in a dusty environment. In these circumstances, the NIRA II cover can be placed over the Sample Spinner when it is positioned on the NIRA II to help keep the surfaces clean (see page 8).

CAUTION

When cleaning the Sample Spinner:

- *Do not attempt to remove the drive unit cover;*
- *Do not use acetone;*
- *Do not submerge the Spinner or place it under running water.*

To clean the Sample Spinner:

1. Grasp the Sample Spinner at the positions indicated with arrows on the drive unit and lift the whole assembly off the top plate of the NIRA II (see Figure 17).

Figure 17 Removing the Sample Spinner from the NIRA II top plate

2. Remove the two retaining screws underneath the front of the Sample Spinner using the 1.5 mm hex key provided (see Figure 18).

CAUTION *Do not remove any other screws from the underside of the Spinner.*

Figure 18 Position of retaining screws for the top cover

3. Lift the turntable cover slightly and slide it firmly towards the front of the Sample Spinner to remove it (see Figure 19).

Figure 19 Removing the turntable cover

4. Lift out the toothed turntable and white plastic bearing (see Figure 20).

Figure 20 Turntable and bearing removed for cleaning

5. Wash the turntable, bearing and cover with water, and dry thoroughly.
   For heavy contamination, use a weak detergent solution or, in extreme cases, a mild solvent such as ethanol or propan-2-ol (iso-propyl alcohol, IPA). Rinse the components thoroughly in water before drying.

CAUTION	*Do not use acetone to clean the NIRA II Sample Spinner.*

6. Wipe the base of the Sample Spinner clean with a damp cloth and dry thoroughly.
   Do not blow spilt powder off the base as it may become lodged in other areas of the Spinner.

7. Reassemble the Sample Spinner by replacing the bearing, turntable and outer cover, and insert the screws to hold the cover in place.
   Push the bearing into place firmly. It can be inserted in either orientation.

CAUTION	*Only reassemble the Sample Spinner once all the components are completely dry.* *Do not apply any grease to the bearing.*

The cleaning procedure is exactly the same for the 60 mm Spinner version, which dismantles as shown in Figure 21.

Figure 21 60 mm Sample Spinner dismantled for cleaning

Additional Parts

The following optional parts are available for use with the NIRA II. Where the part dimensions are 90–100 mm, they are suitable for use with the 100 mm Sample Spinner, and where the dimensions are 55–60 mm, the parts are suitable for use with the 60 mm Sample Spinner (see *NIRA II Sample Spinner* on page 13).

Part Number	Description
L9004575	90 mm plastic Petri dish (pack of 600)
L9004578	55 mm plastic Petri dish (pack of 1620)
L9004574	60 mm soda glass Petri dish (pack of 10)
L9004573	100 mm soda glass Petri dish (pack of 10)
L9004892	Glass vials (pack of 144)
L1281920	99% reflectance polymer (60 mm diameter)
L1281921	99% reflectance polymer (100 mm diameter)
L1281922	99% calibrated reflectance standard (60 mm diameter)
L1281923	99% calibrated reflectance standard (100 mm diameter)
L1281924	Calibrated reflectance standard (nominal 50% reflectivity) (60 mm diameter)
L1281925	Calibrated reflectance standard (nominal 50% reflectivity) (100 mm diameter)
L1281926	Calibrated reflectance standards (set of 4) (60 mm diameter)
L1281927	Calibrated reflectance standards (set of 4) (100 mm diameter)
L1281928	Calibrated reflectance abscissa standards (60 mm diameter)
L1281929	Calibrated reflectance abscissa standards (100 mm diameter)
L1281930	Reflectance standard recalibration service
L1185153	Liquids Transflectance Accessory Kit

We recommend that calibrated standards are sent for recalibration every 12 months. The reflectance standard recalibration service (L1281930) can be used to recalibrate any of the standards listed in the table.

Appendix A: NIRA II Correction Routines

Three correction routines are available specifically for the NIRA II to help ensure that its performance remains optimized throughout its service lifetime. The corrections will be set up by a PerkinElmer Service Representative during the installation and commissioning of your accessory. You can then select whether or not to apply these corrections using Spectrum software.

NOTE: If you choose to apply any of the correction routines, they will be applied to all subsequent measurements on your system until you disable them.

In most cases, you should only need to modify the correction routines if you have to reinstall the Spectrum software. However, you can update the Reference and Stray Light Corrections if required using Spectrum 10 software to collect new spectra.

In addition to the corrections below, you will need to run an AVI calibration specifically for the NIRA II before you can begin scanning samples. You cannot use a transmission AVI calibration.

Window Correction

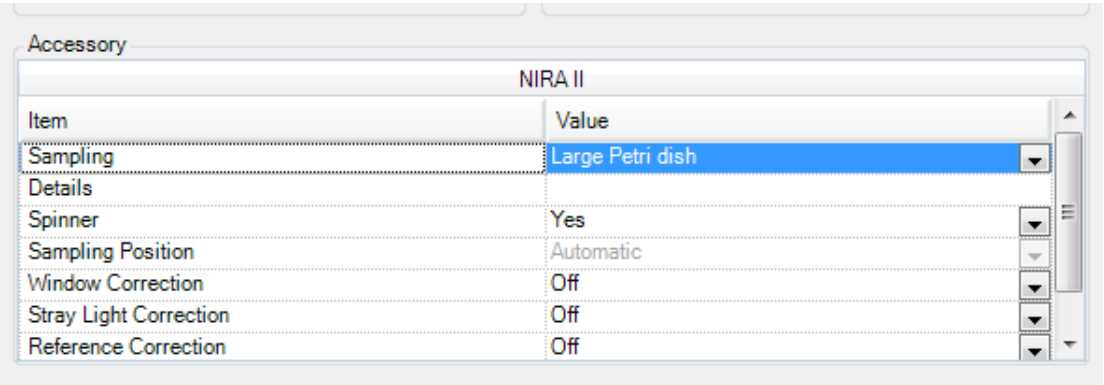
The fused silica window of the NIRA II has very high transmittance in the near-infrared region, absorbing only weakly at 4500 and 7200 cm^{-1}. If you collect sample spectra using a background taken from the internal reference material, then these absorbances may be visible. The Window Correction compensates for this effect by subtracting a reference spectrum of the NIRA II sample window (measured at the PerkinElmer factory) when appropriate, most commonly when the background spectrum is collected from the internal reference material and the sample is placed on the sample window.

NOTE: We recommend that the Window Correction spectrum supplied with your NIRA II top plate is always installed on your system in order for other correction spectra to be collected and applied properly. If you have to reinstall your software on a different PC, contact your PerkinElmer Service Representative for assistance with installing the Window Correction spectrum.

To apply the Window Correction in Spectrum software:

1. Select **Instrument** from the Setup menu.
   The Setup Instrument Basic tab is displayed.

2. In the Accessory section, click the arrow for the row marked **Window Correction** to display the drop-down list.



3. Select **Automatic** to apply the correction when required.
   If you are already applying the correction and want to stop doing so, select **Off**.

NOTE: You cannot change the Window Correction spectrum. If you replace the top plate of your NIRA II, a new correction spectrum will be supplied with the new window to be installed by your PerkinElmer Service Representative.

Stray Light Correction

Stray light is light from the instrument that reaches the detector without having interacted with the sample, which can cause distortion of strong absorptions in your spectrum. Although the NIRA II has been designed to minimize stray light, some light scattering will always occur at the sample window, and this effect is increased if the window becomes dirty or scratched. The Stray Light Correction allows you to compensate for this unavoidable source of error in the spectrum.

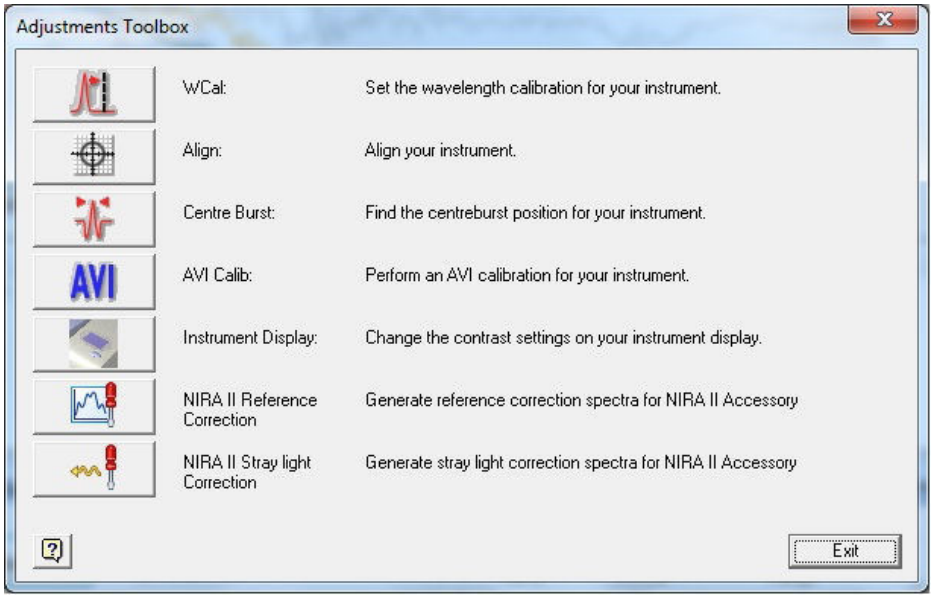
This correction should only be updated if the stray light increases due to light scratching of the sample window. It should not be regarded as a substitute for keeping the window clean and avoiding creating scratches. If the stray light rises above a certain threshold, indicated by a red line in the monitoring screen of the Stray Light Correction Wizard, then the software will indicate that it can no longer adequately correct your results and prevent you from collecting a correction spectrum, at which point a new sample window may be required.

NOTE: The Stray Light Correction does not correct for ambient light in the location of your instrument. This light should have no effect on the results from your samples.

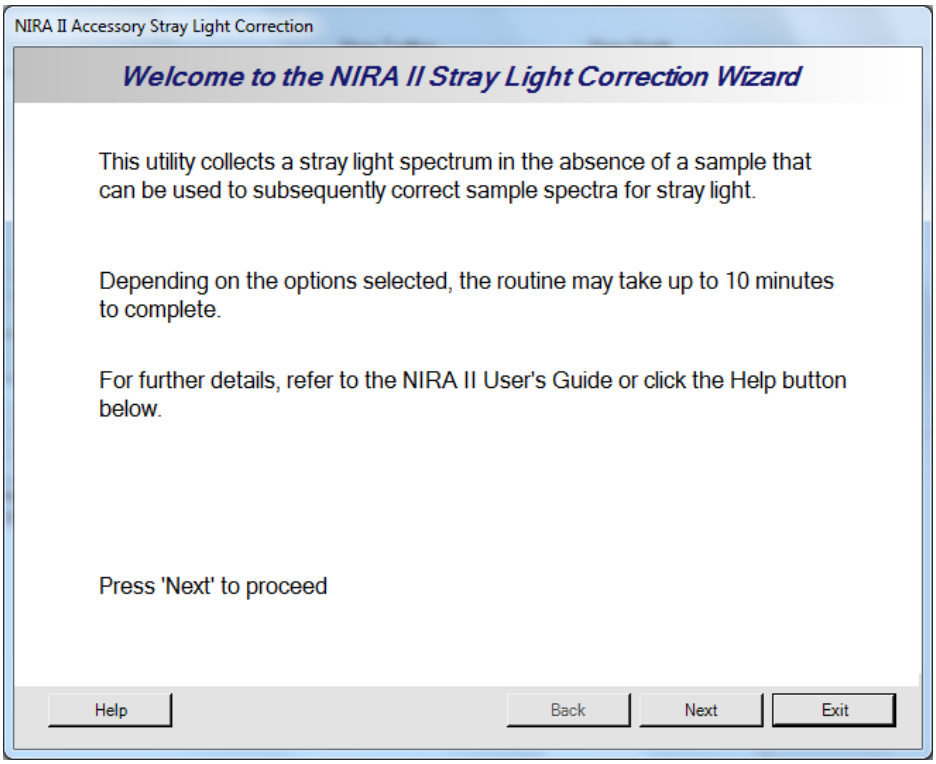
To use Spectrum 10 software to collect a new Stray Light Correction spectrum:

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

2. Select the Setup Instrument Advanced tab and then click **Toolbox**.
   The Adjustments Toolbox dialog is displayed.



3. Click  .
   The Stray Light Correction Wizard is displayed.

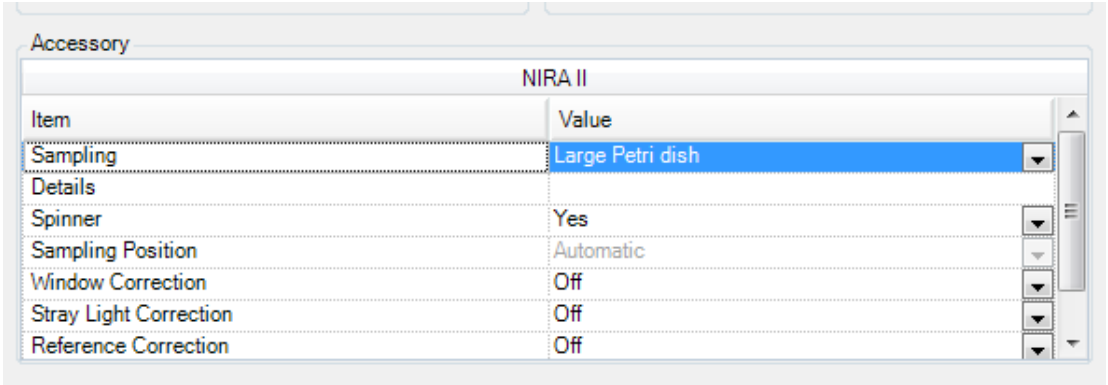


4. Follow the instructions on-screen.
   It is essential to ensure that the sample window is clean and dry, and that the NIRA II cover is placed over the top plate of the NIRA II when you are collecting the stray light correction spectra (see Figure 5).

To apply the Stray Light Correction:

1. Select **Instrument** from the Setup menu.
   The Setup Instrument Basic tab is displayed.

2. In the Accessory section, click the arrow for the row marked **Stray Light Correction** to display the drop-down list.



3. Select **On** to apply the correction.
   If you are already applying the correction and want to stop doing so, select **Off**.

Reference Correction

Diffuse reflectance spectra obtained from the NIRA II (see Figure 3) are measured relative to a white reflector reference material. The NIRA II is supplied with an internal sample of this reference material, or you can use your own external reference material placed over the sample window. The Reference Correction standardizes the chosen reference material against a separate standard, which ensures that reproducible results can be obtained by different systems that use a NIRA II.

The internal reference material is standardized against a standard maintained at the PerkinElmer factory. The corresponding correction spectrum should be copied on to your system when the NIRA II accessory is installed, and should not need to be updated unless you purchase a new accessory.

Alternatively, you can choose to standardize the internal reference material against your own external standard. If you want to use an external reference material instead of the internal reference, it is also possible to standardize this external reference against another external reference material. In this way, multiple NIRA II systems, each with their own external references, can all be standardized against a common reference.

While the factory reference correction spectrum should be suitable in nearly all cases, if you encounter differences between systems when attempting to transfer calibration models, we recommend you obtain an external reference sample (such as PerkinElmer part numbers L1281920/1) and re-standardize the instruments concerned.

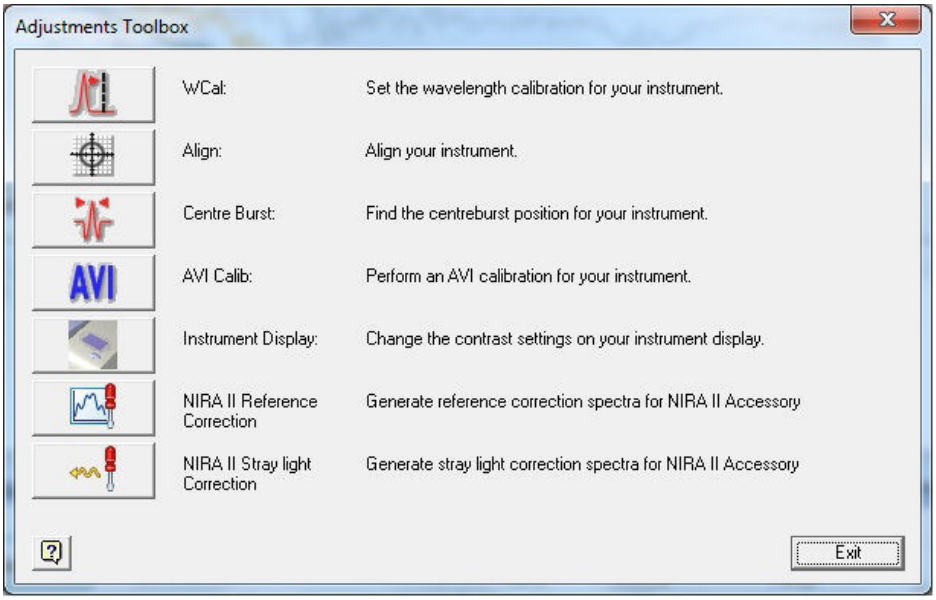
We recommend that the Window Correction spectrum supplied with your NIRA II top plate is always installed on your system **before** you collect a Reference Correction spectrum. If this is not the case, then collecting a Reference Correction spectrum will effectively apply the Window Correction simultaneously; however, you **must** remember to collect a new Reference Correction spectrum if you subsequently change your top plate or install the Window Correction spectrum. Likewise, if you subsequently install a Window Correction spectrum, you **must** collect a new Reference Correction spectrum.

NOTE: To ensure that an accurate correction is applied, it is essential to ensure that any external reference materials that you use are not contaminated. Also check that the top plate of the NIRA II is clean. Refer to *Cleaning the NIRA II* on page 19.

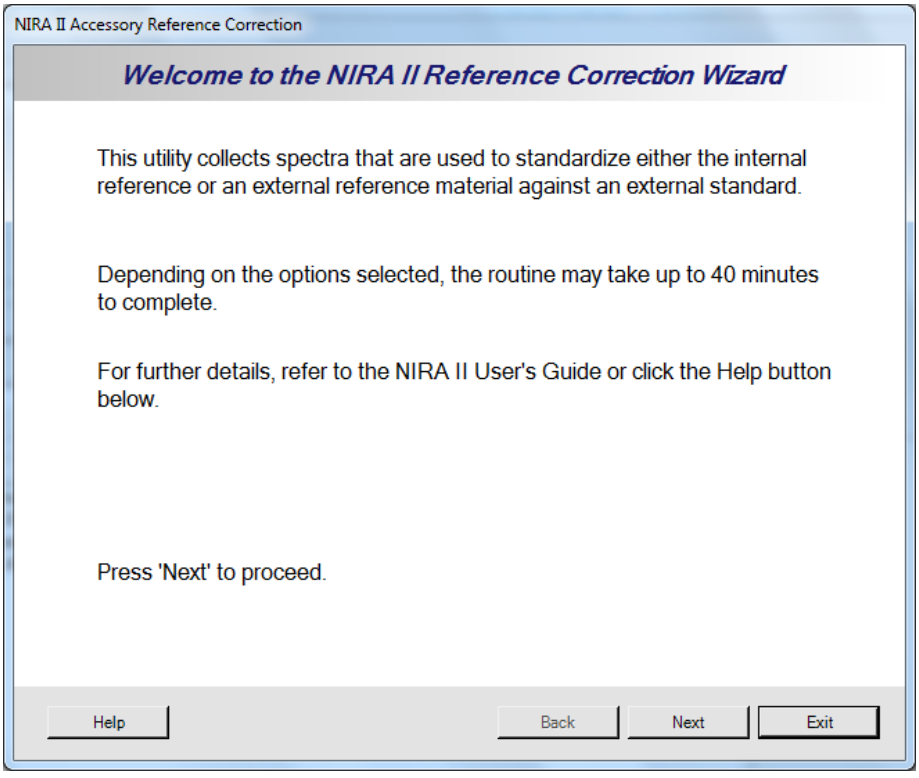
To use Spectrum 10 software to collect a new Reference Correction spectrum:

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

2. Select the Setup Instrument Advanced tab and then click **Toolbox**.
   The Adjustments Toolbox dialog is displayed.



3. Click  .
   The Reference Correction Wizard is displayed.

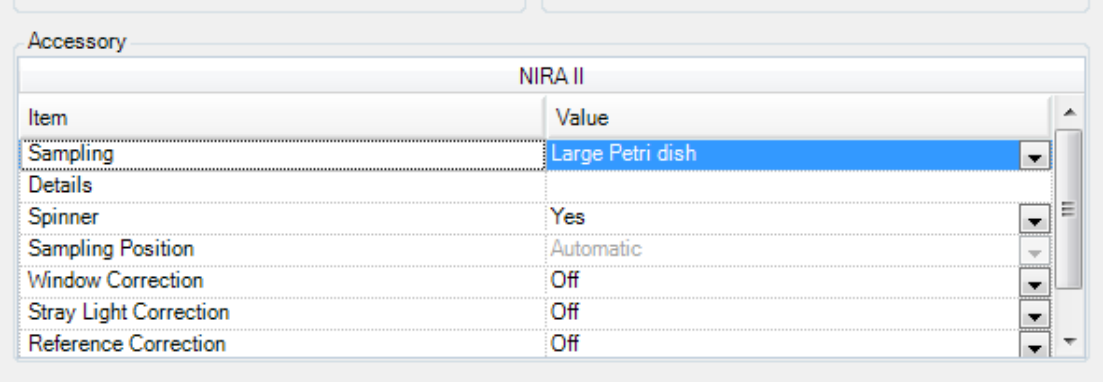


4. Follow the instructions on-screen.

To apply the Reference Correction:

1. Select **Instrument** from the Setup menu.
   The Setup Instrument Basic tab is displayed.

2. In the Accessory section, click the arrow for the row marked **Reference Correction** to display the drop-down list.



3. Select **On** to apply the correction.
   If you are already applying the correction and want to stop doing so, select **Off**.

Returning to Default Corrections

You can use the Reference Correction Wizard to reset the correction spectra to the factory-default settings.

1. On the Choose Reference screen of the wizard, select **Remove User-Generated Standardizations**, and click **Next**.

2. Click **Next** again to confirm that you want to return to the factory-default settings.

You can also remove the stray light correction spectrum from your system using the Stray Light Correction Wizard.

1. On the Choose Reference screen, select **Remove User-Generated Corrections**, and click **Next**.

2. Click **Next** again to confirm that you want to remove the stray light correction spectrum.

No stray light correction is possible once this process has been completed.

Appendix B: Liquids Transflectance Accessory

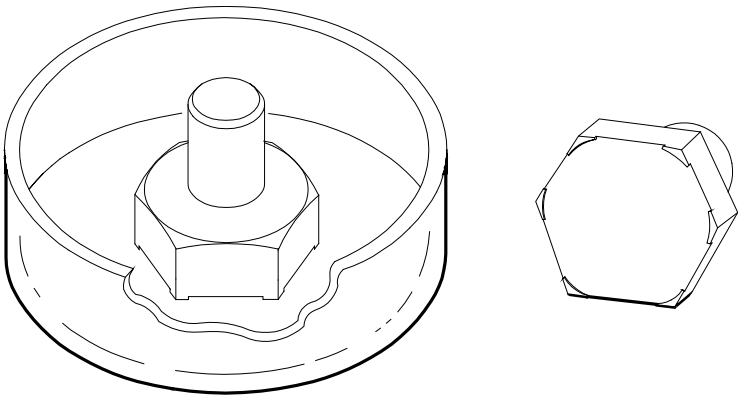


Figure 22 The liquids transflectance accessory

The Liquids Transflectance Accessory (L1185153) extends the range of sampling possible with a NIRA II. This optional accessory facilitates the sampling of liquids and gels, providing users with a near-universal sampling system for the quality control screening of a wide range of solids, pastes, liquids and gels.

This device 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 II sampling position, significantly enhancing the versatility of the NIRA II sampling system.

It can 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 II, without any additional training, other than following the procedures and recommendations given in this User's Guide.

This 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 achieved by removing the NIRA II and replacing with the basic sample slide.

A typical application of the Liquids Transflectance Accessory is shown on page 35.

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.

Item	Quantity	Part Number
Sample Dish	5	L1181106
Machined Diffuse Reflector	5	L1181105

Scanning using the Liquids Transflectance Accessory

For simple identification of liquids or gels, it is usually sufficient to use the normal interleaved scanning mode to collect a spectrum (see *Interleaved Scanning using the Liquids Transflectance Accessory* on page 34). For more demanding measurements, additional corrections need to be applied to take account of the unwanted reflection from the base of the dish.

The procedure for obtaining a spectrum using the Liquid Transflectance Accessory kit involves four stages, described below.

Stage 1 – Obtaining a Correction scan

A correction scan is required in order to compensate for any unavoidable and unwanted reflections from the base of the sample dish.

1. Position the empty sample dish centrally over the sample window of the NIRA II.
2. Pour in water, to a depth of approximately 1 mm.
3. Check the scan and instrument parameters on the Instrument Settings toolbar and enter an appropriate **Sample ID** and **Description** for your correction scan.
4. On the Setup Instrument Basic tab, select **Single Beam** as the **Scan Type**.
5. Click  on the Measurement toolbar to begin collecting data.
6. Once a correction spectrum has been obtained, save it under a suitable name.

Stage 2 – Obtaining a Background scan

1. Handling the diffuse reflector only by its upper section, as shown in Figure 23, place it in the center of an empty sample dish.

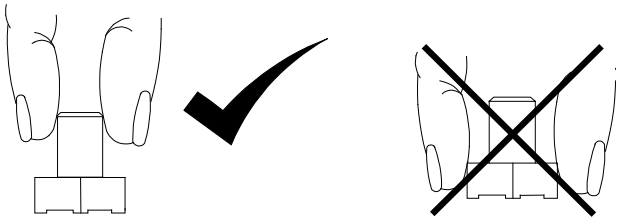


Figure 23 Handling the diffuse reflector

2. Position the sample dish and reflector centrally over the upper sample window, using the sample locator, as shown in Figure 24.

Figure 24 Liquids transflectance accessory in position

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 circles on the top plate or the sample alignment tool if required.

3. Ensure that the scan parameters and instrument settings shown on the Instrument Settings toolbar are the same as those you used to obtain the correction scan.

4. Enter an appropriate **Sample ID** and **Description** for your background scan.

5. On the Setup Instrument Basic tab, select **Background** as the **Scan Type**.

6. Click  on the Measurement toolbar.
   The background scan is collected.

7. Once a background spectrum has been obtained, save it under a suitable name.
   You are now ready to collect a sample scan.

Stage 3 – Obtaining 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 (see Figure 23), 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 (see Figure 24).
   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.

5. Ensure that the scan parameters and instrument settings shown on the Instrument Settings toolbar are the same as those you used to obtain the correction and background scans.

6. Enter an appropriate **Sample ID** and **Description** for your sample scan.

7. On the Setup Instrument Basic tab, select **Single Beam** as the **Scan Type**.

8. Click  on the Measurement toolbar.
   The sample scan is collected.

9. Once the sample spectrum has been obtained, save it under a suitable name.

Stage 4 –Applying the Correction to the Background and Sample

- In the Spectrum software, use the Arithmetic process command to calculate the corrected spectrum, as follows:

$$\text{Corrected spectrum} = \frac{\text{Sample spectrum} - \text{Correction spectrum}}{\text{Background spectrum} - \text{Correction spectrum}}$$

  For details of how to use the Arithmetic process command, see the Spectrum on-screen help.

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 (see Figure 23), 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 (see Figure 24).

   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.

5. Check the scan and instrument parameters on the Instrument Settings toolbar and enter an appropriate **Sample ID** and **Description** for your sample.

6. On the Setup Instrument Basic tab, select **Interleaved** as the **Scan Type**.

   The background spectrum will be taken in the lower position and the sample spectrum will be taken in the upper position.

7. Click  on the Measurement toolbar to begin collecting data.

NOTE: Interleaved scans taken in this way are not corrected for reflections from the base of the sample dish.

Example – Discrimination of Sorbitol, Glycerol and Polyethylene Glycol using the Liquids Transflectance 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 Liquids Transflectance 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}.

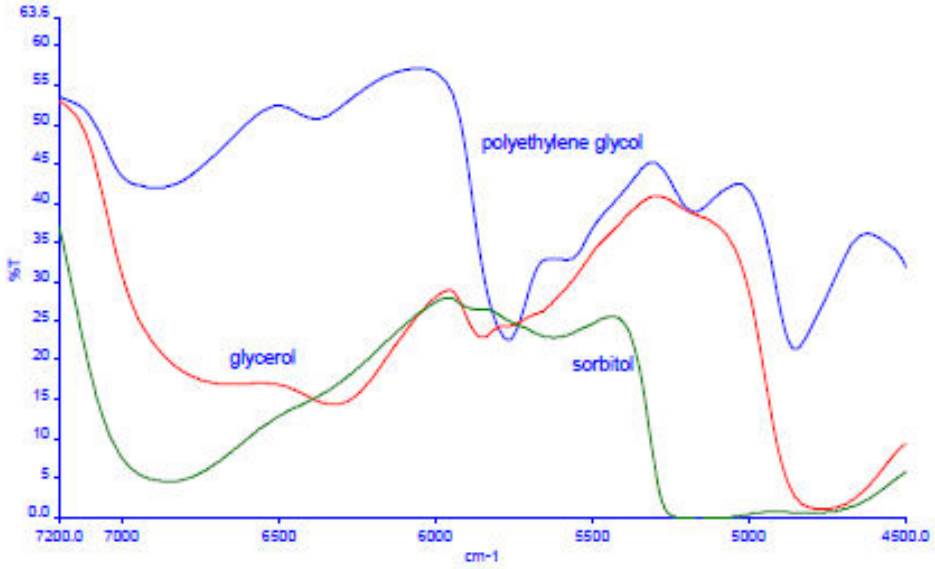


Figure 25 FT-NIR spectra of polyethylene glycol, glycerol and sorbitol

Appendix C: Upgrading a Spectrometer to Control the NIRA II

If you have acquired a NIRA II accessory to be used with a previously purchased Spectrum 100N / Frontier NIR or Spectrum 400 / Frontier MIR/NIR spectrometer, you will need to upgrade the firmware in your spectrometer to provide the capability to control the accessory.

Before upgrading the firmware, you must install Spectrum software version 10.4.1 (or above) on your PC, and use it to connect to your spectrometer.

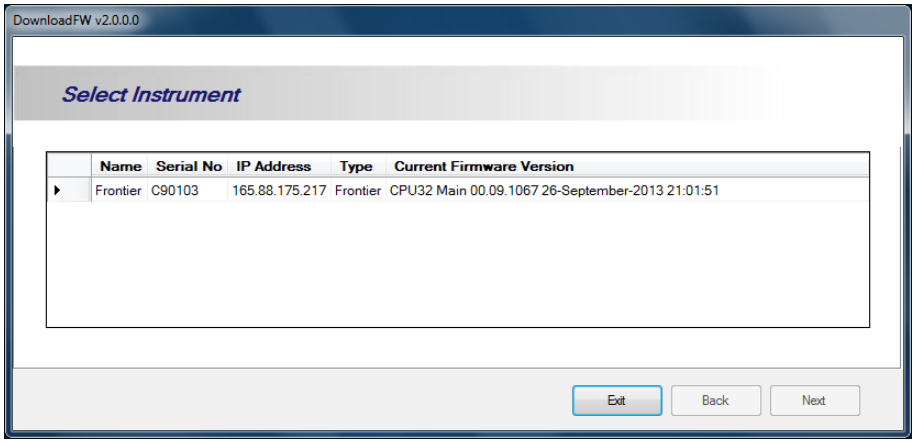
Follow the procedure below to upgrade the firmware:

1. Ensure that Spectrum software or any other software controlling the spectrometer is closed.

2. Using your browser, navigate to 'C:\Program Files (x86)\PerkinElmer\ServiceIR'.

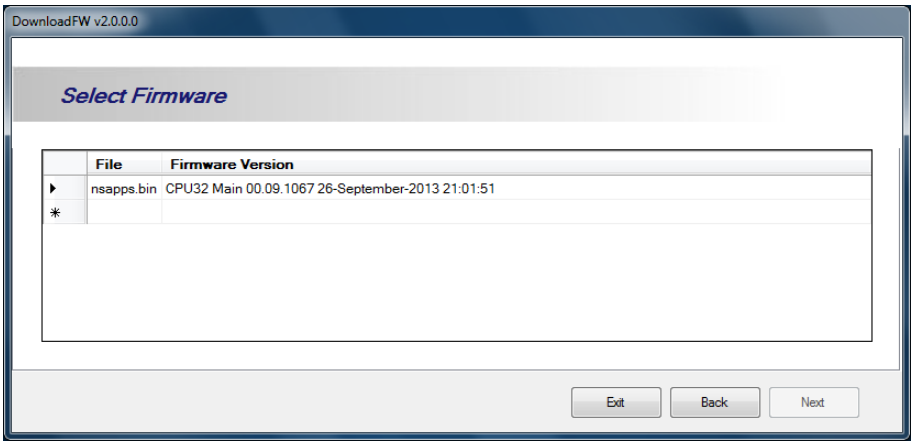
3. Double-click the **Download Firmware** shortcut file.

   Communications with the spectrometers installed on your system are established and the firmware download wizard is initiated.



4. Select the instrument you want to upgrade from the list, and then click **Next**.

   The Select Firmware screen is displayed.



5. Select the firmware file to use for upgrading the instrument, and then click **Next**.

   Normally the default file displayed, **nsapps.bin**, will be the latest version. If you want to use a different version, click the asterisk (*) and browse to the firmware file location on your PC.

6. Check the download details displayed on the Prepare for Download screen, and if they are correct, click **Next**.

   The firmware download is initiated.

7. After the firmware download is complete, check for any errors on the Complete screen, and then click **Exit** to close the wizard.

8. Switch off the spectrometer using the power switch at the rear of the instrument, wait 10 seconds and then switch the instrument back on.

   After the instrument has initialized, it will be capable of controlling the NIRA II accessory.

9. Proceed with the installation of the NIRA II accessory as described in *Installing the Accessory* on page 10.

NOTE: This firmware upgrade will give your spectrometer the capability of controlling the NIRA II accessory. However, it will not ensure transferability of calibrations between systems. This capability requires changes to the spectrometer which cannot be undertaken in the field and involves returning it to the PerkinElmer factory. If transferability of calibrations between systems is required, we recommend that you purchase an enhanced Frontier NIR or dual-range MIR/NIR spectrometer (L1280125 / L1280126).