Smart iTR User Guide

This tutorial introduces the Smart iTR diamond attenuated total reflectance (ATR) accessory. The Smart iTR is a rapid sampling Smart Accessory for use with Thermo Scientific Nicolet x700 and iS10 Series Fourier Transform infrared (FT-IR) spectrometers.

Read this tutorial to learn the basics of sampling with your spectrometer. It explains step by step how to install the Smart iTR 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 iTR is a highly efficient A TR sampling accessory that is easy to install and use. The standard configuration includes a universal high pressure lower and diamond ATR crystal with single-bounce optics. Other pressure lowers, crystal materials and optics are also available.

Use of controls or adjustments or performance of procedures other than those specified herein may result in hazardous radiation exposure.

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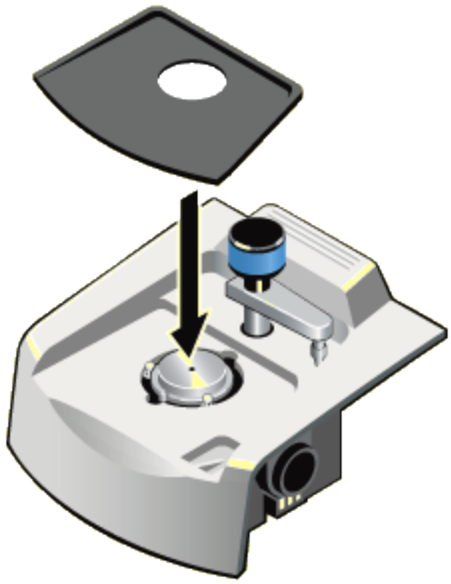
Removable sampling plate

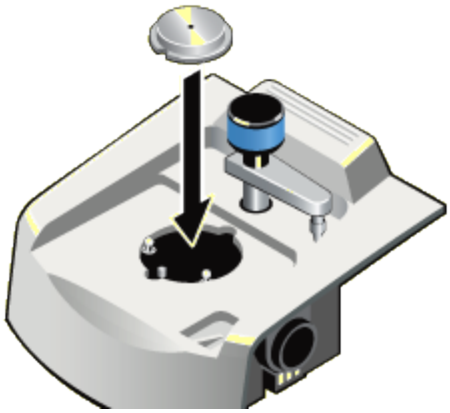
The large open area of the sampling plate permits easy access for loading samples and cleaning. The sampling plate is coated with Teflon' for maintenance and durability with normal use. The sampling plate fits tightly around the stainless-steel crystal plate to protect the accessory's internal optics. The sampling plate is easy lo install and remove for sample disposal and cleaning.



Interchangeable crystal plate

The crystal is mounted in a stainless-steel plate that fits over the optics and seals tightly with the sampling plate. The standard diamond crystal is durable with normal operation and maintenance. Other crystal materials such as germanium and zinc selenide (Zn Se) are available as well as an open-aperture plate for specular reflection sampling.

Crystal plates are pre-aligned and easy to install and remove for cleaning or changing the crystal.



The diamond crystal, while very robust, can be broken if small, very hard materials (like cement or metal beads) are compressed against it at maximum pressure.

Swivel pressure tower

he pressure tower is a mechanical press used lo achieve uniform contact between lhe sample and the ATR crystal. The lower has a swing-away hinge that allows you lo move the tower out of lhe sampling area. This design allows ample room for introducing samples, cleaning the sample area and remo11ing and installing crystal plates.

The standard pressure device applies up to 40 psi of pressure to bring samples into efficient contact with the ATR crystal. The device stops automatically when maximum pressure is achieved to help protect the crystal. Other pressure device options are available.

Exchangeable pressure tips

The pressure tower includes three stainless-steel tip styles that can be used to optimize contact between the sample and the crystal.

Tip	Description
Pointed tip	Use the small pointed tip for flat solids. The pointed tip is ideal for thin samples such as a polymer card or film, and for compressible materials such as urethane foams.

Tip	Description
Concave tip	The concave tip provides better contact with powders and curved solids such as a tablet.
Swivel tip	The swivel tip optimal for solids that have an uneven surface.

Linear absorbance range

The Smart iTR has a short pathlength due to the single-bounce ATR design. This produces absorbance values that are well within the linear absorbance range of the spectrometer, which permits more complete and accurate spectral processing.

This feature also makes the Smart iTR well suited for quantitative measurements of multicomponent mixtures.

Smart Accessory features

The Smart iTR is a member of our extensive family of Smart Accessories for Ni co let Series FT-IR spectrometers. Each Smart Accessory offers 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.

Product specifications

The Smart iTR features:

- Standard crystal material: Laminated diamond mounted in stainless-steel plate Optional crystal material: Ge, ZnSe or open (for specular reflection measurements)
- Sampling plate: Teflon -coated aluminum
- Spectral range: 650-10,000cm
- Refractive index: 2.4@1000cm
- Angle of incidence: 42° (1 bounce)
- Depth of penetration: 2 micrometers at 1000 cm for sample with refractive index of 1.5
- Useful pH range: 1 to 14

Environmental conditions

Store and use your accessory under the environmental conditions explained at the left.

- **Temperature:** Maintain the ambient temperature between 16° and 27°C (60° and SCTF). For better stability, keep the temperature between 20° and 22°C (68° and 72°F). Avoid placing the system near air conditioning or heating ducts or vents or by large windows. Keep other sources of heat, such as hot plates and heating mantles, away from the instrument.
- **Vibration:** Floor vibration or acoustical noise can affect performance. Minimize or eliminate noise and vibration.
- **Dust and particulate matter:** Install the system in a location where it will not be exposed to excessive dust or other particulate matter.
- **Humidity:** Maintain the environmental humidity in the range of 20% to 80% noncondensing. Avoid rapid changes in temperature that may cause condensation. Whenever the accessory has been stored or shipped, immediate exposure to room air can cause condensation damage. Allow 24 hours for the package to slowly warm to room temperature before opening it.
- **Ventilation:** Materials such as hydrochloric acid and hydrofluoric acid, which are highly corrosive, may cause accelerated corrosion of the metallic components in the accessory if the concentration of corrosive gases in the air is excessively high. Be sure to provide storage space for solvents containing halogenated hydrocarbons that is away from the system.

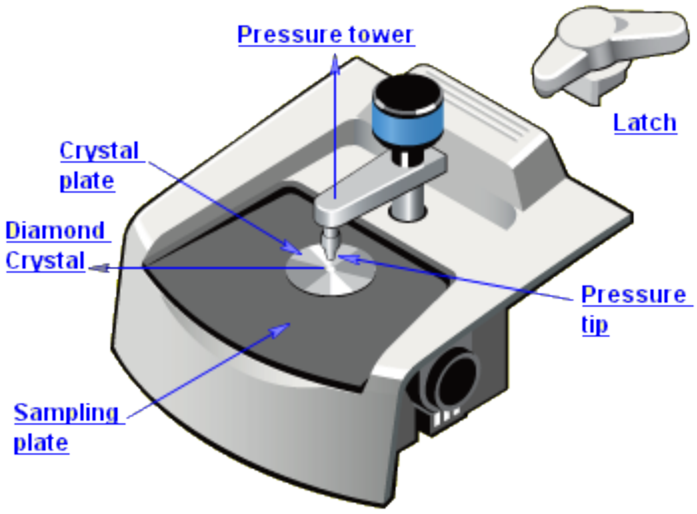
Hydrochloric acid, hydrofluoric acid and phosgene are highly toxic. If you plan to regularly use solvents containing halogenated hydrocarbons, be sure your work area is properly ventilated.

Product components

The Smart iTR can be used to run a variety of samples, including materials that are caustic or abrasive. The accessory comes standard with an optical base, diamond crystal plate, high pressure tower with three tip styles, and an anti-rock clip. The accessory is permanently aligned at the factory, so there is no need for further adjustment.

The Smart iTR interfaces with OMNIC software through the small chip embedded in the right side of the purge tube. The purge tube seals tightly to the sides of the sample compartment to ensure stable operating conditions.

Standard components



Component	Description

Pressure tower	The pressure lower is a mechanical press used to achieve uniform contact between the sample and the ATR crystal. The lower is mounted on a swing-away hinge that allows you to move the lower out of the sampling area. This design allows ample room for introducing samples, cleaning the sample area and removing and installing the sampling plate and crystal plate. The standard pressure device applies up to 40 psi of pressure to bring samples into efficient contact with the ATR crystal. This allows a specified, reproducible amount of pressure to be applied to each measurement. Other pressure device options are available.
Latch	The latch attaches to the spectrometer by a long screw. Before you use the Smart iTR, rotate the latch so that the short stem fits tightly over the back edge of the accessory. The latch ensures the accessory fits securely in the spectrometer, improving reproducibility. Make sure you release the latch before you remove the accessory from the spectrometer sample compartment.
Pressure tip	The standard pressure tower includes three tip styles to maximize contact between the sample and crystal. We provide convenient storage for unused tips in the storage slots under the sampling plate. • Use the small pointed tip for flat solids. The pointed tip is ideal for thin samples such as a polymer card or film, and for compressible samples. • The concave tip provides better contact with powders and curved solids, such as a tablet or polymer bead. • The swivel tip is optimal for solids that have an uneven surface.

Sampling plate	The pressure tower is a mechanical press used to achieve uniform contact between the sample and the ATR crystal. The lower is mounted on a swing-away hinge that allows you to move the lower out of the sampling area. This design allows ample room for introducing samples, cleaning the sample area and removing and installing the sampling plate and crystal plate. The standard pressure device applies up to 40 psi of pressure to bring samples into efficient contact with the ATR crystal. This allows a specified, reproducible amount of pressure to be applied to each measurement. Other pressure device options are available.
Diamond crystal	The single-reflection diamond crystal causes the IR beam to bend and be reflected from its internal surfaces, producing an evanescent wave. When you place a sample in contact with the crystal, the energy from the evanescent wave can obtain absorption information while penetrating only a short distance into the sample. ATR crystals are made from IR-transmitting materials that have a high index of refraction. The diamond crystal has a refractive index of 2.4 @ 1,000 cm and an angle of incidence of 42 degrees. The depth of penetration is approximately 2 micrometers at 1000 cm for a sample with a refractive index of 1.5. The crystal provides a focused, 0.75 mm beam at the crystal surface. For measurements in the mid-IR spectral range, the diamond crystal produces usable data between 10,000 and 650 cm'. Diamond crystals are suitable for both acidic and basic solutions.

Crystal plate	The crystal plate holds the crystal in the proper position for collecting the spectrum of a solid, powder, or liquid sample The crystal plates are stainless-steel to withstand repeated use and cleaning with recommended agents. The crystal plates fit over the optics and seal tightly with the sampling plate to protect the accessory's internal components. Crystal plates are pre-aligned and easy to install and remove for cleaning or changing the crystal. The diamond crystal plate is standard with the Smart iTR. Other crystal plate options are available.

Optional components

The Smart iTR offers a selection of options to customize the accessory for your needs.

Optional crystal plates offer the following ATR crystal choices: ZnSe and Ge. There is also a specular reflection crystal plate that can be used to run 45 degree specular reflectance measurements.

Other options are also available. See your local sales and service office for information.

Maintaining the crystal

Do not use abrasive cleaning agents or pads on any surface of the accessory, including the ATR crystal. Do not use strongly acidic or basic solutions to clean metallic surfaces of the accessory as these will damage the finish.

To maximize the life of the crystal:

- Make sure the samples you analyze and the appropriate solvents will not react with the crystal material.
- Do not scrape the crystal with extremely hard materials such as corundum, or with a knife to remove particles.

Crystal characteristics and compatibility information:

Diamond crystal

Characteristic	Value
Refractive index	2.4
Density (g/cm3)	3.51
Hardness (Knoop number)	7000
Useful pH range	1 to 14
Physical characteristics	hard
Recommended cleaning agents	alcohol, water, toluene
Solvents that attack diamond	concentrated sulfuric acid (h2SO4), K2Cr2O7

Germanium (Ge) crystal

Characteristic	Value
Refractive index	4.0
Density (g/cm3)	5.32
Hardness (Knoop number)	550
Useful pH range	1 to 14
Physical characteristics	hard and brittle, sensitive to temperature, reflection losses

Characteristic	Value
Recommended cleaning agents	alcohol, water, toluene
Solvents that attack diamond	hot sulfuric acid (h2SO4), aqua regia

Zinc selinide (ZnSe) crystal

Characteristic	Value
Refractive index	2.4
Density (g/cm3)	5.27
Hardness (Knoop number)	150
Useful pH range	5 to 9
Physical characteristics	hard, brittle
Recommended cleaning agents	alcohol, water
Solvents that attack diamond	strong acids and alkalies, oxidizing agents, amines, chlorinated solvents such as methylene chloride and chloroform

Using the OMNIC software

Thermo Scientific 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.

After you start OMNIC, the OMNIC window appears on the display. For information about using OMNIC for infrared sampling, open the Help menu in the OMNIC main window and then choose OMNIC Help Topics.

Checking spectrometer 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 or click here to learn how to check individual spectrometer components.

Installing a Smart Accessory

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

Smart Accessories are aligned at the factory and require no further adjustment.

The optical components are mounted in a sealed tube, which automatically connects to the purge system in your infrared spectrometer.

To install a Smart Accessory

1. Prepare the spectrometer.

- Make sure the spectrometer is turned on.
- 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.
- Install the Smart Accessory latch if it is not already installed.

2. Insert the accessory. Lower the accessory into the spectrometer sample compartment and press gently downward until the accessory locks in
3. Rotate the Smart Accessory latch to the lock position. Rotate the latch so the short stem fits tightly over the back edge of the accessory.

   The latch ensures the accessory fits securely in the spectrometer, improving reproducibility. Make sure you release the latch before you remove the accessory from the spectrometer sample compartment.

Other installation options

The optional Thermo Scientific Nicolet iZ10 provides a second sample compartment for the Nicolet iS10 spectrometer. The Nicolet iZ10 can also provide a spectrometer sample compartment for the Nicolet iN10 microscope. The extra sample compartment option can provide flexibility and boost productivity in your laboratory.

The Smart iTR installs in the Nicolet iZ10 module the same way it does in the Nicolet iS10 spectrometer.

Changing the pressure tip

The standard pressure tower includes three tip styles that can be used to maximize contact between the sample and crystal. Follow the instructions at the left if you need to change the tip installed on the pressure tower.

How to change the pressure tip

Do not use tools to install or remove a pressure tip. Using tools may damage the tip.

Do not overtighten the tips.

1. Raise the pressure point by rotating the large pressure control ring counterclockwise.
2. Rotate the pressure tower 90° to the cleaning position.
3. Select the appropriate tip.
4. Install the pressure tip. If a tip is currently installed on the pressure tower, remove it. To remove a tip, use your fingers to turn the tip counterclockwise until it is free of the tower.

   Use your fingers to install the new tip by turning the tip clockwise until it is finger-tight.

   Place the unused tip in one in one of the storage slots under the sampling plate.
5. Reposition the pressure tower.

   Be certain the pressure is fully released before using the hinge to reposition the tower.

   Rotate the pressure tower 90° clockwise until it stops. This is the sampling position.

Installing the crystal plate

Each crystal option for the Smart iTR is mounted on a stainless-steel crystal plate that quickly and easily installs onto the accessory.

Follow the instructions at the left to install a crystal plate. In order for the accessory to pass the ATR performance tests, an ATR crystal must be installed.

Use the color of the crystal to tell the plates apart.

- Diamond is deep red (due to the substrate)
- Zinc selenide (ZnSe) is yellow
- Germanium (Ge) is dark silver

How to install the ATR crystal plate or specular reflectance plate:

1. Raise the pressure point by rotating the large pressure control ring counterclockwise.
2. Rotate the pressure tower 90° to the cleaning position.
3. Remove the sampling plate by lifting the front edge and then raising the plate up and off the accessory.

4. Remove the crystal plate by pressing the plate toward the front of the accessory while you lift the back edge of the plate.

   Then lift the plate up and off the accessory.

5. Install the new crystal plate.

   Align the large notch with the front alignment pin and the small notch with the back pin.

   Lower the front of the plate onto the accessory. Then press forward and down on the back edge until the plate snaps in place. The two ball pins ensure the plate is securely in place.

6. Install the sampling plate.

   Center the sampling plate over the crystal plate.

   Gently press down on the sampling plate until it seals against the O-ring on the crystal plate.

7. Put the pressure device in the sampling position.

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.

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

How to set the background option

If you collected a background spectrum before installing the Smart iTR, the **Use current background** 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.

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 test and is ready to collect data.

Performance test failed

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.

Choose Explain for in-depth information about what went wrong. When you are finished with this dialog box, choose Close.

Rerunning 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 a crystal plate or the specular plate with a gold mirror is installed and then click Redo Test to rerun the performance test.

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 iTR accessory and the spectrometer. Each sample spectrum is ratioed against a background so the final spectrum is free of these features.

How to collect a representative background

Other than Gain, Final Format, Corrections and Number Of Scans, the parameter settings used for the background and sample measurements should be exactly the same. (The settings for the Resolution and Optical Velocity parameters must be the same.)

If you loaded the Smart iTR experiment, all of the data collection parameters have been properly set for collecting background and sample data with your Smart iTR accessory. If you are using another experiment, use the Experiment Setup command in the OMNIC Collect menu to display the Experiment Setup dialog box. Then check the parameters on each of the tabs to make sure they are set to appropriate values for your ATR or specular reflectance experiments.

How to collect a background measurement

1. Prepare the sampling plate to measure the background.
   - ATR

     The same crystal plate must be used to collect the background and the sample. Only the exact same crystal will cancel out the features of the crystal material. If you change crystal plates, you must collect a new background with the new plate.

     Do not place a sample or other material on the crystal when measuring the background. If a pressure device is installed, make sure the pressure point is not touching the crystal. Nothing should be touching the crystal while you are running the background.

     1. Install the crystal plate with the appropriate crystal for your sample.
     2. Install the sampling plate.
     3. Make sure the sampling area is clear.

- Specular reflectance

  Use the gold reference mirror or an uncoated substrate to collect a background spectrum for specular reflectance measurements.

  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 the substrate you have produces undesirable results, use the gold reference mirror.

  1. Install the crystal plate with the open aperture.
  2. Install the sampling plate.
  3. Place the hold reference mirror or other background material face down over the circular opening of the crystal plate. The mirror or background substrate should completely cover the opening.

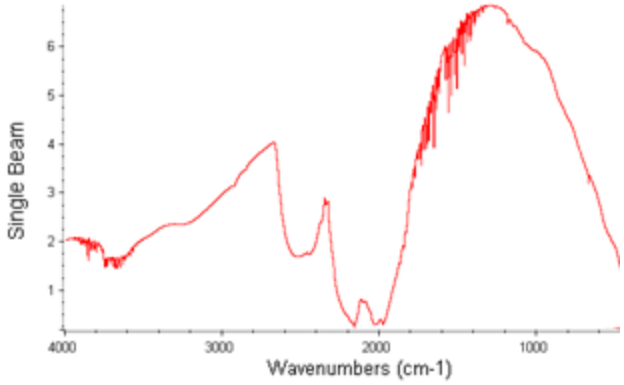
2. Collect a background spectrum

   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.

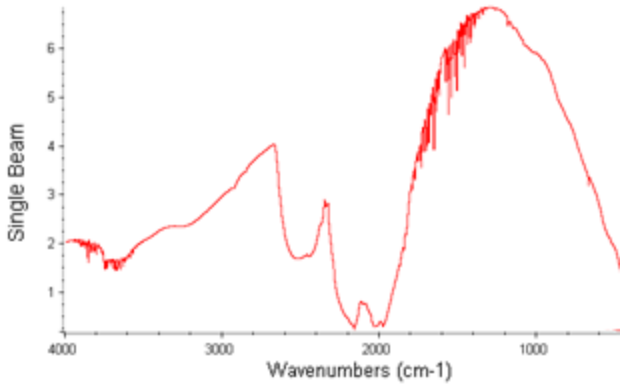
   Use the **Collect background** command in the OMNIC Collect menu or click the Collect Background button the toolbar, if it is displayed.

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.

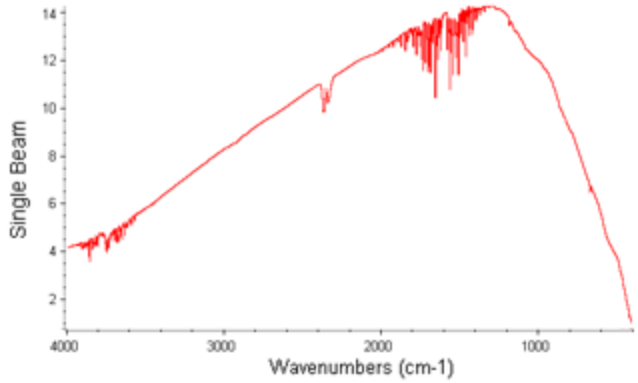
Here is what a background spectrum collected with the diamond crystal looks like:



Here is what a background spectrum collected with the germanium (Ge) crystal looks like:



Here is what a background spectrum collected with the gold 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 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 ofwater or carbon dioxide bands in the IR spectra ofyour samples.
- You see an unexpected change in the spectral baseline.
- The quality ofyour 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.

Installing a solid, powder, or film sample with the pressure tower

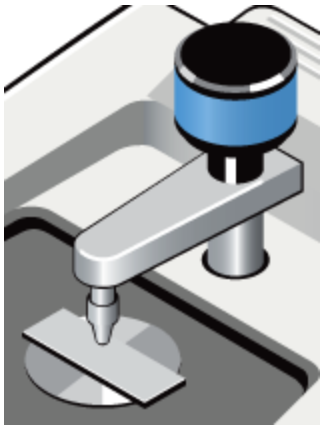
ATR is an excellent technique for measuring the composition of bulk solids or the surface properties of a layered solid. The IR beam from an ATR accessory migrates only a short distance from the surface of the crystal. Because of this, the sample must be placed firmly against the crystal before any sample information will appear in the infrared spectrum.

This does not mean that you simply place the sample on the crystal so that it touches in one or two places. It means you must add pressure so that the surface of the sample conforms to the surface of the crystal. Follow the instructions at the left to accomplish this using the pressure tower.

To install a solid, powder, or film sample with the pressure tower

1. Prepare the accessory for analyzing a solid with the pressure tower.
   - Raise the pressure point until there is sufficient room to insert the sample by rotating the pressure control ring counterclockwise.
   - Make sure the proper tip is installed on the pressure device.
2. Place the sample in the center of the crystal, directly under the pressure point.

   For best results, the sample should cover the crystal completely (required for quantitative analysis).



3. Rotate the pressure control tiny clockwise to press the sample against the crystal.

   The pressure device is designed to apply the same pressure to the sample every time. Rotate the pressure control ring until you feel two or three clicks. The clicking indicates that the internal pressure mechanism has reached the maximum pressure and that no additional pressure will be applied.

Installing a liquid, paste or gel sample

ATR is an ideal technique fot analyzing liquids. Sample preparation is minimal and cleanup is easy and fast. Even highly absorbing liquids, such as aqueous solutions, can be measured accurately without dilution because the IR beam penetrates the sample only a tiny amount.

Liquids are placed directly on the ATR crystal for analysis.

For free-flowing liquids, use a dropper or syringe to form a thin film of sample over the crystal.

If the sample is a viscous liquid, paste or gel, spread the sample so that it covers the crystal.

How to install a liquid, paste or gel sample

1. Rotate the pressure device to the cleaning position.
2. Place the sample on the crystal.

   For best results, the sample should cover the crystal completely (required for quantitative analysis).

Installing a solid sample for specular reflectance

Use the specular plate to perform fast analysis of coatings on reflective substrates using the grazing angle specular reflectance technique. For analysis, samples are simply placed facing down on the specular plate.

Samples must have a flat surface so that they will lie flat on the sampling platform. The sample must completely cover the opening in the specular plate.

How to install a solid sample for specular reflectance

1. Make sure the specular plate is installed and the pressure device is in the cleaning position.
2. Position the sample so the area to he analyzed is over the opening in the specular plate.

Measuring the sample

Once the sample is positioned on the crystal, 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 collect a sample spectrum with OMNIC software

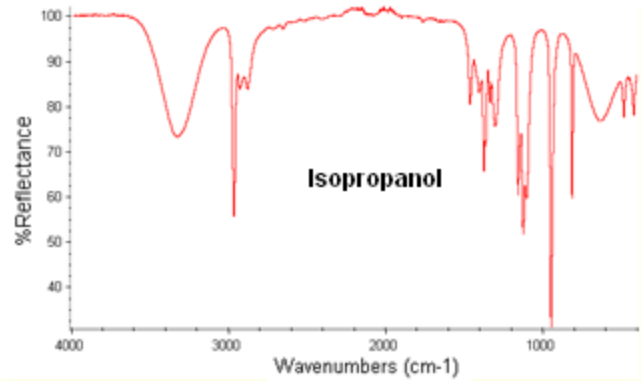
Use the Collect Sample command in the OMNIC Collect menu or choose the Collect Sample button on the toolbar, if it is displayed, to start sample collection.

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

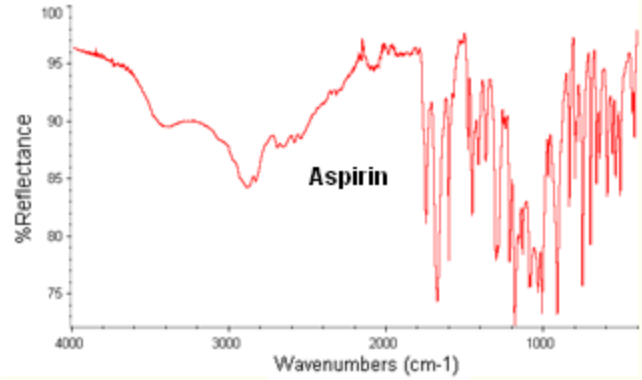
When the system has finished collecting the number of scans specified in the experiment, the final spectrum is displayed in a window.

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

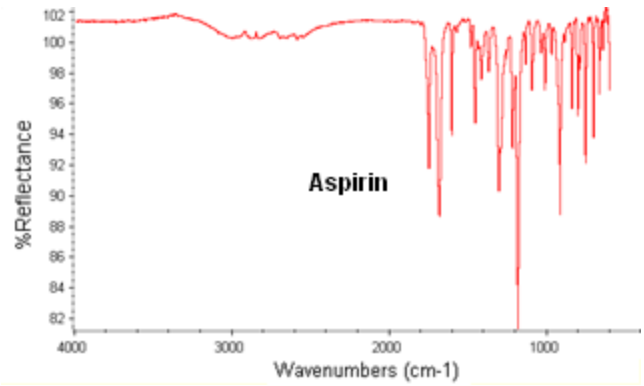
This is a spectrum of isopropanol measured using the Smart iTR with the diamond crystal plate.



This is a spectrum of aspirin measured using the Smart iTR with the diamond crystal plate.



This is a spectrum of aspirin measured using the Smart iTR with the germanium (Ge) crystal plate.



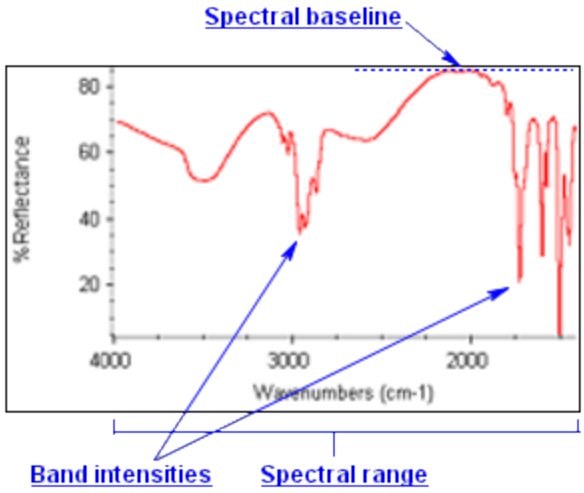
Sample specular reflectance spectrum

Specular reflectance is a type of external reflectance. An external reflection 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.

Specular reflectance works best for thin films and coatings on reflective surfaces.

The illustration at the left highlights the primary characteristics of a specular reflectance spectrum.

This is a specular reflectance spectrum of a coating on metal that was measured using the Smart iTR. The spectrum is shown in % reflectance, which is the typical format for displaying this type of mid-infrared data.



Optimizing the spectrum of a solid

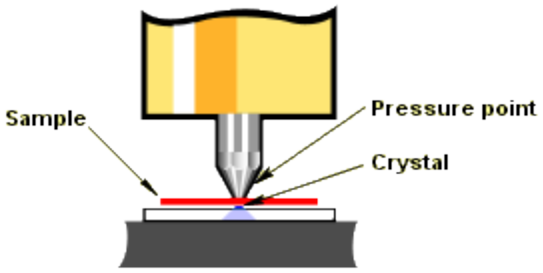
If you are measuring a solid sample and the spectral bands are very small (this happens occasionally with samples that are extremely rigid or if the sample surface is very rough), reposition the sample to ensure good contact directly below the pressure point. Then restart data collection.

If the sample spectrum has no sample peaks, check that the sample material absorbs energy in the infrared region of the spectrum.

If you see extra peaks in the spectrum, clean the crystal (see "Cleaning Up" in this tutorial) and collect the sample spectrum again.

How to optimize the spectrum of a solid

If the peaks in the spectrum of a solid sample are small, reposition the sample and collect the spectrum again. To reposition the sample, raise the pressure point by rotating the pressure control counterclockwise. Then position the sample in the center of the crystal, directly under the pressure point. Lower the pressure point and restart sample collection.



Optimize the spectrum of a liquid

If you see extra peaks in the spectrum of a liquid, residual material from the pervious sample may have been left on the crystal.

If you see extra peaks in the spectrum of a liquid sample, clean the crystal and then then collect the sample spectrum again.

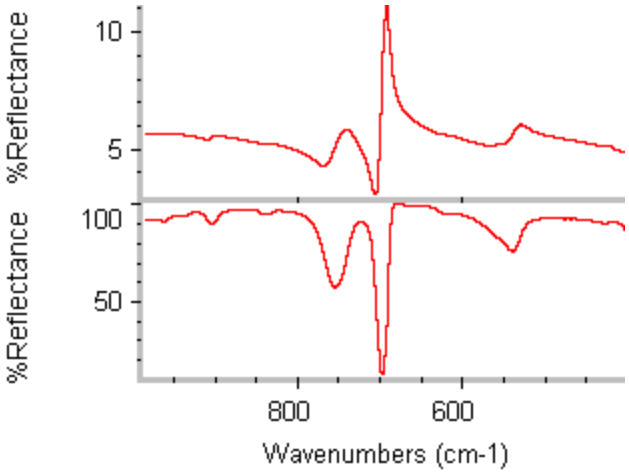
Correcting derivative-shaped peaks

Spectra collected using a reflection technique such as specular reflectance may have derivative-shaped or inverted peaks (see example at left). This is caused by optical dispersion from the specular (non-absorbing) 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 transformation to remove any derivative-shaped peaks. See OMNIC Help Topics for additional information.

This is a specular reflectance spectrum of a polymer.

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



Tips for quantitative analysis

For quantitative measurements using ATR, it is critical that the amount of contact between the sample and crystal be constant for all samples. In order to achieve this for solids, make sure the samples are of equal thickness and use the pressure tower to press the sample against the crystal. Use the same pressure setting for all samples and standards.

- For liquids, make sure the sample completely covers the crystal.
- For solids with the specular reflectance plate, make sure the sample completely covers the opening.

Cleaning up

Remove the sample immediately after you finish the analysis.

If any sample material remains on the crystal, clean the crystal thoroughly before measuring another sample (see the instructions at the left). You can clean the crystal with soap and water or with a suitable solvent. The crystal must be cleaned gently using soft material. You can remove the sampling plate and crystal plate if needed for cleaning. Dry the crystal thoroughly before you continue measuring samples.

To clean the accessory housing, use a clean cloth slightly dampened with a mild soap solution. Do not let liquid seep inside the instrument! Wipe dry.

How to clean the sampling plate and crystal

1. Remove the sample.

   If you were measuring a solid sample, raise the pressure point and remove the sample. You can use a cotton swab or compressed air to remove small particles from the crystal.

   If you were measuring a liquid sample, remove most of the sample by dabbing it with unscented tissues or cotton.

2. Gently clean the sampling plate, crystal plate and crystal.

   Moisten a tissue or cotton ball with water, or soap and water, and use it to clean the crystal.

   Dry the crystal with unscented tissue or cotton. You can remove both plates, if necessaty, for cleaning

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.

Using solvents to clean the crystal

If you need to use a solvent to remove the sample, make sure you choose a solvent that is compatible with the crystal. The following solvents are recommended:

Crystal	Recommended Solvent
Diamond	alcohol, water, toluene
Ge	alcohol, water, toluene
ZnSe	alcohol

How to clean the crystal with solvents

The cotton ortissue may be moistened with a suitable solvent, if necessary, to remove the sample. If you clean the crystal with a solvent, allow enough time for the solvent to evaporate completely. To ensure complete evaporation, dry the crystal with clean, dry air or nitrogen.

Use only recommended solvents to clean the crystal. Do not submerge the crystal or place it in an ultrasonic bath.

Removing the Smart iTR

You can remove the Smart iTR 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 Smart iTR

1. Ensure the crystal area is clean.

   If a sample is positioned on the Smart iTR crystal plate, remove the sample and clean the crystal as specified in the "Cleaning Up" section of this tutorial.

2. Disengage the Smart Accessory latch.

   Rotate the latch so both stems are free of the back edge ofthe accessory.

3. Gently pull up on the Smart iTR to release it.

   Firmly grasp the back edge ofthe accessory with one hand and under the bottom lip of the front ofthe cover with the other. Lift the accessory straight up until it is completely free ofthe sample compartment.

4. 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. Select a title to open the experiment and then choose OK.

Where to go next

Yout OMNIC software includes on-line documentation for many different FT-IR sampling techniques. If you are interested in learning more about ATR, open the Sampling Techniques item in the OMNIC Help menu and then choose ATR Sampling Techniques. It explains the theory of ATR spectroscopy and gives tips on running quantitative experiments and spectral searches

using ATR data. It also discusses common problems with ATR experiments and howto solve them.

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.