

thermo scientific

Nicolet iS50 ATR Module



User Guide

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Avoid an explosion or fire hazard.

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iS50 ATR Module

The optional iSTM50 ATR module offers convenient, reproducible attenuated total reflection (ATR) sampling with the Nicolet[™] iS50 spectrometer.

Figure 1-1: Nicolet iS50 spectrometer with optional iS50 ATR module



The iS50 ATR module provides these enhanced features:

- Built-in design
- Push button configuration
- All-reflective diamond optics
- Dedicated broad-band detector
- Extended spectral range

For information about additional sampling modules and accessories for your Nicolet iS50 spectrometer, contact our sales representative in your area.

NOTICE



Be sure that all persons operating this system read the site and safety manual first.

CONVENTIONS USED IN THIS DOCUMENT OR HELP SYSTEM

This manual uses these conventions for providing safety and other special information:

NOTICE

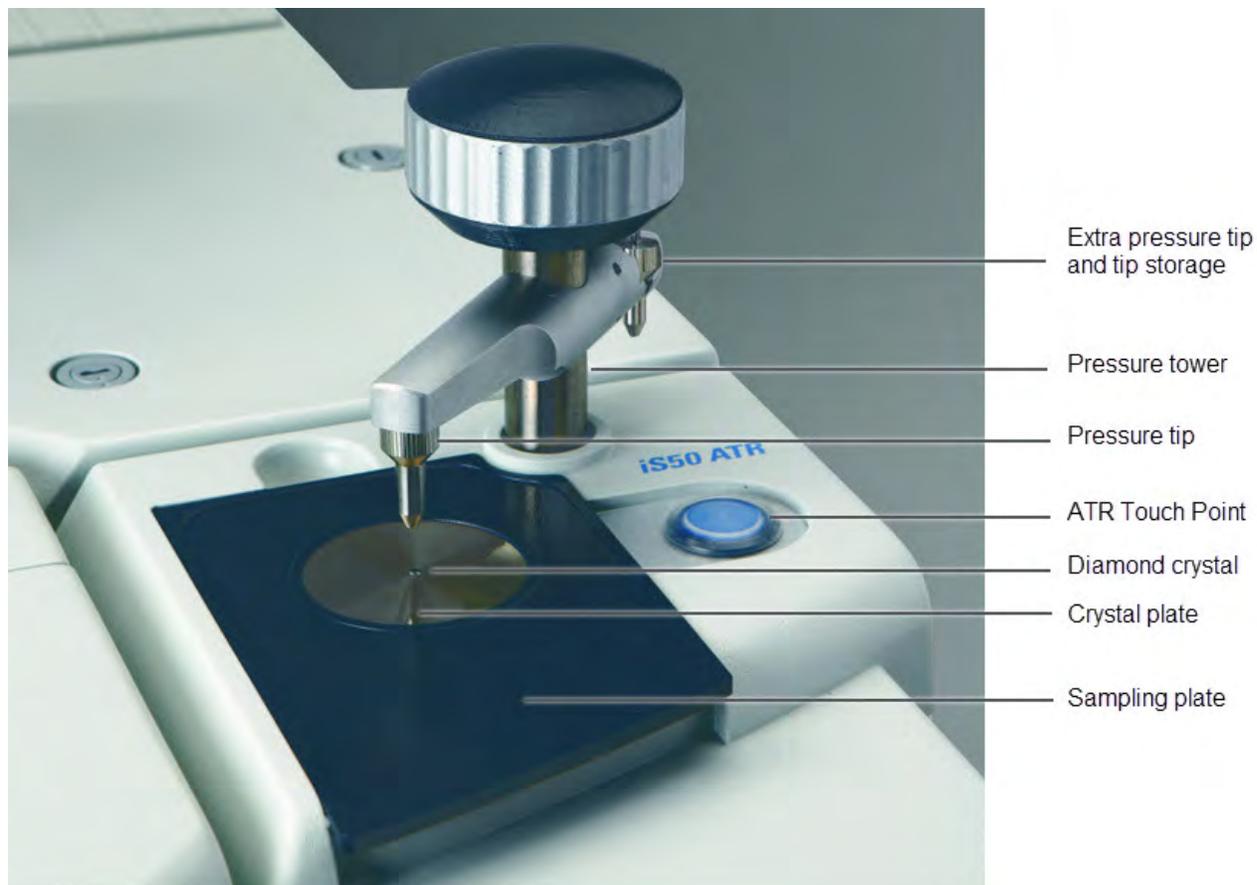
Follow instructions with this label to avoid damaging the system hardware or losing data.

Note Contains helpful supplementary information.

Tip Provides helpful information that can make a task easier.

Important Features

Figure 1-2: iS50 ATR module features



The iS50 ATR module features include:

- **Diamond crystal.** A robust, durable material for ATR sampling. The crystal is brazed to the crystal plate to maximize strength and chemical resistance.
- **Crystal plate.** The stainless-steel mount for the diamond crystal. The crystal plate is permanently sealed to protect internal mirrors and optimize purge performance.
- **Sampling plate.** This plate can be removed for cleaning or sterilizing.
- **Pressure tower.** The pressure tower is a mechanical press used to achieve uniform contact between a solid or powder sample and the ATR crystal (up to 267 N or 60 lbs of force). The arm swings out of the way for easy access to the sampling area. The pressure tower can be removed when analyzing only liquids.

iS50 ATR Module

- Pressure tip. The ATR module comes with two stainless-steel tip styles that can be used to optimize contact between the sample and the diamond crystal. The extra tip can be stored on the pressure arm.
- ATR Touch Point. This button automatically configures the spectrometer for ATR sampling. The Touch Point LED shows the status of the ATR module. The LED has three states described below.

Table 1-1: iS50 ATR Status indicator settings

LED status	Meaning
On	Ready for use
Off	System is not configured for ATR sampling
Blinking	Optics are reconfiguring

The ATR module spectral range options are the same as for the spectrometer and are dependent upon the spectrometer configuration. Refer to the spectrometer help topics or user guide for more information.

Specifications

Table 1-2: iS50 ATR module specifications

Feature	Specification
Crystal material	Diamond, Type IIa
Crystal shape, size	Round, 2.8 mm (0.11 in) diameter
Sampling zone	2 mm (0.08 in)
Refractive index	2.4 @ 1000 cm ⁻¹
Angle of incidence	45 degrees (single bounce)

Table 1-2: iS50 ATR module specifications

Feature	Specification
Depth of penetration	2.0 micrometers at 1000 cm ⁻¹ for a sample material that has refractive index of 1.5
Maximum sample thickness	12.7 mm (0.5 in)
Useful pH range	1 to 14
Pressure arm maximum force	267 Newtons (60 lbs)
Detector	Dedicated broad-band DTGS (deuterated triglycine sulfate)

Compatible Software

Table 1-3: Software compatible with the iS50 ATR module

Software	Use for...
OMNIC™ Paradigm	Measuring and analyzing ATR spectra and running custom workflows. To improve search results using a transmission reference library, use the Advanced ATR Correction feature in OMNIC Paradigm software.
OMNIC Specta™	Analyzing unknowns. This software includes enhanced algorithms, a spectral database and features for using your computer's hard drive as a library. OMNIC Specta also offers a unique multi-component search feature that makes it easy to identify the spectra of mixtures.
TQ Analyst™	Creating quantitative and classification methods that can be used with OMNIC Paradigm and OMNIC software. For more information, see the help system in your TQ Analyst software.

Operating Precautions

NOTICE

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, or if the pressure tip is not centered on the crystal.

Do not use the ATR module with concentrated sulfuric acid (H_2SO_4) or potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$); they will react with the diamond crystal.

To maximize the life of your ATR accessory or module, heed these precautions:

- Do not apply pressure to the bare crystal (the pressure tower should not be used for the background collection).
- Do not use the pressure device to crush a sample.
- Do not apply pressure to the crystal for extended periods.
- Make sure your sample will not react with the ATR crystal or crystal plate. Each material has unique characteristics. Please refer to the "Specifications" section for details.
- Do not scrape the crystal with extremely hard materials such as corundum, or with a knife to remove particles.
- Do not use abrasive cleaning agents, pads or strongly acidic or basic solutions to clean metallic surfaces of the accessory as these will damage the finish.

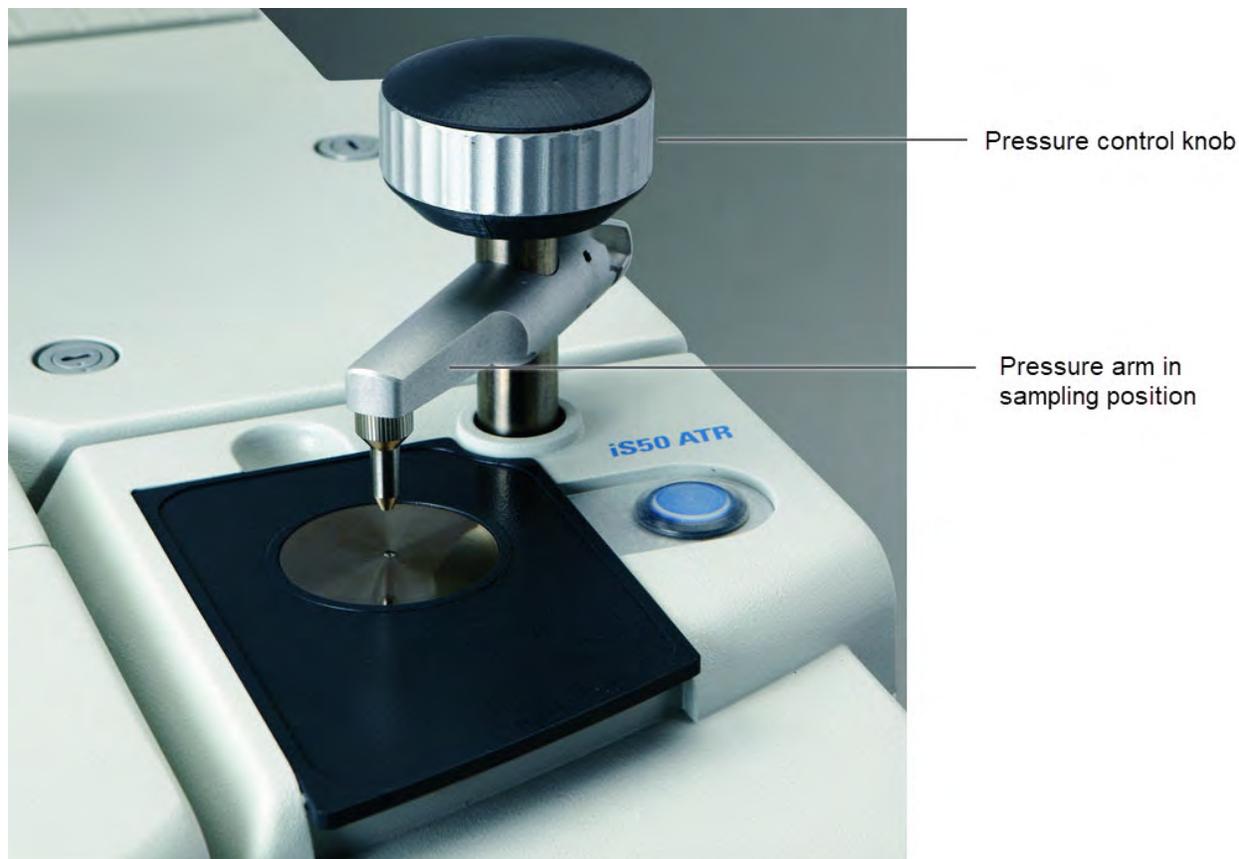
Operation

This section covers the following topics:

- ["Using the Pressure Tower"](#)
- ["Changing the Pressure Tip"](#)
- ["Removing and Installing the Sampling Plate"](#)

Using the Pressure Tower

Figure 1-3: Pressure tower features



NOTICE

Be certain the pressure is fully released before you move the pressure arm horizontally.

◆ To use the pressure tower

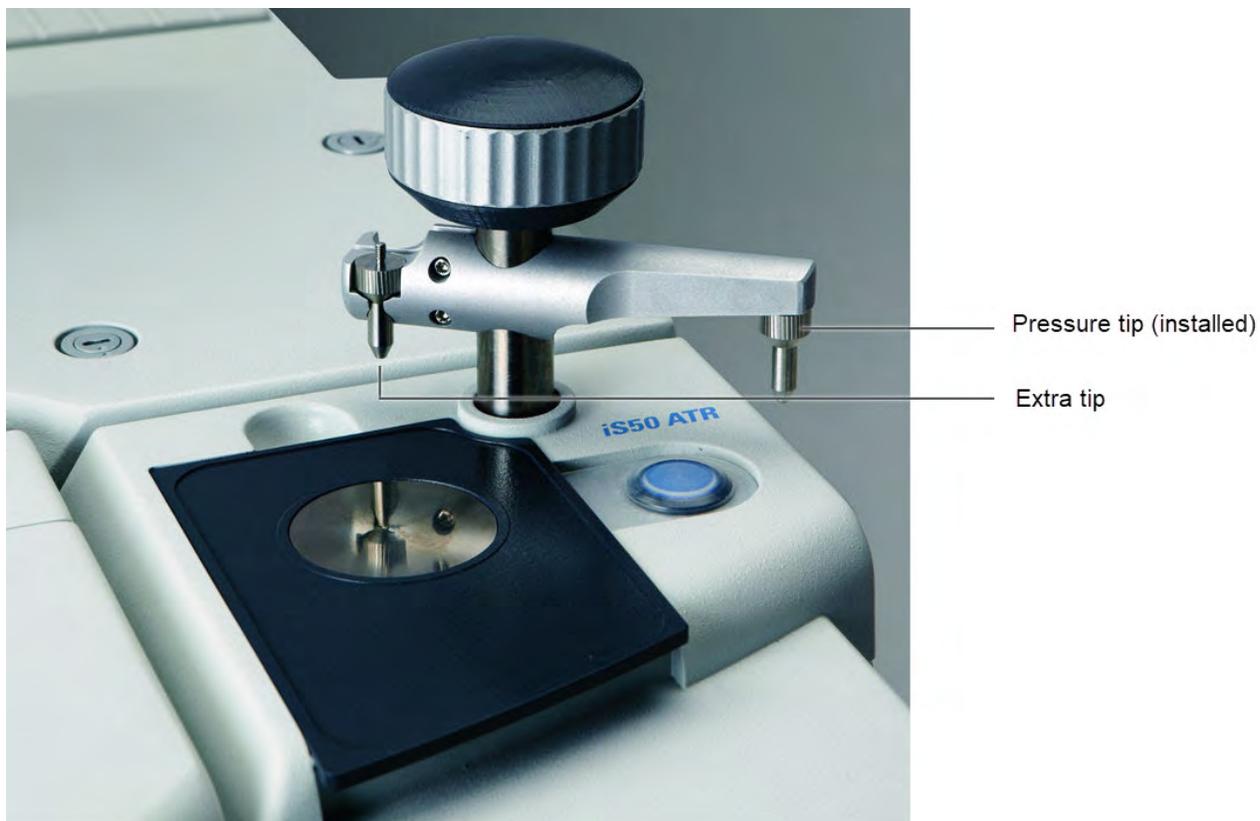
Use the large knob to raise and lower the pressure arm. When using the arm to apply pressure to a sample, rotate the control knob clockwise until you feel and hear a click. At this point the maximum force (267 N or 60 lbs) is achieved and additional rotation of the control knob has no effect. This mechanism is designed to protect the crystal from damage.

To raise the pressure arm, turn the control knob counterclockwise.

The pressure arm rotates to the side for sampling liquids or changing the tip. The pressure tower can be removed if necessary. See [“Installing and Removing the Pressure Arm”](#) for more information.

Changing the Pressure Tip

Figure 1-4: Pressure tip features



NOTICE

- Move the pressure arm away from the sampling area before you change the tip.
- Make sure the tip is properly threaded. The base of the tip should press against the pressure arm when fully installed.
- Do not use tools to loosen or tighten a pressure tip. Using tools may damage the tip.
- Do not overtighten the tips.

❖ To change the pressure tip

The tip can be screwed on and off while the pressure arm is raised. Move the arm to the resting position for easy access. The following tip styles are available.

Tip style	Use with...
<p data-bbox="215 310 272 338">Flat</p>  A close-up photograph of the iS50 ATR module. The device is white with a black sample plate. A silver pressure arm is positioned over the sample plate, holding a flat, circular tip. The text 'iS50 ATR' is visible on the side of the device.	<p data-bbox="862 493 1446 642">The end of this tip is flat, which makes it ideal for measuring films and flat plastics as well as tiny shavings and fibers. This is a general purpose tip style.</p>
<p data-bbox="215 884 337 911">Concave</p>  A close-up photograph of the iS50 ATR module, similar to the one above. The silver pressure arm is holding a concave, circular tip. The text 'iS50 ATR' is visible on the side of the device.	<p data-bbox="862 1087 1438 1194">The end of this tip is slightly concave to allow good contact with powders and curved solids such as a tablet or polymer bead.</p>

The extra tip can be stored on the pressure arm.

Removing and Installing the Sampling Plate

Figure 1-5: Sampling plate features



Move the pressure arm to the sampling position (shown above) before you remove the sampling plate.

To release the sampling plate, lift the back edge. To reinstall it, gently press down on the plate until it seals against the O-ring around the crystal plate.

NOTICE

Make sure the sampling plate is installed before you place a sample on the ATR crystal.

Avoid damaging or contaminating the ATR internal optics. Keep samples and other lab materials away from the ATR module when the sampling plate has been removed.

Compatible Samples

The ATR module can be used to analyze any sample that allows good contact with the surface of the crystal and has an index of refraction less than 2.0 (most organic materials range between 1.3 and 1.7).

ATR is an excellent technique for measuring the composition of liquids, powders and bulk solids or the surface properties of a layered solid. Because the effective pathlength is short, ATR is especially useful for analyzing strongly absorbing or thick samples which often produce large unreadable peaks when measured directly by infrared transmission. The ATR technique also eliminates the sample preparation required to run these types of samples by transmission.

Measuring Your First Sample

This section explains how to use OMNIC Paradigm software with your ATR module to analyze a solid and a liquid sample. These topics are covered:

- Preparing your measurement settings
- Measuring a solid sample
- Measuring a liquid sample

NOTICE

Do not use the ATR module to analyze concentrated sulfuric acid (H₂SO₄) or potassium dichromate (K₂Cr₂O₇); they will react with the diamond crystal.

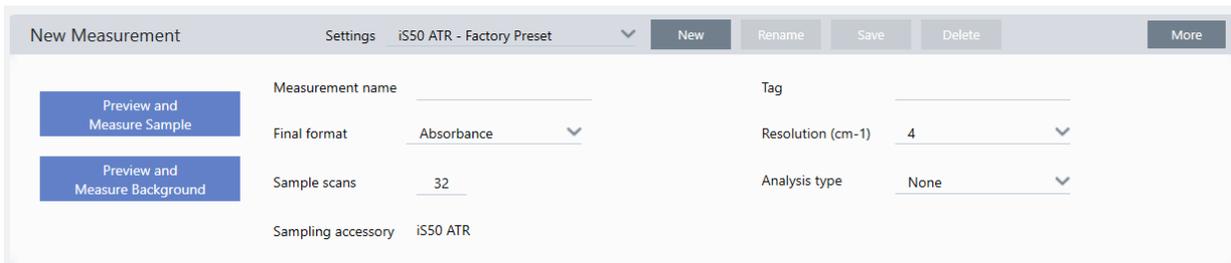
Preparing your measurement settings

Each Touch Point has an associated collection of measurement settings. You can customize and save your own settings or use the factory preset defaults. For information on changing and saving measurement settings, see the OMNIC Paradigm user guide.

◆ To select or edit your measurement settings

1. Start OMNIC Paradigm software.
2. Press the ATR Touch Point to automatically select your most recently used settings for the ATR module, or go to Configure > Sample Location > iS50 ATR Module. The software loads the most recently used settings for this sampling accessory. If you haven't yet created any new settings,

the software loads the iS50 ATR - Factory Preset settings.



Notice that the Settings list shows iS50 ATR - Factory Preset and the Sampling accessory shows iS50 ATR.

Basic measurement settings, such as sample scans and resolution, are shown in the New Measurement pane. Click More to expand this pane to view a live preview of the spectrum and to set advanced measurement settings.

For details on the available settings, see the OMNIC Paradigm software user guide.

Table 1-4: Basic measurement settings

Setting	Description
Measurement name	Set a name for your measurement or use the date and time of the measurement as the default
Final Format	Determines the Y-axis unit used for the collected data. Typically set to log(1/R) or %Reflectance for ATR applications.
Sample scans	The number of scans for the measurement. Scans are averaged.
Sampling accessory	Shows iS50 ATR as your current accessory.
Tag	Associate a tag with the measurement. Tags are useful for finding and sorting measurements later.
Resolution	Sets the spectral resolution of the data you collect. Typically 4 cm ⁻¹ or occasionally 2 cm ⁻¹ are appropriate for most ATR applications. The smaller the resolution value, the higher (better) is the resolution.

Setting	Description
Analysis type	Select Search, QCheck, or Quantify to carry out the analysis automatically after measuring the sample spectrum.

Measuring a Solid Sample by ATR

Before You Begin

- Power on the spectrometer and allow the instrument to stabilize. If your spectrometer has been powered off for more than 20 minutes, this will take at least an hour.
- Select an appropriate film or other solid for your sample.
- Make sure the [sampling plate](#), [pressure tower](#) and appropriate [tip](#) are installed.
- Remove any previous sample from the sampling area and [raise the pressure arm](#).
- Make sure the crystal and pressure point are clean. For cleaning instructions and recommended solvents, see [“Cleaning the Crystal”](#).

Figure 1-6: iS50 ATR module ready for a background measurement



Make sure the pressure point is not touching the crystal during the background measurement.

Note Nothing should be touching the crystal during the background measurement. If you plan to use the pressure arm to take sample measurements, make sure the pressure point is raised or move the pressure arm to the side.

❖ To measure a solid sample by ATR

1. Press the ATR Touch Point to start the measurement. Because there is not yet a background spectrum, you are prompted to measure the background.



2. Press the touch point again to proceed.
3. When the background preview is displayed, press the Touch Point to start the background measurement. The measurement begins and displays the progress and a preview during the measurement.

When the background measurement is complete, the Sample Preview is displayed.

4. Prepare the sample.
 - a. Place the sample in the center of the crystal directly under the pressure point. See “Compatible Samples” for more information. For best results the sample should cover the crystal completely.
 - b. Make sure the pressure arm is in the sampling position.

Lower the pressure arm by rotating the pressure control knob clockwise until you feel and

hear it click. The tip should be pressed firmly against the sample.



5. Press the ATR Touch Point to measure the sample.

You are prompted to enter a name for the measurement.

6. Enter a name for the measurement or press the Touch Point to accept the default name using the current date and time.

The measurement begins and displays the progress and a preview of the sample. When the measurement is complete, the spectrum is shown in the results panel and in the main Spectral view.

7. Raise the pressure arm and remove the sample.
8. Clean the sampling plate, crystal and pressure tip.

For cleaning instructions and recommended solvents, see [“Cleaning the Crystal.”](#)

Measuring a Liquid Sample by ATR

Before You Begin

- Select appropriate liquid, paste or gel sample (we use isopropyl alcohol for this experiment).
- If you have already collected a sample, click Dashboard in the toolbar in the software to return to the dashboard.
- Make sure the [sampling plate](#) is installed.
- [Raise the pressure arm](#) and move it to the resting position or install the [liquid sampling kit](#).
- Make sure the crystal is clean. For cleaning instructions and recommended solvents, see [“Cleaning the Crystal”](#).

Figure 1-7: iS50 ATR module with liquid sampling accessory ready for a background measurement



Note Nothing should be touching the crystal during the background measurement.

iS50 ATR Module

❖ To measure a liquid sample by ATR

1. If the iS50 ATR is not already shown as your current Sampling accessory, press the ATR Touch Point to select the ATR beam path and load the default measurement settings.
2. Press the ATR Touch Point again to start the workflow.



3. If you are prompted to measure the background, press the Touch Point again to proceed.
4. When the measurement is complete, the Sample preview is displayed.
5. Prepare the sample.
 - a. Use a dropper or syringe to place a thin film of sample on the crystal. See "[Compatible Samples](#)" for more information. For best results the sample should cover the crystal completely.

- b. If the sample material is volatile, place the cover from the liquid sampling kit over the sample.



6. Press the ATR Touch Point to measure the sample spectrum. The measurement begins and displays the progress and a preview of the sample. When the measurement is complete, the spectrum is shown in the results panel and in the main Spectral view.
7. Remove the sample cover if used.
8. Raise the pressure arm and remove the liquid sampling kit if used.
9. Clean the sampling plate and crystal.

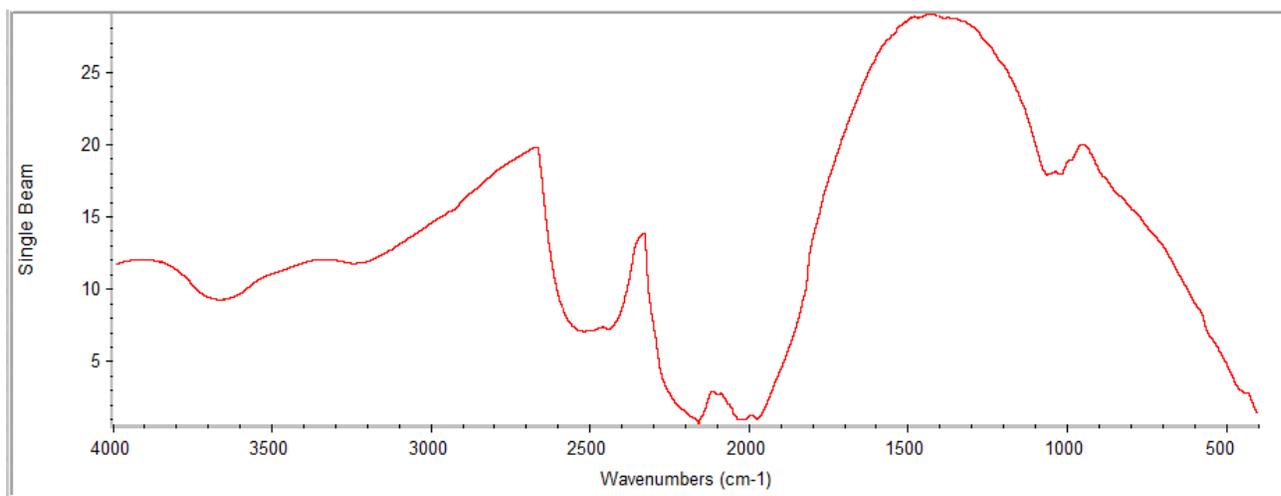
For cleaning instructions and recommended solvents, see ["Cleaning the Crystal"](#)

Typical ATR Spectra

The background spectrum shows the response of the spectrometer and the ATR module without a sample in place. Below is a typical background spectrum collected with the iS50 ATR module.

iS50 ATR Module

Figure 1-8: Typical background spectrum taken with the iS50 ATR module

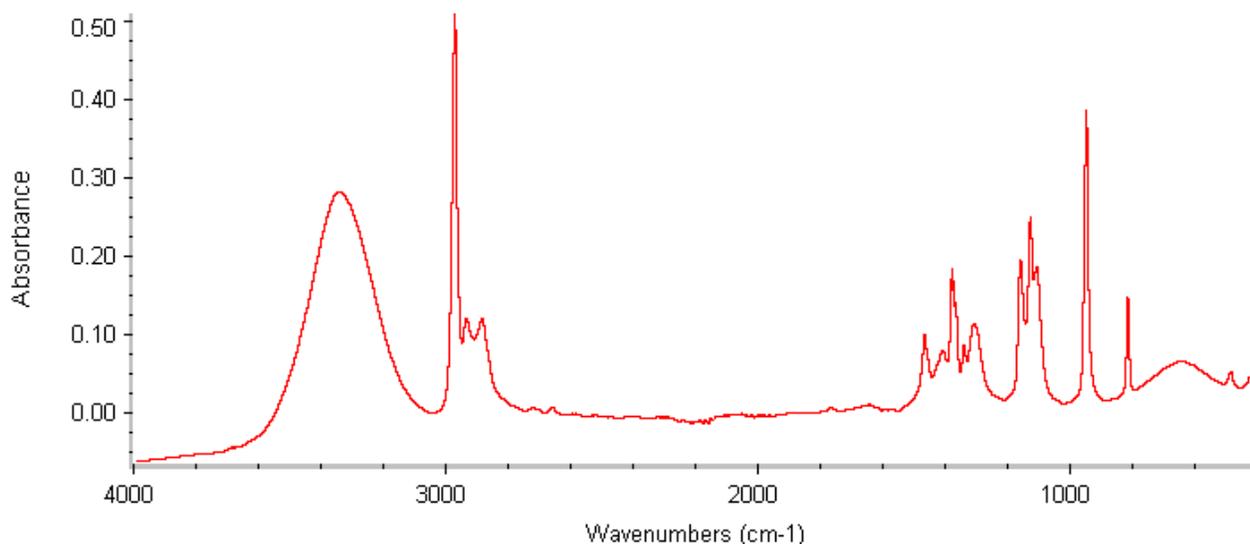


Note that the diamond crystal typically absorbs between 2300 cm^{-1} and 2000 cm^{-1} .

The sample spectrum shows the change in infrared energy (the background energy has been removed). This is an example of the type of spectrum you might obtain using the iS50 ATR module.

Note This spectrum is only an example. The spectra produced from your experiments may vary greatly, depending on the sample material and preparation.

Figure 1-9: Spectrum of isopropanol collected using the iS50 ATR module



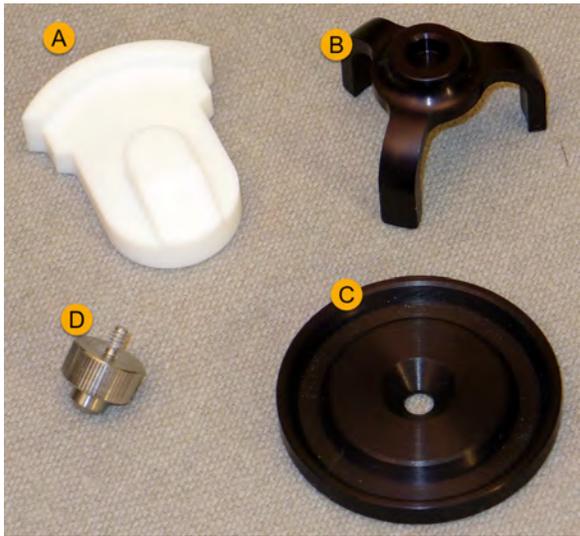
Note that absorptions from the diamond crystal appear in the sample spectrum as low level noise between 2300 cm^{-1} and 2000 cm^{-1} and may mask or interfere with any sample absorptions that

See the section titled “Typical ATR Sample Spectrum” in [“Tips for ATR Sampling”](#) for more information.

Sampling Accessories for the ATR Module

Optional Thermo Scientific accessories for the iS50 ATR module are highlighted below. To order an accessory, call our local sales or service representative or use the information at the beginning of this document or help file to contact us.

Optional Thermo Scientific accessories for the iS50 ATR module

This accessory...	Does this...
<p data-bbox="215 747 469 783">Liquid sampling kit</p>  <p data-bbox="215 1386 337 1612">A. Cover B. Bridge C. Base D. Tip</p>	<p data-bbox="868 821 1448 1014">The recessed base fits over the ATR crystal to hold a liquid sample. The bridge sits on the base and has a recess for the pressure tip. Applying pressure to the bridge seals the base against the crystal to prevent leakage.</p> <p data-bbox="868 1045 1398 1115">The cover can be placed over the base to prevent evaporation.</p> 

Maintenance

This section describes maintenance routines that you can perform on the ATR module to keep it running efficiently. These topics are covered:

- ["Cleaning the ATR Module Housing""Cleaning the Crystal"](#)
- ["Cleaning the Crystal"](#)
- ["Cleaning the Sampling Plate"](#)
- ["Installing and Removing the Pressure Arm"](#)

Cleaning the ATR Module Housing

To clean the ATR module housing, use a clean cloth slightly dampened with a mild soap solution. Do not use excessive amounts of liquid or let it seep inside the instrument! Wipe dry.

Cleaning the Crystal

NOTICE

- Use only water, mild soap, alcohol (isopropyl or methanol) or toluene to clean the crystal.
- Apply the cleaning solution to a lens cleaning tissue, cotton swab, or napkin and use that to clean the crystal. (Do not use scented tissue or tissues that contain added lotions or oils.)
- Avoid spilling solvent onto the ATR module or instrument.

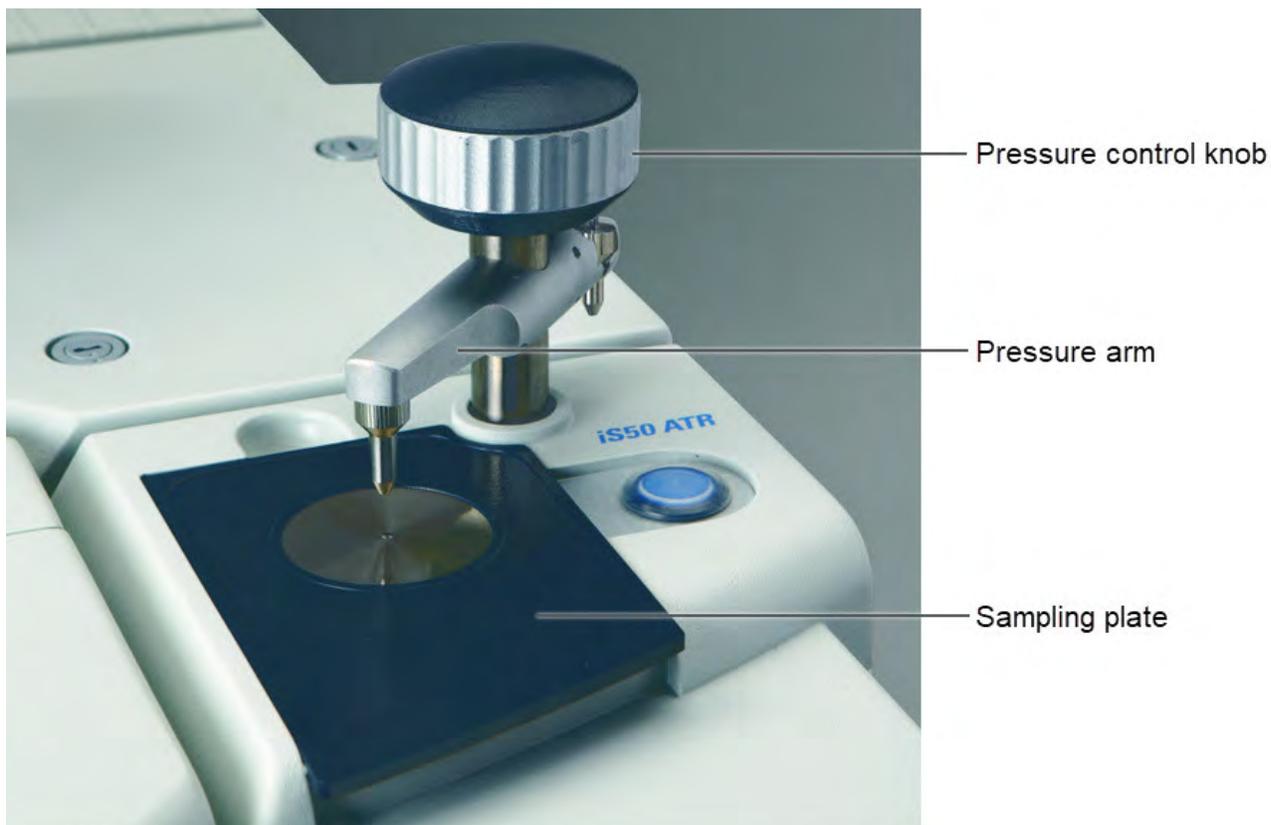
❖ To clean the crystal after measuring a sample

1. Remove the sample cover if used.
2. Remove the liquid sampling kit bridge and base if used.
3. Dab the crystal with a white paper napkin to remove most of the sample.
4. Dampen another clean paper napkin with alcohol and use it to wipe the crystal.
5. If the sample is oily or viscous, place a drop of alcohol on the crystal and wipe it with another clean napkin.
6. Dry the crystal with another clean napkin.

Cleaning the Sampling Plate

You can remove the sampling plate if necessary for cleaning.

To remove the sampling plate, raise the pressure arm by turning the control knob counterclockwise and then lift the plate off the assembly.



Use mild soap and water or, if needed, alcohol, toluene or a similar organic solvent to clean the sampling plate. The plate can be disinfected with a mild bleach solution.

NOTICE

Prolonged exposure to strong acids or bases may stain the sampling plate.

When the sampling plate is clean, install it on the ATR module. See [“Removing and Installing the Sampling Plate”](#) for more information.

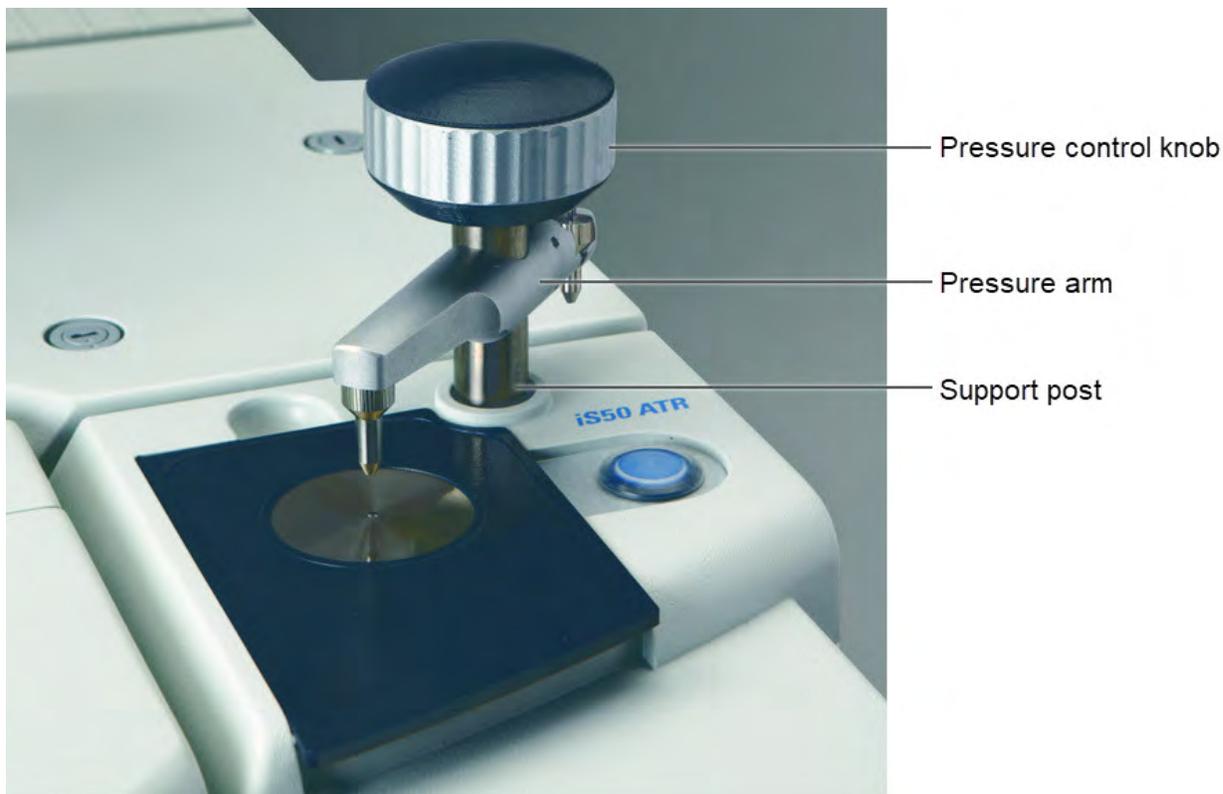
Installing and Removing the Pressure Arm

The pressure arm can be removed if needed.

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❖ To remove the pressure arm

1. Turn the pressure control knob counterclockwise until the support post disengages from the threads.



2. Pull up on the pressure arm until the support post is free of the ATR module housing.

❖ To install the pressure arm

1. Place a sample on the diamond crystal (a piece of paper will suffice).
2. Fit the end of the pressure arm support post into the hole in the ATR module housing.
3. Align the tip of the pressure arm with the diamond crystal.
4. Gently lower the post into the housing.
5. Turn the pressure control knob clockwise until the pressure arm lowers to the desired height.

Troubleshooting

The troubleshooting information will help you solve problems you may have with the ATR module. The information is divided as follows:

Hardware problems Steps you can take to resolve problems with the ATR module.

Applications problems How to fix problems in the collected spectra.

Error messages How to deal with error messages you may see.

Hardware Problems

This section lists common problems with ATR module components and how to resolve them.

NO INTERFEROGRAM SIGNAL

If you see an interferogram signal when you are using the main sample compartment but not seeing it when you press the ATR Touch Point, the ATR flipper mirror or detector may be malfunctioning. Contact our local service representative for technical support.

INTERFEROGRAM SIGNAL IS WEAK

If you switch to the ATR sample location and you notice a sudden drop in the strength of the interferogram signal displayed on the dashboard when you click the More button, the ATR crystal may be damaged or broken, the seal is broken or the ATR detector has failed. Contact our local service representative for technical support.

Applications Problems

BACKGROUND SPECTRUM HAS UNKNOWN FEATURES

If your background spectrum is significantly different from the background spectrum shown in [“Typical ATR Spectra”](#), clean the crystal thoroughly using the instructions in [“Cleaning the Crystal”](#), and then collect another background spectrum.

If the features are still there, the ATR mirrors may be contaminated. Contact our local service representative for technical support.

QUALITY OF SAMPLE SPECTRUM IS REDUCED

If the quality of your spectral data is reduced, collect a new background and then remeasure the sample. The background should be measured periodically (at least two times per day during continuous use) and after the following significant events:

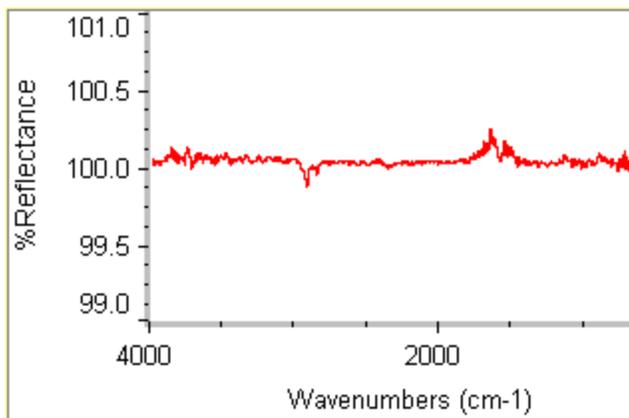
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- You changed the beam path by selecting another Touch Point or experiment file.
- You changed a component in your spectrometer or the ATR module.
- You changed one of the measurement settings other than Gain, Final Format, or Number Of Scans.
- You see a change in the amount of water or carbon dioxide bands in the sample spectra.
- 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).

SAMPLE BANDS ARE TOO SMALL

The sample bands should be large enough that you can distinguish them from the spectral baseline. The image below shows a spectrum with bands that are too small.

Figure 1-10: ATR spectrum with weakly absorbing bands



If your spectrum looks like the one shown above, the following may have occurred:

- Not enough contact between the sample and crystal. Check the following:
 - The pressure arm is applying adequate pressure to the sample. Place a flat, relatively soft material such as a credit card on the crystal and tighten the pressure control knob. If the pressure control knob spins freely or feels loose or stops prematurely, contact our local service representative for technical support.
 - You are using the correct tip for your sample material. See [“Changing the Pressure Tip”](#) for more information.
- The spectral range is set incorrectly.

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- The sample concentration is low or the sample is weakly absorbing. If you are measuring a liquid, concentrate it if possible. If you are measuring a solid, the sample may not be compatible with the diamond ATR technique.

SAMPLE SPECTRUM HAS NEGATIVE PEAKS

If peaks in your sample spectrum go below the baseline, your ATR crystal may be contaminated with layers of dried sample. This can occur after measuring a number of similar materials in succession, especially when using the pressure arm. Clean the crystal thoroughly a few times following the instructions in "[Cleaning the Crystal](#)," and then collect another background spectrum. If the problem persists, contact our local service representative for technical support.

SAMPLE SPECTRUM HAS EXCESSIVE NOISE

If your sample spectrum has excessive amounts of noise (sharp peaks that go above and below the baseline), the ATR detector may have failed. Contact our local service representative for technical support.

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