

iD7 ATR Accessory User Guide

Introduction

The iD7 Attenuated Total Reflectance (ATR) accessory is a single-bounce ATR sampling device designed for use with Thermo Scientific™ Nicolet™ iS™5 Fourier Transform Infrared (FT-IR) spectrometers. It offers the following features:

- Pinned-in-place prealigned all-reflective optics
- Interchangeable crystal plates including
 - Solid Diamond for full spectral range
 - Zinc Selenide (ZnSe) for high energy throughput
 - Germanium (Ge) for strongly absorbing samples
- Built-in swiveling slip-clutch pressure tower
- Fully integrated design
- Snap-in installation
- Automatic recognition, performance verification and experiment setup

This document explains how to install and use the features of the iD7 ATR accessory. It also describes how to operate the accessory to analyze solids, powders, and liquids. And you will learn how to properly maintain and store the accessory when it is not being used.

Product Overview

The iD7 ATR accessory can be used to run a variety of samples. It consists of an optical base, and a selection of four single-bounce crystal plates; high throughput diamond (AR-coated), extended range diamond (uncoated), zinc selenide (ZnSe), or germanium (Ge). A built in high-pressure tower with two removable tips and a volatiles cover are also included. The iD7 ATR provides seamless operation with OMNIC™ software using a chip embedded in the base of the accessory for automatic recognition and parameter setup.



Linear Absorbance Range

The iD7 ATR accessory provides a short sample pathlength due to the single-bounce ATR design. This produces absorbance values that are within the linear absorbance range of the spectrometer, which makes it ideal for rapid material identification. This linear absorbance range feature also makes the iD7 ATR accessory well suited for quantitative measurements.

Interchangeable Crystal Plate

The iD7 ATR crystal plate design accurately holds the ATR crystal in position to reproducibly collect spectra of solid, powder, or liquid samples. The plates are prealigned and easy to install and remove for cleaning or exchanging. Crystal plates are made of stainless steel to withstand repeated use and cleaning with recommended agents. The crystal plates fit securely over the optics to protect the accessory's internal components.



ATR crystals are made from high refractive index infrared transmitting materials. The high refractive index combined with the 45 degree incident angle of the infrared beam causes the light to reflect from the crystal's upper surface. When you place a sample in contact with the crystal, energy from the infrared beam is absorbed by the sample, which provides the spectrum we observe. Since the depth of penetration of the infrared energy into a sample is very shallow (1 to 4 micrometers), intimate contact must be made between the sample and the crystal. For solid samples, this requires putting pressure on the sample using the accessories built-in pressure tower. With the exception of the extended range diamond which is uncoated, the crystals use an anti-reflectance coating to maximize energy throughput and improve signal-to-noise. For crystal and crystal plate specifications, refer to the [Technical Details](#) section of this guide.

Pressure Tower

The pressure tower is a mechanical press used to achieve intimate contact between the sample and the ATR crystal, which is needed to obtain good quality spectra of solid samples. The tower is mounted on a swiveling hinge that allows you to move the arm out of the way to install samples, clean the sampling area, and remove the crystal plate.



The pressure tower is designed to apply consistent pressure to the sample every time. It applies approximately 40 pounds of pressure to ensure maximal contact of the sample to the ATR crystal. Using a slip clutch mechanism, the device automatically stops applying more pressure once the maximum pressure is achieved. This protects the crystal from damage and provides consistent sampling results.

Exchangeable Pressure Tips

The pressure tower includes two removable pressure tips to maximize contact between the sample and crystal for different sample types. A volatiles cover is also included.



Self Leveling flat pressure tip



Concave pressure tip



Volatiles cover

- The flat tip is ideal for thin samples such as a polymer film, and for compressible materials such as urethane foams. The self-leveling feature accommodates samples of variable thickness.
- The concave tip provides the best contact with powders and curved surfaces such as a polymer bead.
- The volatiles cover can be used to prevent evaporation of a volatile liquid sample during analysis.

The following sections describe how to set up the iD7 ATR and verify its performance. Refer to the [Accessory Features](#) section for details on accessory operation.

Installing the Accessory



Setting Up the Accessory

❖ To set up the Accessory

1. Unpack accessory and remove packing material.
Volatiles cover and concave tip are stored on the back side of the iD7 ATR.
2. Install the crystal plate (shipped separately).
 - a. Align the plate so that the notch on the underside matches up with the small pin on the top and to the front of the Optical Unit.
 - b. Ensure that the plate is seated into position.
3. Make sure the spectrometer is turned on and is operating correctly.
Refer to the Nicolet iS5 User Guide or the Spectrometer Help Topics in the OMNIC Help menu for assistance.
4. Insert the accessory.
Hold the front and back hand-holds located near the bottom faces of the accessory and install the iD7 ATR into the spectrometer.

Opening an ATR Experiment

Running an ATR Performance Test

5. If your instrument is purged, attach the purge line labeled “Accessory” to the purge inlet on the back of the accessory.

See “Installing a Purge Kit” in the Nicolet iS5 user guide and the [Purge](#) section of this manual for more information.

When you install an accessory, OMNIC™ software automatically shows 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. You can select and save the sampling defaults using Experiment Setup in the OMNIC software.

❖ To select the default experiment

After you install an accessory, the name of the associated experiments are displayed in a dialog box. Click **OK**.

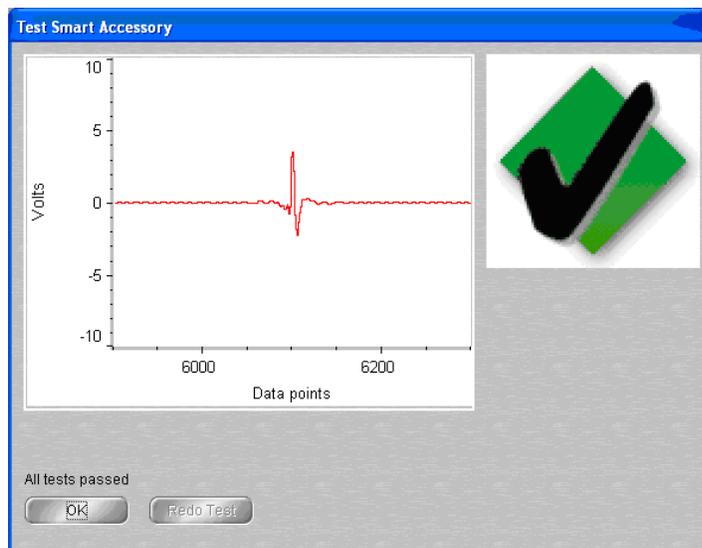
If multiple experiments exist for the accessory, they appear in a list box. Select the appropriate experiment.

Each time you install an accessory, the system automatically runs a diagnostic test to evaluate its performance. The spectral-quality feature of OMNIC software specifies the criteria used for the performance test. The performance diagnostics work in the background while the accessory is installed, ensuring high-quality spectra every time. The performance test starts automatically when you select an experiment. When the test is completed, one of the following two dialog boxes appear, displaying the results.

Note While the default accessory diagnostic test verifies the basic performance of the accessory, we recommend that you create a System Suitability test using the OMNIC System Performance Verification feature. The System Suitability test provides a more thorough evaluation of the accessory’s performance and allows you to track its performance over time. For more information, view the System Performance Verification topic in the OMNIC Help Topics from the Help menu.

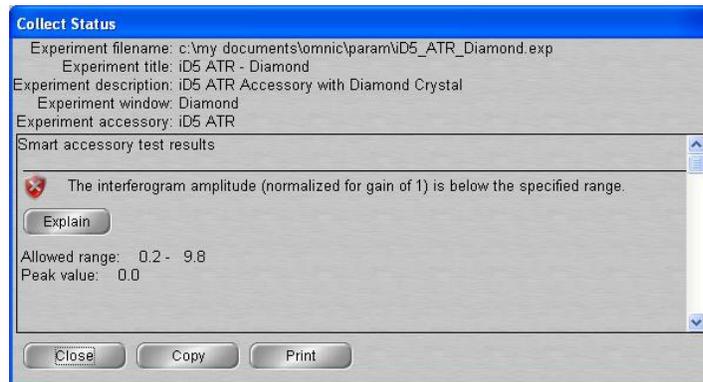
- Performance test passed

Look for the check mark in the green box as shown below, which tells you the system passed the performance test and is ready to collect data. Click **OK** to continue.

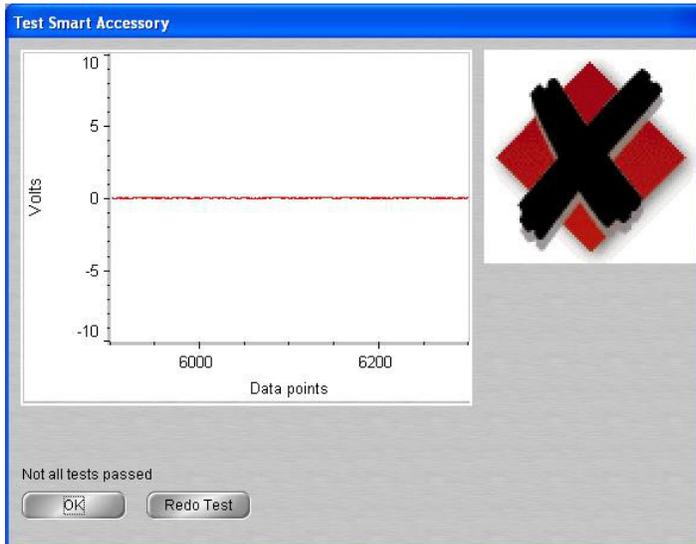


- Performance test failed

If the accessory is set up incorrectly or the system does not meet the performance criteria, a message appears indicating there is a problem, as shown below. Click **Explain** for instructions on how to verify and fix the problem.



Click **Close**. The Test Accessory screen indicates that a problem occurred with the performance test and allows you to restart the test. Make sure a crystal plate is installed, then click **Redo Test** to rerun the performance test.



If the test still fails, there may be an issue with the accessory. See [Troubleshooting](#) for more information.

Accessory Features

Using the Pressure Tower

The following sections describe how to use the iD7 ATR accessory features. For detailed information, refer to the [Technical Details](#) section.

The pressure tower arm can be positioned to easily change the crystal plate and pressure tips.

❖ To raise the pressure tower

Rotate the pressure control knob counterclockwise. The pressure tower and arm will rise.

Turn counterclockwise to raise tower



❖ To lower the pressure tower

Rotate the pressure tower knob clockwise until you have applied the maximum pressure. The slip clutch mechanism will allow the pressure tower knob to freely rotate when maximum pressure is reached.

Turn clockwise to lower tower



NOTICE Do not apply pressure directly to the crystal without a sample in place.

❖ To put the pressure tower arm in the cleaning position



1. Raise the pressure tower by turning the pressure control knob counterclockwise.
Make sure the tip is high enough to avoid hitting the crystal plate or accessory housing.
2. Swing the pressure tower arm to the left or right 90 degrees until it stops.
This is the cleaning position. See [Cleaning the Accessory after an ATR Experiment](#) for cleaning instructions.

❖ **To put the pressure tower in the sampling position**



Rotate the pressure tower arm toward the center of the accessory until the tip stops above the crystal. This is the sampling position.

NOTICE Ensure that the pressure is completely released (the tower is raised and the tip is not touching anything) before repositioning the tower.

Changing the Pressure Tips

Both pressure tips are installed in the same way.

❖ **To install a pressure tip**

1. Move the tower arm to the cleaning position.



2. Select the appropriate tip for the type of sample being analyzed.



The spare tip and volatiles cover are stored in the back of the accessory.

3. Install the tip by carefully screwing it into the end of the tower arm.

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

Changing the Crystal Plate

Each crystal plate is labeled on the back for easy identification. Plates may also be identified by crystal color and relative crystal size, as described:

- The diamond crystals are smaller in size than the others
 - High throughput (AR-coated) diamond appears yellow
 - Extended range (uncoated) diamond is clear
- Zinc selenide (ZnSe) is yellow
- Germanium (Ge) is silver

❖ To change an ATR crystal plate

1. Put the pressure tower in the cleaning position.
2. Remove the crystal plate by grasping the plate edge with your fingertips and pulling straight up.

NOTICE Do not use tools to install or remove the ATR crystal plate. Using tools may damage the crystal plate or the optical unit.

3. Install the new crystal plate:
 - a. Align the plate so that the Notch on the underside matches up with the small pin on the top and to the front of the Optical Unit. The crystal plate is held in position by three magnetic catches around the top aperture of the Optical Unit.



- b. If required, slightly rotate the plate to ensure that it is seated in position.

Failure to completely install the plate will result in lower energy throughput and could damage the accessory.

Maintaining the Crystal

To maximize the life of the crystal:

- Make sure the samples you analyze and the cleaning solvents will not react with the crystal.
- Do not use abrasive cleaning agents or pads on the crystal.
- Do not scrape the crystal with extremely hard materials such as sandpaper or a knife.
- Follow the recommended cleaning instructions in [Cleaning the Accessory after an ATR Experiment](#).

Running Samples

Collecting a Background Spectrum

Follow these instructions to run samples using an ATR crystal plate.

The background data used to process each sample measurement to an infrared spectrum must be collected under the same conditions as the sample, but without the sample in place. If you change crystal plates, you must collect a new background with the new plate. You can use the same background to analyze multiple samples. However, we recommend collecting a new background at least every two hours.

Note It is critical for the ATR crystal to be clean before a background is collected! The shallow depth of penetration of the ATR technique (1 to 4 micrometers) makes it sensitive to surface contaminants. Residues left on the crystal may show up as contaminants in your sample spectrum.

If any condition described below is true, collect a new background immediately.

- You changed a component in your spectrometer or sampling accessory.
- You changed one of the Collect, Bench, or Advanced settings in the selected experiment (except Gain, Final Format, Number Of Scans, or Correction).
- You see a change in the amount of water or carbon dioxide bands in the infrared 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 an ATR background spectrum

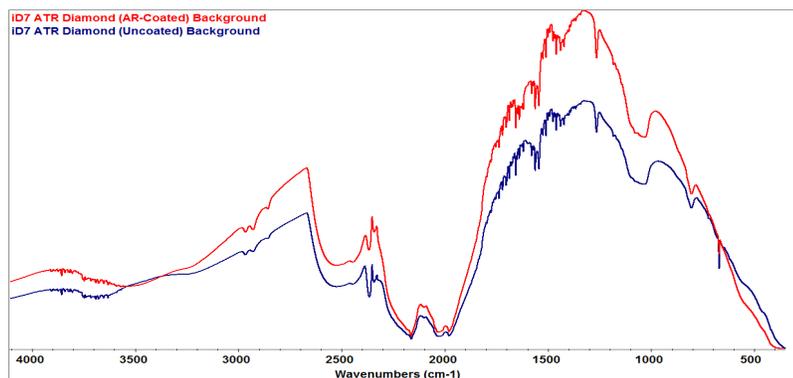
1. Prepare the accessory to collect the background spectrum.
 - a. Move the pressure tower to the cleaning position.
 - b. Make sure the crystal surface is clean. Refer to [Cleaning the Accessory after an ATR Experiment](#).
2. Collect the background spectrum.

Choose **Collect > Collect Background** or click **Collect Background**  on the toolbar, if it is displayed.

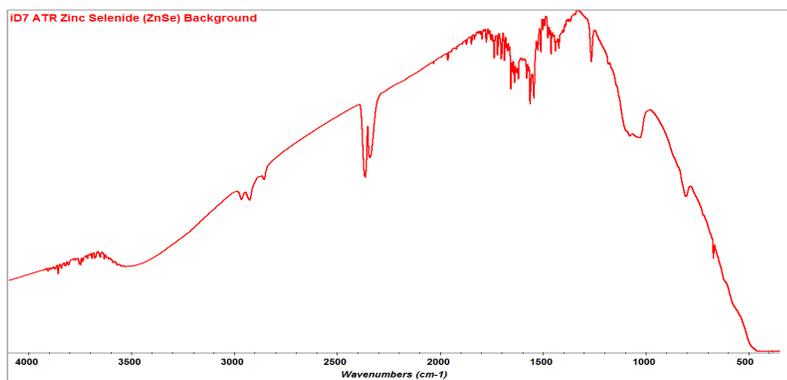
The new background spectrum is displayed in the Collect Background window. Whether or not you add it to the active window, the background remains in memory as the current background. It will be used to process all of the sample spectra you collect until you replace it by collecting another background.

The following images show typical background spectra collected using different crystal materials.

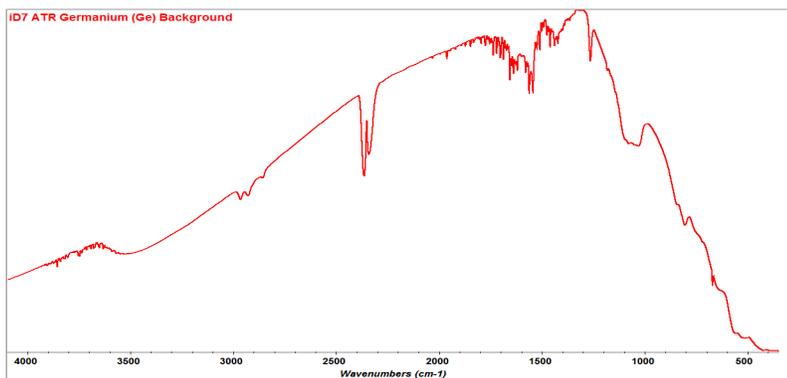
Background collected with AR-coated and uncoated diamond crystals:



Background collected with a ZnSe crystal:



Background collected with a Ge crystal:



Note For troubleshooting purposes and checking for contamination, it is a good idea to save a background spectrum when the crystal plate is new before it has been used for sample analysis.

Installing an ATR Sample

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

Solid, Powder, or Film Sample

ATR is an excellent technique for measuring the composition of bulk solids or the surface properties of a layered solid. Since the infrared beam only penetrates a short distance into the sample, the sample must be placed firmly against the crystal before an infrared spectrum is obtained. The pressure tower typically applies sufficient pressure so the sample conforms to the surface of the crystal.

❖ To install a solid, powder, or film sample

1. Ensure the correct pressure tip is installed on the pressure tower.
2. Move the pressure tower into the sampling position, leaving enough space between the tip and the crystal to insert your sample.



3. Place the sample onto the crystal, directly under the pressure tip.
4. Lower the pressure tower to press the sample against the crystal.
The pressure tower knob will click and freely rotate when the maximum pressure is reached.



5. Proceed to [Collecting a Sample Spectrum](#).

Liquid, Paste, or Gel Sample

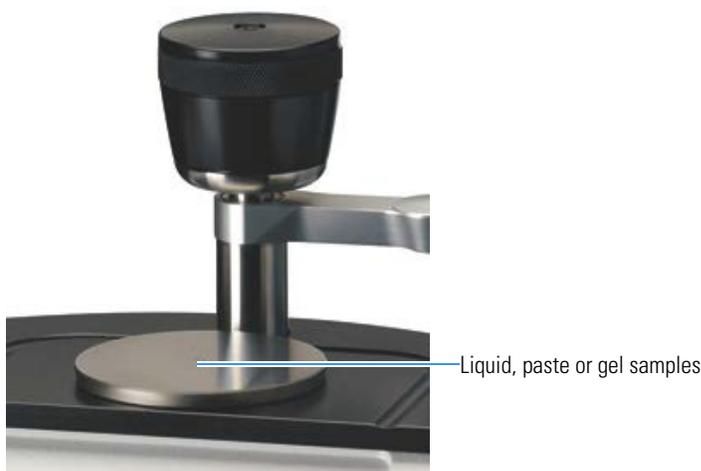
ATR is an ideal technique for analyzing liquids. Sample preparation is minimal and clean-up is easy and fast. Even highly absorbing liquids such as aqueous solutions can be measured accurately without dilution.

❖ To install a liquid, paste, or gel sample

1. Move the pressure tower into the cleaning position.
2. Place the sample onto the crystal.

The sample should cover the crystal completely. Do not overfill so the sample runs off the crystal plate.

Collecting a Sample Spectrum



A volatiles cover is available for containing liquid samples and reducing evaporation, if needed. The volatiles cover is a flat circular plate with an O-ring on the underside. To eliminate evaporation it can be sealed by applying pressure using the flat pressure tip.

3. Proceed to [Collecting a Sample Spectrum](#).

Once the sample is positioned on the crystal, you are ready to start collecting sample data.

❖ To collect a sample spectrum

1. Choose **Collect > Collect Sample** or click **Collect Sample**  , if it is displayed.
2. Follow the on-screen instructions to collect the sample spectrum.

The instructions depend on how Background Handling and other parameters and options are set in OMNIC and in the selected experiment.

OMNIC collects a few scans and then calculates and displays a spectrum. The spectrum is updated as new data is collected.

When the system has finished collecting, the final spectrum is displayed.

Note OMNIC software includes a preview data collection option, which allows you to view the spectrum before starting to collect data. This is useful for ATR spectral collection so that you can ensure good contact of the sample with the crystal before starting the data storage. See OMNIC Help topics for details.

Features of ATR Spectra

An ATR spectrum is similar to a transmission spectrum in that the locations and intensities of the spectral bands will be unique for a particular material. You must be careful, however, when comparing ATR spectra with transmission spectra, because the shapes and intensities of the bands can be quite different.

Spectral Baseline

In ATR sampling, depth of penetration (and thus, sample absorption) is wavelength dependent. This can cause the baselines of ATR spectra to slope up in the long wavelength (low wavenumber) region of the spectrum.

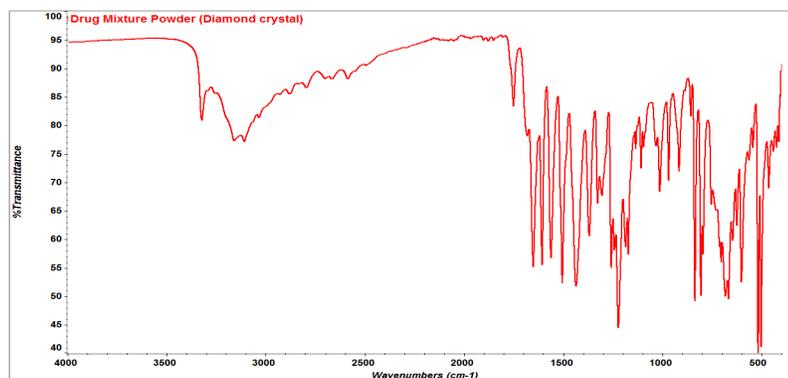
Band Intensities

If you compare the ATR spectrum to a transmission spectrum of the same material, the peaks in the short wavelength (high wavenumber) portion of the ATR spectrum will be smaller than those in the same region of the transmission spectrum. This happens because in ATR sampling, depth of penetration is wavelength dependent. The longer the wavelength of energy, the deeper the energy penetrates the sample.

Spectral Range

Infrared spectra collected using a mid-IR source, KBr beamsplitter and DTGS detector are typically displayed in the range between 4000 and 400 cm^{-1} . Some crystals, however, don't transmit infrared energy below about 650 cm^{-1} . Any peaks that appear in the spectral region beyond the transmission range should be ignored. See [Technical Details](#) for the spectral range of the crystal type you are using.

The following example shows an ATR spectrum of a drug mixture powder.



Optimizing the ATR Spectra

❖ To optimize the spectrum of a solid

If you are measuring a solid sample and the spectral bands are very small (possibly due to an extremely rigid sample or very rough sample surface), reposition the sample to improve contact between the sample and the pressure tip. The sample should be placed directly below the tip. Collect the sample spectrum again.

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 the Accessory after an ATR Experiment](#) for instructions) and collect the sample spectrum again.

❖ To optimize the spectrum of a liquid

If you see extra peaks in the spectrum of a liquid sample, residual material from the previous sample may have been left on the crystal. Clean the crystal (see [Cleaning the Accessory after an ATR Experiment](#) for instructions) and collect the sample spectrum again.

Remove the sample and clean the crystal immediately after the analysis. Clean the crystal plate and accessory housing as needed, to remove any contaminants or sample residue.

❖ To clean the crystal

1. Remove the sample.

For a solid sample, use a cotton swab or compressed air to remove small particles from the crystal.

For a liquid sample, remove as much of the sample as possible by dabbing it with lotion-free tissues or cotton.

2. Clean the crystal.

Gently clean the crystal with a suitable solvent using a soft material such as a lotion-free tissue or cotton ball. Do not use abrasive cleaning agents or pads on the crystal (this includes laboratory wipes, which are abrasive). Isopropanol or other alcohols are generally good cleaning solvents. Depending on the type of samples you run, water or an organic solvent such as iso-octane or heptane are also appropriate.

NOTICE

- Do not use ammonia, chlorine-based cleaners, acetone or other aggressive solvents to clean the crystal.
- Avoid contacting, contaminating or cleaning the bottom of the crystal.
- Do not submerge the crystal or place it in an ultrasonic bath.
- 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.
- Use only recommended solvents to clean the crystal.

3. Dry the crystal thoroughly with lotion-free tissue or cotton. Let the solvent evaporate completely before you continue measuring samples.

Cleaning the Accessory after an ATR Experiment

Removing the Accessory

❖ To clean the crystal plate and accessory housing

1. Use a cotton swab or compressed air to remove particles from the crystal plate and accessory housing.
2. Remove the crystal plate.
Do not let particles fall inside the accessory housing.
3. Use a clean cloth slightly dampened with a mild soap solution to clean the crystal plate and exterior of the accessory housing.
Do not use abrasive cleaning agents or pads on any surface of the accessory. Do not let liquid seep inside the accessory or instrument. Wipe dry.
4. Reinstall the crystal plate.

When you are finished using the iD7 ATR accessory, you can easily remove it from the spectrometer. When the accessory is not in use, store it in a dust-free environment such as a cabinet or box.

❖ To remove the accessory

1. Ensure the crystal area is clean and free of sample material.
2. Gently pull up on the accessory using the front and back recesses near the bottom of the accessory as hand holds.
The following dialog box is displayed:



3. Install another accessory if desired.

Troubleshooting

The iD7 ATR accessory has been aligned and tested in the factory to ensure that it performs to specifications. When it is new, it is ready to run without the need for adjustment. If you experience an accessory performance test failure, first remove the accessory and run a Performance Verification (PV) test to ensure the spectrometer is working correctly. See the Running a Performance Test topic in the OMNIC Help for instructions.

If the spectrometer is performing as expected, the problem is likely with the accessory. The information in this section is provided to help you determine what may be wrong with the accessory.

If the accessory performance test fails, follow these steps to resolve the problem:

1. Ensure that the accessory is fully seated on the sample compartment pins.
2. Check that the crystal plate is properly seated on the alignment pins on top of the accessory.
3. Examine the crystal to make sure it is not excessively scratched or cracked.

Note Infrared energy throughput of your accessory will decrease slowly over time as the crystal surface is exposed to the environment and due to normal wear from cleaning the ATR surface. After time, the crystal may need to be replaced.

If you see unusual spectral data, view the ATR Sampling Techniques tutorial in the Sampling Techniques group in the OMNIC Help menu. Note that you should save a background spectrum from any new crystal as a reference because different crystal types exhibit different spectral features. This allows you to check for possible build-up of contaminants on the crystal. You can do this easily by setting up a System Suitability test for your accessory using the System Performance Verification feature in the OMNIC software.

Infrared Energy Throughput Measurement

A simple measurement can be used to determine the infrared energy throughput of your iD7 ATR accessory.

❖ To measure the energy throughput

1. Collect a background spectrum with the accessory removed from the sample compartment.
2. Install the accessory into the instrument.
 - a. When the Accessory Change dialog box appears, select **Use Current Background**.
 - b. When the Performance Test appears, click **Skip Test**.
3. Collect a spectrum of the clean crystal and display the results in %T units.

The %T value at 1000 cm^{-1} will typically be above 10%. The percentage will vary with the crystal type being used.

Purge

The iD7 is equipped to handle a purge line to help limit the spectroscopic effects of water vapor or carbon dioxide within the accessory. Dry air or nitrogen are both appropriate gases that can be used for purging moisture and other environmental contaminants from the accessory. When using purge, set the purge gas controls for the accessory as indicated below:

- Accessory pressure, 5psig (1.34 atm)
- Accessory flow, 4 to 10 scfh (1.9 to 4.7 l/min)
- Dried to a dew point of -70°C (-94°F) or below

See the spectrometer user guide for more information regarding purging the spectrometer and accessory.



WARNING Never use a flammable, combustible, or toxic gas to purge this instrument. The purge gas must be free of oil and other reactive materials. Heat from the source or from laser absorption may ignite flammable gases or reactive materials in purge gas. Use only dried air or nitrogen to purge your instrument.

Maintaining the Accessory

To order parts, contact us.

Information about installing and replacing hardware is available in the Nicolet iS5 user guide. All other maintenance, troubleshooting or repair must be performed by one of our trained and certified service engineers. If you need to send the instrument or an accessory to us for repair, call or e-mail us first for any shipping requirements or other instructions.

Technical Details

Accessory

Angle of Incidence	45°
ATR Plate Assembly	Crystal sealed with Indium into a hardened steel plate
Cleaning Agents	Methanol, iso-propanol, water, iso-octane
Pressure Tower Applied Force	40 lbs (nominal)
Pressure Tower Maximum Travel	18 mm
Weight	1.6 kg

Crystal

	ZnSe	Germanium	Diamond
Active Area	3.4 mm	3.4 mm	1.8 mm
Spectral Range* (cm ⁻¹)	7,800 to 550 (AR coated)	5,000 to 650 (AR coated)	7,800 to 400 (AR coated) 7,800 to 350 (Uncoated)
Penetration depth (1000 cm ⁻¹)	2.0 μm	0.7 μm	2.0 μm
Hardness (Knoop #)	137	550	7000
pH Range	5 to 9	1 to 4	1 to 14
Refractive Index (1000 cm ⁻¹)	2.43	4.0	2.40

* Spectral range based on Nicolet iS5 KBr spectrometer with spectral range from 7800 - 350 cm⁻¹

NOTICE Do not use laboratory wipes to clean ZnSe or Ge crystal surfaces. This may severely scratch the crystal surface and degrade its performance.

All trademarks are the property of Thermo Fisher Scientific Inc. or its subsidiaries.