

NEAR INFRARED REFLECTANCE MODULE

FOR USE WITH SPECTRUM TWO N FT-NIR



User's Guide

Release History

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Warnings and Safety Information

General Safety

The Near Infrared Reflectance Module (NIRM) is intended for use with a PerkinElmer Spectrum Two N 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 2014/35/EU.

Radiation Emitted by the Instrument NIR Source

The NIR source in the PerkinElmer Spectrum Two N FT-NIR spectrometer uses a tungsten 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

AVERTISSEMENT

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

La carte principale contient des périphériques statiques. Ne touchez pas la carte principale ou les composants électriques lors de la modification du filtre à air de la carte principale.

For further safety and warning information, refer to the appropriate User's Guide for your instrument. These are distributed, as .pdf files, on the **Spectrum Two Manuals CD** (L1050242).

Introduction

The NIRM Near Infrared Reflectance Module is used for collecting diffuse reflectance spectra of solids, granules, pastes, powders and turbid liquids. Clear liquids can also be analyzed using the liquids transmittance accessory (see page 37). The technique is non-destructive. It is particularly useful for undiluted powders and granular materials. You can make both qualitative and quantitative measurements.

The NIRM 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 20).
- Minimizing the amount of stray light entering the detector, and correcting for the effect of any stray light that is measured (see page 29).
- Introducing a reference correction to standardize the internal reference material of the NIRM against a chosen reference standard (see page 31).

The use of the optional Sample Spinner (see page 16) allows results of optimum precision to be obtained. The use of the optional Iris (see page 10) allows for proper alignment and secure positioning of sample vials on the sample window.



Figure 1 The Near Infrared Reflectance Module (NIRM) 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.

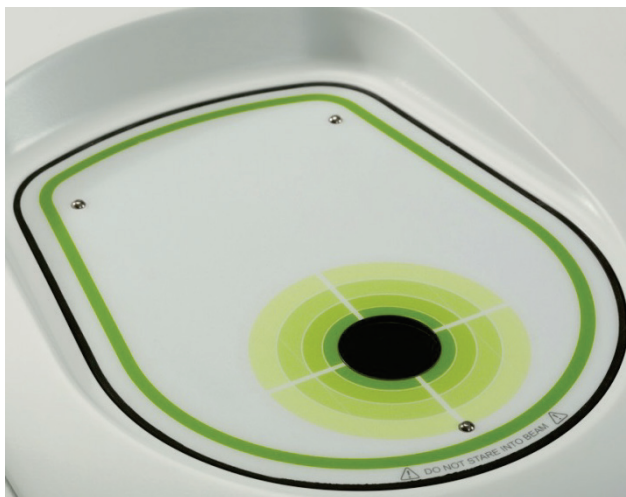


Figure 2 NIRM top plate

The infrared beam from the instrument enters the NIRM 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.

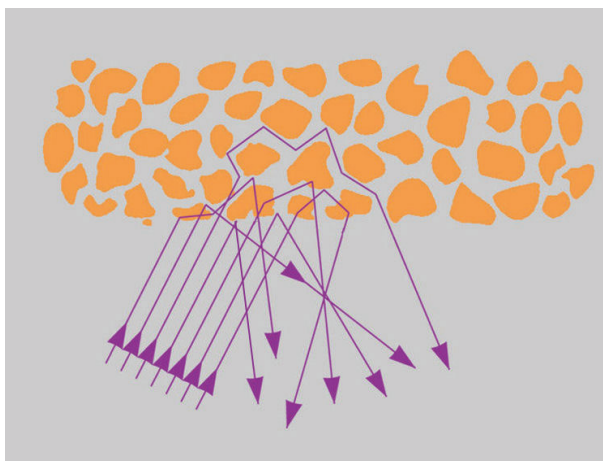


Figure 3 Schematic of diffuse reflectance

The NIRM 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 NIRM 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 31 for more information.

Sample Options

Because it is a non-destructive technique, near infrared diffuse reflectance is useful for a wide range of samples, from emulsions and powders to tablets. For many types of samples, 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 a 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) are available from PerkinElmer that can be either placed directly on the NIRM top plate or (in the case of the Petri dishes) used with the optional Sample Spinner. An optional Iris attachment is also available to securely hold and align vials on the sample window. See *Additional Parts* on page 27 for more information.



Figure 4 Sample containers available from PerkinElmer

Packing List

The following items are provided with the NIRM:

| Part Number | Description | Quantity |
|-------------|------------------------------|----------|
| L1100711 | NIRM Accessory Shipping Disk | 1 |
| L1281032 | NIRM Stray Light Cover | 1 |
| L1280452 | Sample Alignment Tool | 1 |
| L1285013 | Standard Sample | 1 |
| L1600813 | Power Splitter Cable | 1 |

NIRM Accessory Shipping Disk

This **Accessory Shipping Disk** (L1100711) contains an electronic copy of the test certificate for your NIRM 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: NIRM Correction Routines* on page 28 for further information).

NIRM Stray Light Cover

The **NIRM Stray Light Cover** (L1281032) helps protect the top plate and sample window of the NIRM 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 29 for further information.



Figure 5 NIRM Cover in position

Sample Alignment Tool and Sample Vial Holder

The **Sample Alignment Tool** (L1280452) is a device that helps you to reproducibly position vials, beakers and other containers on the NIRM sample window. It is magnetically held in position on the top plate (see Figure 6a). 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.

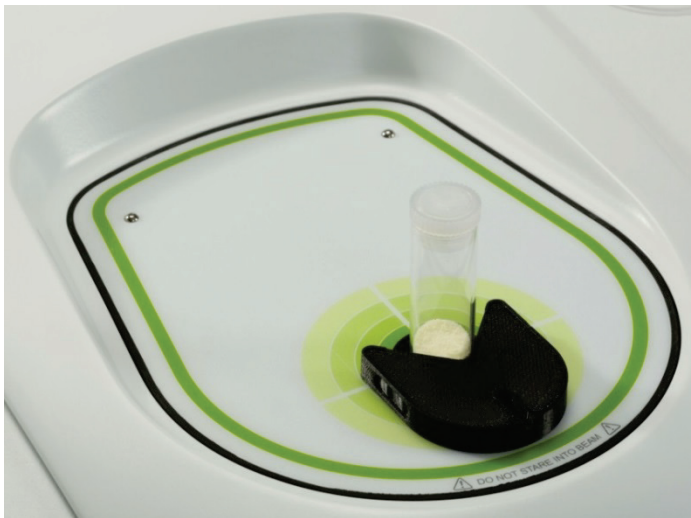


Figure 6a Sample Alignment Tool in use

The optional **Sample Vial Holder** (L1390122) allows for multiple sizes of vial types to be accurately and reproducibly held for reflectance measurements. The vial holder is auto aligned using magnetic fastening allowing for easy attachment and removal (see Figure 6b). Squeeze the levers to open and close the holder to center the vial.



Figure 6b Sample Vial Holder

Standard Sample

The **Standard Sample** (L1285013) 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 NIRM.

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



Figure 7 NIRM Standard Sample

Power Splitter Cable

The power supply used to power the Spectrum Two N FT-NIR is also used to power the NIRM accessory. A **Power Splitter Cable** (L1600813) is used to provide power to both the spectrometer and NIRM accessory simultaneously.



Figure 8 Power Splitter Cable

Installing the Module

The NIRM 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 NIRM:

1. Raise the sample cover to the vertical position and lift the cover upwards, clear of the spectrometer.
Store sample cover in a safe place for future use.
2. Pull the baseplate of the current sample accessory towards you, and slide the accessory out of the sample area (see Figure 9).
Store this accessory in a safe place for future use.

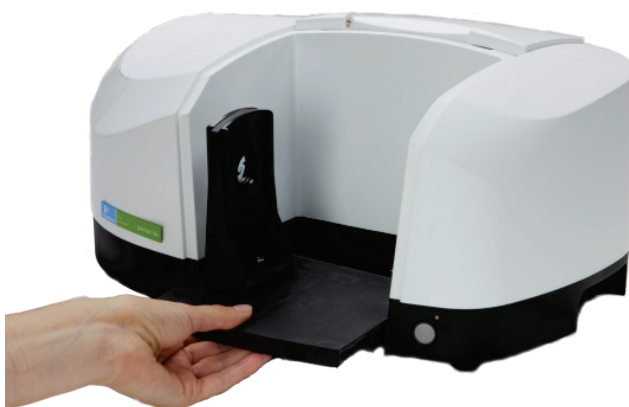


Figure 9 Removing the standard baseplate

3. Connect the long end of the Power Splitter Cable (L1600813) to the bottom of the NIRM.

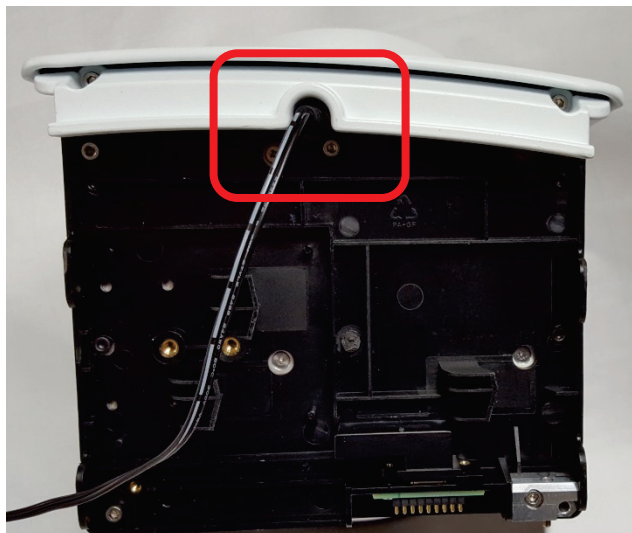


Figure 10 The power in plug connected to the NIRM

The power cable should then be routed out of the front of the module.

4. Slide the NIRM into the Spectrum Two N by resting the back of it on the ledge in the sample area and slide it into position. Push it firmly in to ensure that the multi-way

connector on the rear of the accessory engages properly with the spectrometer connector. There should not be any significant gaps between the NIRM and the Spectrum Two N FT-NIR.

When correctly installed, a seal around the inside of the NIRM cover protects the optics in the sample area from contamination if samples are spilt.

NOTE: Secure the NIRM within the Spectrum Two N sample compartment using the underside M3 screw supplied with the NIRM accessory.



Figure 11 Splitter Power Cable with the NIRM installed

5. Connect the splitter power cable to the rear of the Spectrum Two N then connect the power supply cable to the power splitter cable. Make sure the cable is safely routed to prevent accidental pulling.

NOTE: Only the power splitter cable (L1600813) provided with the NIRM should be used to supply power to the NIRM.

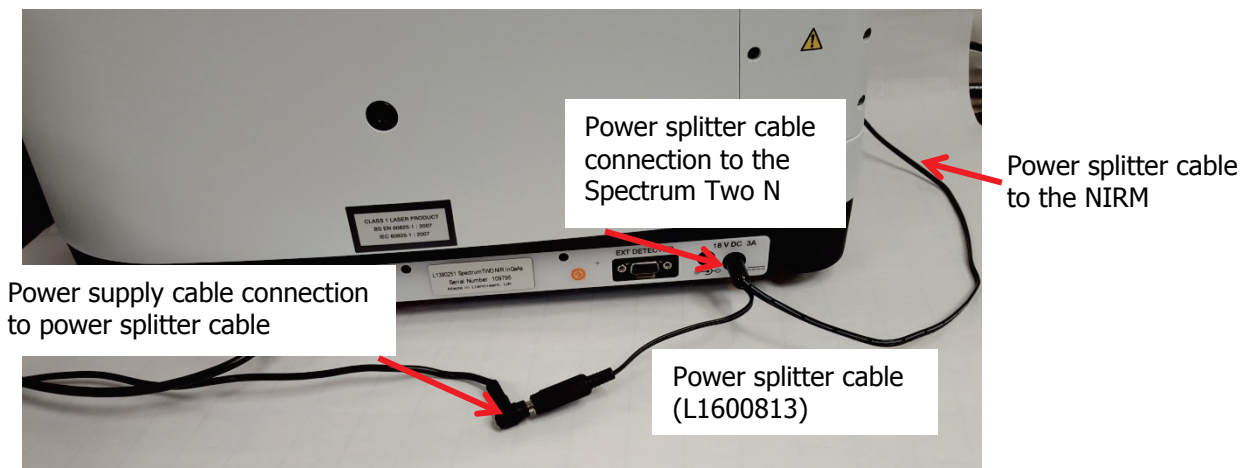


Figure 12 Splitter Power Cable connection to the power supply and Spectrum Two N




Figure 13 NIRM installed in a Spectrum Two N FT-NIR 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 NIRM will only be detected and operate correctly when used with Spectrum software version 10.6.0 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 NIRM* on page 39 for further details.

The Spectrum 10 software detects the presence of the accessory, and the NIRM icon  is added to the toolbar and NIRA II is displayed on the instrument setup tabs. The Setup Instrument Basic (see Figure 14) and Setup Instrument BeamPath (see Figure 15) tabs are updated to show that the NIRM is in position.

Setup Instrument Basic

Actions

Restore Defaults

Load and Save...

Settings

Abscissa Units: Wavenumber

Start (cm-1): 10000

Ordinate Units: A

End (cm-1): 4000

Scan Settings

Resolution (cm-1): 16

Scan Type: Interleaved

☐ Data Interval (cm-1): 2

Accumulations: 64 Scans

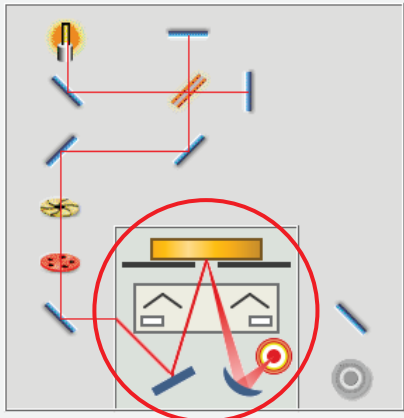
Accessory

NIRA II

| Item | Value |
|------------------------|------------------|
| Sampling | Large Petri dish |
| Details | |
| Spinner | Yes |
| Sampling Position | Automatic |
| Window Correction | Off |
| Stray Light Correction | Off |
| Reference Correction | Off |

Figure 14 Setup Instrument Basic tab with NIRM

Setup Instrument Auto-Name | Setup Instrument Data Collection | **Setup Instrument BeamPath** | Setup Instrument Advanced | Setup Instrument Basic



| Setting | Value |
|--------------------|----------------------------|
| Source | NIR (15000 - 2000) cm-1 |
| Beamsplitter | CaF2 (15000 - 1250) cm-1 |
| Detector | InGaAs (10000 - 4000) cm-1 |
| Window | CaF2 |
| Optimum Scan Range | (10000 - 4000) cm-1 |

| Setting | Value |
|--------------------------------------|-------------------------|
| J-Stop Image Size (mm) (1.58 - 8.94) | 8.94 |
| J-Stop Wavenumber (cm-1) | 16000.01 |
| Filter Wheel | None (15000 - 0 cm-1) ▼ |
| Desiccant change due in (days) | 169 |
| Instrument service due in (days) | 349 |
| Accessory | NIRA II |

Figure 15 Setup Instrument BeamPath tab with NIRM (circled)

NIRM Sample Spinner

The NIRM 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 NIRM Reflectance Accessory. It also optimizes the precision of the results obtained for apparently homogeneous samples.

Particularly suited for samples with particle sizes exceeding 1 mm, the NIRM 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 17) Without Spinner – With Spinner.

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

Two versions of the NIRM Sample Spinner are available for Petri dishes with approximate diameters of 100 mm (L1280417) and 60 mm (L1280410) (see Figure 16).



Figure 16 NIRM Sample Spinners for 100 mm (left) and 60 mm (right) Petri dishes

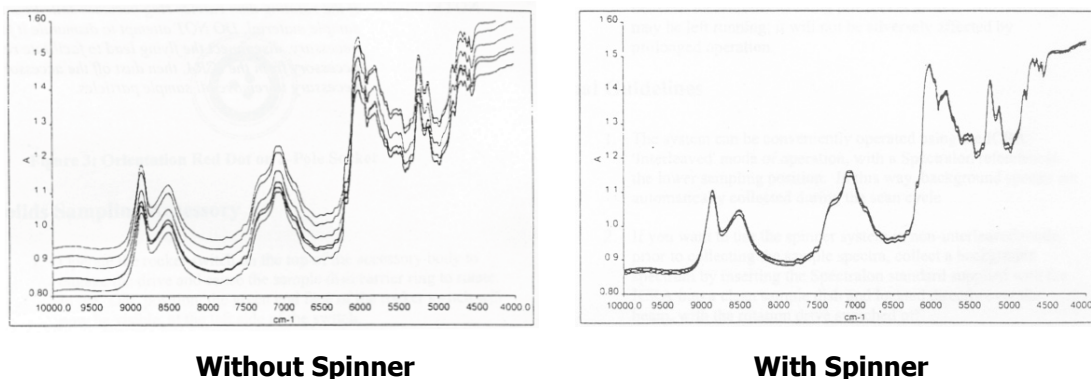


Figure 17 Replicate spectra comparison

Using the NIRM Sample Spinner

The NIRM 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 NIRM Sample Spinner locates on three studs in the top plate of the NIRM and receives electrical drive power wirelessly via a transmitter coil located in the top plate of the NIRM. When the Sample Spinner is properly positioned, Petri dishes placed onto the turntable will be located above the sample window (see Figure 18).

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.

ATTENTION

Pour éviter la possibilité de pertes dans la transmission de puissance sans fil et le fonctionnement intermittent du Sample Spinner, gardez le Spinner et la plaque supérieure exempte d'objets métalliques.

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 28) to remove any material clogging the drive mechanism.

ATTENTION

N'utilisez pas le Sample Spinner pendant plus de quelques minutes avec la LED clignotante. Vérifiez que le poids de l'échantillon est inférieur à 300 g. Si la LED clignotante persiste, nettoyez le Sample Spinner (voir Nettoyage du Spinner d'échantillonnage à la page 27) pour supprimer tout matériau bloquant le mécanisme d'entraînement.

The NIRM Sample Spinner is intended to be used by operators of the NIRM 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 NIRM 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 |
|--|----------|
| NIRM Sample Spinner Diameters of 100 mm (L1280417) and 60 mm (L1280410) | 1 |
| Spinner Cover Hex Key and Screws Kit (L1280427) | 1 |

Installing the NIRM Sample Spinner

The NIRM Sample Spinner should be fitted after the NIRM is installed in the spectrometer (see page 12).

1. Check that the top plate of the NIRM is clean and dry (see *Cleaning the NIRM* on page 22 for further information).
2. Holding the drive unit, locate the NIRM Sample Spinner in position on the top plate, using the thick green line as a guide (see Figure 18).

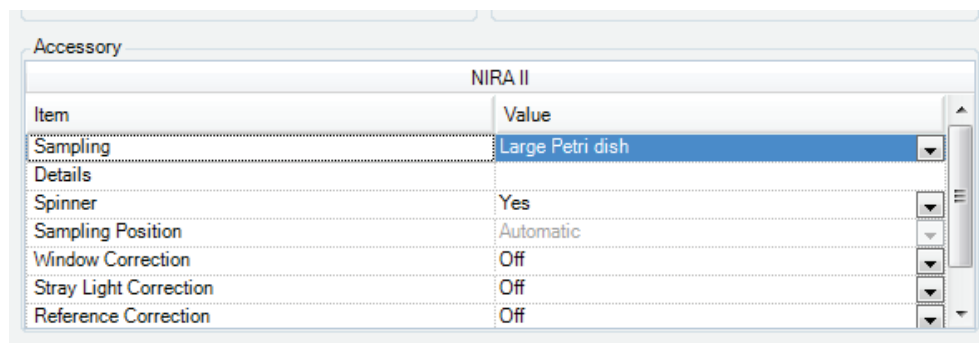
The Sample Spinner should "click" into place when correctly positioned. The Spinner is designed to sit in exactly the same position each time it is placed on the top plate.



Figure 18 NIRM Sample Spinner in position over sample window

Using the NIRM 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 Sample Spinner, fill the sampling dish on a surface away from the NIRM and check that the outside of the dish is clean before placing it onto 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.

| Start (cm-1) | End (cm-1) | Accumulations | Sample ID | Description | Mode |
|--------------|------------|---------------|-----------|-------------|------|
| 10000 | 4000 | 32 Scans | Sample 1 | | NIR |

Figure 19 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 NIRM 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.

NOTE: **Window Correction** must be **enabled** for interleaved mode to function correctly.

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.

| Setup Instrument Auto-Name | | Setup Instrument Data Collection | | Setup Instrument BeamPath | | Setup Instrument Advanced | | Setup Instrument Basic | |
|--|--|---|--|---|--|--|--|------------------------|--|
| Actions Restore Defaults Load and Save... | | Settings Abscissa Units: Wavenumber Start (cm-1): 10000 Ordinate Units: A End (cm-1): 4000 | | Scan Settings Resolution (cm-1): 16 Data Interval (cm-1): 2 Accumulations: 64 Scans | | Accessory NIRA II Item: Sampling Value: Large Petri dish Details: Spinner Value: Yes Sampling Position: Automatic Window Correction: Off Stray Light Correction: Off Reference Correction: Off | | | |

Figure 20 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, SingleBeam 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 NIRM.

- **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 NIRM 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 20) and then click .

If you switch on one or more of the NIRM correction routines (see page 28) 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 NIRM

The outer casing of the NIRM 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 (isopropyl alcohol, IPA).

CAUTION
ATTENTION

Do not use acetone to clean the NIRM.
Ne pas utiliser d'acétone pour nettoyer le NIRM.

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
ATTENTION

Do not submerge the NIRM or place it under running water when cleaning it.
Ne pas submerger le NIRM ou le placer sous l'eau courante lors du nettoyage.

When the accessory is not in use, the NIRM cover can be used to reduce the need for frequent cleaning of the top plate and window (see page 9).

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 29). 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 NIRM 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 NIRM 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 NIRM cover can be placed over the Sample Spinner when it is positioned on the NIRM to help keep the surfaces clean (see page 9).

When cleaning the Sample Spinner:

CAUTION

- *Do not attempt to remove the drive unit cover;*
- *Do not use acetone;*
- *Do not submerge the Spinner or place it under running water.*
- *Do not blow spilt powder off the base as it may become lodged in other areas of the Spinner.*

Lors du nettoyage du Sample Spinner:

ATTENTION

- N'essayez pas d'enlever le capot de l'unité d'entraînement;
 - Ne pas utiliser d'acétone;
 - Ne pas immerger le Spinner ou le placer sous l'eau courante.
- Ne pas faire tomber la poudre répandue hors de la base car elle peut être logée dans d'autres zones du Spinner.

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 NIRM (see Figure 21).

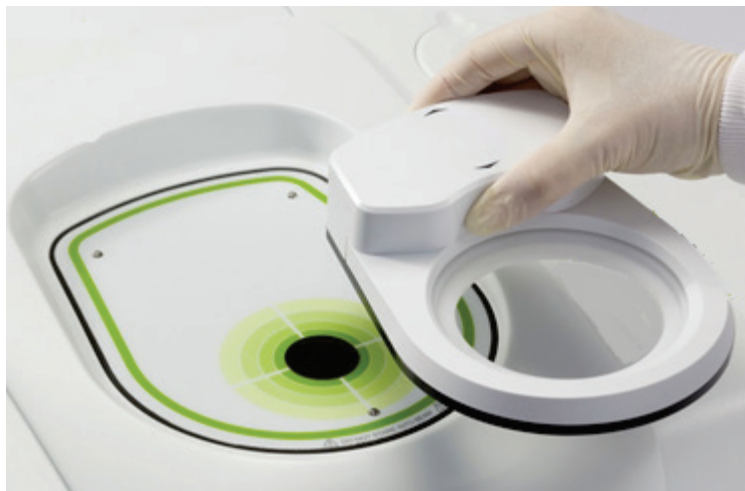


Figure 21 Removing the Sample Spinner from the NIRM top plate

2. Remove the two retaining screws underneath the front of the Sample Spinner using the 1.5 mm hex key provided (see Figure 22).

**CAUTION
ATTENTION**

*Do not remove any other screws from the underside of the Spinner.
Ne retirez pas d'autres vis de la partie inférieure du Spinner.*



Figure 22 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 23).



Figure 23 Removing the turntable cover

4. Lift out the toothed turntable and white plastic bearing (see Figure 24).



Figure 24 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 NIRM Sample Spinner.

Do not blow spilt powder off the base as it may become lodged in other areas of the Spinner.

ATTENTION

N'utilisez pas d'acétone pour nettoyer le filtre à échantillon NORM.

Ne pas faire tomber la poudre répandue hors de la base car elle peut être déposée dans d'autres zones du 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.

ATTENTION

Remettre le Sample Spinner une fois que tous les composants sont complètement secs.

Ne pas appliquer de graisse sur le roulement.

The cleaning procedure is exactly the same for the 60 mm Spinner version, which dismantles as shown in Figure 25.



Figure 25 The 60 mm Sample Spinner dismantled for cleaning

Additional Parts

The following optional parts are available for use with the NIRM. Part dimensions ranging from 90-100 mm are suitable for use with the 100 mm Sample Spinner, and part dimensions ranging from 55-60 mm are suitable for use with the 60 mm Sample Spinner (see NIRM *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) (2 dram; diameter 19mm; 51mm height) |
| 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: NIRM Correction Routines

Three correction routines are available specifically for the NIRM 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 NIRM before you can begin scanning samples. You cannot use a transmission AVI calibration.

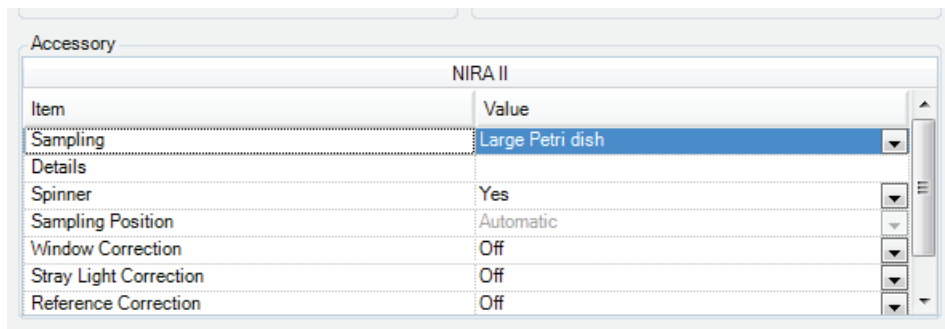
Window Correction

The fused silica window of the NIRM 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 absorption features may be visible in the final sample spectrum. The Window Correction compensates for this effect by subtracting a reference spectrum of the NIRM 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 NIRM 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 NIRM, a new correction spectrum will be supplied with the new window to be installed by your PerkinElmer Service Representative.

Stray Light Correction

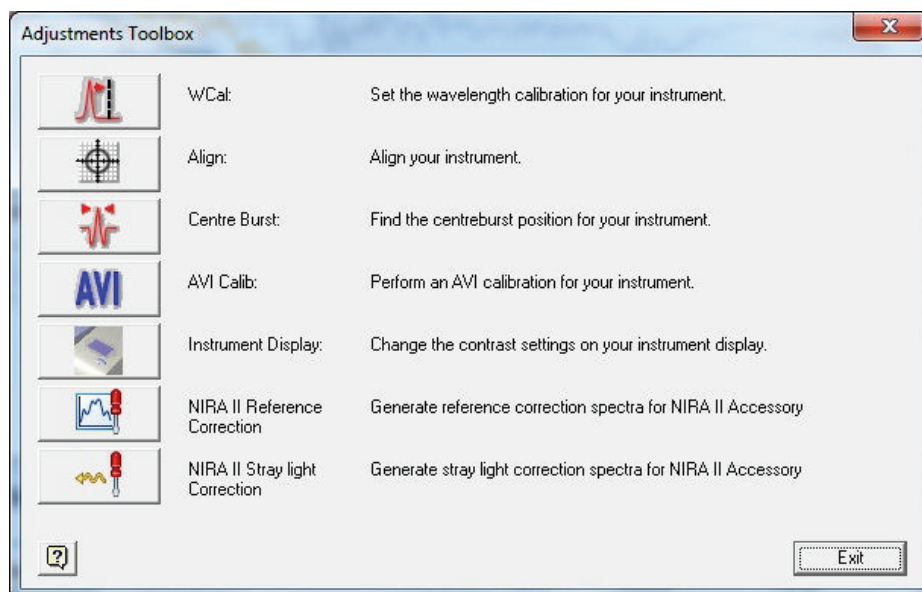
Stray light is light from the instrument that reaches the detector without having interacted with the sample, which can cause distortion of strong absorptions in your spectrum. Although the NIRM has been designed to minimize stray light, some light scattering will always occur at the sample window, and 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, as 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 will 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:

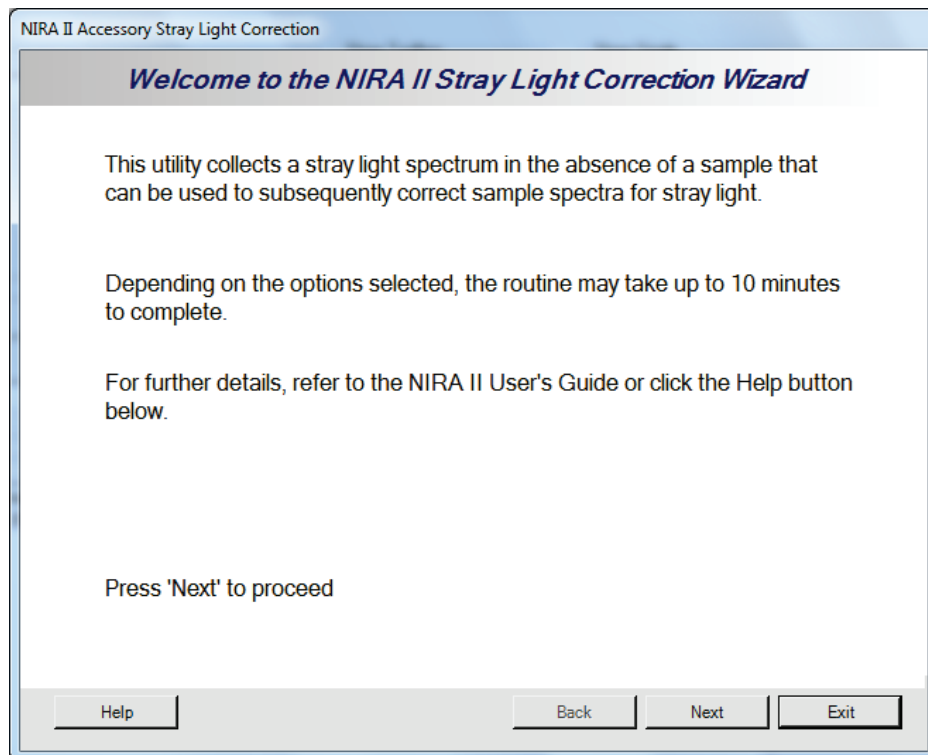
1. Select **Instrument** from the Setup menu.
The Setup Instrument tabs are displayed.
2. Select the **Setup Instrument Advanced** tab and then click **Toolbox**.
The Adjustments Toolbox dialog is displayed.





3. Click

The Stray Light Correction Wizard is displayed.



4. Follow the instructions on-screen.

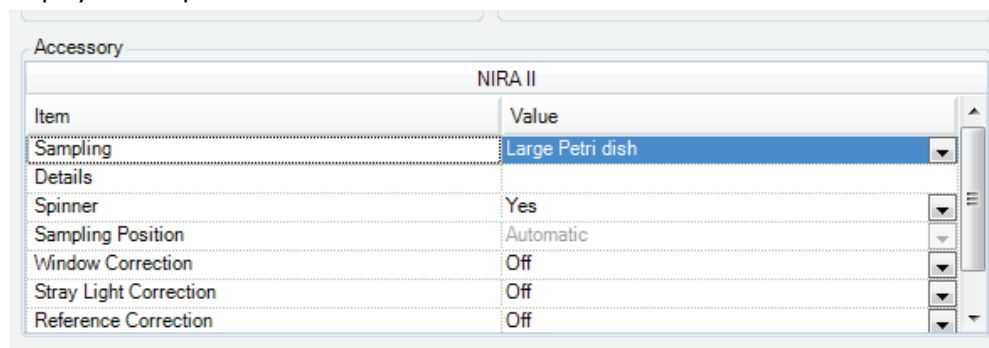
It is essential to ensure that the sample window is clean and dry, and that the NIRM cover is placed over the top plate of the NIRM when you are collecting the stray light correction spectra (see Figure 5).

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 NIRM (see Figure 3) are measured relative to a white reflector reference material. The NIRM 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 NIRM.

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 NIRM 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 NIRM systems, each with their own external references, can all be standardized against a common reference.

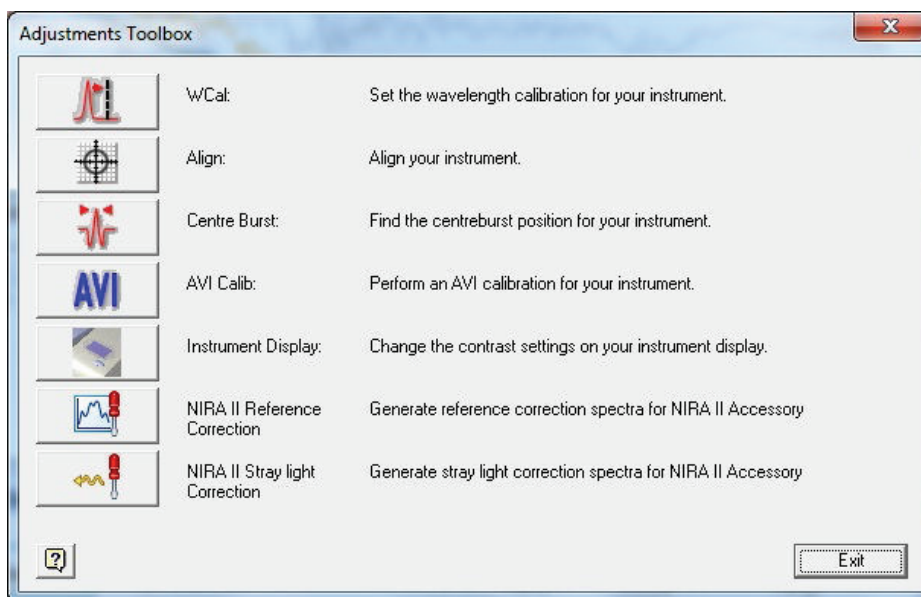
While the factory reference correction spectrum should be suitable in nearly all cases, if you encounter differences between systems when attempting to transfer calibration models, we recommend you obtain an external reference sample (such as PerkinElmer part numbers L1281920/1) and re-standardize the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

We recommend that the Window Correction spectrum supplied with your NIRM 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.

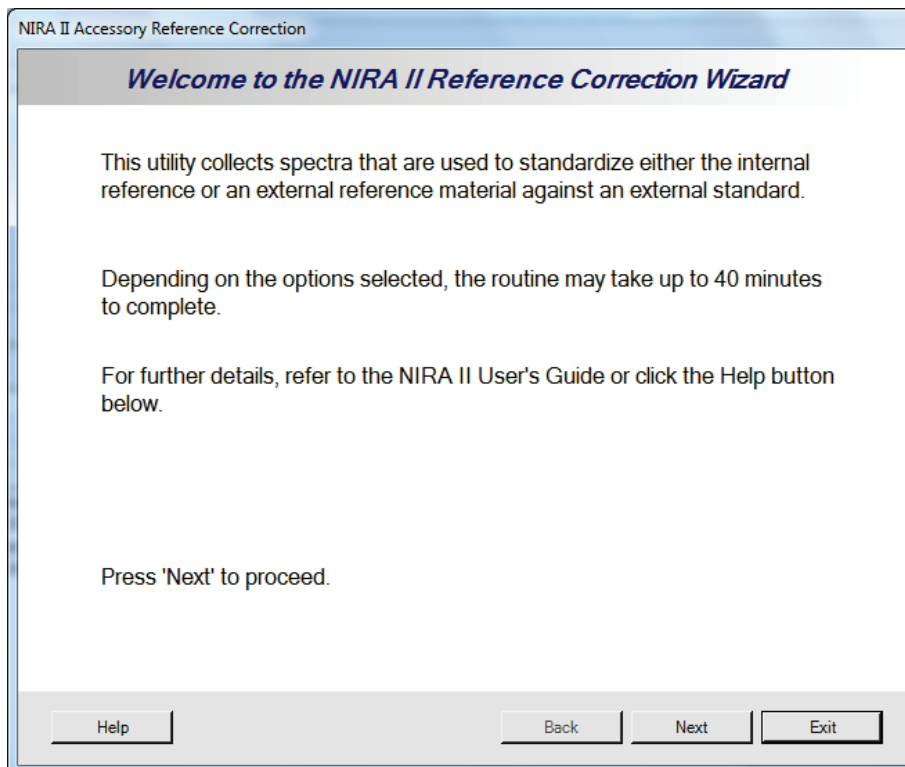
NOTE: To ensure that an accurate correction is applied, it is essential to ensure that any external reference materials that you use are not contaminated. Also check that the top plate of the NIRM is clean. Refer to *Cleaning the NIRM* on page 22.

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.



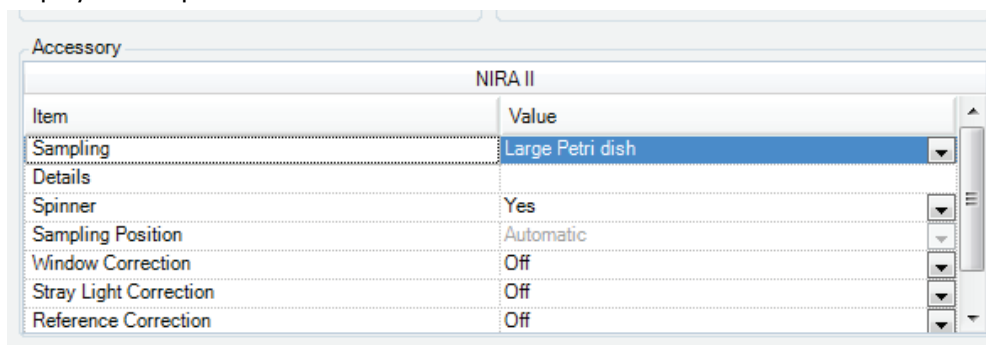
3. Click  .
The **Reference Correction Wizard** is displayed.



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

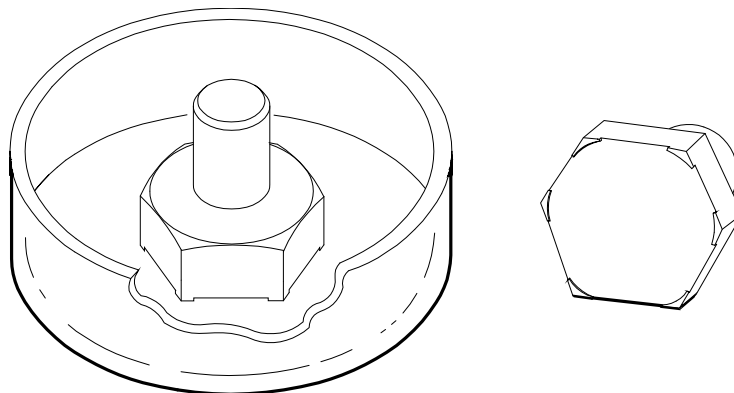


Figure 26 The liquids transflectance accessory

The **Liquids Transflectance Accessory** (L1185153) extends the range of sampling possible with a NIRM. 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 NIRM sampling position, significantly enhancing the versatility of the NIRM 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 NIRM, without any additional training, other than following the procedures and recommendations given in this User's Guide. Feel free to contact technical support for additional guidance on this accessory.

This accessory is intended for simple qualitative screening of liquids and gels. For more demanding quantitative measurements, we recommend the standard transmission configuration achieved by removing the NIRM and replacing with the basic sample slide.

A typical application of the Liquids Transflectance Accessory is shown on page 38.

List of Parts

As soon as you receive the Liquids Transflectance Accessory kit (L1185153), make sure that it includes, at minimum, 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 37). 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 NIRM.
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 27, place it in the center of an empty sample dish.

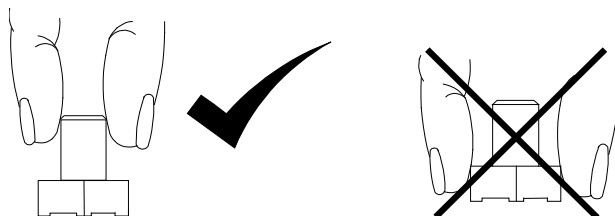


Figure 27 Handling the diffuse reflector


2. Position the sample dish and reflector centrally over the upper sample window, using the sample locator, as shown in Figure 28.




Figure 28 Liquids transreflectance 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 27), 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 28). 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 27), 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 28).
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} .

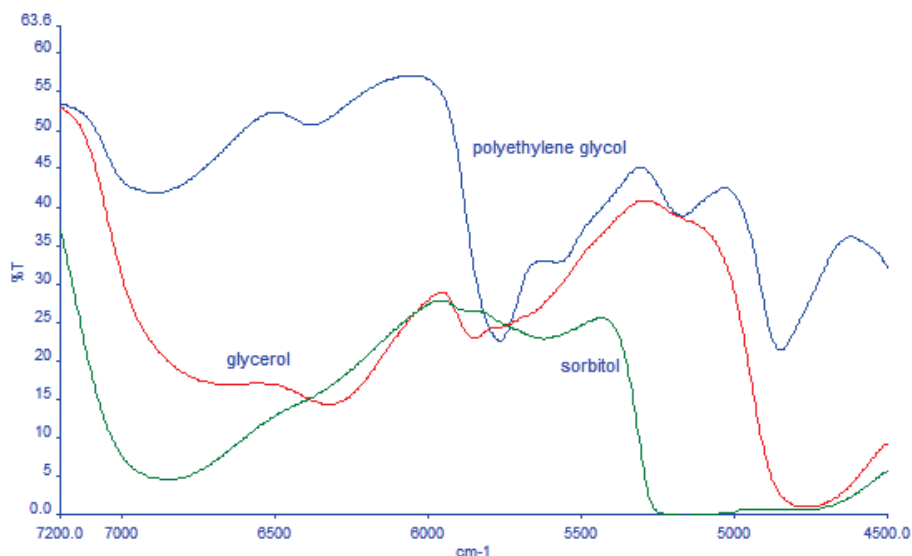


Figure 29 FT-NIR spectra of polyethylene glycol, glycerol and sorbitol

Appendix C: Upgrading a Spectrometer to Control the NIRM

If you have acquired a NIRM accessory to be used with a previously purchased Spectrum Two N spectrometer, you may need to upgrade the firmware in your spectrometer to provide the capability to control the accessory. This can be confirmed with technical support (email address)

Before upgrading the firmware, you must install Spectrum software version 10.6.0 (or above) on your PC, and use it to connect to your spectrometer.

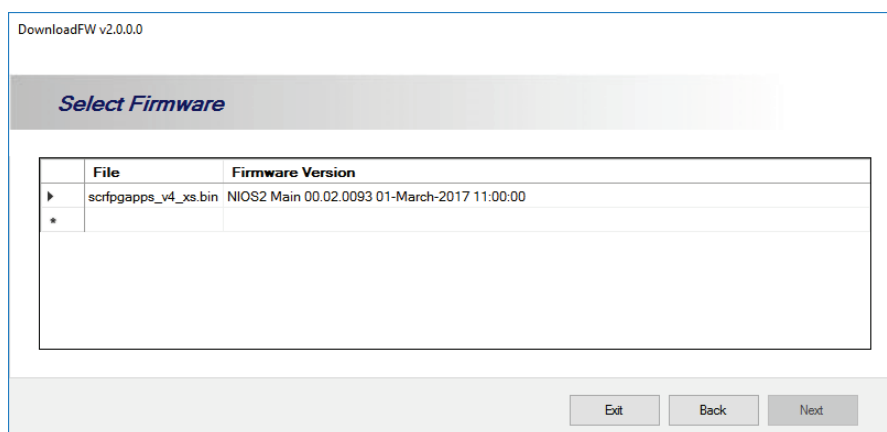
Follow the procedure below to upgrade the firmware:

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.

| | Name | Serial No | IP Address | Type | Current Firmware Version |
|---|---------------------------|-----------|----------------|--------------|---|
| ▶ | PerkinElmer FT-IR C105290 | C105290 | 167.116.185.71 | Spectrum Two | NIOS2 Main 00.02.0098 05-September-2017 13:1... |

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, **scrfpgapps_v4_xs.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 NIRM accessory.
9. Proceed with the installation of the NIRM accessory as described in *Installing the* on page 12.

