

# DIFFUSE REFLECTANCE SAMPLING ACCESSORY



## User's Guide



## Release History

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# ***Contents***

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Conventions Used in this Manual .....	4
Notes, Cautions and Warnings .....	4
Introduction .....	5
How it works .....	5
Installing the Accessory .....	6
Accessory detection by Spectrum software .....	7
Preparing Samples .....	9
Using the sample holder .....	9
Using the sample cup and filler .....	9
Using abrasive pads .....	10
Using abrasive sticks .....	11
Using the Accessory with Spectrum Software .....	12
Replacement Parts List .....	14

## ***Conventions Used in this Manual***

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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 IR System, Spectrum 400 Series or Spectrum 100 Series 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.*

## ***Introduction***

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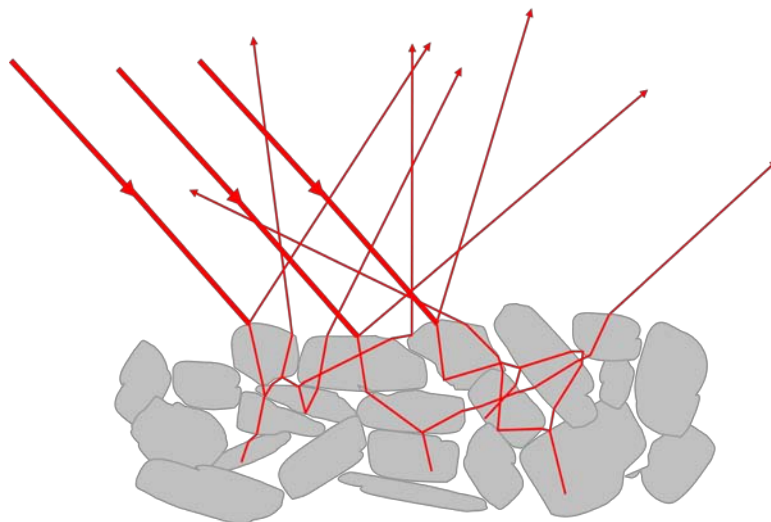
Diffuse reflectance enables you to collect spectra from samples of powders, fine particles, and highly scattering materials not suitable for transmittance measurements. The same method can be used to check solid samples, by grinding them up and treating them as powders, or by scratching a sample from the surface using abrasive pads or sticks, which are then inserted direct into the sample holder.

In the Diffuse Reflectance Sampling Accessory, an infrared beam is reflected by an ellipsoid mirror onto the surface of the sample. The same mirror then collects the reflected beam and directs it onto the detector.

### ***How it works***

Diffuse reflection occurs when light is incident on a scattering material such as a powder or a roughened surface. Rather than being reflected specularly (as from a mirror), the light is scattered in all directions (as from a sheet of white, non-glossy paper).

As the light is scattered by randomly oriented particles, it undergoes specular reflection from the facets of some particles and is partially absorbed as it passes through others. This is illustrated schematically in Figure 1.



**Figure 1 Schematic representation of diffuse reflection by a powder**

Diffusely reflected light has, therefore, undergone both reflection and absorption, and characteristic features of both of these processes may be visible in the spectrum. When the specularly reflected component is small, the diffuse reflectance spectrum has a similar appearance to a transmittance spectrum.

Hence, when obtaining the diffuse reflectance spectrum of a sample, the aim should be to maximize the diffuse reflection and to minimize the specular reflectance. Grinding the sample to produce a small particle size does this. The energy is scattered but not fully absorbed and this increases the amount of diffuse reflectance.

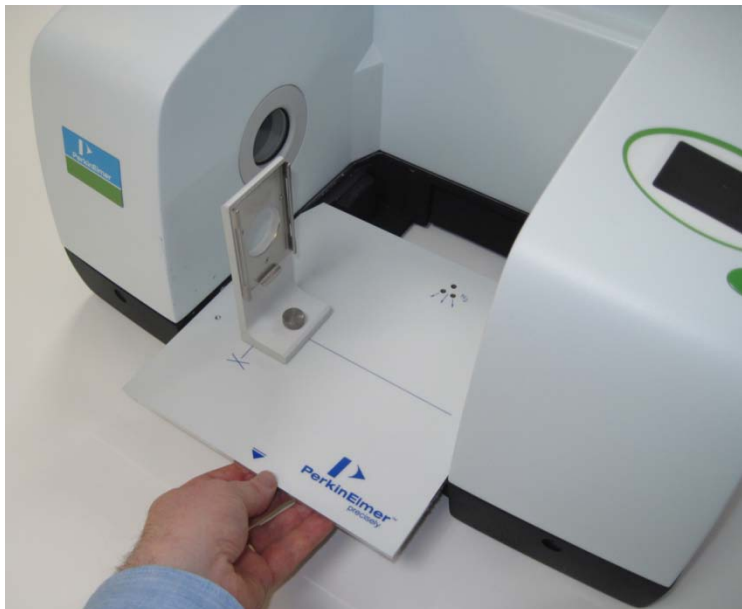
You can further increase the diffuse reflectance by mixing the sample with a non-absorbing diluent, such as KBr. This increases the scattering of the energy; in addition, the decrease in concentration of the sample decreases the amount of energy that is absorbed.

## ***Installing the Accessory***

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The Diffuse Reflectance Sampling Accessory can be simply installed into the sample compartment of the spectrometer, first removing any other sampling accessory currently fitted.

Figure 2 shows the basic sample slide being withdrawn.



**Figure 2 Removing the basic sample slide**

To remove the current accessory and install the Diffuse Reflectance Sampling Accessory:

1. Raise the sample cover to the vertical position, press the release clip and lift the cover upwards, clear of the spectrometer.  
Store it in a safe place for future re-use.
2. Reach in under the base of the current accessory and pull the release handle towards you to release the accessory.
3. Now simply slide the accessory out of the sample area.  
Store it in a safe place for future re-use.
4. Slide in the Diffuse Reflectance Sampling Accessory as shown in Figure 3, rest the back of it on the ledge in the sample area and slide it into position. Push it firmly home to ensure that the multi-way connector on the rear of the Diffuse Reflectance Sampling Accessory mates properly with the spectrometer connector, and the release handle clicks into the engaged position.



**Figure 3 Installing the accessory**

The Diffuse Reflectance Sampling Accessory is now ready for use.

**NOTE:** When the Diffuse Reflectance Sampling Accessory is installed, a connection is made between the accessory and the purge system used in the instrument. To purge the accessory, use the left-hand (Sample) purge connector on the rear of the instrument. The right-hand purge connector purges the instrument.

### ***Accessory detection by Spectrum software***

**NOTE:** If you do not have an instrument installed, see the administrator's guide for your software for details of the Instrument Install Wizard.

The Spectrum software detects the presence of the accessory, and a toolbar of accessory-specific controls is displayed (Figure 4).



**Figure 4 Diffuse reflectance accessory toolbar**

The Setup Instrument Basic (Figure 5) and Setup Instrument BeamPath (Figure 6) tabs are updated to show that the Diffuse Reflectance Sampling Accessory is in position.

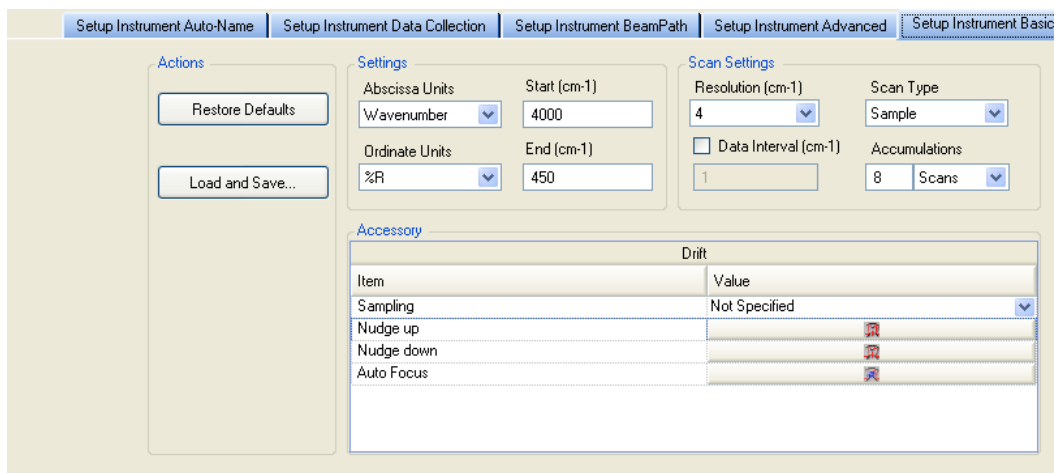


Figure 5 Setup Instrument Basic tab with Diffuse Reflectance Accessory controls

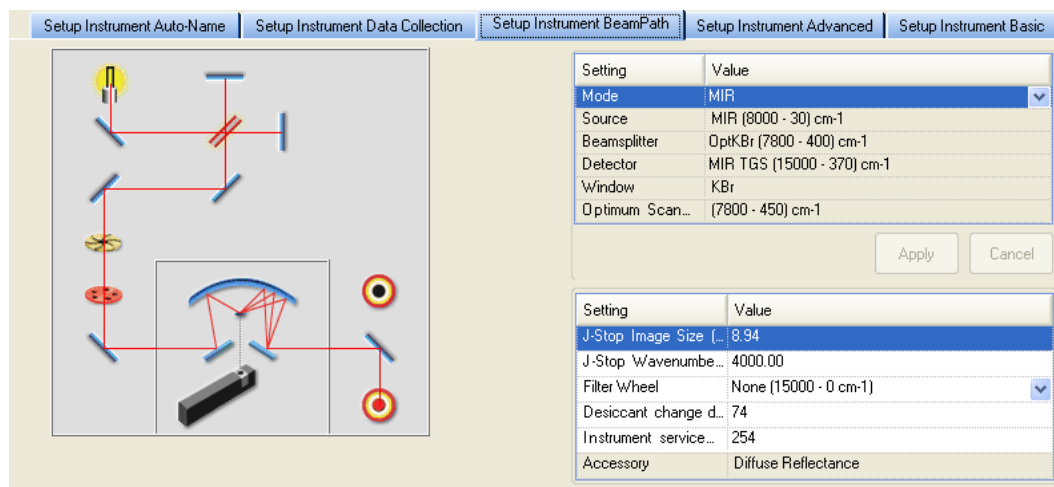


Figure 6 Setup Instrument BeamPath tab with Diffuse Reflectance icon

**NOTE:** To use your accessory, you must first have added your instrument to the list of available instruments. If you have Spectrum (version 10 or later) software and do not have an instrument installed, see the *Spectrum Administrator's Guide* (L1050095) for details of the Instrument Install Wizard.



## Preparing Samples

### Using the sample holder

The sample holder takes cups, pads and sticks in different positions and has two sampling positions for each type of sample. In each case, the position nearest the end of the sample holder is usually used for a background reading and the second position for the sample.

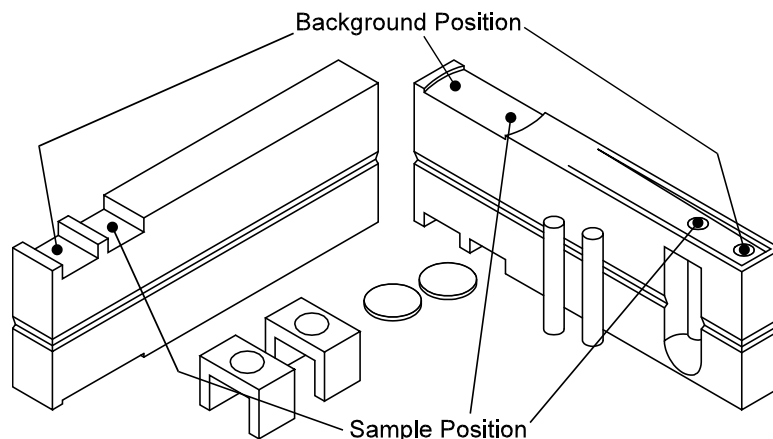


Figure 7 The sample holder

### Using the sample cup and filler

The sample cups are used to measure powdered samples, usually diluted with KBr. The sample cup is placed in the sample filler (see Figure 8) and used as follows:

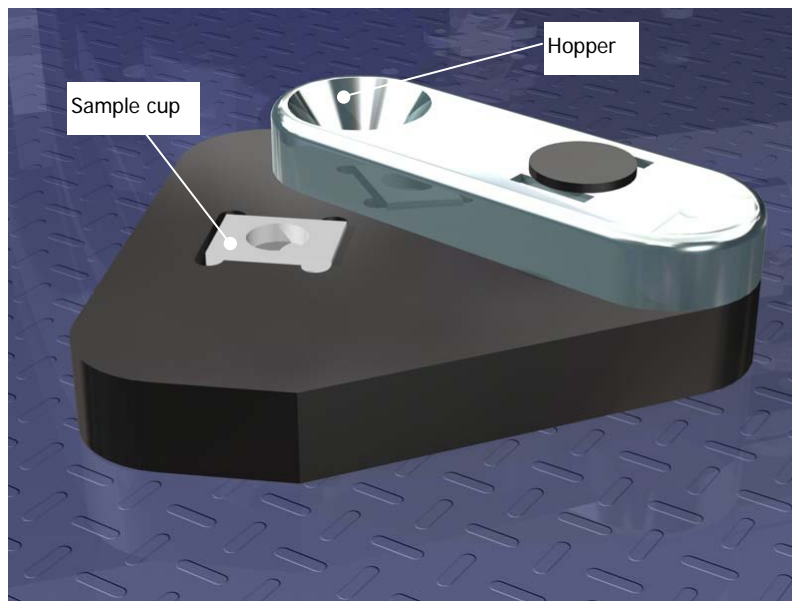


Figure 8 Sample cup and filler

1. Place the sample cup in the slot in the sample filler.
2. Fill the hopper with the sample powder, and then swing the hopper over the top of the sample cup.
3. Tap the sides of the hopper until the cup is filled.

## 10 . Diffuse Reflectance Sampling Accessory User's Guide

- Swing the hopper away from the cup (leveling the powder in the cup in the process).  
Do not pack down the powder. Loosely packed powder increases the scattering effect necessary for diffuse reflectance measurements.
- Place the filled cup on the sample holder in the second position.
- If a background spectrum measurement is required:  
In the first (front) position of the sample holder, place a cup filled with the same material used to dilute the sample in the front position.  
Alternatively, you can collect a generic background spectrum by sticking an abrasive pad over a sample cup and placing it in the front position.

To obtain the best possible results:

- Grind coarse samples before use;
- If the pure sample produces specular distortions, the sample must be diluted by mixing it with KBr, typically to give less than 5% of sample in the mixture. Make sure the sample is well mixed;
- Make sure that you fill sample cups the same way each time, for reproducible results;
- If you have only a small amount of sample, use one of the micro-sample cups.

Clean the hopper and sample cups with tissue, or tissue dipped in mild detergent or alcohol. Make sure they are fully dry before re-use.

### ***Using abrasive pads***



**Figure 8 Abrasive Pads**

Abrasive pads are used to remove a sample from the surface of a solid.

When you use an abrasive pad, a thin layer of particles from the surface of the solid is deposited on the surface of the pad.

- Rub the pad against the sample and then place it on the sample holder in the second position.
- Place an unused pad in the front position for a background spectrum measurement, if required.

To obtain the best possible results:

- The sample should be in the center of the pad;
- You can peel the backing off a pad and stick it to the holder in the sample position before rubbing it on the sample;
- Do not try to load too much sample onto the pad: a light loading is similar to diluting the sample with KBr.

Abrasive pads are disposable items and are not designed to be re-used.

## Using abrasive sticks



**Figure 9 Abrasive Sticks**

Abrasive sticks are used to remove a sample from the surface of a solid.

When you use an abrasive stick, a thin layer of particles from the surface of the solid is deposited on the end of the stick.

1. Rub the abrasive end of the stick against the sample, and then place it on the sample holder in the second position.
2. Place an unused stick in the front position for a background spectrum measurement, if required.

To obtain the best possible results:

- Do not try to load too much sample onto the stick: a light loading is similar to diluting the sample with KBr/KCl;
- Sticks are better than pads for sampling very hard materials;
- To remove the sample stick from the holder, first put your finger or thumb in the recess and slide the stick up. Remove the background stick by turning the holder upside down;
- An accessory shaped like the end of a pen is provided to hold the abrasive stick when sampling.

Abrasive sticks are disposable items and are not designed to be re-used.

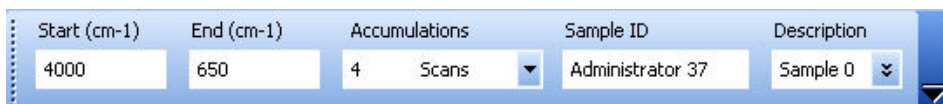
## Using the Accessory with Spectrum Software

Once the sample is placed in the sampling accessory, you are ready to start collecting spectra.

To perform a scan:

1. 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.




**Figure 9 The Instrument Settings toolbar**

2. If required, select your sample type from the **Sampling** drop-down list on the Setup Instrument Basic tab.

The options are: **Abrasive Pad**, **Abrasive stick**, **Macrocup**, **Microcup** and **Custom**.

3. If you need to collect a background spectrum, ensure that the Sample Holder is in the background position.


When sliding the Sample Holder into the Sample Port on the front of the accessory, the first "click" position is the background position and the second "click" is the sample position.

4. Use the **Autofocus**  and/or **Nudge up**  and **Nudge down**  buttons functions to focus the beam and maximize the energy throughput.

The beam can be automatically focused by clicking on the **Autofocus** button.

A dialog is displayed to tell you that the beam is being focused.

OR

Select **Monitor**, by clicking  on the Measurement toolbar to show the energy reaching the detector.




Click on the **Nudge up** and **Nudge down** buttons to maximize the energy.

5. Click  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).

**NOTE:** If you want to collect a background that will be added to the Sample View and can be saved separately, then select **Background** as the **Scan type** on the Setup Instrument Basic tab.


6. Slide the Sample Holder to the sample position.

7. Use the **Autofocus**  and/or **Nudge up**  and **Nudge down**  buttons functions to focus the beam and maximize the energy throughput. The beam can be automatically focused by clicking on the **Autofocus** button. A dialog is displayed to tell you that the beam is being focused.

OR

Select **Monitor**, by clicking  on the Measurement toolbar to show the energy reaching the detector.

Click on the **Nudge up** and **Nudge down** buttons to maximize the energy.

8. Click  on the Measurement toolbar to collect a spectrum. The spectrum is added to the Samples View. The **History** tab will contain information about the Accessory and Sampling Type.

**NOTE:** In weakly absorbing samples, including most organic compounds, specular reflectance can be seen as derivative-shaped bands. These can be avoided by grinding of the sample.

In strongly absorbing samples, pure inorganic compounds for example, specular reflectance can be seen as totally inverted bands. These bands can usually be avoided by diluting the sample to 5%, or less, with KBr.

By default, diffuse reflectance spectra are measured in percent reflectance (%R). However, for quantitative analysis, you should convert spectra to Kubelka-Munk units. Kubelka-Munk units are linear with concentration for weakly absorbing samples with low specular reflectance.

If you select the **Preview** checkbox on the **Sample Table**, you have the option to preview the sample and adjust the energy throughput using the accessory toolbar buttons upon clicking the Scan button.

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.

## ***Replacement Parts List***

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<b>Part Number</b>	<b>Description</b>	<b>Quantity</b>
L1201654	Macro cup	1
L1201655	Micro cup	1
L1201865	Sample holder	1
L1275105	Metal-coated abrasive sticks	100
L1275102	Diamond abrasive sticks	100
L1275106	Metal-coated abrasive pads	100
L1271021	Silicon carbide abrasive pads	100