Everest ATR Accessory User Guide

Introduction

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

- Pinned-in-place pre-aligned all-reflective optics
- Interchangeable crystal plates, including
  - Monolithic AR-coated or uncoated extended range diamond
  - Zinc selenide (ZnSe) for high energy throughput
  - Germanium (Ge) for strongly absorbing samples
- Built-in swiveling pressure tower with slip clutch
- Fully integrated design
- Snap-in installation
- Automatic recognition and measurement setup

This document explains how to install and use the features of the Everest ATR accessory, how to operate the accessory to analyze solids, powders, and liquids, and how to properly maintain and store the accessory when it is not in use.

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Product Overview

The Everest ATR accessory can be used to measure 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 Everest accessory provides seamless operation with Thermo Scientific™ OMNIC™ Paradigm software, using a smart chip embedded in the base of the accessory to automatically recognize the accessory and optimize measurement settings.

Exchangeable Crystal Plates

The Everest ATR crystal plate design securely holds the ATR crystal in position to reproducibly collect spectra of solid, powder, or liquid samples. The plates are pre-aligned 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 solvents. The crystal plates fit securely over the optics to protect the accessory's internal components.

ATR crystals are made from materials that transmit infrared light and have a high refractive index. 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 accessory's 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 firm contact between the sample and the ATR crystal, which is needed to obtain good quality spectra of solid samples. The pressure tower should not be used when measuring liquid samples. The tower is mounted on a swiveling hinge that allows you to move the arm out of the way to introduce samples, to clean the sampling area, and to 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 between the sample and 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 the crystal for different types of samples. 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.

Installing the Accessory

The following sections describe how to set up the Everest accessory. Refer to the Accessory Features section for details on accessory operation.

Setting Up the Accessory

❖ **To set up the accessory**

1. Unpack the accessory and remove the packing material.

   The volatiles cover and concave tip are stored on the back side of the accessory.

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 base unit.

   b. Ensure that the plate is seated into position.

3. Ensure that the spectrometer is turned on and is operating correctly.

4. Insert the accessory.

   Hold the front and back hand-holds located near the bottom faces of the accessory and install the accessory into the spectrometer.

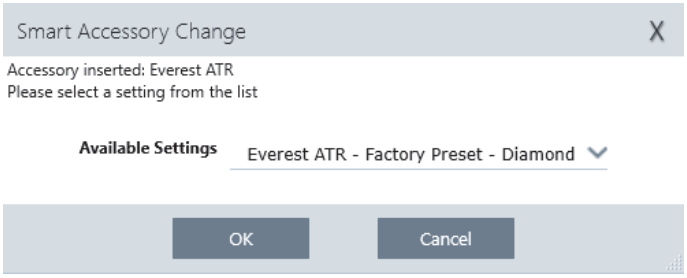
5. If your instrument is purged, attach the purge line labeled “To Accessory” to the purge inlet on the back of the accessory.

   See “Install and Maintain a Purge Kit” in the Nicolet Summit user guide and the Purge section of this manual for more information.

Updating Measurement Settings

When you install an accessory, OMNIC™ Paradigm software automatically updates the measurement settings to those that are optimized for the accessory.

When you install the accessory, the Smart Accessory Change dialog opens to allow you to choose your measurement settings. Choose from among the factory preset options to match your crystal plate, or choose a custom ATR setting that you have previously saved.



Handling the Accessory

Using the Pressure Tower

The following sections describe how to use the pressure tower, change the pressure tips, and change and maintain the crystal plates. For detailed information, refer to the Technical Details section.

Re-position the pressure tower to more easily change the crystal plate or pressure tips and to place or remove a sample.

❖ **To raise the pressure tower**

Rotate the pressure control knob counterclockwise to raise the pressure tower and arm.

❖ **To lower the pressure tower**

Rotate the pressure tower knob clockwise until you have applied the maximum pressure. When you have reached maximum pressure, the slip clutch mechanism allows the pressure tower knob to freely rotate.

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

❖ To move the pressure tower arm to 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 Measuring a Sample for cleaning instructions.

❖ To move the pressure tower to 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.

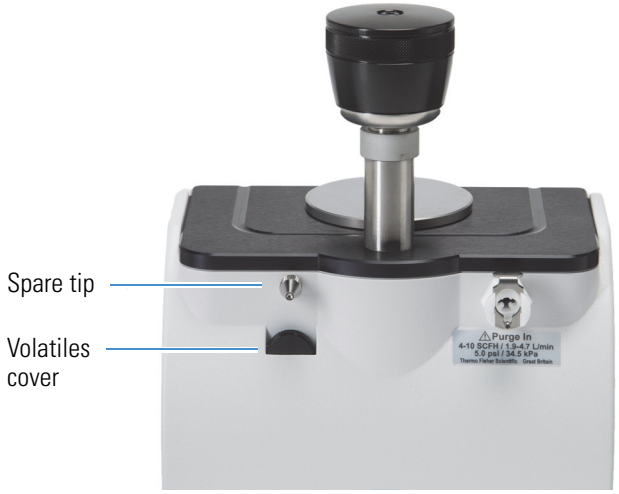
NOTICE Before repositioning the tower, ensure that the pressure is completely released (the tower is raised and the tip is not touching anything).

Changing the Pressure Tips

Pressure tips screw into the end of the tower arm.

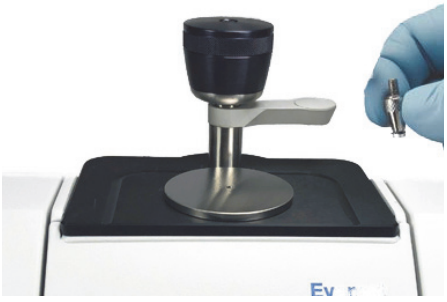
❖ To install a pressure tip

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. Move the pressure tower to 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 base. 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, follow these recommendations:

- Ensure that your samples or cleaning solvents will not react with the crystal.
- Use only non-abrasive cleaning agents or pads on the crystal. Do not clean the crystals with Kimwipes wipes, as they contain abrasives.
- 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 Measuring a Sample.

Measuring Samples

After placing the Everest ATR accessory in the sampling compartment, measuring a sample requires you to collect a background spectrum, to introduce the sample, and then to measure the sample.

This section also introduces common features of spectra collected with ATR techniques and how to optimize the spectra.

Collecting a Background Spectrum

You must measure the background environment under the same conditions used to measure the sample, but without the sample in place. For example, if you change the crystal plate, you must collect a new background spectrum with the new plate in place. You can measure multiple samples using the same background spectrum, but because the background environment can change slightly over time, we recommend collecting a new background spectrum every few hours.

See “Measure a Background” in the OMNIC Paradigm Guides and Tutorials on www.thermofisher.com for more on measuring background spectra.

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 the measurement settings in OMNIC Paradigm software
- You see a change in the amount of water vapor 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 measure the background with OMNIC Paradigm software for touchscreen

1. Move the pressure tower to the cleaning position and ensure that the crystal surface is clean.
2. From the home screen, select your analysis type (Measure, Search, QCheck, or Quantify).
3. To review or edit your background settings, select the **Background** tab.
4. To proceed, touch **Measure Background.** A preview of the background spectrum is displayed.
5. Touch **Start Background Measurement** to measure the background or **Cancel** to return to the analysis setup.

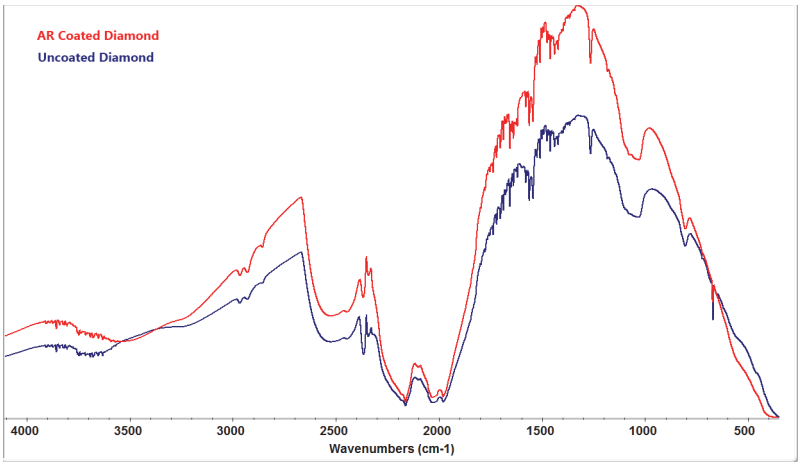
❖ To measure the background with OMNIC Paradigm software for desktop

1. Move the pressure tower to the cleaning position and ensure that the crystal surface is clean.
2. To review or edit your background settings, click **More** in the New Measurement pane and scroll to the Background group.
3. To proceed, click **Preview and Measure Background.** A preview of the background spectrum is displayed.
4. Click **Start Background Measurement** to measure the background or click Dashboard in the toolbar to cancel and return to the dashboard.

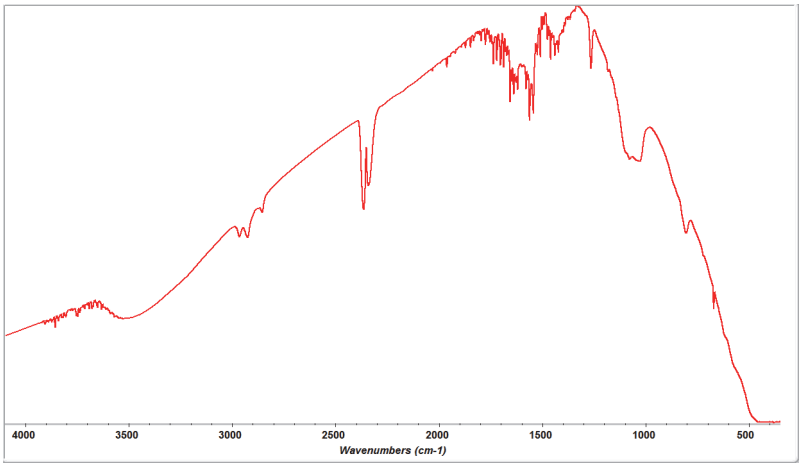
During the measurement, you can pause, restart, or stop and save the measurement. If you select Stop and Save, the spectrum will be saved with the number of scans that were completed.

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

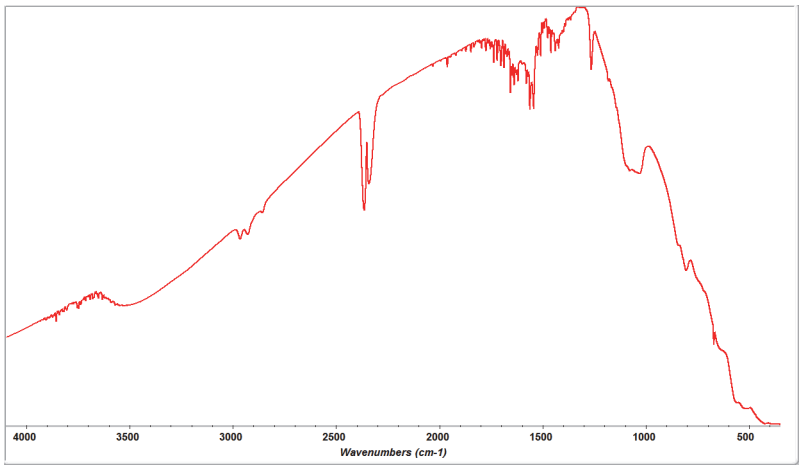
Background collected with AR-coated and uncoated diamond crystals:



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.

Introducing an ATR Sample

After you have measured the background, you are ready to introduce 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. The pressure tower typically applies enough pressure to ensure that the sample conforms to the surface of the crystal.

❖ To introduce 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 Measuring a Sample Spectrum.

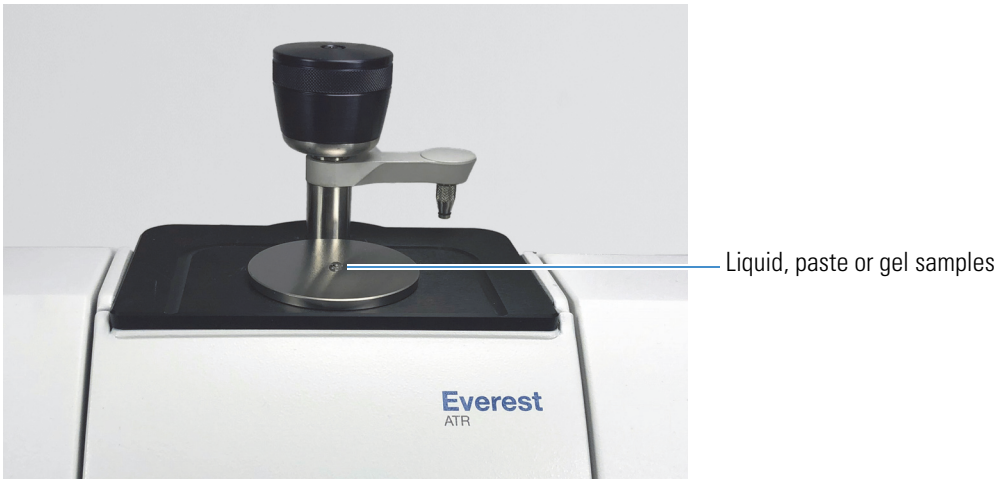
Liquid, Paste, or Gel Sample

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 introduce 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 or the sample may run off the crystal plate.



   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.

Measuring a Sample Spectrum

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

❖ To measure a sample spectrum with OMNIC Paradigm software for touchscreen

1. From the home screen, select an analysis type (Measure, Search, QCheck or Quantify).
2. Edit your measurement and analysis settings. For details on measurement settings, see "Measurement Settings" in the OMNIC Paradigm Guides and Tutorials on www.thermofisher.com.
3. Touch **Measure Sample**. If your background spectrum is incompatible or needs to be updated, you may be prompted to measure the background first. A preview of the sample spectrum is displayed.

   During the measurement, you can pause, restart, or stop and save your measurement. If you select Stop and Save, the data will be saved with the number of scans that were completed before stopping.

❖ To measure a sample spectrum with OMNIC Paradigm software for desktop

1. From the dashboard, review or edit your measurement settings in the New Measurement pane. For details on measurement settings, see "Measurement Settings" in the OMNIC Paradigm Guides and Tutorials on www.thermofisher.com.
2. Click **Preview and Measure Sample** to begin. Depending on your Background settings, you may be prompted to measure a new background before proceeding. A preview of the sample spectrum is displayed.
3. Click **Start Sample Measurement** to proceed.

   During the measurement, you can pause, restart, or stop and save your measurement. If you select Stop and Save, the data will be saved with the number of scans that were completed before stopping.

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

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. However, the ATR spectra can be corrected to mimic transmission spectra by using Advanced ATR Correction in OMNIC Paradigm software.

Spectral Baseline

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

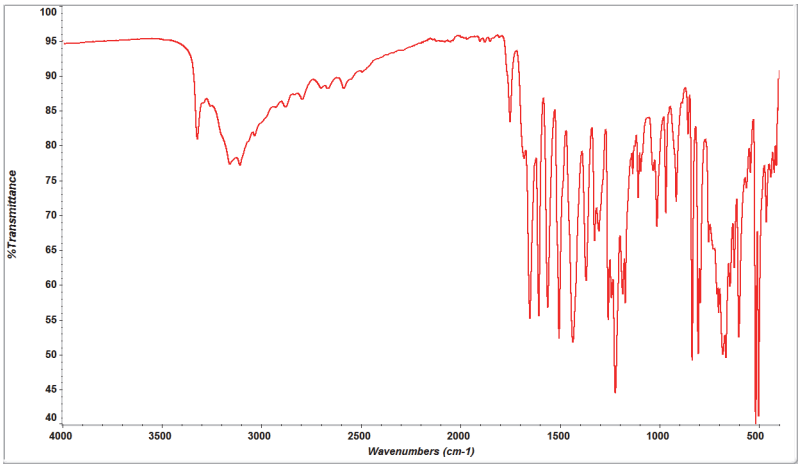
Band Intensities

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 depends on the wavelength. 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 of the crystal 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 Measuring a Sample 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 Measuring a Sample for instructions) and collect the sample spectrum again.

Cleaning the Accessory after Measuring a Sample

Remove the sample and clean the crystal immediately after the analysis. Clean the crystal plate and optical base 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 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 a cotton swab.

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. Use only non-abrasive cleaning agents or pads on the crystal. Isopropanol or other alcohols are generally good cleaning agents. Depending on the type of samples you measure, water or an organic solvent such as isooctane or heptane are also appropriate.

   NOTICE

   - Do not wipe the crystal with laboratory Kimwipes as they are abrasive.
   - 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 plate.
   - 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 swab. Let the solvent evaporate completely before you continue measuring samples.

Removing the Accessory

When you are finished using the Everest 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 plate 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.
3. Install another accessory if desired.

Troubleshoot

The Everest 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 qualification method, such as the Nicolet FTIR -PV workflow to ensure the spectrometer is working correctly.

If the spectrometer is performing as expected, the problem is likely with the accessory. Install the accessory and run the ATR Accessory - PHEUR workflow to test the performance of 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** The infrared energy throughput will decrease slowly over time because the crystal surface is exposed to the environment and to normal wear from cleaning the crystal surface. Eventually, the crystal may need to be replaced.

Purge

The Everest accessory 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 purging the accessory, set the purge gas controls for the accessory as indicated below:

- Accessory pressure: 5psig (1.34 atm)
- Accessory flow: 4 to10 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 Summit User Guides and Tutorials at www.thermofisher.com. 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	Isopropanol, methanol, water, isooctane, heptane
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[a] (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 to14
Refractive Index (1000 cm^{-1})	2.43	4.0	2.40

[a] Spectral range based on Nicolet Summit 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.