

Smart Integrating Sphere

This tutorial introduces the Smart Integrating Sphere, a back-scatter sampling Smart Accessory for use with Thermo Scientific FT-IR and FT-NIR spectrometers.

This tutorial includes:

- a description of the Smart Integrating Sphere features and components.
- step-by-step installation instructions.
- tips for analyzing various sample materials.
- maintenance and storage information.

Product Features

Smart Accessory Features

The Smart Integrating Sphere is part of Thermo Scientific's extensive family of Smart Accessories. All Smart Accessories offer the following features:

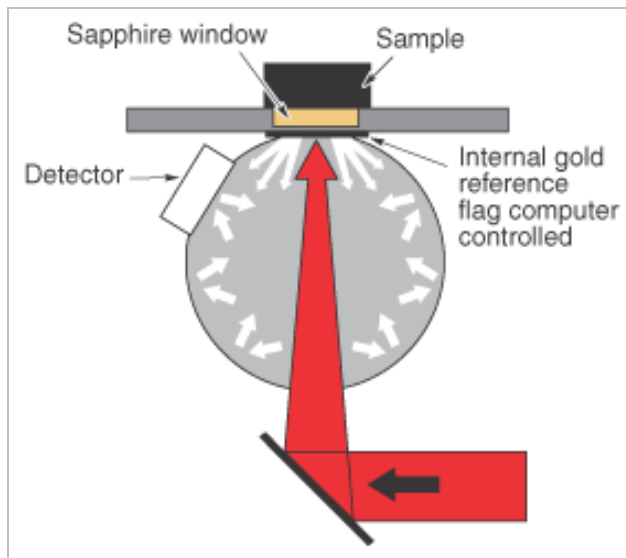
- Pinned-in-place, permanently aligned optics.
- Fully integrated design.
- Snap-in installation.
- Automatic purging.
- Automatic recognition.
- Automatic experiment setup.
- Automatic performance checking.
- Multimedia tutorials.

How does it work?

Light enters the integrating sphere from the bottom. The light then strikes the sample and is diffusely reflected. The reflected light is collected by the sphere and directed onto the detector.

The Smart Integrating Sphere can be used with Nicolet x700, Nicolet iS10, Nicolet 380, Avatar , and Nexus model spectrometers.

Figure 1-1: The Smart Integrating Sphere is a highly efficient collector of diffuse scattered radiation



Sampling features

For near-infrared (NIR) applications, the accessory contains an Indium-Gallium-Arsenide (InGaAs) detector. NIR analysis of samples in their original glass or plastic containers is possible, as NIR light passes through these materials without affecting spectra. You can collect spectral data without touching the material, protecting your samples from contamination and yourself from exposure to hazardous materials.

No pressure device or other mechanism is needed to bring samples into contact with the window. This patented design ensures efficiency in collecting the back-scattered radiation.

Standard sampling interface

The large top surface of the Integrating Sphere allows unobstructed access to the sampling window. Light from the spectrometer passes through the window to the sample. Back-scatter light returns to the detector through the sampling window, as well.

CAUTION



Use of this accessory and its controls in a manner other than specified by the manufacturer may result hazardous radiation exposure or impaired protection

Optional sample spinner

The optional sample spinner allows a wide area from inhomogeneous materials, like pellets or powders, to be analyzed.

A switch on the front panel of the Integrating Sphere enables rotation. The internal flag position and data collection functions are controlled through the OMNIC software. Enabling the "Sample Shuttle" option enables the automatic controls.

Compliance and validation

The integrating sphere can be validated to confirm USP 1119 compliance.

For NIR applications, a Spectralon™ sample is used to validate intensity and wave length accuracy. These checks ensure the unit can operate in regulated environments.

Specifications

The Smart Integrating Sphere features:

- Crystal material: Sapphire.
- Spectral range: 4000 to 10,000 cm
- Detector: Indium Gallium Arsenide (InGaAs) for NIR applications

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- Internal coating: All gold
 - Top plate material: Cast aluminum
 - Reference material: Internal gold flag
 - Option: Sample spinner

Temperature and humidity

Maintain the ambient temperature between 16° and 27°C (60° and 80°F). For best performance, keep the temperature between 20° and 22°C (68° and 72°F).

Maintain the environmental humidity between 20% to 80% noncondensing. Avoid rapid changes in temperature that may cause condensation.

Whenever the integrating sphere has been stored or shipped, immediate exposure to room air can cause condensation. Allow 24 hours for the package to slowly warm (or cool) to room temperature before opening the protective packaging.

Static electricity

If you have trouble with static electricity in your laboratory, follow these guidelines:

- Maintain the ambient humidity in the between 20% o 80% noncondensing.
- Use conducting carpet or place antistatic mats over conventional
- Avoid plastic and synthetic furniture that allows a build-up of large static potentials.
- Wear natural fiber clothing.
- Use a grounding strap.

Vibration and magnetic fields

Neither floor vibration or acoustical noise will damage your system, but they can affect performance. Minimize or eliminate noise and vibration.

Intense magnetic fields, such as those produced by superconducting magnets, can affect performance. The system should be at least 5.5 meters (18 feet) away from these fields. Minimize or eliminate exposure to magnetic fields.

Ventilation

Many standard spectroscopy methods use solvents during sample preparation. The pyrolysis of these solvents by an infrared source may produce hydrochloric acid, hydrofluoric acid, or phosgene.

Sample materials dissolved in solvents can be measured using an integrating sphere, but take care to prevent damage by caustic agents or their vapors. Be sure your work space is well ventilated, particularly if you are working with toxic or volatile samples and/or solvents. Damage to the spectrometer and/or accessories caused by the use of caustic agents is not covered by the spectrometer warranty.

WARNING

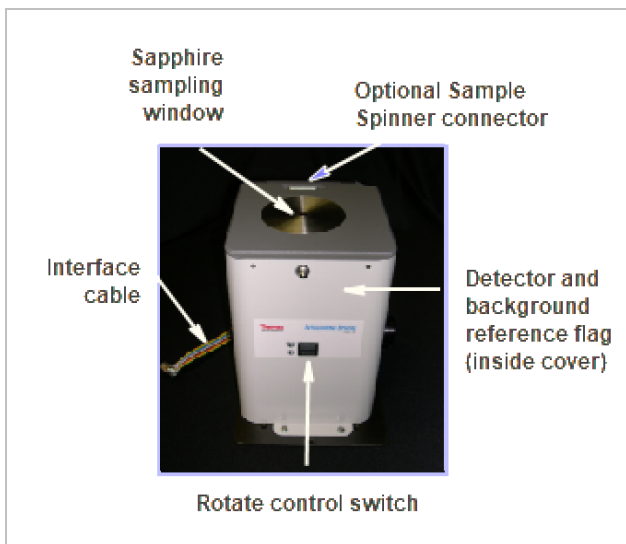


Hydrochloric acid, hydrofluoric acid and phosgene are toxic. If you use solvents containing halogenated hydrocarbons, be sure your work area is well ventilated

Components

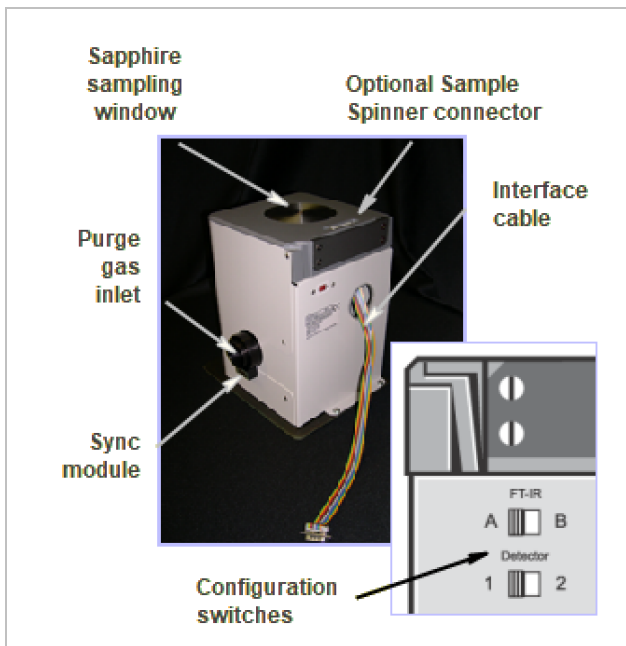
Standard components Take a minute to familiarize yourself with the Integrating Sphere components. From the front, you will find

- the sampling window
- detector and background reference flag
- optional Sample Spinner connector and rotate control switch
- spectrometer interface connector and cable



On the rear and side panel, you will find:

- purge gas inlet
- optional sample spinner connector
- spectrometer model and detector configuration switches
- spectrometer interface connector and cable
- Sync™ module



Operating Precautions

Maximizing accessory life

The Integrating Sphere is very robust, but a few precautions are necessary to maximize its life:

- Use a damp (not wet), soft cloth and a mild soap to clean the cover and other metal parts.
- Do not use harsh detergents, solvents, chemicals or abrasives; these can damage the finish.
- Use solvents that do not react with sapphire crystals when cleaning the sampling window.
- Use care when installing samples. Avoid scraping the window with samples or sample containers.

OMNIC Software Basics

Using OMNIC software

Thermo Scientific's OMNIC™ software is an advanced software package for FT-IR spectroscopy that runs on the Microsoft® Windows® operating systems.

With OMNIC you can perform a wide range of tasks, from collecting infrared spectra to performing quantitative analysis. The commands needed to collect and process spectra are conveniently arranged in menus and can also be entered from the keyboard.

For instructions on starting OMNIC, refer to the box at the left.

How to start the OMNIC software:

The methods you can use to start OMNIC depend on the version of Windows you are using.

[Click here to learn how to start OMNIC.](#)

For information on other methods of starting applications and more detailed instructions on using Windows features, see your Windows documentation.

After you start OMNIC, the OMNIC window appears on the display.

Installing a Smart Integrating Sphere

Checking spectrometer performance

Before you install the integrating sphere for the first time, we recommend that you check the spectrometer performance.

Going forward, for optimal performance, we recommend that you run the performance test at least once a week.

These tests are designed to verify your system's performance without an accessory installed.

Smart Integrating Sphere

If the spectrometer performance test results are acceptable, follow the instructions at the left to install the Smart Integrating Sphere.

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

Before you can use the Smart Integrating Sphere, it must be configured for the spectrometer you are using. Two switches, labeled FT-IR and Detector, on the back panel configure the accessory.

The Smart Integrating Sphere receives power and control signals through the spectrometer interface cable which is connected to the spectrometer through the Detector/Motors connector on the rear wall of the sample compartment.

The connector is keyed so you cannot plug it in backwards. Be sure the connector is firmly seated before continuing on to the next step.

The integrating sphere fits snugly into the sample compartment. If necessary, gently rock the accessory until it is firmly seated in the cradle and the Sync module is in contact with the connector on the side panel of the sample compartment.

Once it is in place, the Integrating Sphere requires no adjustment. The optical components are pinned in place and permanently aligned.

How to install a Smart Integrating Sphere:

1. Prepare the spectrometer.
 - Turn on the spectrometer power.
 - If the sample compartment cover is attached, remove it.
 - If the side wall adapters are in place inside the sample compartment, remove them.
 - If you were using any of the connectors at the back of the sample compartment, remove the cables.
 - Remove the Snap-in™ baseplate or any other accessories installed inside the sample compartment.
2. Configure the integrating sphere for your model. Locate the spectrometer model configuration switch on the back of Integrating Sphere. The switch is labeled FT-IR.

Switch setting	Spectrometer model
A	Avatar (all), Nicolet 380, Nicolet x700, Nicolet 8700 USB
B	Nicolet iS10, Nexus (all), Nicolet 8700

3. Configure the integrating sphere for your detector. Locate the detector configuration switch on the back of the Integrating Sphere. The switch is labeled Detector. Set the switch:

Switch setting	Spectrometer model
1	Nexus 870, Nicolet 8700
2	Avatar (all), Nexus (all other), Nicolet iS10, Nicolet 380, Nicolet x700, Nicolet 8700 USB

4. Connect the spectrometer interface cable. Set the accessory near the spectrometer sample compartment with the ribbon cable toward the rearwall of the sample compartment. Align the pins on the cable with the connector on the back panel and press straight in.

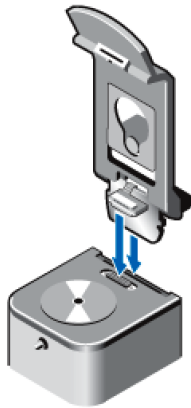
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5. Lower the accessory in the sample compartment. The Integrating Sphere fits into guides on the sidewalls of the sample compartment. As you lower it into place, be sure that the accessory is firmly seated and that the cable is not been stretched or crimped.

How to install an optional sample spinner

If your Smart Integrating Sphere is equipped with an optional sample spinner, follow the instructions at the left to install it.

To install the spinner you will:

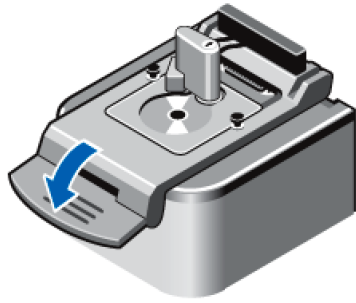
- Insert the hinge tab
 - Lower the spinner onto the sampling surface
 - Latch the spinner in place
1. Install the tab on the spinner into the slot on the Integrating Sphere. Make sure the hinge tab on the spinner is aligned with the slot on the sampling surface.



2. Carefully lower the spinner onto the sample surface.



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3. Latch the spinner in place. Press down on the latch until it clicks into place.



Opening an Experiment

When you install a Smart Accessory, the system automatically opens the experiment files that are associated with the accessory.

Each experiment file contains a complete set of parameters, which have already been optimized for collecting data with the accessory. There is no need for you to set the parameters individually.

To select the default experiment

After you install a Smart Accessory, its name and the title of its associated experiment are displayed in a dialog box. If multiple experiments exist for the accessory, a list box is provided so you can select the experiment you want to open.

Choose a background option

If you collected a background spectrum before installing your Smart Integrating Sphere, the Use current background option appears in the Smart Accessory Change dialog box.

This option lets you associate the current background with the new experiment.

Leave the check box blank if you plan to collect a new background (recommended). The current background spectrum will be deleted.

When you are finished selecting the experiment and setting the background option, choose OK to close the Experiment Accessory Change dialog box.

Running the performance test

Each time you install a Smart Accessory, the system automatically runs a series of diagnostic tests to evaluate its performance with the new accessory.

The spectral quality checking features of your OMNIC software specify the criteria used for the performance test.

The performance diagnostics continue working in the background while the accessory is installed, ensuring high quality spectra time after time.

The performance test starts automatically when you are finished opening an experiment. When the test is completed, a dialog box shows you the results.

Look for the check mark in the green box, which tells you the system passed the performance test and is ready to collect data.

If the accessory is set up incorrectly or the system does not meet the performance criteria, a message appears along with instructions on how to verify and fix the problem.

When a problem occurs, the Redo Test button allows you to restart the test.

Be sure to follow the instructions to correct the problem before you click Redo Test.

Measuring a Background

What is a 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 Smart Integrating Sphere accessory and the spectrometer. Each sample spectrum is ratioed against a background so the final spectrum is free of these features.

Follow the steps at the left to collect a background spectrum.

How to collect a background spectrum:

1. Confirm that the Sample Shuttle option on the Bench tab in Experiment Setup is enabled.

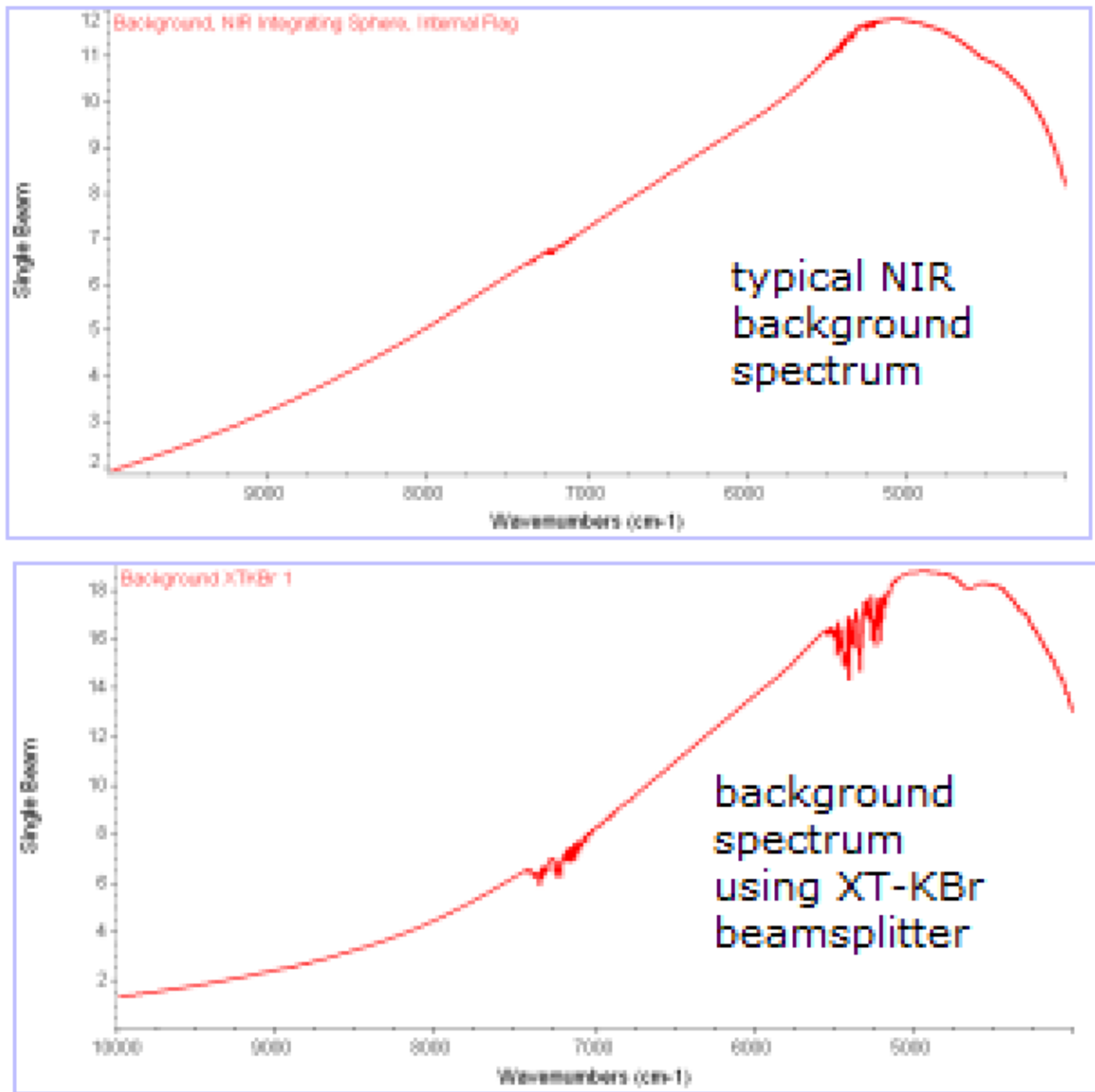
With the Sample shuttle option enabled, an internal gold reference automatically moves into the beampath during background data collection. When you are ready to collect sample data, the flag automatically moves to its storage position.

2. Collect a background spectrum. Click the Collect Background button on the toolbar. If the toolbar is not displayed, choose Collect Background from the OMNIC Collect menu.

Background spectrum

The new background spectrum is displayed in the Collect Background window. The background spectrum remains in memory and is selected as the current background. It will be used to process all of the sample spectra you collect until you replace it by collecting another background.

Figure 1-2: Background spectra collected through the sapphire sampling window using the internal gold reference



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 conditions described at the left are true, remeasure the background immediately.

If one of the following statements 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 Collect, Bench or Advanced settings in the selected experiment (except Gain, Final Format, Number Of Scans or Correction).
- 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).

Installing a Sample

When background collection is completed, the software prompts you to insert the sample. Installing the sample is quite simple - just place the sample atop the sapphire window and start data collection.

Powders, pellets, gels and granulated samples work well. Clear liquids do not. Tablets and other solids can also be analyzed easily.

Installing a solid homogeneous sample

When prompted to install the sample, simply place the sample atop the Integrating Sphere. The sample need not be removed from any transparent (plastic or glass) container.

Installing a nonhomogeneous powder, gel, pellet, or liquid sample

The sample spinner allows a larger surface area of the sample to be examined.

To use the optional sample spinner, fill the cup at least half full. Before you begin data collection, move the switch on the front panel to the rotating sample position. The cup will begin to rotate.

When using the sample spinner, you should use a longer collection time, as a short collection time negates the effect of the spinning.

Measuring the Sample

Collecting the spectrum

Once the sample is positioned on the crystal, you are ready to start collecting the sample data. The sample measurement shows how the energy you started with was reduced by the sample.

Use the Collect Sample button on the toolbar or spectrometer to start data collection. Then follow the instructions displayed on your screen to measure the sample.

The instructions that appear differ depending on how the Background Handling and other parameters and options are set in OMNIC and in the selected experiment.

How to collect a sample spectrum: Choose the Collect Sample button on the toolbar.

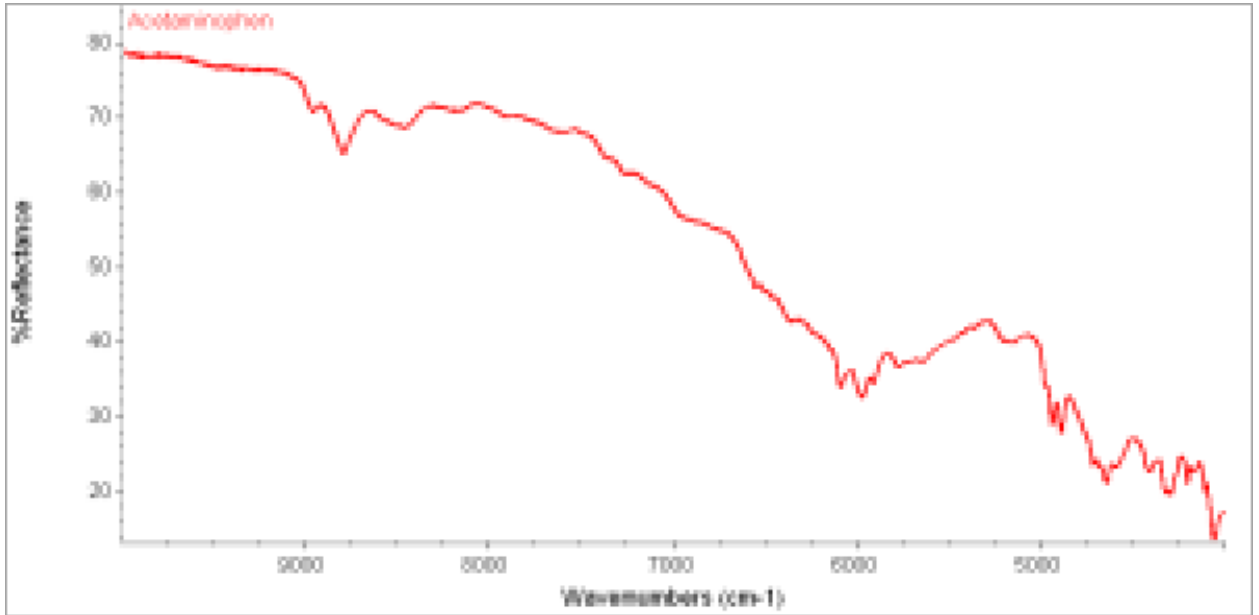
If the toolbar is not displayed, choose Collect Sample from the Collect menu.

Sample NIR spectrum

When % reflectance is selected as the final format of your sample spectra— that's how it's set up in the standard Smart Integrating Sphere NIR experiment—OMNIC collects a four scans and then calculates and displays a reflection spectrum. The spectrum is updated every four scans, as new data are collected.

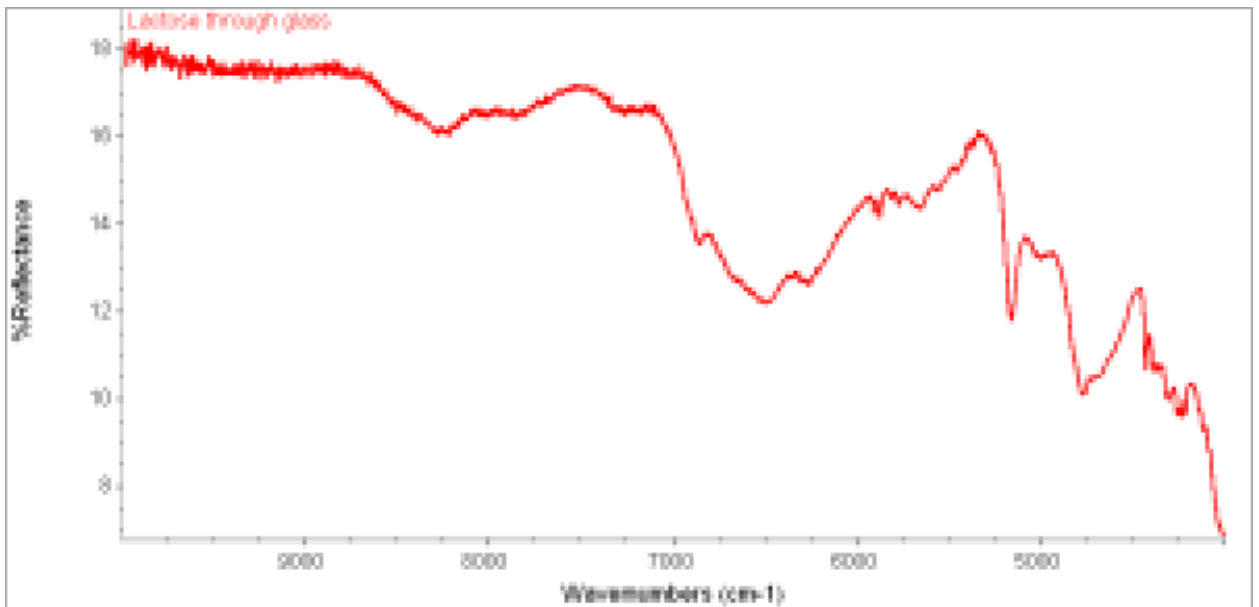
When the system has finished collecting the number of scans specified in the experiment, the final spectrum is displayed in a window.

Figure 1-3: NIR spectrum of acetaminophen



The final spectrum shows only the change in the IR energy relative to the background. You can then use the various processing options within OMNIC to prevent artifacts from overwhelming the signals.

Figure 1-4: Lactose spectrum collect while sealed in a glass bottle



Optimizing a spectrum

The Smart Integrating Sphere should work very well with powders or granular materials.

Solid pieces with highly reflective coatings may not work as well.

Liquids tend to have insufficient back scatter to produce useful spectra.

Good test samples include aspirin tablets and small containers with sugar or other granular solid.

How to optimize the spectrum:

When a sample is analyzed through its container, be sure the sample is in a transparent glass or plastic container.

While water vapor interferes to a much lesser extent in the NIR range, purge the instrument if water vapor or other atmospheric contaminants interfere with your data.

To prevent variations in the atmospheric conditions from corrupting your data, collect backgrounds regularly.

Where spectral data is improved by sampling a wider area of the sample material, use an optional sample spinner.

Quantitative Analysis

The Smart Integrating Sphere is an excellent tool for quantitative analysis.

The key is to properly model the variations in path length due to sample granularity. Consistency in sampling is also required.

When you are doing quantitative analysis, it is best to use chemometrics to correct for variations in path length due to different particle sizes and penetration depths.

Cleaning Up

Cleaning the sampling surface

In many cases, the sample does not touch the sampling surface or the sampling window. The material remains in its container. In these cases, there is no need to clean the sample window. In most cases, poorly executed cleaning is more damaging than no cleaning at all.

Good cleaning agents include methanol, ethanol, IPA, and water. Use of soaps or other surfactants is discouraged, as these can leave a residue bound to the sapphire. Acetone is not a good choice long term, as it may dissolve adhesives and allow the crystal to come loose. Chemical wipes are usually not a good idea, as embedded clays can make these slightly abrasive.

How to clean the sampling plate and crystal:

1. Remove the sample. If you were using the spinner, move the switch to the stationary position. Gently push the motor assembly toward the back of the sample compartment until the spinner disengages. Lift the cup out of the spinner.
2. Gently clean the sampling surface and sapphire window. Moisten a tissue or cotton ball with an appropriate cleaning agent, then clean the crystal. Dry the crystal with unscented tissue or cotton. Avoid tissues that have been treated with aloe or other additives.

NOTICE

The cleaning fluid should be at or close to room temperature (or the temperature of the previous sample). Applying extremely hot or cold liquids may crack the crystal.

Removing a Smart Integrating Sphere

You can remove the Smart Integrating Sphere accessory as easily as you installed it. Follow the instructions at the left.

When not in use, your accessory should be stored in a dust-free environment such as a cabinet or box. Do not use adhesive tape to protect the sampling window, as the adhesive may absorb radiation and contaminate your sample.

How to remove the Smart Integrating Sphere:

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1. Ensure the crystal area is clean. Remove the sample and clean the sampling window, as described in the "Cleaning Up" section of this tutorial.
 2. Remove the Smart Integrating Sphere. Firmly grasp the sides of the accessory and then lift the accessory straight up until it clears the cradles. Remember that ribbon cables connect the accessory to the connectors on the back wall of the sample compartment. Without stretching or crimping the cable, carefully set the Integrating Sphere near the sample compartment. Disconnect the ribbon cable from the sample compartment connector. Grasp the connector and not the cable wires and pull straight out.
 3. Open an experiment. After you remove a Smart Accessory, a dialog box appears allowing you to select the experiment for your next analysis. Select a title from the drop-down listbox to open the experiment and then choose OK.

Where to go Next

OMNIC software includes on-line documentation for many different sampling techniques.

If you need information on another sampling technique or accessory, open Sampling Techniques from the OMNIC Help menu and then choose the technique or accessory name.