

# EXTERNAL NEAR INFRARED REFLECTANCE ACCESSORY



## User's Guide

## Release History

Part Number	Release	Publication Date
L1050088	B	February 2011

Any comments about the documentation for this product should be addressed to:

User Assistance  
PerkinElmer Ltd  
Chalfont Road  
Seer Green  
Beaconsfield  
Bucks HP9 2FX  
United Kingdom

Or emailed to: [info@perkinelmer.com](mailto:info@perkinelmer.com)

### Notices

The information contained in this document is subject to change without notice.

**Except as specifically set forth in its terms and conditions of sale, PerkinElmer makes no warranty of any kind with regard to this document, including, but not limited to, the implied warranties of merchantability and fitness for a particular purpose.**

### Copyright Information

This document contains proprietary information that is protected by copyright.

All rights are reserved. No part of this publication may be reproduced in any form whatsoever or translated into any language without the prior, written permission of PerkinElmer, Inc.

Copyright © 2011 PerkinElmer, Inc.

### Trademarks

Registered names, trademarks, etc. used in this document, even when not specifically marked as such, are protected by law.

PerkinElmer is a registered trademark of PerkinElmer, Inc.  
Frontier and Spectrum are trademarks of PerkinElmer, Inc.  
Spectralon is a trademark of Labsphere Ltd.

# Contents

---

<b>Warnings and Safety Information .....</b>	<b>5</b>
General Safety .....	6
Electrical Safety.....	7
Radiation Emitted by the Instrument NIR Source .....	8
<b>Introduction.....</b>	<b>9</b>
Overview .....	10
Diffuse Reflectance Measurements Using the NIRA.....	11
How it Works.....	11
Sample Options .....	12
<b>Installing the Accessory.....</b>	<b>13</b>
Electrical Requirements.....	14
Fitting the Accessory.....	15
Accessory Detection by Spectrum Software .....	15
The Sampling Kit.....	17
<b>Using the Accessory with Spectrum Software.....</b>	<b>21</b>
Selecting the Accessory.....	22
Contamination Ready Check .....	23
Performing a Scan.....	24
Manual Scanning .....	24
<b>Appendices.....</b>	<b>27</b>
Appendix 1: NIRA Liquids Transflectance Accessory .....	28
List of Parts.....	29
Scanning using the Liquids Transflectance Accessory .....	29
Interleaved Scanning using the Liquids Transflectance Accessory.....	31
Example – Discrimination of Sorbitol, Glycerol and Polyethylene Glycol using the NIRA Liquids Sampling Accessory .....	32
Appendix 2: External NIRA Sample Spinner .....	33
Typical Sample Spectra .....	33
Using the Sample Spinner.....	34
Installing the Sample Spinner.....	35
Using the NIRA Sample Spinner with Spectrum Software.....	37
Preparing your Sample .....	37
Cleaning.....	37
The Quartz Base Sample Dish .....	37
Using the Quartz Base Sample Dish .....	38
Cleaning the Quartz Base Sample Dish.....	38
Appendix 3: Removing and Refitting the External NIRA.....	39
Removing the External NIRA.....	39
Refitting the External NIRA .....	40



# **Warnings and Safety** **Information**

## ***General Safety***

---

The External Near Infrared Reflectance Accessory (External NIRA) is intended for use with a PerkinElmer Frontier FT-NIR, Frontier FT-IR/FT-NIR, Spectrum 100N FT-NIR, or a Spectrum 400 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.

## ***Electrical Safety***

---

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

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



**WARNING**

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

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

## ***Radiation Emitted by the Instrument NIR Source***

---

The NIR source used in PerkinElmer Frontier FT-NIR, Frontier FT-IR/FT-NIR, Spectrum 100N FT-NIR, and Spectrum 400 FT-IR/FT-NIR spectrometers is a quartz halogen bulb that emits ultraviolet, visible and infrared radiation. The majority of this energy is in the infrared region. Do not stare into the beam produced by this bulb.

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



**WARNING**

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

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**

## Overview

---

The External Near Infrared Reflectance Accessory (External NIRA) is used for gathering diffuse reflectance spectra. It is an external accessory for a Frontier FT-NIR, Frontier FT-IR/FT-NIR, Spectrum 100N FT-NIR or a Spectrum 400 FT-IR/FT-NIR spectrometer.



**Figure 1 Frontier FT-NIR Spectrometer with External Near Infrared Reflectance Accessory (External NIRA) fitted**

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

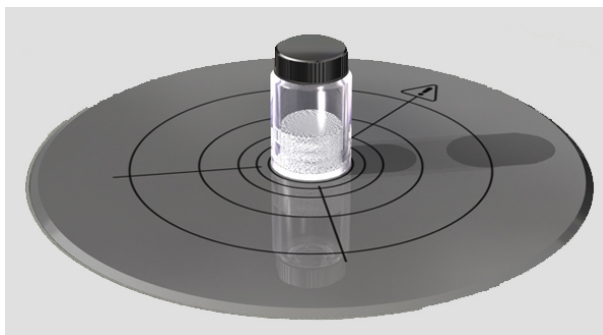
## ***Diffuse Reflectance Measurements Using the NIRA***

---

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

### ***How it Works***

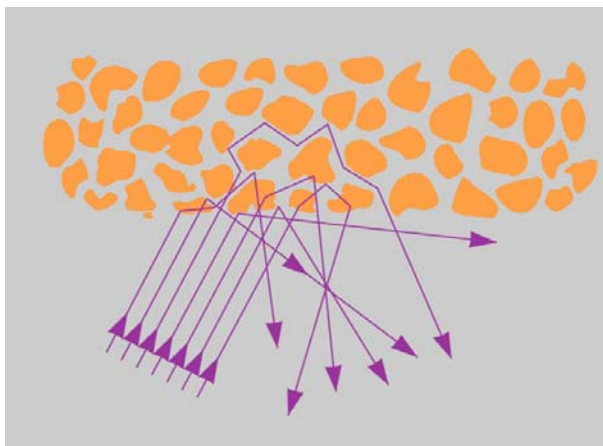
The sample is placed on a sapphire window located on the top of the accessory. The circles and lines on the plate help you center the sample over the window.



**Figure 2** Vial placed on the NIRA top plate

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

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



**Figure 3** Diffuse reflectance

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

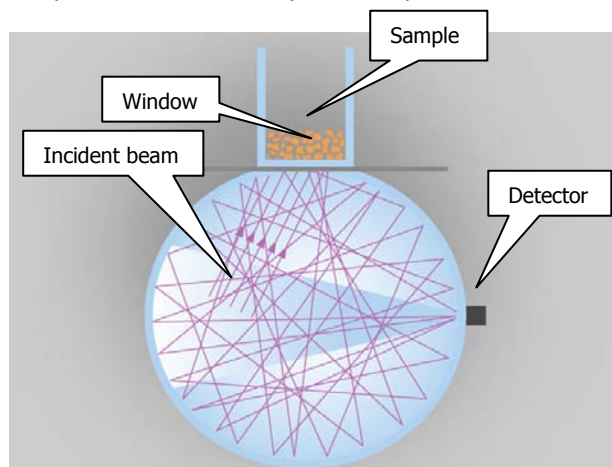


Figure 4 Diffuse reflectance within a NIRA

## ***Sample Options***

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

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

A loose powder cup is provided with the NIRA Sampling Kit (see page 17) that can be used as a container for loose powders. You are also supplied with a tablet holder with three different sized inserts.

## **Installing the Accessory**

## ***Electrical Requirements***

---

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

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

## ***Fitting the Accessory***

---

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

For Frontier IR Systems, these components form part of the External Beam Path Kit which provides support for a wide range of external beam accessories. See the User's Guide supplied with your instrument for details.

The accessory should not be removed unless:

- You want to use the external beam port on the right of the instrument with a different external accessory.  
See *Appendix 3: Removing and Refitting the External NIRA* on page 39.
- The spectrometer is moved to a new location.  
For information about moving the spectrometer, refer to the appropriate User's Guide for your instrument. These are distributed, as .pdf files, on the *IR & Raman Manuals CD* (L1050002).



**WARNING**

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

**CAUTION**

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

## ***Accessory Detection by Spectrum Software***

A new External NIRA is installed by a PerkinElmer Service Engineer who will, if necessary, install the appropriate software on a PC that is connected to the instrument.

**NOTE:** To use your accessory with Spectrum software, you must first install an instrument. See the *Spectrum Administrator's Guide* (L1050095) on the *IR & Raman Manuals CD* (L1050002) for details of the Instrument Install Wizard.


Before using the External NIRA with Spectrum software:

1. If you have removed and refitted the External NIRA (see *Appendix 3: Removing and Refitting the External NIRA* on page 39), make sure that the accessory data cable is fitted correctly.

- Switch the accessory power on, wait for a few seconds, and then switch the instrument on.

OR

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

The Spectrum software detects the presence of the accessory, and the NIRA icon  is added to the toolbar. The Setup Instrument Basic (Figure 5) and Setup Instrument BeamPath (Figure 6) tabs are updated to show that the NIRA is in position.

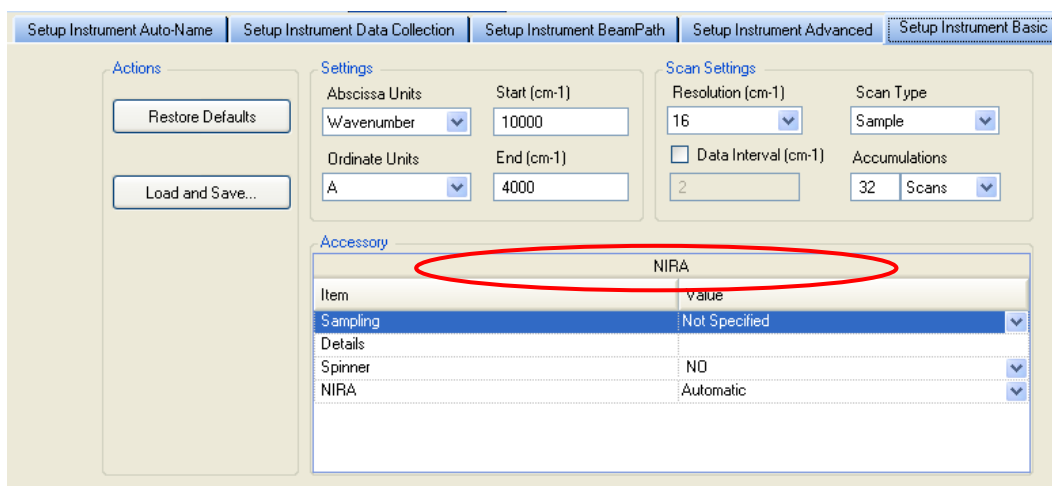


Figure 5 Setup Instrument Basic tab with NIRA (circled)

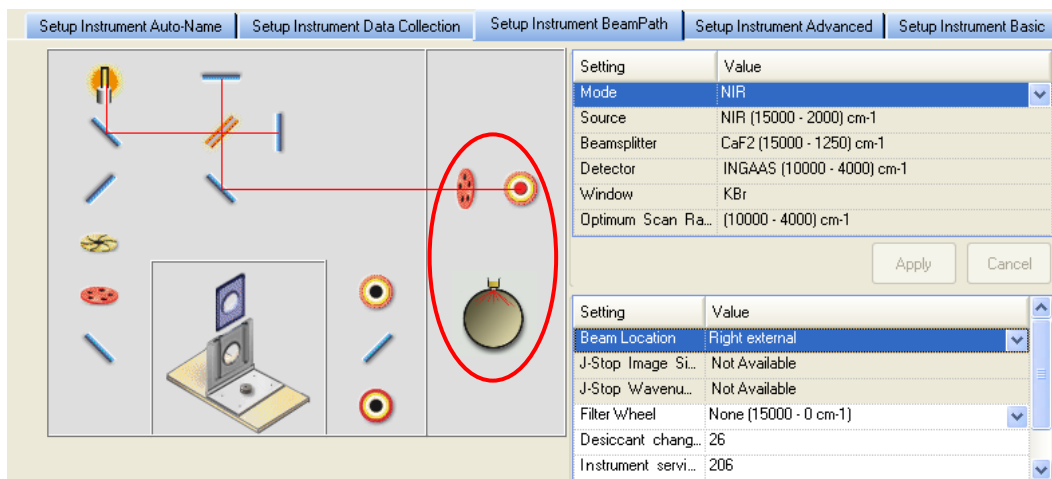


Figure 6 Setup Instrument BeamPath tab with the beam path directed to the External NIRA (circled)



## ***The Sampling Kit***

---

The following items are provided with the External NIRA:

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

### ***Loose Powder Sample Holder***

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

**NOTE:** When analyzing powders ensure that you use a depth of at least 1 cm, and that your sample is positioned directly over the window in the center of the NIRA top plate.

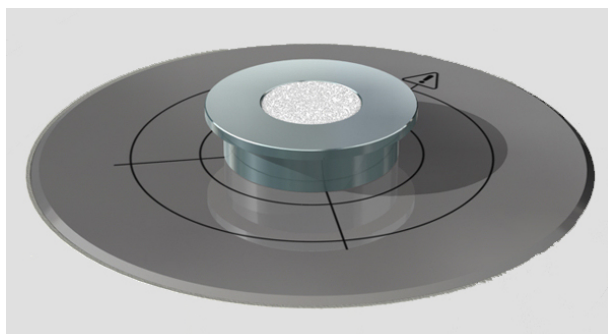


Figure 7 Loose powder sample holder

### ***Powder Sampling Plate***

This is a metal plate used when filling sample cups.

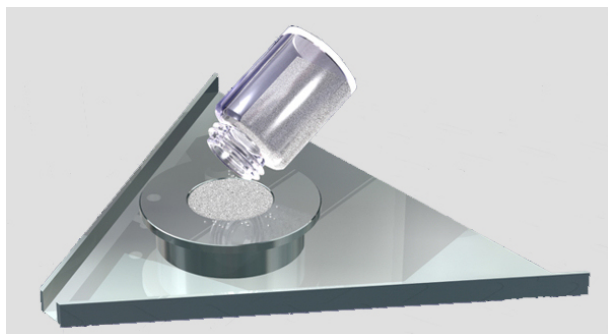


Figure 8 Powder sampling plate

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

### ***Sample Alignment Tool***

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

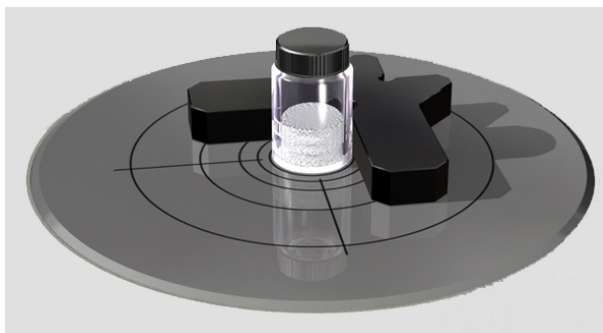


Figure 9 Sample alignment tool

### ***Cleaning Brush***

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

### ***Spatula***

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

### ***Tablet Holders***

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

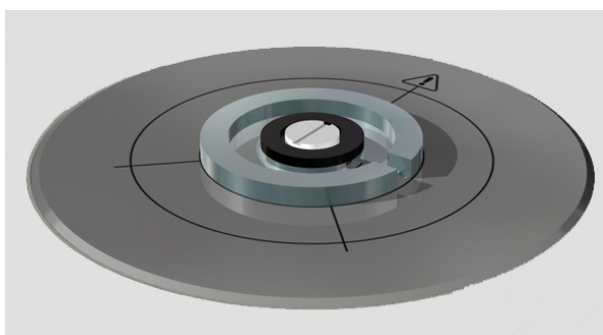


Figure 10 Tablet holder

**NOTE:** The correct size of holder should be chosen; such that the sample tablet completely covers the aperture.

### ***Spectralon Reference***

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



**Using the Accessory with**  
**Spectrum Software**

## Selecting the Accessory

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

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

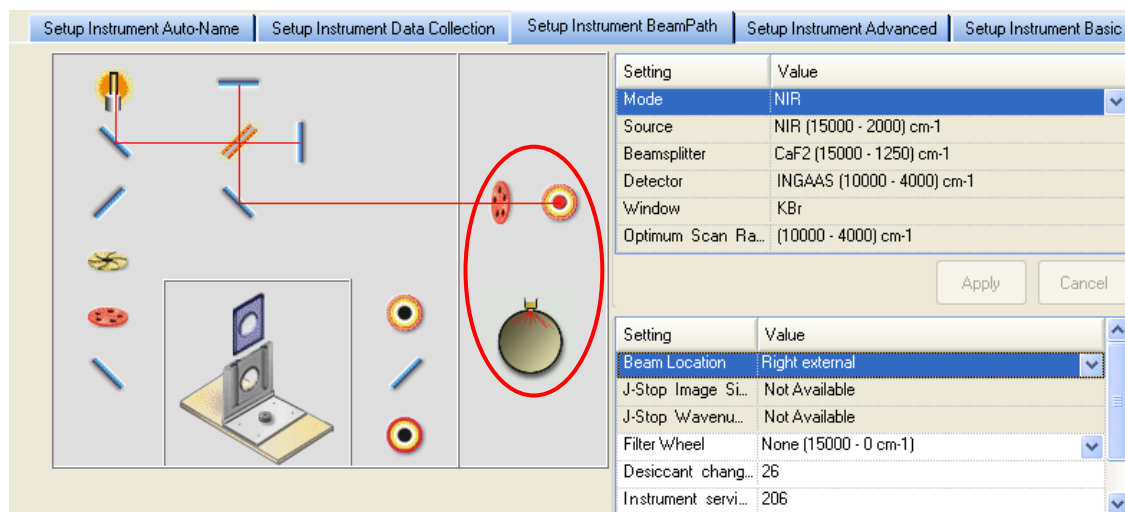


Figure 11 Setup Instrument BeamPath tab with the beam path directed to the External NIRA (circled)

## ***Contamination Ready Check***

---

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

To perform a Ready Check:

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

## Performing a Scan


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 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	Administrator 12	Sample 0	NIR

Figure 12 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 Help file for more information.

2. Click  on the Measurement toolbar to collect a spectrum.  
If Spectrum detects that a Background spectrum is required, the Scan icon will display a

flag at the bottom right-hand corner . Click on the icon and a background scan will be collected before the sample spectrum.

If at any time you wish to collect a background scan but do not wish to save it, click



If you wish to collect a background and save the spectrum, select **Background** as the

**Scan Type** on the Setup Instrument Basic tab and then click .

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

## Manual Scanning

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



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

- Actions:** 'Restore Defaults' and 'Load and Save...' buttons.
- Settings:**
  - Abscissa units: Wavenumber
  - Start (cm-1): 7800
  - Ordinate Units: AbsY
  - End (cm-1): 4000
- Scan Settings:**
  - Resolution (cm-1): 2
  - Scan Type: Interleaved
  - Data Interval (cm-1): 0.5
  - Accumulations: 30 Scans
- Accessory:**

NIRA	
Item	Value
Sampling	Not Specified
Details	
Spinner	NO
NIRA	Automatic

**Figure 13 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**. In this mode, background scans are automatically collected from inside the NIRA sphere as part of the sample scan: you do not need to take a separate background.

4. Select the **NIRA** setting from the drop-down list.

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

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

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

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

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

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

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

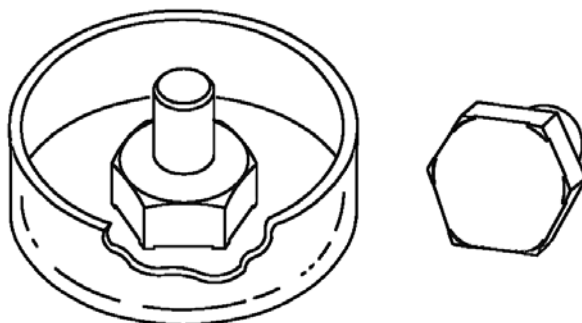
**NOTE:** The Spectrum Help file describes how to format, process and report your results. To open the Help file, select **Contents** from the Help menu.



## **Appendices**

## **Appendix 1: NIRA Liquids Transflectance Accessory**

---



**Figure 14 The NIRA liquids transfectance accessory**

The Liquids Transflectance Accessory extends the range of sampling possible with a NIRA. 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 sampling position, significantly enhancing the versatility of the NIRA 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, without any additional training, other than following the procedures and recommendations given in this User's Guide.

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

A typical application of the Liquids Transflectance Accessory is shown on page 32.

## 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
NIR Liquid Sample Procedure disc (For use with Spectrum 6 software only)	1	L1100609

## Scanning using the Liquids Transflectance Accessory

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.
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.

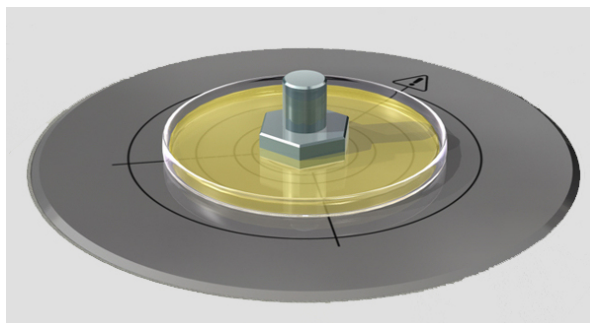
### Stage 2 – Obtaining a Background scan

1. Handling the diffuse reflector only by its upper section, as shown in Figure 15, place it in the center of an empty sample dish.



Figure 15 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 16.



**Figure 16 Liquids transreflectance accessory in position**

To ensure consistency between results, always take care to position the reflector in the centre of the dish, and the dish centrally over the sample window.

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, as shown in Figure 15, place it into the center of the sample dish.
3. Apply sufficient downward pressure to ensure that the integral spacers of the reflector are in contact with the base of the dish, and that there are no air bubbles trapped between the reflector and the sample.
4. Position the sample dish and reflector centrally over the sample window, as shown in Figure 16.

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 – Using Spectrum Arithmetic Functions to Apply 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.

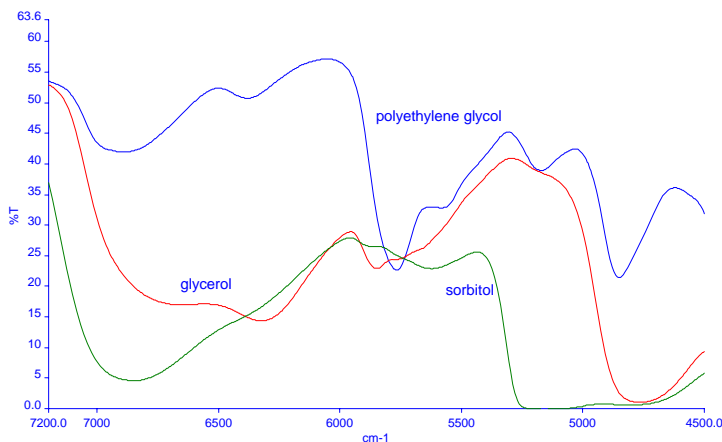
#### ***Interleaved Scanning using the Liquids Transflectance Accessory***

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, as shown in Figure 15, place it into the centre of the sample dish.
3. Apply sufficient downward pressure to ensure that the integral spacers of the reflector are in contact with the base of the dish, and that there are no air bubbles trapped between the reflector and the sample.
4. Position the sample dish and reflector centrally over the sample window, as shown in Figure 16.  
To ensure consistency between results, always take care to position the reflector in the centre of the dish and the dish centrally over the sample window.
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 NIRA Liquids Sampling Accessory***

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



**Figure 17 FT-NIR spectra of polyethylene glycol, glycerol and sorbitol**



## ***Appendix 2: External NIRA Sample Spinner***

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

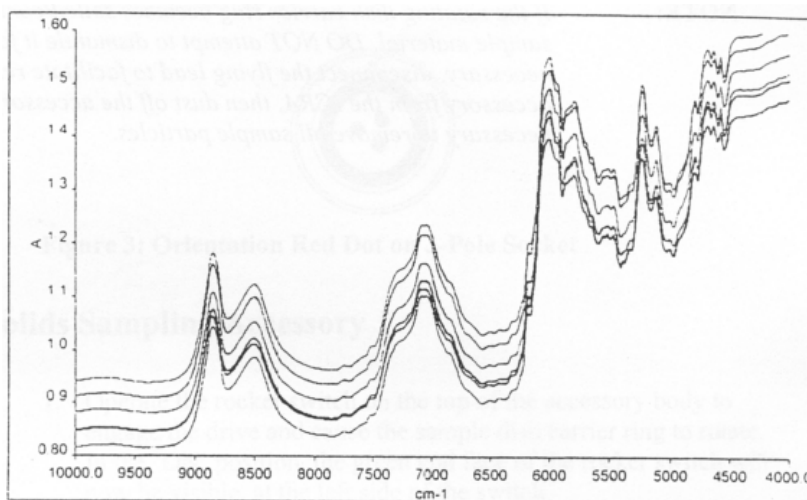
Particularly suited for samples with particle sizes exceeding 1 mm, the sample spinner obviates the need for collecting spectra of representative batches and then calculating a mean spectrum from the series of results obtained.

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

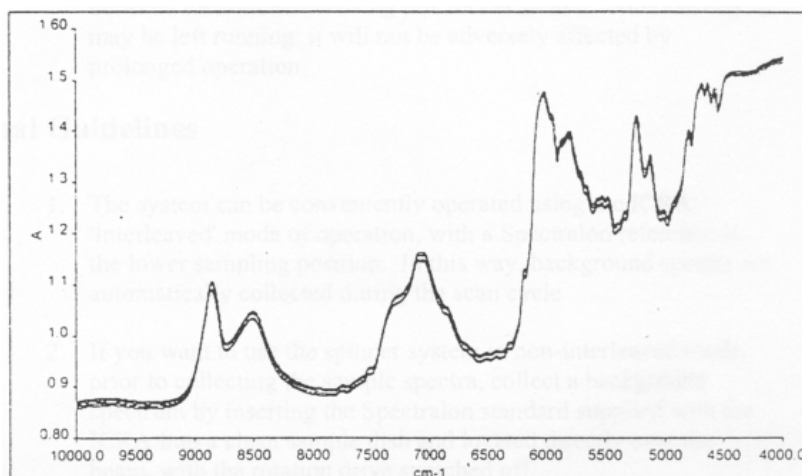
The sample spinner also offers the benefit of non-destructive sampling, as no grinding is required.

**NOTE:** A comparison between two methods of collecting spectra is shown on page 33.

### ***Typical Sample Spectra***



**Figure 18** Representative spectra collected on a NIRA (traditionally used to calculate a mean)



**Figure 19** Replicate spectra (collected using the sample spinner)

## ***Using the Sample Spinner***

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

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

The sample spinner locates on top of the External NIRA and receives electrical drive power via a connector that is plugged into a connector on the accessory (See *Installing the Sample Spinner* on page 35).

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

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

### ***List of Parts***

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

Description	Quantity
External NIRA Sample Spinner	1
Sampling Dish (90 mm diameter)	5
Quartz Base Sample Dish (optional)	1

**NOTE:** For more information about the Quartz Base Sample Dish, see page 37.

## Installing the Sample Spinner

### CAUTION

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

1. Remove the interface slot cover on the External NIRA (Figure 20).  
Store it in a safe place for future re-use.



**Figure 20 Interface slot cover on the External NIRA**

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

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



**Figure 21** Locating feet of sample spinner in the interface slot

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



**Figure 22** Spinner positioned over the sampling window

The spinner is now correctly positioned over the sampling window.

## ***Using the NIRA Sample Spinner with Spectrum Software***

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

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

## ***Preparing your Sample***

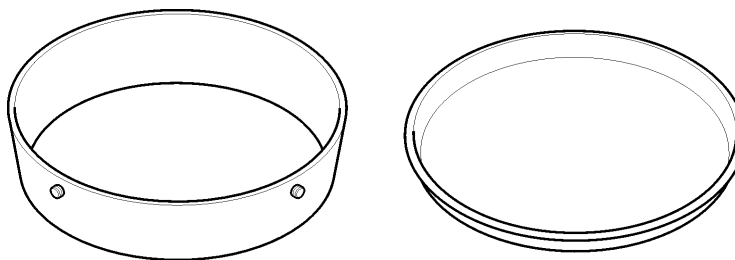
It is good practice to use a consistent quantity of sample. Ensure that the entire sampling dish is filled to a depth of at least 5–10 mm. Sampling dishes may be used with or without a lid as required.

## ***Cleaning***

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

## ***The Quartz Base Sample Dish***

The optional Quartz Base Sample Dish L1185305 (Figure 23), enables quantitative and qualitative measurements of large granular materials.



**Figure 23** The quartz base sample dish

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

### ***Care of the Quartz Base Sample Dish***

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

### ***Using the Quartz Base Sample Dish***

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

### ***Cleaning the Quartz Base Sample Dish***

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

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

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

## Appendix 3: Removing and Refitting the External NIRA

---

In the first instance, the External NIRA is fitted by a PerkinElmer Service Engineer.



**WARNING**

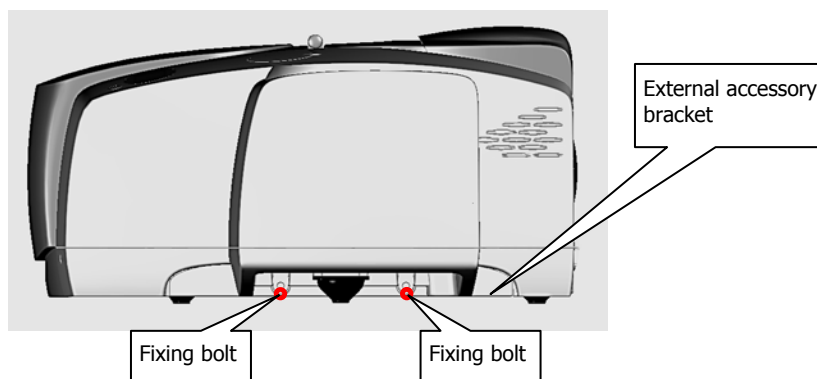
*The spectrometer is a heavy precision instrument, so two people are required for safe handling.*

**CAUTION**

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

### Removing the External NIRA

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



**Figure 24** Location of external accessory fixing bolts

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

3. Use the 6 mm hex key supplied to carefully undo the two 100 mm M8 socket-headed bolts that attach the accessory to the external accessory bracket. These bolts are accessible from under the baseplate of the accessory, looking from the right of accessory (Figure 24). Each bolt is fitted with a spacer (between the accessory baseplate and the accessory bracket), a plain washer and a spring washer.

**CAUTION**

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

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

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

OR

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

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

## ***Refitting the External NIRA***

To fit the External NIRA:

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



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



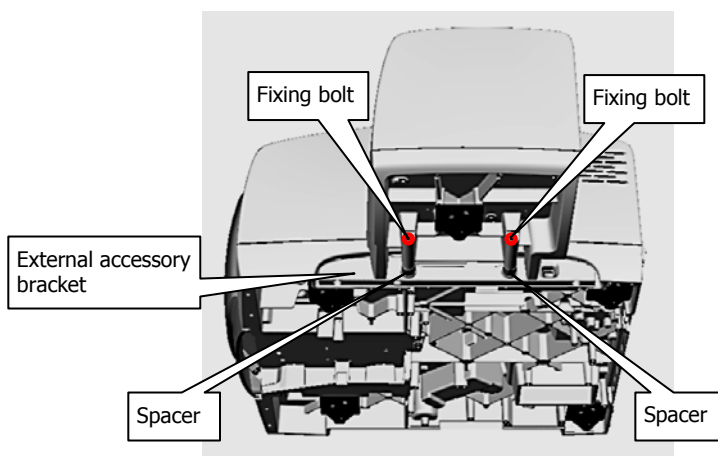
Figure 25 External accessory bracket

**CAUTION**

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

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

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



**WARNING**

*DO NOT lift the spectrometer when fitting the External NIRA.*

*The underside of the spectrometer is shown for illustration only.*

Figure 26 Location of external accessory spacers

2. Add a spacer to each bolt and then carefully align the bolts with the threaded holes in the external accessory bracket.
3. Using the 6 mm hex key supplied, turn each bolt until it engages in the external accessory bracket, make sure the bolts are perpendicular to the bracket, and then carefully and evenly tighten the bolts.

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

Do not over-tighten the bolts.

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

OR

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

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