FIBER OPTIC INTERFACE SAMPLING ACCESSORY

HUMAN HEALTH | ENVIRONMENTAL HEALTH

User's Guide

PerkinElmer®

Release History

Part Number	Release	Publication Date
L1050097	B	November 2010

User Assistance
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Notices

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Conventions Used in this Manual

Normal text is used to provide information and instructions.

Bold text refers to text that is displayed on the screen.

UPPERCASE text, for example ENTER or ALT, refers to keys on the PC keyboard. '**+**' is used to show that you have to press two keys at the same time, for example, ALT+F.

All eight digit numbers are PerkinElmer part numbers unless stated otherwise.

The term 'instrument' refers to a Frontier FT-NIR, Frontier FT-IR/FT-NIR, Spectrum 100N FT-NIR, or Spectrum 400 FT-IR/FT-NIR spectrometer, and any sampling accessory fitted.

Notes, Cautions and Warnings

Three terms, in the following standard formats, are also used to highlight special circumstances and warnings.

NOTE: A note indicates additional, significant information that is provided with some procedures.

CAUTION	*We use the term CAUTION to inform you about situations that could result in **serious damage to the instrument** or other equipment. Details about these circumstances are in a box like this one.*

⚠️ **WARNING**	*We use the term WARNING to inform you about situations that could result in personal injury to yourself or other persons. Details about these circumstances are in a box like this one.*

Radiation Emitted by an Instrument NIR Source

The NIR source used in PerkinElmer Frontier FT-IR/NIR, Frontier 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.

⚠️
WARNING

> *Do not look directly into the beam emitted from the sampling aperture. 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* (part number L1050002).

Introduction

The Fiber Optic Interface Sampling Accessory is designed for attaching a variety of NIR solids or liquids remote sampling probes. For best results, PerkinElmer recommends separate probes for solids or liquids sampling.

The interface fits into the sampling area of the spectrometer and is automatically recognized by Spectrum software.

For solids sampling, the system (part number L1240063) uses a fiber bundle to direct the light to the sample and collect the reflected light from the sample. For liquids sampling, the system (part number L1240064) uses a single fiber to send light to and from the probe.

The system can be coupled with a range of other probes via SMA connectors.

The system includes a high-performance dedicated detector for use in near-infrared analysis.

How to Install the Accessory

The Fiber Optic Interface Sampling Accessory can be simply installed into the sample compartment of the spectrometer.

First, remove any other sampling accessory fitted:

1. If the sample compartment cover is fitted, remove it by opening the cover, pressing the clip and then pulling the cover vertically.

2. Store it in a safe place for future re-use.

Figure 1 Removing the Sample Compartment Cover

3. Remove the current sampling accessory by pulling the release handle (under the baseplate of the accessory) towards you and then sliding the accessory towards you.

4. Store it in a safe place for future re-use.

Figure 2 Removing the Sampling Accessory

5. Install the Fiber Optic Interface Sampling Accessory by sliding it onto the ledge and pushing it into the sample compartment until it engages with the connector.

Fitting the probe:

1. Taking care not to damage the faces of the fibers, screw the fiber optic cables into the connectors (labeled A in Figure 3).

NOTE: The Fiber Optic Interface Sampling Accessory is supplied with the gain set for either a solids or liquids probe. If you wish to change the operation of the interface from solids to liquids or vice-versa, we recommend that you reset the electric gain. For further information see the *Appendix* on page 15.

2. Pass the fiber optic cables through the cut-out slot in the front of the sampling accessory baseplate.

NOTE: Take care not to stress or compress the quartz fibers; they are relatively fragile and easily fractured, degrading performance.

3. Place the front clamp over the two holes (labeled B) and secure in place using the two screws provided (labeled C).

4. Place the rear clamp on the other side of the fiber optic cables and secure to the front clamp using the screws provided.

NOTE: The screws must be screwed into place from the holes in the back of the rear baseplate (labeled D) into the front clamp. If the screws are fixed from the front to the rear clamp the sample compartment lid will not close.
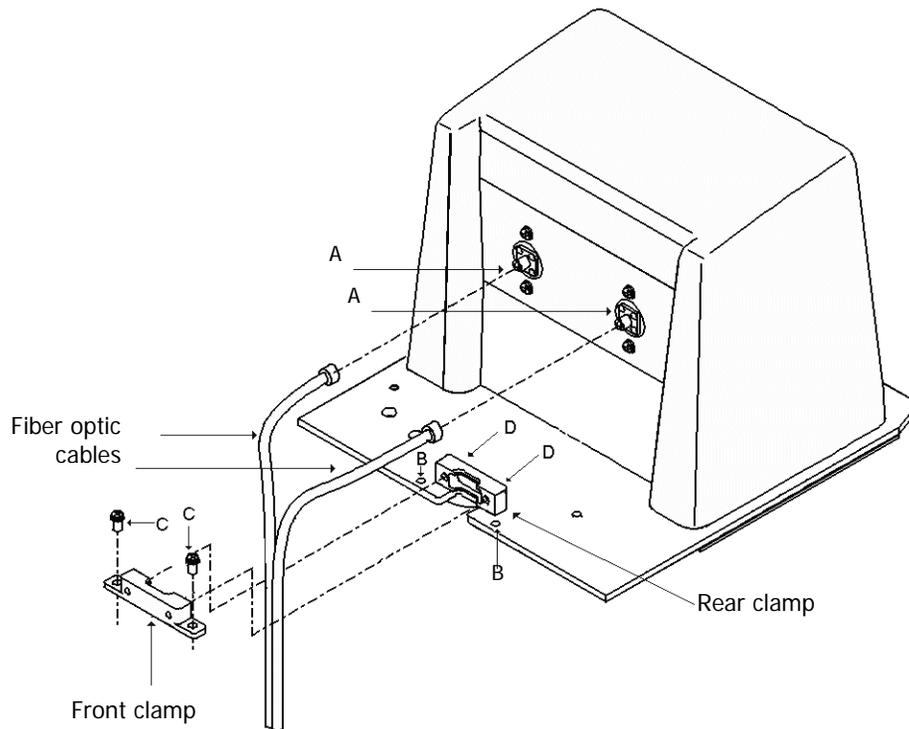
Figure 3 Installing the accessory

5. Fit the sample cover supplied with the fiber optic sampling accessory.

Once assembled, the entire system comprising probe, fibers and sampling accessory can be left assembled. It can be removed and re-fitted into the sample compartment as a single unit.

Changing the connectors on the Fiber Optic Interface Sampling Accessory

If you have purchased the Sample Area Fiber Interface Kit (part number L1240067) you will have connectors for both a solids probe and a liquids probe. The Fiber Optic Interface Sampling Accessory will be fitted with either the connectors for a solids probe or the connectors for a liquids probe.

In order to use the probe for which the Fiber Optic Interface Sampling Accessory is not currently configured, the connector (shown in Figure 4) must be changed using the procedure described below.

1. Loosen the grub screw shown in Figure 4 using the hexagonal wrench supplied.

Grub screw

Solids connector

Liquids connector
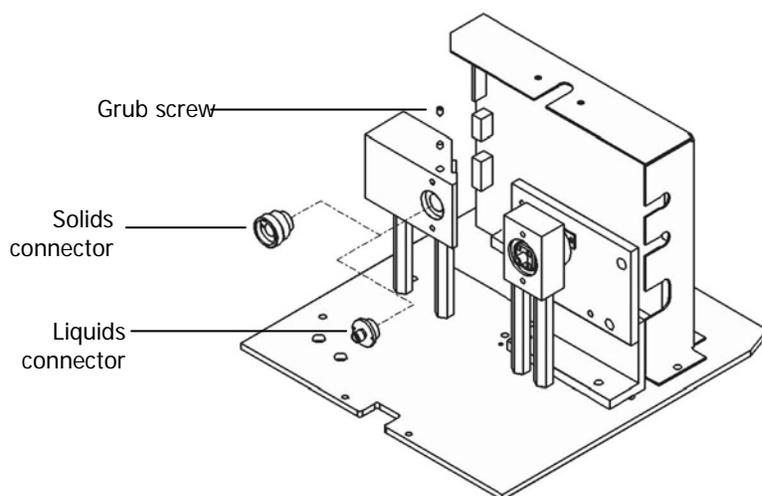
Figure 4 Replacing the connectors

2. Unscrew the connector currently fitted.
    Finger pressure should be sufficient to remove the connector.

3. Screw the other connector into place until finger-tight.

4. Tighten the grub screw using the hexagonal wrench.

Checking that the PerkinElmer Solids Probe and Fiber Optic Interface Sampling Accessory is working

1. Ensure that the probe tip is clean, as described in *Maintenance* on page 14.

2. Place a piece of Spectralon at the tip of the probe, or use a PerkinElmer probe cap (part number L1245218).

3. Start Spectrum and select **Monitor** on the Measurement menu.
    The energy should be in the range 1000–3000 units.

Checking that the PerkinElmer Liquids Probe and Fiber Optic Interface Sampling Accessory is working

1. Check that the probe tip is clean.

2. Place the probe in a suitable holder with no sample present.

3. Start Spectrum and select **Monitor** on the Measurement menu.
   The energy should be 1000–3000 units

Checking that the Fiber Optic Interface Sampling Accessory is working if you are using a custom probe

1. Connect a suitable fiber loop between the input and output SMA connectors.

2. Start Spectrum and select Monitor on the Measurement menu and monitor for the presence of a signal.

NOTE: If the signal level is too high and the system is overloaded this can appear as zero energy. Place an attenuating sample in the beam to check to see if any signal appears when monitoring. If it does, the signal is too high and either the electric gain needs to be reset and/or signal attenuation used. Optical signal attenuation can be achieved by placing an appropriate neutral density filter in the filter wheel (or reducing the J-stop).

Software update

When the Fiber Optic Interface Sampling Accessory is installed, the software detects the presence of the accessory. The Fiber Optic Interface Accessory toolbar is displayed and the Setup Instrument BeamPath is updated to show the accessory in position (Figure 5).
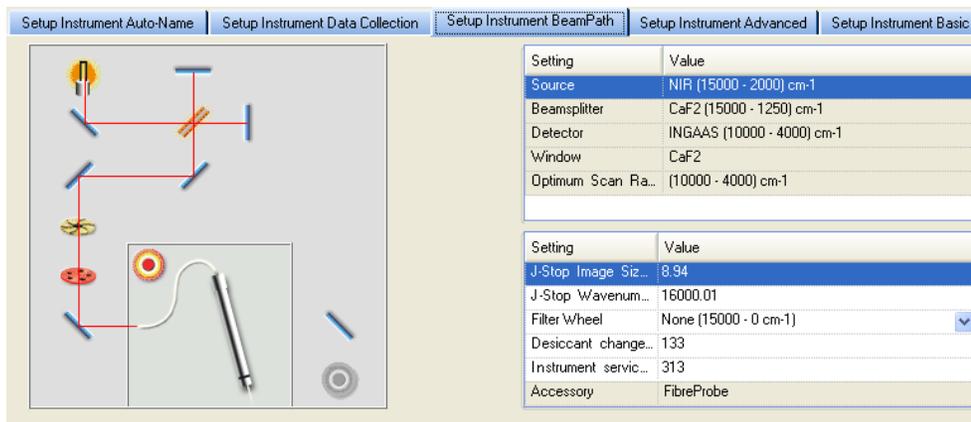
Figure 5 Setup Instrument BeamPath tab with Fiber Optic Probe

Using the Accessory

Optimizing the performance of your remote solids probe

- Ensure as far as possible that consistent pressure is applied at the tip of the probe when analyzing each sample.

- In general, if placing the probe directly into the sample, ensure that there is at least 1 cm depth of sample below the probe tip.

- In order to minimize the risk of cross contaminants, you should check whether it is feasible to measure the sample through thin glass vial walls (ideally less than 1 mm thick) or through thin polymer bags.

- Keep the probe tip clean at all times.

- Do not drop the probe.

Collecting a spectrum

Spectra are collected with a remote solids probe by first collecting a background spectrum using a suitable reflectance reference material such as Spectralon.

If you are using the liquids probe, background spectra are usually collected with the clean probe tip suspended in air.

> **NOTE:** Although Spectrum automatically alerts you when you need a new background, and can be configured to request a new background at set intervals (on the Setup Instrument Data Collection tab), if you want to collect a background, use the **Scan Type** option on the Setup Instrument Basic tab.

3. Enter the required scan and instrument parameters in the Instrument Settings toolbar.

   When your accessory is installed in the instrument, Spectrum will default to the instrument settings 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	
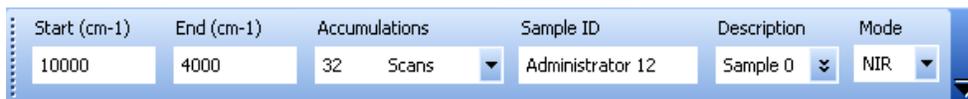
Figure 6 The Instrument Settings toolbar

> **NOTE:** The **Sample ID** and **Description** are automatically supplied by the AutoName function. Search for "AutoName" in the Spectrum on-screen help for more information.

   To amend any value, select the parameter and enter your new value.

4. If you need to collect a background spectrum, place the reference at the tip of the probe.

5. Click ![icon] on the Measurement toolbar.

   Spectrum automatically alerts you when you need a new background, and can be configured to request a new background at set intervals (on the Setup Instrument Data Collection tab).

6. Position the probe on or in the sample.

7. Click **Scan** ![icon] to begin collecting data.

   By default, during scanning the sample data is displayed on the Live tab in the Viewing Area.

   The completed spectrum is displayed on the Graph tab, and added to the current Sample View in the Data Explorer.

If you want to set additional instrument parameters that are not displayed in the Scan toolbars, use the Setup Instrument tabs in the Dialog Pane at the bottom of the workspace.

The Spectrum on-screen Help describes how to format, process and report your results. To view the Help, select **Contents** from the Help menu.

Operational guidelines

- Use a resolution of 16 cm^{-1}.

- Scan times of approximately 1 minute for background and sample scans are sufficient for most routine solvent identification applications.

- It is possible to use shorter scan times, depending on the application.

- Take care to route the fibers through the slot at the front of the modified baseplate. Ensure that there are no sharp bends in the fibers before closing the modified sample compartment lid.

- Keep the fibers as still as possible during measurement.

- As far as possible, collect the background and sample spectra with the fibers and probe in the same position.

- Leave the modified sample compartment lid closed at all times.

- Results can sometimes be improved by clamping the probe in a fixed position while taking measurements.

- When installed, but not in use, keep the probe parked in the holder provided, or keep the probe cap fitted.

When sampling with a clamped probe:

- Raise the sample to the probe, rather than moving the probe to the sample (if possible).

- If measuring a powder directly, make sure that the probe tip is immersed before starting a scan.

- Make sure that air bubbles are not trapped in the light path at the probe tip (liquids probe only).

Maintenance

- No maintenance of the Fiber Optic Interface Sampling Accessory is required, except to keep it clean.

- The probe can be left connected to the Fiber Optic Interface Sampling Accessory.

- Clean the Fiber Probe by gently wiping with ethanol or isopropanol.
  Ensure the tip is completely dry before use.

- If using the solids probe, leave the tip cover on when the probe is not in use. Park it in the holder supplied.

- No alignment or adjustment is possible with the PerkinElmer probes. If the energy reading of the system suddenly changes:
  - Remove the Fiber Optic Interface Sampling Accessory and first check the instrument without the probe interface. If the basic system is OK, check the probe interface using a fiber loop. If this shows a high energy reading it suggests that the probe itself, or the probe fibers have developed a fault.
  - If a loop is available check the Fiber Optic Interface Sampling Accessory with the loop.
  - If checking with the loop does not show any problem, and there is still no signal with the probe it is possible that the probe has been damaged. Contact your PerkinElmer Service Engineer.

Appendix: Resetting the electric gain

CAUTION	*Anti-static precautions must be taken when following the procedure described below to avoid damaging components on the board.*

The Fiber Optic Interface Sampling Accessory is supplied with the electric gain set for either a solids or a liquids probe. If you wish to change the operation of the interface from a solids to a liquids probe, or vice-versa, we recommend that you reset the electric gain, by replacing the resistor as described below.

1. Remove the fiber optic interface sampling accessory from the instrument.

2. If attached, remove the fiber optic probe.

3. Undo the two front screws and two rear screws on the sampling accessory.

4. Remove the lid of the sampling accessory.

5. Remove the resistor (R6) shown in Figure 7 from the electronics assembly by pulling the resistor carefully.

    If necessary the electronics assembly can be unscrewed from the sampling assembly to help access the resistor.

6. Replace the resistor with the other resistor supplied.

    Ideally the gain should be set to provide an energy reading of between 1000 and 3000 units with the probe connected.

    If the resistors supplied are inappropriate for your application please contact your PerkinElmer Service Engineer.

7. Replace the lid and the four screws.

8. Attach the required fiber probe.

9. Fit the sampling accessory back into the sample compartment of the instrument.
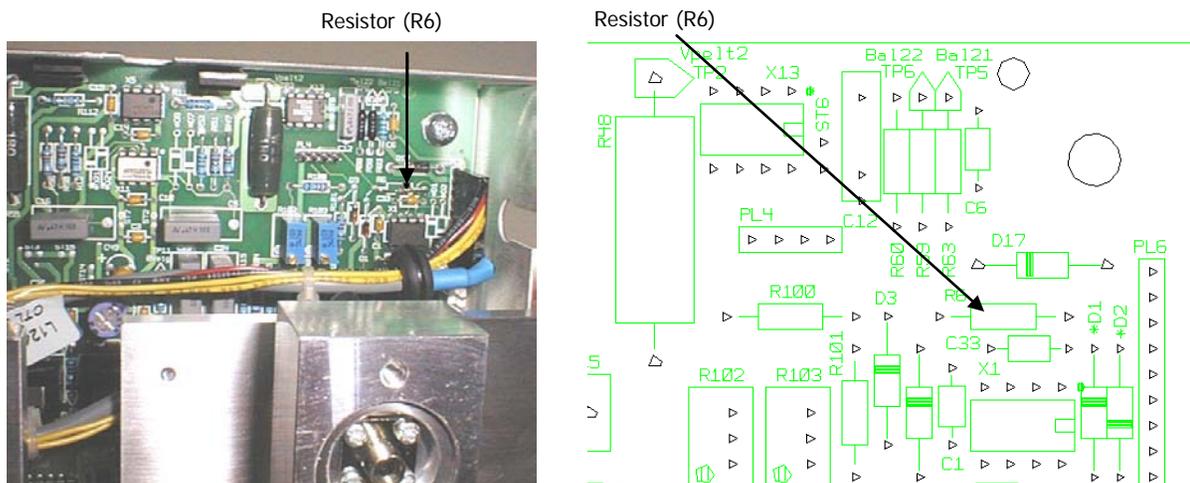
Figure 7 Resistor (R6) to be replaced