

Smart Diffuse Reflectance

This tutorial introduces the Smart Diffuse Reflectance accessory from Thermo Fisher Scientific. This accessory is designed for fast analysis of powder samples using the standard diffuse-reflectance technique and is compatible with Thermo Scientific Nicolet Series Fourier Transform Infrared (FTIR) spectrometers.

In this tutorial, you will learn the basics of diffuse-reflectance sampling with your spectrometer. It explains how to install the accessory in the spectrometer sample compartment and use the accessory to analyze powders. You will also learn how to maintain and store the accessory when it is not in use.

Product features

The Smart Diffuse Reflectance accessory is a general-purpose sampling accessory for basic diffuse-reflectance analyses. This accessory offers the throughput, ruggedness, and stability needed for most quality control and research applications using standard diffuse-reflectance optics and ambient conditions. The Smart Diffuse Reflectance accessory is designed for fast analysis of organic and inorganic solids and powders using the standard diffuse-reflectance technique. A special tool for collecting and analyzing surface samples is also included.

Smart accessory features

The Smart Diffuse Reflectance accessory is part of our extensive family of Smart Accessories for Nicolet Series spectrometers. All Smart accessories offer the following features:

- Pinned-in-place, permanently aligned optics.
- Fully integrated design.

- Snap-in installation.
- Automatic purging.
- Automatic recognition.
- Automatic measurement settings.

Smart Diffuse Reflectance specifications

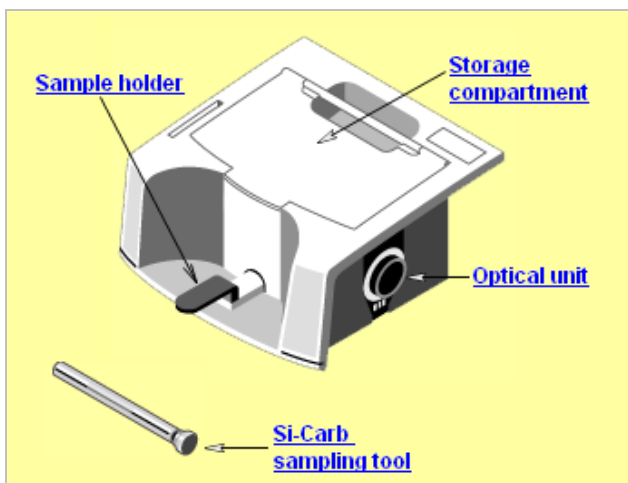
The Smart Diffuse Reflectance accessory features:

- Sample holders: Si-Carb and powder
- Sample cup capacity: ~0.25 gram
- KBr packets: 25 premeasured 0.5 gram packets of high quality KBr powder, ground to the correct particle size for diffuse-reflectance measurements
- Silicon-carbide disks: adhesive-backed, silicon-carbide disks; 100 each 320-grit and 400-grit disks



Product components

The Smart Diffuse Reflectance accessory effectively maximizes diffusely scattered radiation while minimizing specular reflection, which is a source of spectral interference. The accessory comes with two sample holders, a Si-Carb sampling kit, and premeasured packets of KBr.



Component	Description
Sample holder	<p>The sample holder slides into the sample port in the Smart Diffuse Reflectance accessory and presents the sample surface at the focal point of the infrared beam. Two types of sample holders are provided with the accessory.</p> <p>Double-cup holder: The standard sample holder has two cups, one for the sample material and one for the material used to measure the background. Each cup holds approximately 0.25 gram.</p> <p>Single-cup holder: The other sample holder has a trough in the front position to hold the platen from the Si-Garb sampling tool. A gold disk mounted in the back position is used for the performance test and some background measurements.</p>

Component	Description
Storage compartment	The Smart Diffuse Reflectance accessory provides a handy compartment for storing tools and accessories when they are not being used. The compartment is large enough to hold two sample holders, the Si-Garb sampling kit and several premeasured packets of KBr.
Optical unit	The optical unit is a sealed tube that contains an aluminum base supporting a parabolic mirror. The input mirror focuses the beam from the IR spectrometer on the surface of the sample. The output mirror collects the scattered IR energy emitted from the sample and directs the energy to the detector in the IR spectrometer. The optics tube is connected to the purge system in the IR spectrometer. This design maintains a controlled environment around the IR beam as it travels through the accessory and eliminates water and carbon dioxide peaks in the infrared spectra.
Si-Carb sampling tool	The Smart Diffuse Reflectance accessory comes with a Si-Carb sampling kit. The kit includes a probe, two platens, and two packets of silicon-carbide papers in 320- and 400-grit particle sizes. The tool is used to sample large intractable solids such as the painted surface of a piece of equipment or an automobile. To collect a surface sample, you simply rub the sample surface with silicon-carbide paper and then measure the particles adhering to the paper. A reflective disk is used for the performance test and some background measurements.

Starting the software

To measure and analyze samples, start OMNIC Paradigm software.

For instructions on using OMNIC Paradigm software, see the guides and tutorials at www.knowledge1.thermofisher.com

Installing the Smart Diffuse Reflectance accessory

The accessory fits into guides on the side walls of the sample compartment.

These accessories are aligned at the factory and they require no further adjustment. The optical components are mounted in a sealed tube, which automatically connects to the purge system in your IR spectrometer.

❖ To install the Smart Diffuse Reflectance accessory

1. Make sure the spectrometer is turned on.
2. If the sample compartment cover is attached, remove it.
3. If the side wall adapters are in place inside the sample compartment, remove them.
4. If you were using any of the connectors at the back of the sample compartment, remove the cables.
5. Remove the Snap-in baseplate or any other accessories installed inside the sample compartment.
6. Insert the accessory. Lower the accessory into the spectrometer sample compartment and press gently downward until the accessory locks in place.

Installing a sample holder

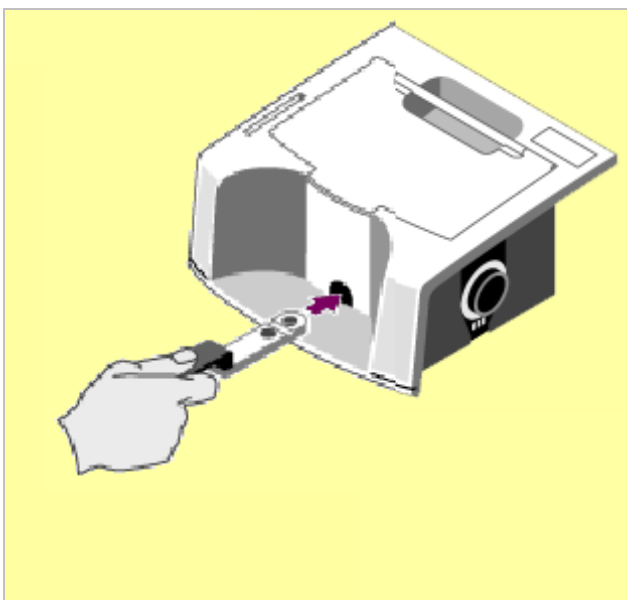
Two types of sample holders are provided with the Smart Diffuse Reflectance accessory:

- a double-cup sample holder for measuring a powder sample and background and
- a single-cup sample holder with a reflective (gold) disk for use with the Si-Carb sampling tool.

Both sample holders fit in the Smart Diffuse Reflectance accessory in the same way.

❖ To install a sample holder

Slide the single-cup sample holder (the one with the reflective disk) into the sample port at the front of your Smart Diffuse Reflectance accessory. The reflective disk will be used to run the performance test.



Note To position the reflective disk in the infrared beam, push the sample holder all the way in and then pull it out slightly until you feel the holder click into position.

Changing your measurement settings

When you install a Smart Accessory, the system automatically updates the measurement settings to those associated with the accessory. The default settings for the accessory have already been optimized for collecting data with the accessory.

After you install the accessory, the accessory name is shown in the Settings list on the dashboard of OMNIC Paradigm software.

Running the performance test

Run the Accessory Performance Test workflow to verify the performance of your accessory.

1. From the dashboard of OMNIC Paradigm software, right-click the Accessory Performance Test workflow and select Run.
2. Follow the prompts to complete the workflow.

When the workflow is complete, you can find the test report in the Reports section of the dashboard.

Preparing a sample for analysis

Preparing a powder sample

Powders are easier to run by diffuse-reflectance than by IR transmission because little or no preparation is required. When analyzed by IR transmission, powders must be mixed with the proper amount of KBr and pressed into a pellet. The pellet is then placed in the IR beam. When a diffuse-reflectance accessory is used, powders are placed directly in the sample holder or mixed with a premeasured packet of KBr and then poured into the sample holder. The sample holder is then inserted into the sample port of the diffuse-reflectance accessory and data collection can begin.

◆ To prepare a powder sample

1. Grind the sample as finely as you can. A particle size of 2 to 5 micrometers is recommended. You can use a mortar and pestle or an electric grinder to grind the sample. One to two minutes of grinding is usually sufficient. Grind the sample slowly to avoid creating heat due to friction; vigorous grinding may change the chemical structure of some samples.

The WIG-L-BUG electric grinder, available from Thermo Fisher Scientific, is designed to grind samples to the correct particle size for diffuse-reflectance measurements.

2. Dilute the sample with a diffusely scattering matrix material (optional). Some powders can be analyzed neat (without dilution). If the sample is highly absorbing or has a high refractive index (it's difficult to tell just by looking at it), it should be mixed with a diffusely scattering matrix material such as KBr, KCl, or diamond powder. Make sure you use a high quality (ground and dried) matrix. If the particles are bigger than 5 micrometers, use a clean mortar and pestle or an electric grinder to grind the matrix material. Don't grind the sample and matrix together.

Note The premeasured packets that come with your Smart Diffuse Reflectance accessory contain high quality KBr that has been thoroughly dried and ground to the correct particle size. As a general rule, mix the sample with the matrix material in the following ratio (by weight):

- For organic samples: 10% sample to 90% matrix
- For inorganic samples: 2 to 5% sample to 95 to 98% matrix

The sample cup can hold about 0.25 gram of powder. However, you should make enough to fill the cup to overflowing. For example, to prepare 0.5 g of organic sample, mix 0.05 g of sample material to 0.45 g of matrix.

3. Open the storage compartment on top of the Smart Diffuse Reflectance accessory and remove the sample holder that has two sample cups.
4. Fill the front cup with your sample. Fill the cup to overflowing. Be careful not to tap the sample holder when pouring in the powder. Tamping packs the particles too closely and may reduce the sensitivity of the analysis. When the cup is filled, place the sample holder on a flat surface and level the sample surface with a spatula. This reduces reflections off the sample surface and helps ensure reproducible results.
5. Fill the back cup with the reference material. If the sample is undiluted, fill the back cup with a non-infrared absorbing matrix such as potassium bromide (KBr). If you diluted your sample in a matrix material, such as KBr, KCl or diamond powder, fill the back cup with the pure matrix. Be sure to follow the same procedure when placing the background material in the sample cup as you used for the sample.

Grinding a solid sample

The diffuse-reflectance technique can be used to measure homogeneous solids by grinding a representative sample and analyzing the powdered material. This works for any organic or inorganic solid that can be ground into a fine powder (particle size of 2 to 5 micrometers).

◆ To grind a solid sample

1. Prepare the sample by grinding it to a fine powder. You can use a mortar and pestle or an electric grinder to grind the sample (2 to 5 micrometer particle size is recommended). Grinding for 1 to 2 minutes is usually sufficient.
2. Follow the instructions for preparing a powder sample.

Dissolving a solid sample

The diffuse-reflectance technique can also be used to analyze solids that are dissolved in a solvent by mixing with a powdered supporting matrix such as potassium bromide and then evaporating the solvent and analyzing the residue.

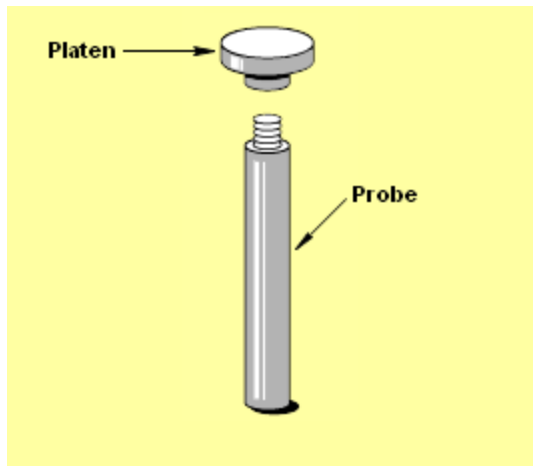
1. Prepare a 10 to 25% solution of sample to solvent and mix the solution well.
2. Fill the front sample cup with a powdered supporting matrix such as KBr. Fill the cup to overflowing and then level the surface with a spatula.
3. Use a syringe to drop 100 micrometers or so of the sample mixture onto the powdered matrix.
4. Wait for the solvent to evaporate completely and then follow the instructions for preparing a powder sample.

Collecting a surface sample

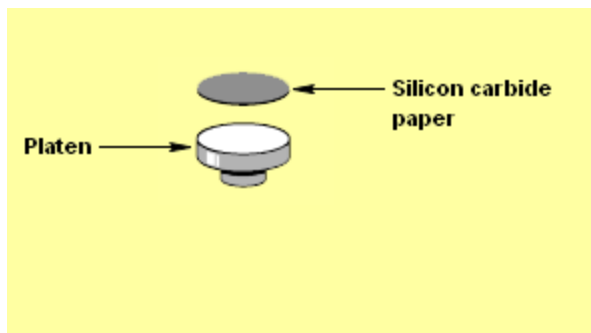
The Si-Carb (silicon carbide) sampling tool can be used to sample the surface layer of any solid. This works well for sampling large, intractable solids, such as the painted surface of a piece of equipment or an automobile

◆ To collect a surface sample

1. Open the storage compartment on top of the Smart Diffuse Reflectance accessory and remove the sample holder that has one sample cup and a gold disk.
2. Remove the probe and platen from the storage compartment and screw the platen onto the probe.



3. Select a silicon carbide paper with the appropriate grit size. Use the 320-grit paper for soft coatings such as paint. Harder materials may require a more abrasive paper such as diamond paper. The higher the grit size the smaller are the particles adhering to the paper. Handle the paper gently to avoid contaminating the surface.
4. Remove the protective cover from the back of the paper and lightly touch the platen with the adhesive.



5. Use the protective cover to press the paper firmly onto the platen.
6. Use the tool to abrade the sample surface. Gently rub the paper against the sample surface for 1 to 2 seconds.
7. Unscrew the platen from the probe. Be careful not to touch the paper with your fingertips.
8. Place the platen, with the paper side up, into the sample cup. When the platen is seated properly, the surface of the platen will be flush with the edges of the sample holder.

Measuring the background

A background spectrum is needed to process the sample data to an infrared spectrum. The background is a reference spectrum which accounts for the unique optics of the diffuse-reflectance accessory and spectrometer. Each sample spectrum is ratioed against a background so that the final spectrum is free of these features.

Choosing the background material

When measuring powders that are mixed with a diffusely-scattering matrix such as KBr, the pure matrix is used for the background measurement. Use KBr for the background measurement if the sample is undiluted. When the silicon-carbide sampler is used, the gold disk or a clean Si-Carb paper is used for the background. If you use the gold disk to measure the background, your spectra will have a large silicon-carbide band around 850 cm^{-1} .

Eliminating the silicon-carbide band

If your sample absorbs near 850 cm^{-1} , you may be able to eliminate the silicon-carbide band in your sample spectrum by using a blank silicon-carbide paper for the background. Be sure to use the same grit size as you used for the sample and collect a thin layer of sample.

Measuring the background

The background data used to process each sample measurement to an IR spectrum must be measured under exactly the same conditions as the sample.

❖ To measure the background

1. Insert the sample holder into the Smart Diffuse Reflectance accessory. To position the background material under the IR beam, push the sample holder all the way in and then pull it out slightly until you feel the holder click into position.
2. Collect a background spectrum. Click Preview and Measure Background on the dashboard.

Background spectrum

After you collect a new background, the background spectrum will be used to process all of your sample measurements until you replace it by collecting another background spectrum.

Figure 1-1: Figure: Background of potassium bromide

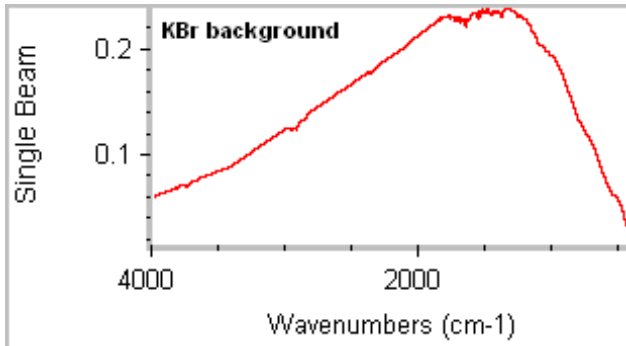


Figure 1-2: A background spectrum collected using the gold disk.

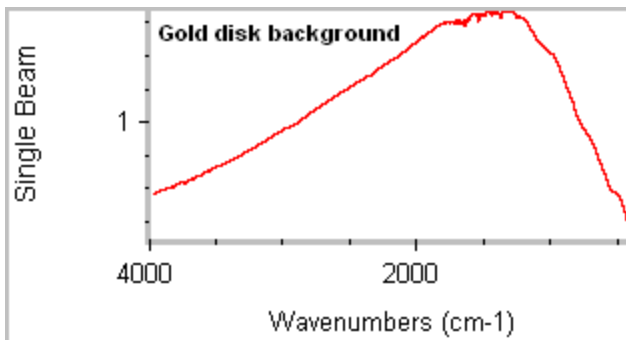
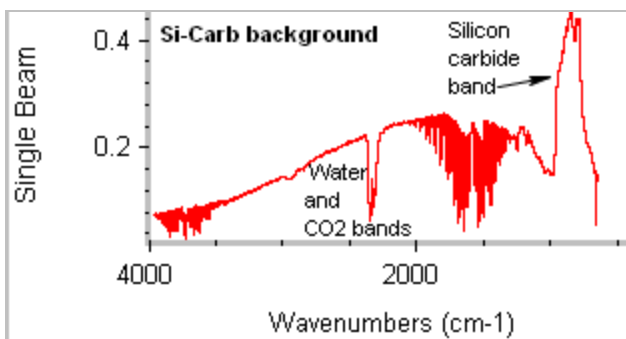


Figure 1-3: A background spectrum collected using a blank 320-grit silicon-carbide paper



When to collect a new background

The background data used to process each sample measurement to an IR spectrum must be measured under exactly the same conditions as the sample. For best results, we recommend collecting a background spectrum before each sample.

If you use the same sampling technique and instrument settings to analyze all of your samples, you can use the same background to process multiple samples. However, we recommend collecting a new background at least once every two hours.

If any of the following is true, you should immediately remeasure the background and use the new background to process your sample data.

- You changed a component in your spectrometer or sampling accessory
- You changed one of the measurement settings other than Gain, Final Format, or Sample Scans.
- You see a change in the amount of water or carbon dioxide bands in the IR spectra of your samples.
- You see an unexpected change in the spectral baseline.
- The quality of your spectral data is reduced (more noise or spurious peaks in the spectrum).

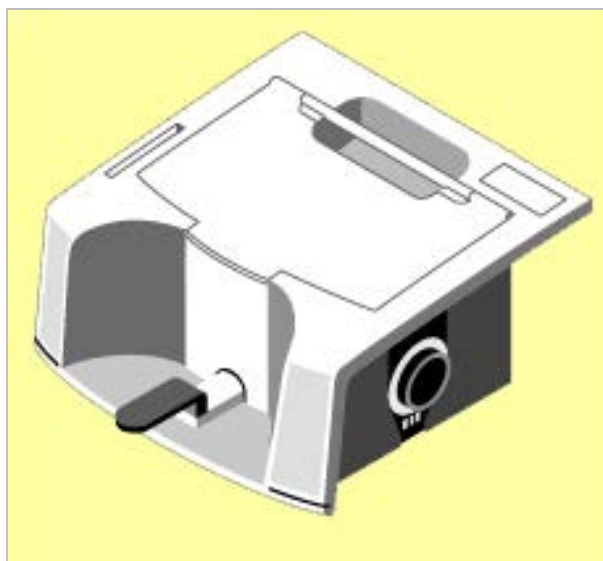
Measuring the sample

Once the background data are collected, move the sample into the infrared beam and measure the sample.

The sample measurement will show how the energy you started with was reduced by the sample.

◆ To measure the sample

1. Move the sample into the infrared beam. Slide the sample holder out about 3 cm (1 inch). You should feel it click into position.



2. Measure the sample.

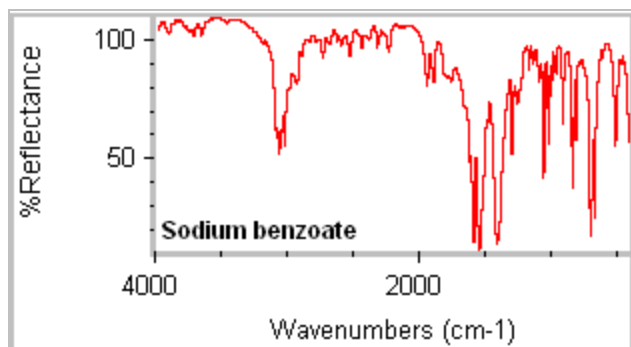
Sample spectrum

When % Reflectance is selected as the final format of your sample spectra, the software displays a reflection spectrum. A reflection spectrum looks similar to a transmission spectrum. The spectrum is updated as new data are collected.

When the system has collected the number of scans specified, the final spectrum is displayed in a window.

The final spectrum shows only the change in IR energy (the background energy has been removed).

Figure 1-1: A spectrum of sodium benzoate diluted with potassium bromide

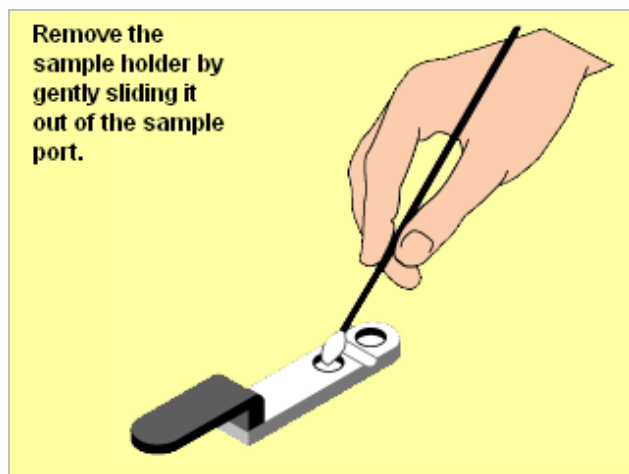


Cleaning between samples

Clean the sample holder after you finish measuring each sample.

You can clean the holder with soap and water or with a solvent such as acetone. If you clean the holder with a solvent, allow enough time for the solvent to evaporate completely. To ensure complete evaporation, dry the sample cups with clean, dry air or nitrogen.

When the cups are clean and dry, you are ready to pour in the next sample.



Removing the accessory

You can remove the Smart Diffuse Reflectance accessory as easily as you installed it.

When not in use, your accessory should be stored in a dust-free environment, such as a cabinet or box.

❖ **To remove the accessory:**

1. If a sample holder was left in the accessory, remove and clean the sample holder and then place it in the storage compartment on top of the Smart Diffuse Reflectance accessory.
2. Gently pull up on the accessory to release it. Continue lifting the accessory straight up until it is completely free of the sample compartment.

Next Steps

For more information on using OMNIC Paradigm software to measure and analyze samples, see the guides and tutorials at www.knowledge1.thermofisher.com