

SPECTRUM TWO DIFFUSE REFLECTANCE ACCESSORY



User's Guide

Release History

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Introduction

The Diffuse Reflectance (DRIFT) Accessory 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 material from the surface using abrasive pads or sticks, which are then inserted direct into the sample holder.

The accessory is mounted on a baseplate for quick and accurate installation into your Spectrum Two instrument.

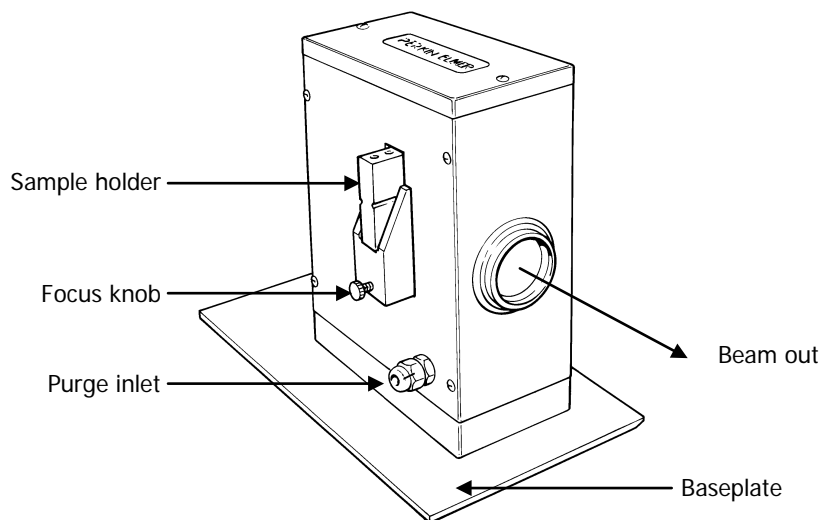


Figure 1 Spectrum Two Diffuse Reflectance Accessory

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 2.

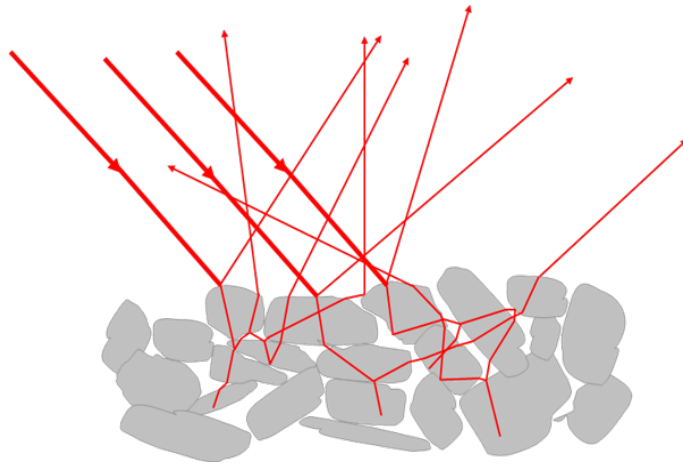


Figure 2 Schematic representation of diffuse reflection by a powder

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 reflection. 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.

In the Diffuse Reflectance Accessory, an infrared beam is reflected by an ellipsoid mirror onto the surface of the sample (M4). The same mirror then collects the reflected beam and directs it onto the detector.

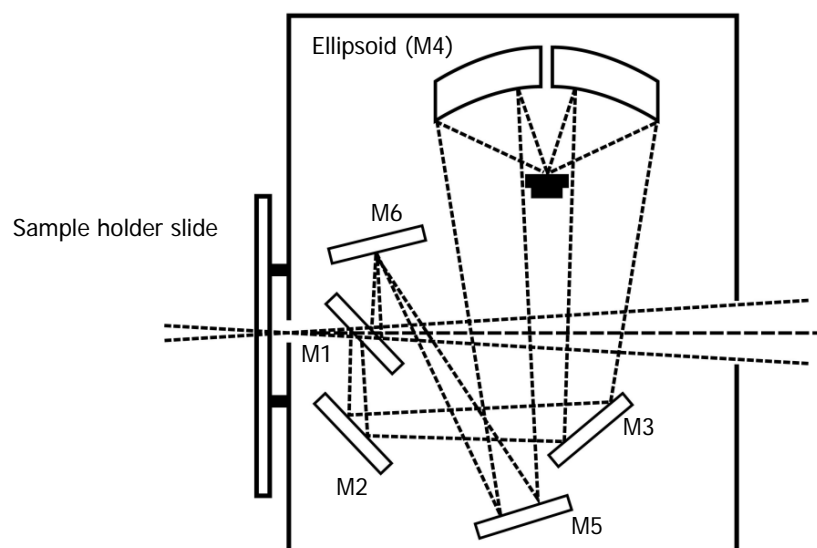


Figure 3 Optical diagram of the accessory; "M" and "S" represent mirror and sample, respectively

Unpacking the Accessory

As soon as you receive your Diffuse Reflectance Accessory, make sure that it includes the parts listed below. If anything is missing or damaged, contact PerkinElmer.

Part Number	Description	Quantity
L1605044	Alignment tool	1
09919187	Loading rod	1
L1271012	Sample cup (macro)	2
L1271013	Sample cup (micro)	2
L1271019	Funnel	1
L1271022	Abrasive stick holder	1
L1275105	Abrasive sticks	25
L1275106	Abrasive pads	25

Installing the Accessory

The Diffuse Reflectance Accessory is pre-mounted on a baseplate for quick installation into the sample compartment of the spectrometer.

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

1. Open the sample cover of the Spectrum Two, if fitted.
2. Pull the baseplate of the current sample accessory towards you, and slide the accessory out of the sample area (Figure 5).
Store it in a safe place for future re-use.



Figure 4 Removing the sampling accessory

3. Start Spectrum software and connect to the instrument.
4. Select **Monitor** from the Measurement menu in Spectrum software and record the energy throughput of the open beam.
5. Slide in the Diffuse Reflectance Accessory baseplate (Figure 5). Push it in firmly to ensure that the connector on the rear of the baseplate connects properly with the spectrometer connector.



Figure 5 Diffuse reflectance accessory installed in the Spectrum Two

We recommended that you maximize the energy throughput of the system as described in *Aligning the Diffuse Reflectance Accessory* on page 9 after installing the accessory.

Accessory detection by Spectrum software

When the accessory is installed in the sample compartment, Spectrum software detects it, and the Setup Instrument Basic (Figure 6) and Setup Instrument BeamPath (Figure 7) tabs are updated to show that the Diffuse Reflectance Accessory is in position.

The Accessory toolbar displays the DRIFT icon  .

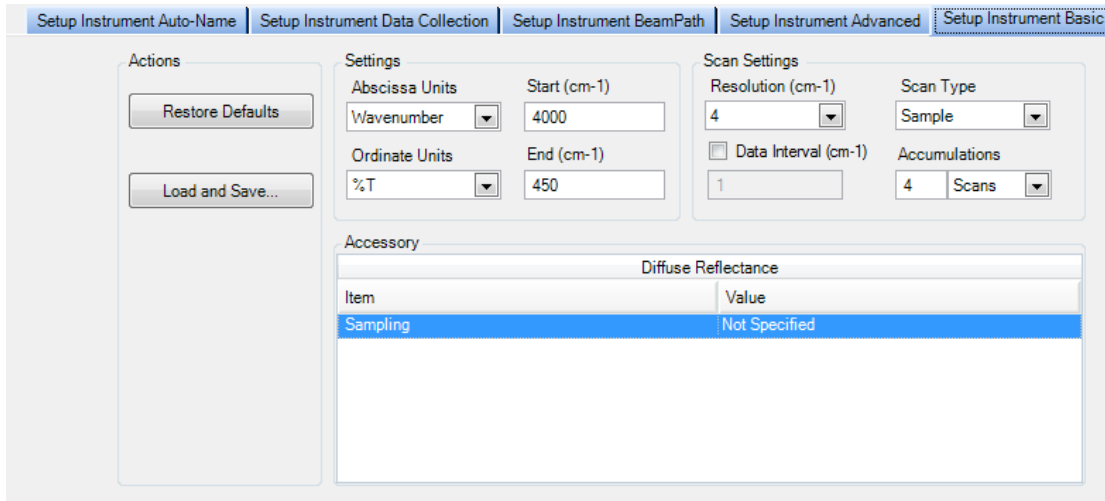


Figure 6 Setup Instrument Basic tab with Diffuse Reflectance Accessory

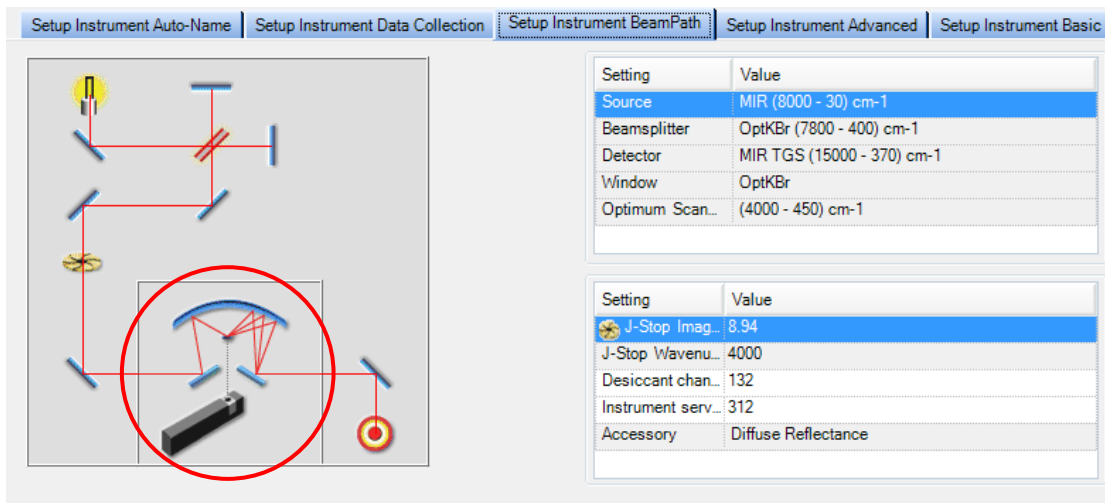


Figure 7 Setup Instrument BeamPath tab with Diffuse Reflectance Accessory icon

Aligning the Diffuse Reflectance Accessory

Before you collect spectra using your Diffuse Reflectance Accessory, we recommend that you align the accessory and make sure that the accessory is operating correctly.

1. If the sample holder is in the accessory, remove it.
2. Place one of the (coated) abrasive pads supplied with the Diffuse Reflectance Accessory in the background (front) indentation in the sample holder (Figure 8).

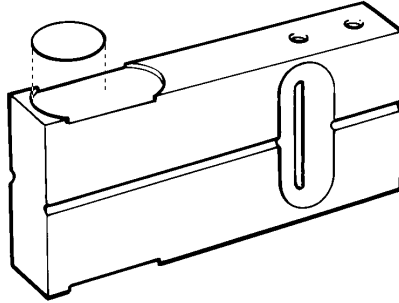


Figure 8 The background position

3. Slide the sample holder into the accessory and push as far as the first click (Figure 9).

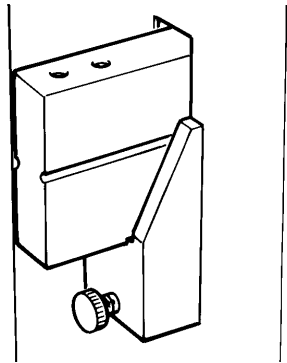


Figure 9 The sample holder in the background position

4. In Spectrum software, select **Monitor** from the Measurement menu.
The Live tab is displayed.
If you do not observe an energy throughput, or if you are aligning your accessory for the first time, continue at Step 5.
If you do observe an energy throughput, and your accessory has been aligned previously, continue at Step 14.
5. Click **Halt** to exit the Live tab.
6. Using a 2.5 mm Allen key, undo the four M4 cap-head screws to remove the front plate from the accessory.
7. Insert the alignment tool in position on the baseplate (Figure 10).
Press the alignment tool mount firmly to locate the two pins on the base of the alignment tool into the holes on the baseplate.

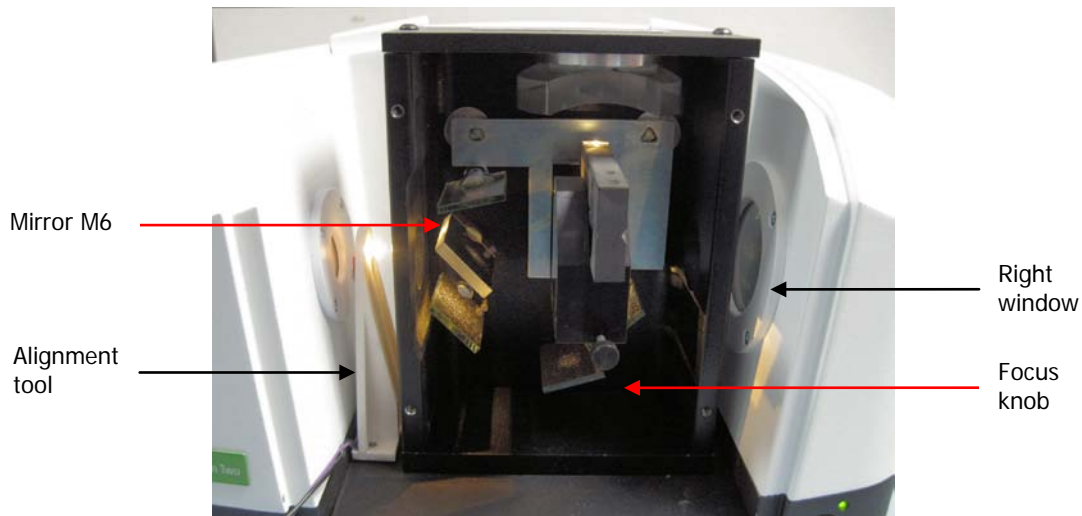


Figure 10 Alignment tool in position on the Diffuse Reflectance Accessory baseplate (accessory front plate removed)

8. Connect the 15-way connector on the cable of the alignment tool into the EXT DETECTOR port on the rear of the spectrometer (Figure 11).
The white LED on the alignment tool should be lit.



Figure 11 Alignment tool connected to EXT DETECTOR port

9. Adjust the height of the sample holder using the focus control until the white light is in the center of the abrasive pad.
10. Place a card between the exit port of the Diffuse Reflectance Accessory and the window on the right of the sample compartment.
11. Adjust the top left mirror (mirror M6 in Figure 3) until you observe the output beam in against the paper on the right of the sample compartment.

NOTE: You may need to adjust mirror M6 and the height several times to find the optimum position.

12. Remove the white-light alignment tool from the baseplate and disconnect from the EXT DETECTOR port.
13. Select **Monitor** from the Measurement menu.
The Live tab is displayed.

14. Adjust the focus height until you obtain an energy maximum. Then adjust M6 again maximize signal by iteratively adjusting height and M6.

NOTE: You may need to adjust mirror 6 and the height several times to find the optimum position.

15. Record the maximum throughput energy and then click **Halt**.
16. Compare this value with the value for the energy throughput of the open beam (recorded in Step 4 of the installation procedure on page 7).
If the energy throughput is 10% or more of the energy throughput of the open beam, it is sufficient for you to collect spectra. Further adjustment of the height of the sample in the accessory may increase this energy level; for a pad this may be approximately 15% of the open beam energy.
17. Replace the front panel of the accessory.

NOTE: Once the accessory is aligned, only use the focus knob is on the front of the accessory to adjust the energy throughput. Do not adjust the nuts on the back of the accessory because this may cause serious errors in alignment.

Preparing Samples

After you have fitted the Diffuse Reflectance Accessory, you are ready to collect spectra using the sample cups, abrasive pads or abrasive sticks.

To reduce the effect of water vapor and CO₂ on your spectra, we recommend that you purge the accessory with clean, dry air or nitrogen (see *Purging the Diffuse Reflectance Accessory* on page 21).

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.

Using a sample cup

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.

To fill the sample cups:

1. If the sample holder is in the accessory, remove it.
2. Fill a sample cup with KBr or KCl powder.
You may find it helpful to use the funnel that is supplied with the Diffuse Reflectance Accessory.
3. Tap the sample cup on the bench to level the powder.
4. Place the sample cup containing KBr/KCl powder in the background (front) well in the sample holder (Figure 12).
5. Fill a sample cup with your sample.
6. Tap the sample cup on the bench top to level the sample.
To minimize adjustment between background and sample measurements, the level of powder in the sample cups should be the same.
7. Place the sample cup containing your sample in the sample (rear) well in the sample holder (Figure 12).

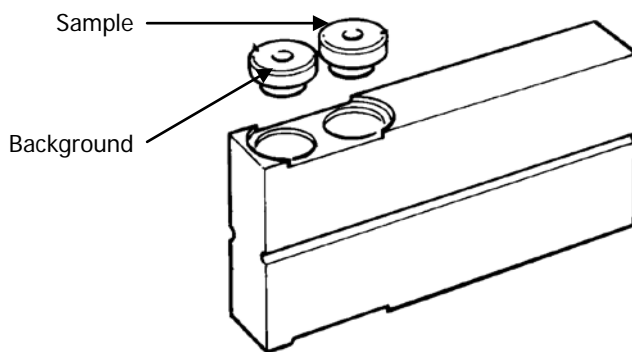


Figure 12 Background and sample positions for the sample cups

To collect the background spectrum, push the accessory as far as the first click (Figure 13).

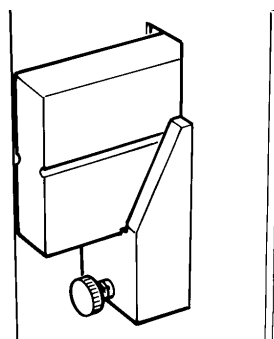


Figure 13 The sample holder in the background position

To collect the sample spectrum, push the accessory as far as the second click (Figure 14).

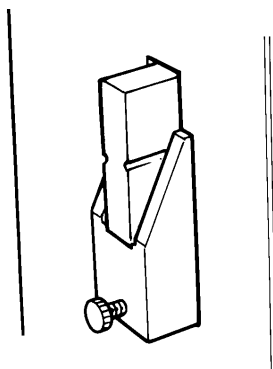


Figure 14 The sample holder in the sample position

Abrasive sampling techniques

The abrasive pads and sticks in the Diffuse Reflectance Accessory kit enable you to collect diffuse reflectance spectra from solid samples with no sample preparation. Abrading the surface of a solid sample produces a sample that can be measured directly and gives spectra that are similar to transmission spectra.

The abraded sample consists of a thin layer of small particles, so that each ray will have a short pathlength through the absorbing material, making it similar to a diluted powder sample.

The spectra collected are similar to those collected from KBr disks; this makes them suitable for qualitative analysis. The technique is not so easily used for quantitative analysis because it is sometimes difficult to control the amount of material on the abrasive pad or stick.

Abrasive sampling can also be used for waxy or viscous materials that are not easily prepared as thin films.

The abrasive pads and sticks supplied are coated with a reflective aluminum coating that greatly increases the diffusely reflected energy that is collected, and gives a spectrum of higher signal-to-noise ratio than one collected using an uncoated abrasive pad or stick. The coated stick or pad also provides an ideal material for collecting a background spectrum.

NOTE: If your samples are very hard, there is a danger that the sample may remove the aluminum coating on the abrasive pads or sticks. This may lead to features in the sample spectrum that are not compensated for by ratioing with the background spectrum. In this case, you may prefer to use uncoated abrasive pads (L1271021) or sticks (L1275102).

Using an abrasive pad

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.

The infrared beam is focused onto the center of the pad. For the technique to work correctly, your sample must therefore be in the target area in the center of the abrasive pad (Figure 15).

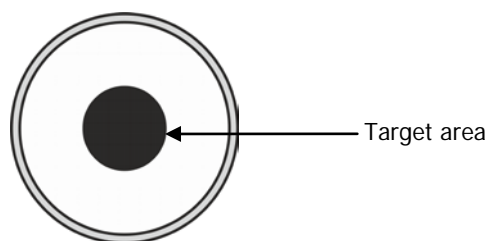


Figure 15 The target area on the abrasive pad

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.

The quality of the spectrum collected from an abrasive pad depends on the level of loading of the sample on the pad: if there is too much sample on the pad, the peaks in the spectrum will be too strongly absorbing. We recommend that you load the pad so that no loose dust is visible on the pad. Loading a pad more lightly has the same effect as diluting a powder sample with KBr or KCl.

The abrasive pads supplied with the Diffuse Reflectance Accessory are coated with aluminum to increase the reflectance of the pad and therefore the energy reaching the instrument. If your samples are very hard, and there is a danger that the sample may remove the aluminum coating, we recommend that you use abrasive pads that are not coated with aluminum (L1271021).

Loading the samples into the sample holder

1. If the sample holder is in the accessory, remove it.
2. Gently rub the abrasive pad over the surface you want to sample.
You may find it helpful to you stick an abrasive pad in the sample position on the sample holder, this enables you to easily collect a sample from a surface.
3. Put the pad that you have collected your sample on into the sample (rear) indentation in the sample holder (Figure 14).
It is important that the back edge of the pad is aligned with the back edge of the indentation in the sample holder.

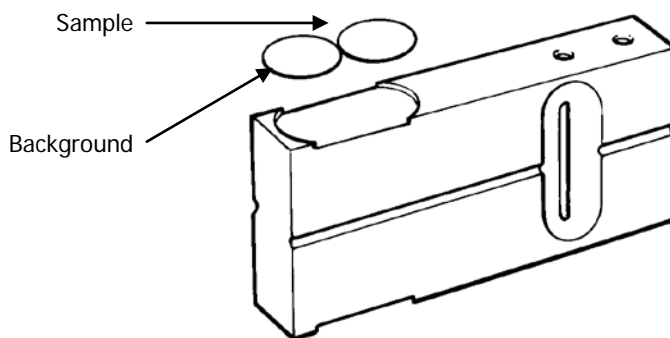


Figure 16 Background and sample positions for the abrasive pads

4. Put a clean abrasive pad in the background (front) indentation in the sample holder (Figure 14).
It is important that the front edge of the pad is aligned with the front edge of the indentation in the sample holder.

To collect the background spectrum, push the accessory as far as the first click (Figure 17).

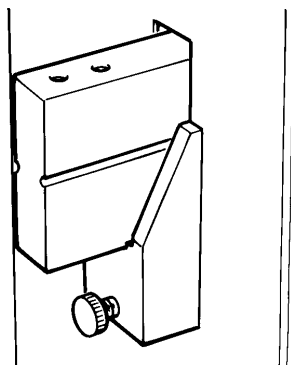


Figure 17 The sample holder in the background position

To collect the sample spectrum, push the accessory as far as the second click (Figure 18).

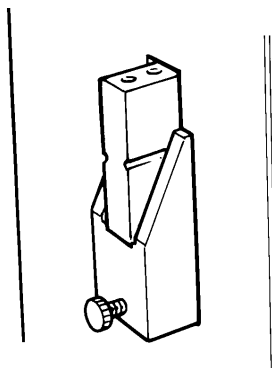


Figure 18 The sample holder in the sample position

Using an abrasive stick

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.

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.

Collecting the sample

Abrasive sticks enable you to collect samples from solids. The infrared beam is focused onto the center of the end of the abrasive stick. One end of an abrasive stick is slightly domed. For the technique to work correctly, your sample must be on the domed area of the abrasive stick.

The quality of the spectrum collected from an abrasive stick depends on the level of loading of the sample on the stick. If there is too much sample on the stick, the peaks in the spectrum will be too strongly absorbing. We recommend that you load the stick so that no loose dust is visible on the stick. Loading a stick more lightly has the same effect as diluting a powder sample with KBr or KCl.

The abrasive sticks supplied with the Diffuse Reflectance Accessory are coated with aluminum to increase the reflectance of the stick and therefore the energy reaching the instrument. If your samples are very hard, and there is a danger that the sample may remove the aluminum coating, we recommend that you use abrasive sticks that are not coated with aluminum (L1275102).

Loading the samples into the sample holder

1. Gently rub the abrasive stick over the surface you want to sample.
You may find it helpful to use the abrasive stick holder (L1271022).
2. Remove the sample holder from the Diffuse Reflectance Accessory.
3. Put a clean abrasive stick in the background (front) hole in the sample holder (Figure 19).
4. Put the stick that you have collected your sample on into the sample (rear) hole in the sample holder (Figure 19).

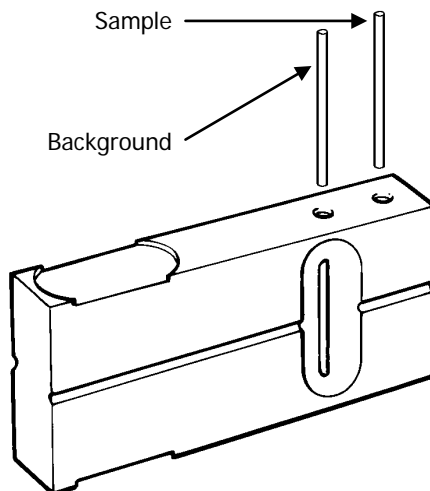


Figure 19 Background and sample positions for the abrasive sticks

To collect the background spectrum, as far as the first click (Figure 20).

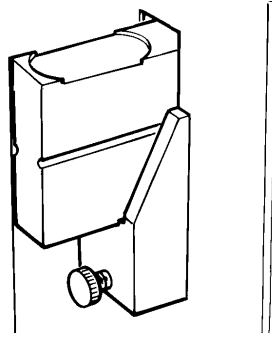


Figure 20 The sample holder in the background position

To collect the sample spectrum, push the sample holder into the accessory as far as the second click (Figure 21).

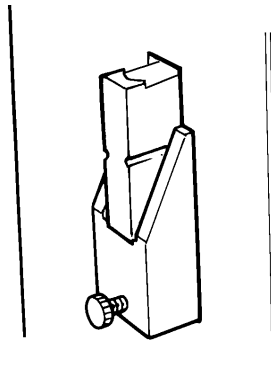


Figure 21 The sample holder in the sample position

Removing the abrasive sticks from the sample holder

The recess in the side of the sample holder enables you to remove the abrasive stick in the sample position, without contaminating the abrasive surface of the stick.

1. Using a finger or thumb, push upwards the shaft of the abrasive stick that is in the recess.
The stick protrudes above the sample holder.
2. Using the thumb and a finger of your other hand, pull the abrasive stick from the sample holder.
Take care not to touch the abrasive surface of the stick.

Using the Accessory with Spectrum Software



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

The Measurement bar (Figure 22) displayed by default at the top of the workspace includes the tools you need to collect a spectrum from a sample. You can also select these commands from the Measurement menu.



Figure 22 Spectrum Two Measurement bar

To perform a scan:


1. Ensure that the required scan and instrument parameters have been set.
When you first fit the accessory, the software automatically sets default scan parameters to values that are appropriate to the Diffuse Reflectance Accessory. If you want to set any of the instrument parameters that are not included on the toolbars select **Instrument** from the Setup menu to display the Setup Instrument tabs.
2. If you want, enter a unique **Sample ID** and **Description** for the sample on the Measurement toolbar.
3. 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**.
4. 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.
Refer to *Preparing Samples* on page 12 for information on preparing your samples.
5. Select **Monitor** from the Measurement menu to view the energy reaching the detector.
6. Slowly turn the focus knob until the energy throughput is maximized.
For a power in a sample cup, the maximum energy level should be approximately 10% of the open beam energy level. For a coated abrasive pad or abrasive stick the maximum energy level should be approximately 15% of the open beam energy level.
7. Record the maximum energy level.
8. Click  to stop monitoring.
9. Click  on the Measurement toolbar to collect the background spectrum.
10. Slide the Sample Holder to the sample position.


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11. Monitor the energy.

The level should be within 90% of the energy you measured for the background.

12. If required, slowly turn the focus knob to maximize the energy.

13. Click  to stop monitoring.

14. Click  on the Measurement toolbar to collect the spectrum.

The spectrum is added to the Samples View. The History tab will contain information about the Accessory and Sampling Type.

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

Purging the Diffuse Reflectance Accessory

You can purge the Diffuse Reflectance Accessory, rather than the whole sample compartment. Purging the Diffuse Reflectance Accessory has two advantages: the purge is maintained when you change from background to sample (unless you remove the sample holder), and the purged volume is smaller.

Use polyethylene tubing with a 6 mm external diameter and a 4 mm internal diameter to carry the gas to the purge inlet on the Diffuse Reflectance Accessory.

1. Remove the nut on the Diffuse Reflectance Accessory and remove the blanking disc (Figure 23).

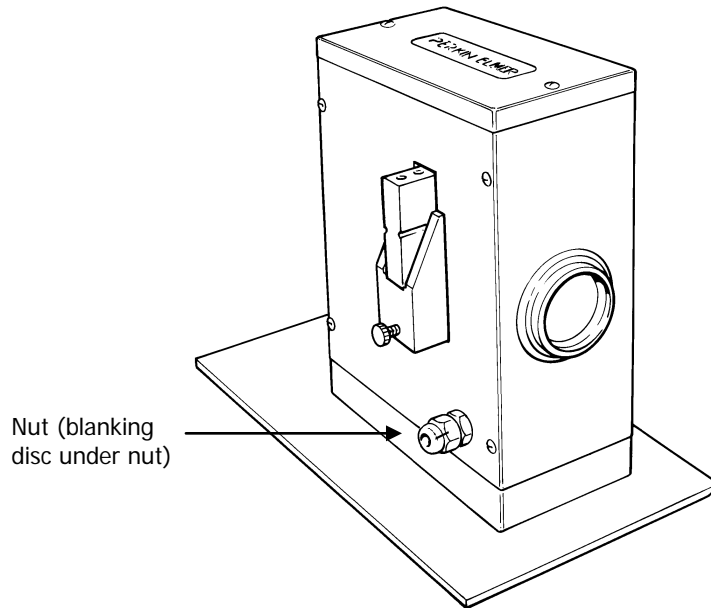


Figure 23 The purge inlet

1. Re-fit the nut on the thread.
2. Push the tubing through the nut until it rests against the interior connector.
3. Tighten the nut to clamp the tubing in place.
4. Purge the Diffuse Reflectance Accessory using a flow rate of approximately 1 to 2 l/min.



WARNING

Never connect the purge tubing directly to a gas cylinder or other high pressure supply. Always use a pressure regulator and set the pressure to a maximum of $6.9 \times 10 \text{ kPa}$ (1 lb in²)

Troubleshooting

The spectrum in Figure 21 was collected from a sample holder in the Diffuse Reflectance Accessory. You may collect a similar spectrum if:

- you do not put a sample cup, abrasive pad or abrasive stick in the sample holder,
- or you collect a spectrum with sample holder between the background and sample positions.

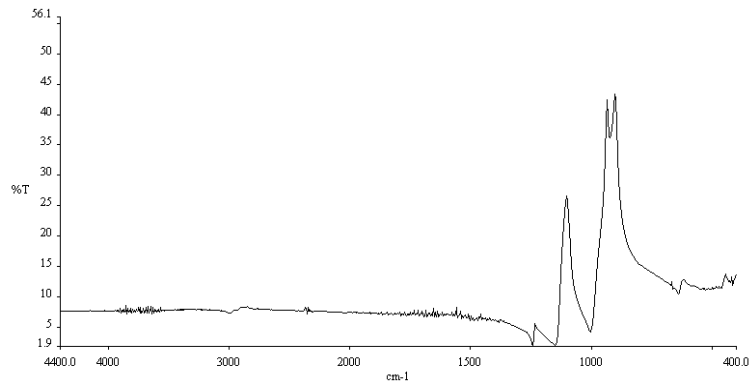


Figure 24 Ratio Spectrum of the sample holder

Replacement Parts List

Part Number	Description	Quantity
L1201654	Macro cup	1
L1201655	Micro cup	1
L1201865	Multi-purpose sample holder	1
L1275102	Diamond abrasive sticks	100
L1275105	Metal-coated abrasive sticks	100
L1275106	Metal-coated abrasive pads	100
L1271021	Silicon carbide abrasive pads	100

