

Smart Golden Gate

This tutorial introduces the Smart Golden Gate from Thermo Fisher Scientific. The Smart Golden Gate is a horizontal single-reflection diamond ATR Smart Accessory™ for Thermo Scientific Nicolet™ Series FTIR spectrometers.

Run this tutorial to learn the basics of attenuated total reflection (ATR) sampling with your Nicolet Series spectrometer. It explains step by step how to install the Smart Golden Gate in the spectrometer sample compartment and use the accessory to analyze solids, powders and liquids. You will also learn how to properly maintain and store the accessory when it is not being used.

Product features

The Smart Golden Gate is designed to handle a wide range of sample types, including samples that are normally difficult to analyze by ATR such as hard or brittle solids, corrosive liquids and hard powders. The platform is large enough to support macro samples, such as a polymer sheet, yet it provides easy access for measuring samples as small as a single particle or fiber.

The accessory features a diamond ATR crystal bonded at high temperature to a tungsten carbide support. The crystal is extremely durable and stable. The tungsten carbide support can withstand up to 200 pounds of pressure, providing unparalleled sensitivity for a single-reflection ATR element

Smart accessory features

The Smart Golden Gate 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.

Product components

The Smart Golden Gate offers unprecedented sensitivity, ruggedness and versatility for a single-reflection ATR accessory. Samples are placed directly on the diamond ATR crystal for analysis.

The accessory includes two anvils for pressing solid samples against the crystal and a torque wrench for applying pressure reproducibly. An optional air-sensitive anvil is also available for analyzing samples that are hygroscopic or that react with other components in ambient air.

A volatile liquid cover is also included for preventing evaporation of volatile samples during the analysis.

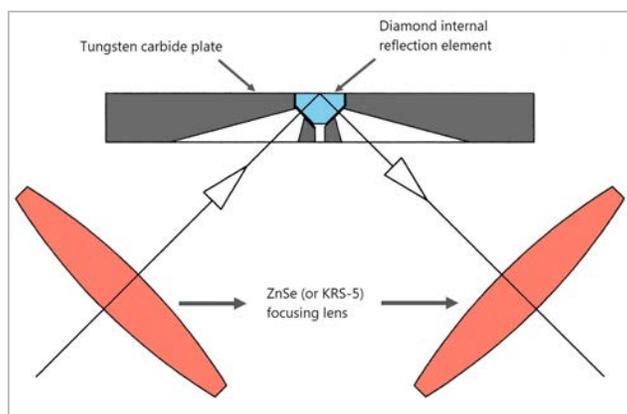
- Anvil screw: This screw is used to apply pressure from the anvil to a solid sample.
- Bridge locking screw: This screw is used to lock the bridge in position for analyzing solid samples.
- Sampling area: The sampling area is a type IIa diamond crystal brazed into a tungsten carbide disc. The sample should be placed in the center of the crystal.
- Anvil: Two anvils are provided with the Smart Golden Gate. The anvils are used to press solid samples against the ATR crystal.
 - Use the self-levelling sapphire anvil to measure powders and solids that have a flat surface.
 - The stainless-steel anvil is designed for irregularly-shaped solids and polymer pellets.
- Torque wrench: A solid sample is initially held in position on the ATR crystal by gently tightening the anvil screw. The torque wrench is then used to apply a specific load to the sample. The torque wrench can be set to apply between 20 and 120 centi-Newton meters of torque (between 33 and 200 pounds of force).

- Top plate removal screws: These screws are used to secure the top plate to the optics. Normally, the top plate does not need to be removed, but when using the optional air-sensitive anvil, a sample can be mounted remote from the instrument.
- Volatile liquid cover: The cover can be placed over a volatile liquid sample to minimize evaporation during the analysis.

Optical design

The Smart Golden Gate's innovative optical design combines a ZnSe (or KRS-5) focusing lens with a type IIa diamond head that is mounted on a tungsten carbide support. The sample material contacts only the diamond portion of the optical unit.

This configuration provides a (diamond) sampling surface that is extremely durable and chemically resistant. The tungsten carbide support adds superior strength (can withstand up to 200 pounds of pressure). This allows intimate contact with any kind of sample, including materials that are extremely hard or brittle. The focusing lens reflects the infrared beam at the proper angle for ATR analysis.



Operating precautions

Care of the diamond crystal

The diamond crystal is extremely durable and scratch resistant so minimal precautions are needed for operating the accessory. It is possible with certain samples to achieve sufficient

contact with the diamond crystal by hand tightening the anvil screw. Heed the precaution described below to avoid damaging the accessory when applying high pressure.

NOTICE

Use only the torque wrench provided with the Golden Gate to apply pressures up to the specified maximum for solid samples (120 cNm or 200 lbs).

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 Golden Gate

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

The Smart Golden Gate 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.

NOTICE

When installing or removing the Golden Gate, lift or press only on the Golden Gate bridge.

❖ To install the Smart Golden Gate

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.

Opening and closing the bridge

Samples are placed directly on the sampling area for analysis. The sampling area is located under the bridge.

Use the bridge locking screw to lock and unlock the bridge.

◆ To open and close the bridge

To open the bridge, turn the locking screw counter clockwise until it swings freely. Then lift the bridge to expose the sampling area.

To close and lock the bridge, lower the bridge and tighten the locking screw fingertight.

Installing the anvil

In order to see features in an ATR spectrum, the sample must be in close contact with the ATR crystal. The Golden Gate anvil is used to apply pressure to powders, pellets, fibers, and other solids to achieve the required contact.

Two types of anvils are provided with the Golden Gate. Use the smaller sapphire anvil to measure powders and solids that have a flat surface. The larger stainless-steel anvil is designed for irregularly-shaped solids and polymer pellets.

The anvil fits on the underside of the bridge. To install an anvil Lift the bridge and press the appropriate anvil onto the anvil mount on the underside of the bridge. The anvil is held in place by the o-ring on the anvil mount.

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.

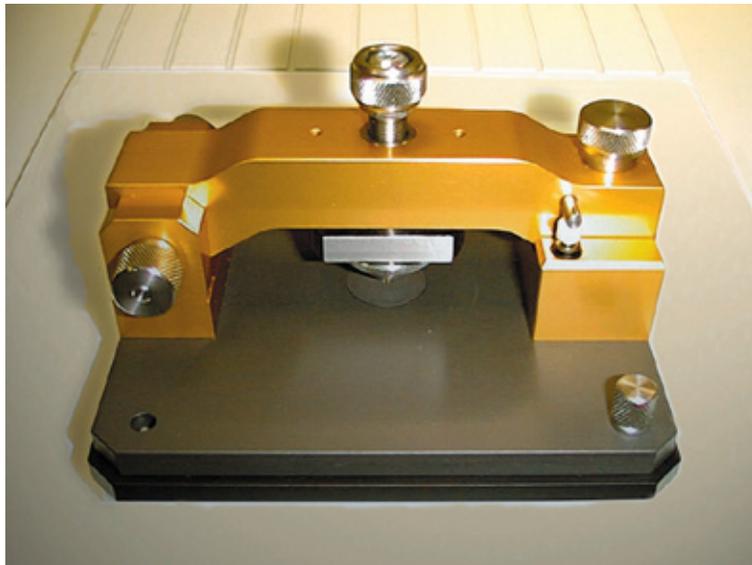
Measuring the background

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 Smart Golden Gate accessory and the spectrometer. Each sample spectrum is ratioed against a background so that the final spectrum is free of these features.

DO NOT place a sample on the crystal when measuring the background. The Golden Gate bridge can be up or down but the anvil should not be touching the ATR crystal during background collection.

❖ **To measure the background**

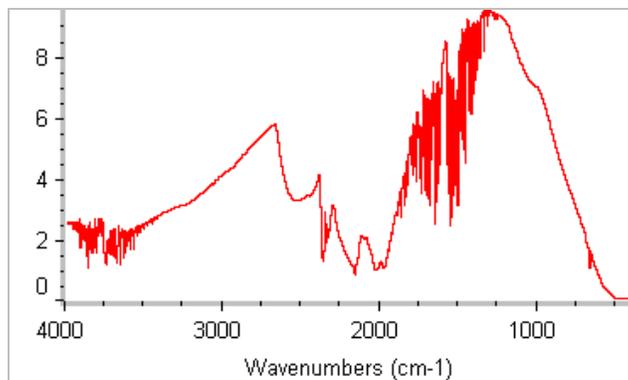
1. If a sample is positioned on the Golden Gate sampling area, remove the sample.
2. Make sure the anvil isn't touching the crystal. Turn the anvil screw counter clockwise to lift the anvil off the crystal."



3. Collect a background spectrum. Click Preview and Measure Background on the dashboard.

The background spectrum will be used to process all of the sample spectra you collect until you replace it by collecting another background.

Figure 1-1: Smart Golden Gate ATR background spectrum



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 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 (especially the Smart Golden Gate crystal).
- 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).

To collect a representative background

Other than Gain, Final Format, Corrections and Sample Scans, the settings used for the background and sample measurements should be exactly the same.

Measuring the sample

Installing a solid sample

When background collection is completed, you are ready to insert the sample.

Solid and powder samples are placed directly on the ATR crystal in the center of the sampling area. The anvil is used to apply pressure to powders, pellets, fibers, and other solids to achieve adequate contact with the ATR crystal. Use the smaller sapphire anvil to measure powders and solids that have a flat surface. The larger stainless-steel anvil is designed for irregularly-shaped solids and polymer pellets.

Follow the instructions at the left to prepare a solid or powder sample for analysis.

❖ **To install a solid sample**

1. Release the bridge by turning the locking screw counter clockwise.
2. Raise the bridge and install the appropriate anvil by pressing it onto the anvil mount.
3. Place the sample on the ATR crystal in the center of the sampling area. For best results, use enough sample to cover the crystal completely (required for quantitative analysis). For micro samples and fibers, place the sample in the center of the crystal."
4. Close the bridge and hand tighten the locking screw.
5. Press the anvil against the sample by turning the anvil screw clockwise by hand.
6. Use the torque wrench to apply a specific pressure to the sample. The torque wrench can be set to apply between 20 and 120 centi-Newton meters of torque (between 33 and 200 pounds of force).

NOTICE

Use only the torque wrench provided with the Golden Gate to apply pressures up to the specified maximum for solid samples.

Installing a liquid sample

The anvil is not required for the analysis of liquids.

To analyze a liquid sample, open the bridge and use a pipette or syringe to place a drop or two of the liquid directly onto the ATR crystal. For best results, the sample should cover the crystal completely (required for quantitative analysis).

If the sample is volatile, place the cover over the sampling area to prevent evaporation during the analysis

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.

❖ **To measure the sample**

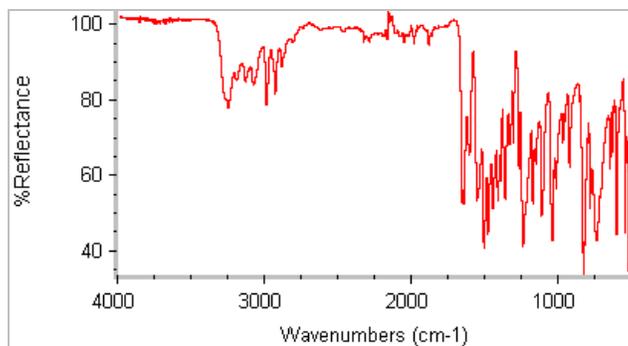
1. Move the sample into the infrared beam. Slide the sample holder out about 3 cm (1 inch). You should feel it click into position.
2. Measure the sample.

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.

This image shows a spectrum of phenacetin powder collected using the Smart Golden Gate.



Cleaning up

Remove the sample and clean the crystal after you finish the analysis. If any sample material remains on the anvil, clean the anvil as well.

When the crystal and anvil are clean and dry, you are ready to continue measuring samples.

❖ **To clean the crystal and anvil**

Wipe the crystal and anvil with a clean cloth. Use soap and water or a suitable solvent, if necessary, to completely remove the sample. Then dry the crystal and anvil with a clean cloth. When cleaning with a solvent, allow enough time for the solvent to evaporate completely.

Since the diamond is very durable, it is okay to use abrasive paper or even a sharp blade to remove a stubborn sample. Follow with a thorough cleaning using a suitable solvent.

Removing the Smart Golden Gate

You can remove the Smart Golden Gate 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 your accessory**

1. If a sample is positioned on the Smart Golden Gate sampling area, remove the sample.
2. Close and lock the bridge by turning the locking screw clockwise.
3. Gently pull up on the Golden Gate bridge to release the accessory. Continue lifting the accessory straight up until it is completely free of the sample compartment.

NOTICE

When installing or removing the Golden Gate, lift or press only on the Golden Gate bridge.

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

