

Smart SAGA

This tutorial introduces the Smart Apertured Grazing Angle (SAGA) sampling accessory. The SAGA is an advanced grazing angle Smart Accessory™ for Thermo Scientific's Nicolet™ Series FT-IR spectrometers.

Run this tutorial to learn the basics of monolayer/grazing angle sampling with your spectrometer. It explains step by step how to install the SAGA and use the accessory to analyze thin coatings and adsorbed species on metal substrates. You will also learn how to properly maintain and store the accessory.

Product features

The SAGA accessory is designed for the analysis of thin films on reflective substrates. The 80 degree angle of incidence for this reflection-absorption accessory allows sensitive measurements of submicron films (1 micrometer down to 10 angstroms).

A built-in polarizer reduces the 's' polarized light which does not contribute to the spectral data resulting in even greater sensitivity.

The SAGA includes an integrated aperture assembly with four different openings for the isolation of small samples. All optics are gold-coated for maximum reflectivity.

The SAGA is part of Thermo Scientific's 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 experiment setup
 - Automatic performance checking
 - Multimedia tutorials.

Product specifications

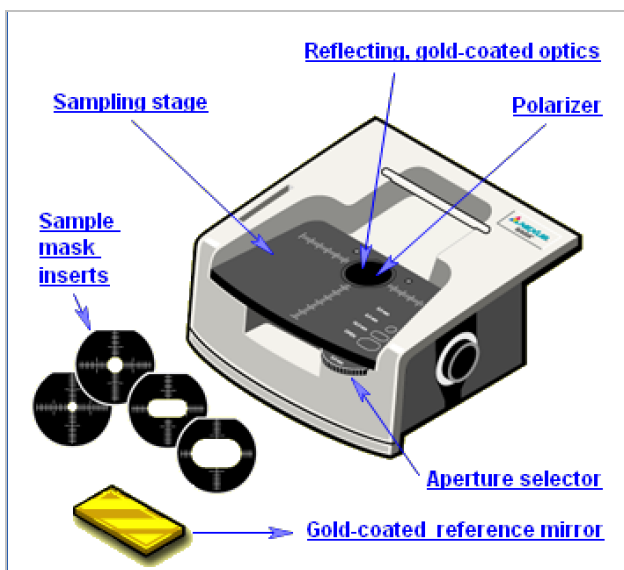
The Smart Apertured Grazing Angle accessory features:

- **Angle of incidence:** 80 degrees
- **Aperture settings:** circular active areas of 5 mm and 8 mm, oval active area of 16 mm by 8 mm and open size of 18 mm by 8 mm
- **Sampling masks:** sizes correspond to above aperture sizes
- **Spectral range:** 4000 to 475 cm^{-1}
- **Integrated Polarizer:** enhances sensitivity for grazing angle measurements

Product components

The SAGA features all reflecting, goldcoated optics. The aperture assembly provides two circular active areas, one oval and an open setting. This new design eliminates the need to sputter the apertures, as well as the undesired scattering effect of other grazing angle accessories. Three sample mask inserts are included for use as guides in the isolation of sample areas.

The integrated polarizer is positioned within the accessory to protect it from contamination and atmospheric conditions. A laser engraved grid on the sampling stage allows reproducible positioning for quantitative measurements.



Component	Description
Sampling stage	The sample stage is engraved with a grid that allows reproducible positioning for quantitative measurements. The sampling stage is coated with Teflon* to protect it from sample materials that may be corrosive.
Reflecting, gold-coated optics	The optics within the SAGA direct the beam to the sample at an 80 degree angle of incidence. All of the optics within the SAGA are gold-coated for maximum reflectivity
Polarizer	The build-in polarizer reduces the 's' polarized light which does not contribute to the spectral data, resulting in even greater sensitivity. Because the polarizer is internal, it is not exposed to contamination from being handled in the external environment.

Component	Description
Aperture selector	The aperture selector allows you to select the size and shape of the active area. Four possible settings allow you to choose from two circular active areas of varying sizes, one oval active area and an open setting. Decreasing the aperture setting focuses the beam on a smaller area of the sample. By focusing the beam so that only the area of interest is sampled, you can analyze samples whose size or area of interest is smaller than the opening in the sampling stage with excellent results. There is no need to sputter the apertures, nor will your results be degraded by the scattering effects of other grazing angle accessories.
Gold-coated reference mirror	The SAGA includes a gold-coated reference mirror. The reference mirror is useful for collecting background data when an uncoated substrate is unavailable or produces anomalous results. The reference mirror is also used to test the SAGA's performance.
Sample mask inserts	The sample mask inserts allow you to define the area of interest on the sample. This allows you to analyze samples whose size or area of interest is smaller than the opening in the sampling stage. For best results, the sample should completely cover the opening in the sample mask. The sampling masks, used with the corresponding aperture setting, direct the beam so that only the area of interest is sampled.

*Teflon is a registered trademark of Du Pont.

NOTICE

Do not allow dust or other contaminants to enter the sample opening, which can permanently damage the optics within the accessory. Cover the opening with a sheet of paper or cardboard when not in use.

Never touch the mirrored surface of the gold-coated reference mirror. This will permanently damage the mirror.

OMNIC Software

Thermo Scientific's OMNIC™ Software is an advanced software package for FT- IR spectroscopy that you can run using Windows®.

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.

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.

System performance

Run the performance test on your spectrometer at least once a week to verify your system's performance without an accessory installed.

See the documentation that came with your spectrometer for instructions on how to check individual spectrometer components.

Installing the SAGA

If the spectrometer performance results are acceptable, follow the instructions to install the SAGA Accessory in the sample compartment of your Nicolet Series spectrometer. The accessory fits into guides on the side walls of the sample compartment.

These accessories are aligned during the manufacturing process and they require no further adjustment. The optical components are mounted in a sealed unit, which automatically connects to the purge system in your Nicolet Series spectrometer.

To install the SAGA

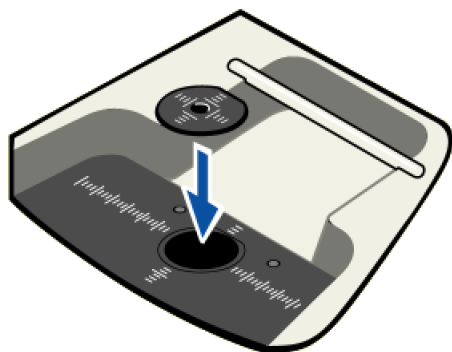
1. Make sure the spectrometer is turned on.
2. If the sample compartment cover is attached, remove it.
3. If the side wall adapters are in place inside the sample compartment, remove them.
4. If you were using any of the connectors at the back of the sample compartment, remove the cables.
5. Remove the 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 the sample masks

Masks are provided to help guide you in targeting the desired area on your sample. The mask inserts fit into the recessed area on the sampling stage. The sample should completely cover the opening in the mask. The area of the sample that shows through the opening of the mask will be sampled. You should choose the mask with the largest sample area which your sample is able to cover. The smaller masks are useful for small samples. The mask inserts correlate with the size chosen on the aperture dial. When no aperture is required, the largest mask insert may still be used.

How to install the sample mask

Place the sample mask over the circular opening of the sampling stage. The mask should fit securely in the recessed area around the opening.



Selecting an aperture

The dial on the front of the sampling stage allows you to select an aperture for your experiment. The aperture focuses the beam, determining the active area where the beam interacts with the sample. Four settings allow you to choose from circular active areas of 5mm or 8mm, and an oval active area of 16mm by 8mm or open. You should choose the largest active area that your sample allows. The smaller settings are used when the size or condition of your sample allows only a small area to be sampled. Make sure you install the sample mask that corresponds with the aperture you select.

How to select an aperture

Rotate the dial on the front of the sampling stage to select the desired aperture. The labels on the dial indicate the four possible settings: 5mm, 8mm, 16mm x 8mm or open. When the dial clicks into place, the indicated aperture is engaged.

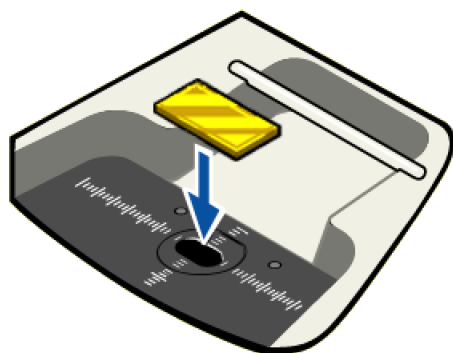


Installing the reference mirror

The gold-coated reference mirror must be installed for the performance test that follows. For the performance test, set the aperture to open with the largest mask or no mask installed. Once the performance test is passed, change the aperture and mask to what will be used to measure the sample.

How to install the reference mirror

Place the reference mirror face down on the sampling stage. The mirror should completely cover the opening in the mask or sampling stage if no mask is being used.



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.

How 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.

How to select from a list of experiments

If multiple experiments exist for the accessory, a list box is provided so you can select the experiment you want to open.

Click the arrow in the list box to see the titles of the experiments for the currently installed accessory.

Click a title to select the experiment

How to set the background option

If you collected a background spectrum before installing your SAGA Smart Accessory, the following option appears in the Smart Accessory Experiment 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 choosing the experiment and setting the background option, choose OK.

Running the performance test

After a Smart Accessory is installed, 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.

Performance test passed

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 tests and is ready to collect data.

Performance test failed

If the system does not meet the performance criteria (it won't if you didn't install the gold-coated reference mirror and set the aperture to open), a message appears along with instructions on how to verify and fix the problem

How to rerun the performance test

The next screen indicates that a problem occurred with the performance test and allows you to restart the test. Follow the instructions to correct the problem. Then choose Redo Test to rerun the performance test.

Make sure the mirror is installed face down on the SAGA and the aperture is set to open. Then [click here](#) to rerun the test.

Measuring the background

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

For best results when measuring coated samples, use the same substrate as the sample, but without the coating to eliminate the spectral features of the substrate. If no substrate is available or produces undesirable results, use the gold reference mirror.

The quality of the sample spectra you collect will be enhanced if the sample spectra are processed with an appropriate background. [Click here for tips on collecting representative backgrounds.](#)

How to measure the background

With OMNIC software

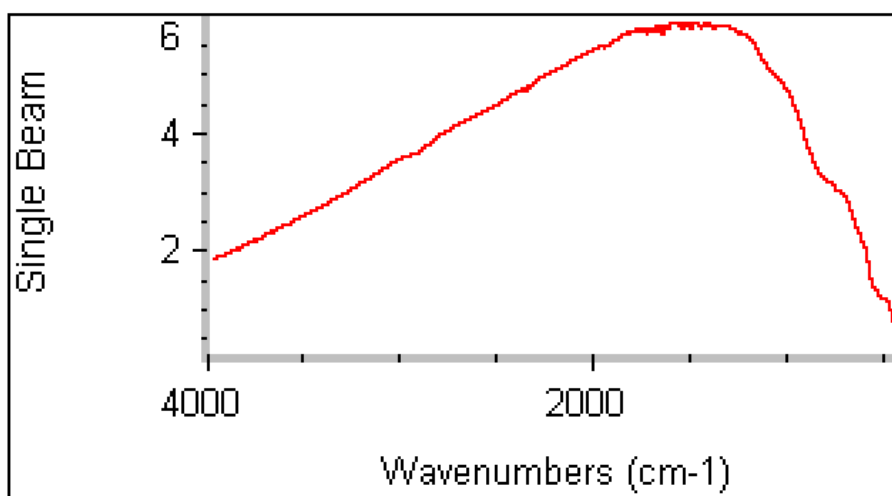
1. Select the desired aperture for the experiment and insert the corresponding sample mask.
2. Place the gold-coated reference mirror or other background material face down on the sampling stage.

The reference mirror or substrate should completely cover the active area.

3. Collect a background spectrum. Use the Collect Background command in the OMNIC Collect menu or click the Collect Background button on the toolbar, if it is displayed.

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.

Here is what a background spectrum collected with the gold-coated reference mirror looks like.



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

- You changed a component in your spectrometer or sampling accessory (especially the aperture or mask).
- 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, you are ready to insert the sample.

Samples must have a flat surface so they lie flat on the sampling stage. For best results, the sample should completely cover the opening in the sample mask. If you need to change to a different mask, stop and collect a new background spectrum before measuring the sample. Make sure the aperture setting matches the sample mask when collecting background and sample data.

The grid on the sampling stage allows reproducible sample placement.

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

Use the Collect Sample command in the OMNIC Collect menu to start data collection. Then follow the instructions displayed on your screen to measure the sample.

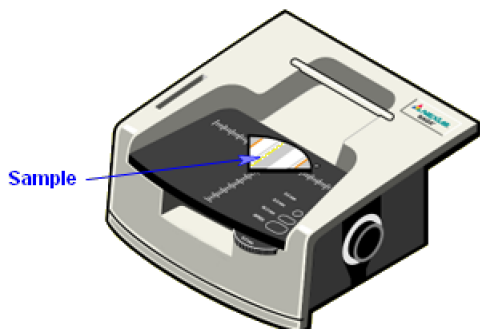
The instructions that appear will differ depending on how the Background Handling and other parameters and options are set in OMNIC and in the selected experiment.

How to install and measure a sample

With OMNIC software

1. Position the sample so the area to be analyzed is over the opening in the sample mask.

The sample should completely cover the opening in the sample mask.



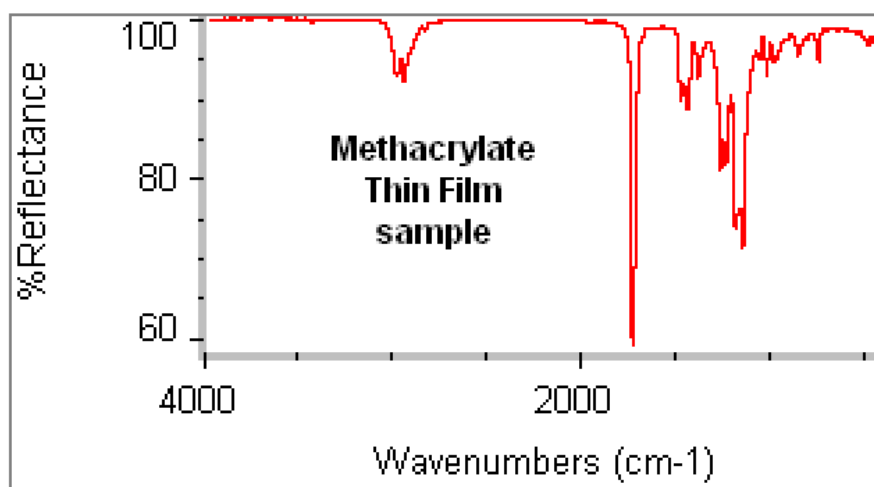
-
2. Use the Collect Sample command in the OMNIC Collect menu or click the Collect Sample button on the toolbar, if it is displayed, to start sample collection.

The sample spectrum

When % reflectance is selected as the final format of your sample spectra—that's how it's set up in the standard SAGA experiment—OMNIC collects a few scans and then calculates and displays a % reflectance spectrum. The spectrum is updated 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. The final spectrum shows only the change in IR energy (the background energy has been removed).

This is a spectrum of a methacrylate thin film sample. The gold-coated reference mirror background spectrum was used to process the sample data.

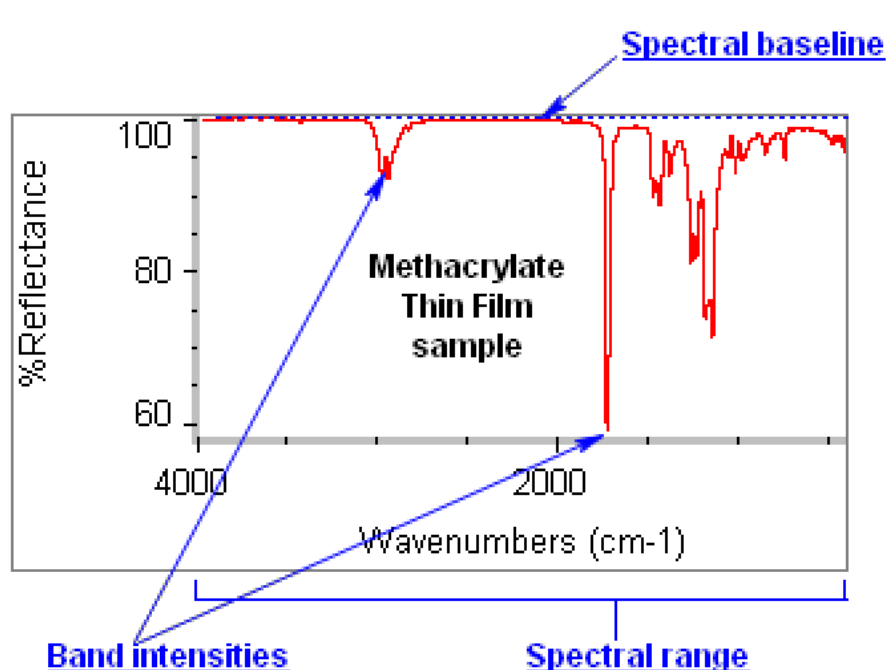


External reflectance spectrum

Grazing Angle reflection-absorption is a type of external reflectance. An external reflectance spectrum is similar to a transmission spectrum in that the locations and intensities of the spectral bands will be unique for a particular sample material. You must be careful, however, when comparing spectra because the shapes and intensities of the bands can be different.

The illustration highlights the primary characteristics of a grazing angle reflectance-absorption spectrum.

Here is the spectrum of a methacrylate thin film sample. The spectrum is shown in %reflectance, which is the typical format for displaying this type of mid-infrared data.

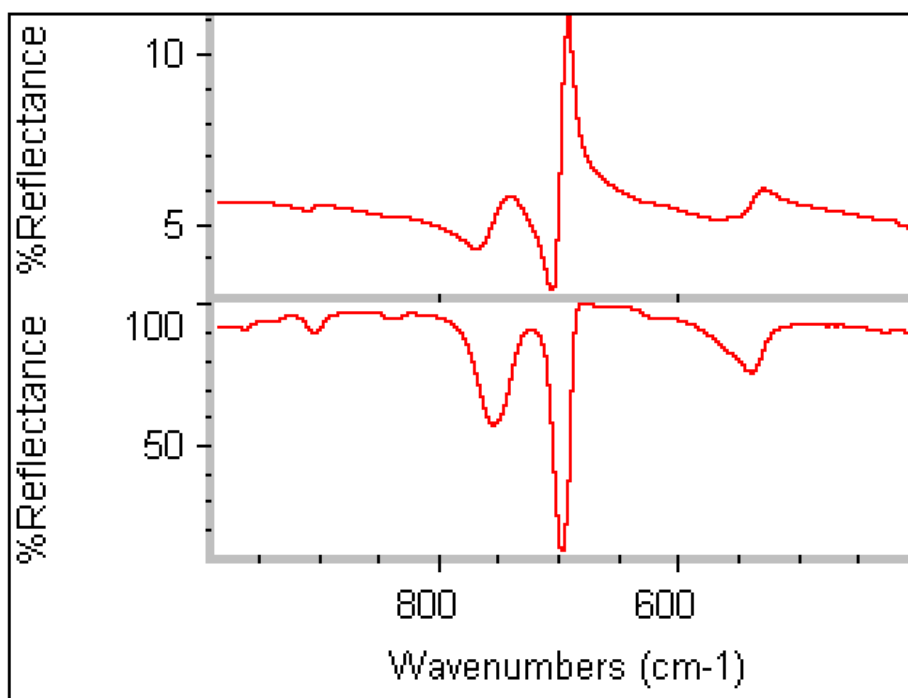


Correcting the derivative-shaped peaks

Spectra collected using a reflection technique such as specular reflection or diffuse reflection may have derivativeshaped or inverted peaks (see example at left). This is caused by optical dispersion from the specular (nonabsorbing) component of the reflected light.

Kramers-Kronig in OMNIC's Other Corrections dialog box removes the effects of optical dispersion. If you will be comparing your sample spectrum to other spectral data or searching against a spectral library, use the Kramers- Kronig correction to remove any derivative-shaped peaks. See OMNIC help topics for additional information.

This is a specular reflectance spectrum of a plastic:



The top spectrum shows the raw sample data with a derivative-shaped peak. The bottom spectrum shows the same data after the Kramers-Kronig correction was applied.

Quantitative analysis

Careful, reproducible sampling is crucial to accurate quantitative analysis. For best results, place all samples in the same position on the grid.

The grid on the sampling stage allows precise placement of samples for quantitative analysis

Cleaning the sampling stage

If the sampling stage is dirty or contaminated with sample or other material, use the instructions at the left to clean it.

When the stage is clean and dry, you are ready to continue measuring samples.

How to clean the sampling stage

Moisten a towel with acetone or methyl ethyl ketone and wipe the sampling stage. Do not over wet the towel since the solvent may drip inside the accessory and contaminate the polarizer.

NOTICE

Do not allow solvent to drip into the sampling stage opening.

If you clean the sampling stage, allow enough time for the solvent to evaporate completely. To ensure complete evaporation, dry the stage with clean, dry air or nitrogen.

Removing the SAGA

You can remove the SAGA 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.

How to remove the SAGA

1. If a sample is positioned on the SAGA sampling area, remove the sample.
2. Gently pull up on the SAGA accessory to release it. Continue lifting the accessory straight up until it is completely free of the sample compartment.
3. Open an experiment. After you remove a Smart Accessory, a dialog box appears allowing you to select the experiment for your next analysis.

Click the arrow in the list box to see the titles of the experiments available in OMNIC. Click a title to open the experiment.

Where to go next

Your OMNIC software includes many kinds of on-line documentation to give you easy access to useful information.

To learn more about FT-IR spectrometers and how they work, see [Getting Started and Learning OMNIC](#) in the OMNIC Help menu.

For detailed information about your spectrometer or OMNIC software, see [Spectrometer Help Topics](#) or [OMNIC Help topics](#) in the OMNIC Help menu.

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.

