

# Slide-On ATR User's Guide

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# Introduction

This manual provides background information about ATR spectroscopy, instructions for installing the Slide-On™ ATR, and a typical analysis with a Slide-On ATR. If you have any questions, please contact a Thermo Electron Technical Representative.

## ATR spectroscopy

Attenuated Total Internal Reflectance (ATR) spectroscopy is a versatile and powerful technique for infrared sample analysis. Coupled with an infrared microscope, ATR spectroscopy is a highly accurate system for obtaining infrared spectra from small spots on both large and small samples with effortless sample preparation. As such, micro-ATR methods permit rapid spectral acquisition from a broad variety of materials and surfaces. Typically, samples which are too thick or strongly absorbing to be attempted by transmission spectroscopy, such as carbon-filled elastomers, can be routinely approached with ATR spectroscopy. ATR is inherently a surface technique and is therefore ideal for analyzing the surface of materials.

## The Slide-On ATR

The Slide-On ATR is intended to be used with the Infinity Corrected 15X Replachromat™ Objective and extends the capability of this objective to micro-ATR measurements while still permitting its use for transmission and external reflection measurements. The high refractive indices of the germanium (4.0) and silicon (3.4) crystals are ideal for micro-ATR analysis of carbon-filled polymers, paints, coatings, contaminants, inclusions and glass materials.

The Slide-On ATR has two modes of operation: View and ATR. With the View mode selected, the sample is positioned in the center of the field of view. To acquire a spectrum, the ATR mode is selected and the stage raised to establish contact between the sample and the crystal. To facilitate sample contact, the Slide-On accessory comes with the Contact Alert system, a force-sensing device.

The Ge Tip ATR is conical in shape and allows for deeper physical placement into sampling areas. Its design also allows for deeper IR penetration into the sample and higher energy throughput.

## Manual conventions

The following conventions are used in this manual to draw your attention to important information:

**Note** Notes contain helpful supplementary information. ▲

**Important** Follow instructions labeled “Important” to avoid damaging the system hardware or losing data. ▲

**▲ Caution** Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. It may also be used to alert against unsafe practices. ▲

**▲ Warning** Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury. ▲

**▲ Danger** Indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury. ▲

## Questions or concerns

In case of emergency, follow the procedures established by your facility. If you have questions or concerns about safety or need assistance with operation, repairs or replacement parts, use the information below to contact Thermo Electron. Outside the U.S.A., contact the local Thermo Electron sales or service representative.

**Phone:** 1-800-642-6538 (U.S.A.) or +608-273-5015 (worldwide)

**Fax:** +608-273-5045 (worldwide)

**E-mail:** techsupport.analyze@thermo.com

**World Wide Web:** <http://www.thermo.com/spectroscopy>

# Installing the Slide-On ATR

This chapter provides instructions for installing the Slide-On ATR. The topics covered include:

- Installing the ring mount
- Aligning the Hemispherical ATR crystal
- Aligning the Tip ATR crystal

**Note** If your system was delivered from the factory with the Slide-On ATR installed, you may skip this chapter. ▲

**Note** Your system is shipped in a protective foam filled cardboard box. On arrival please check the box to ensure that all pieces have been received and that no pieces are damaged. Save the box for storage and possible shipment of the kits. ▲

## Installing the ring mount

Installing the ring mount is the first procedure you perform when installing the Slide-On ATR. Use the instructions that follow to install the ring mount.

**Important** Do not touch the lens in the objective with your bare fingers. Skin oils can damage the optical surface. ▲

**Important** Do not handle the nosepiece by the objectives. This can cause the objectives to become misaligned. ▲

### 1. Remove the nosepiece with the objective still attached.

Use a 3/32 inch hex wrench or ball driver to loosen the set screw in the access hole on the right side of the microscope base. Hold the nosepiece by the ring on the top, and slide it off the mount. Once the nosepiece is removed, turn it over and lay it down on a clean, stable surface.



**Note** The black purge baffle comes in two styles, so yours may not look like the one shown below. If this is the case, do not be concerned, these instructions are still applicable. ▲

## 2. Remove the black purge baffle.

Use a 3/32 inch hex wrench or ball driver to remove the three screws that secure black purge baffle to the 15X Refflachromat objective. Do not discard the ring and screws; you will need them later.



### 3. Install the ring mount.

Use the 3/32 inch ball driver to secure the ring mount to the 15X objective with the three screws that are provided. Align the index mark on the ring mount to the zero compensation index mark located on the body of the Reflachromat objective. The orientation of the slide holder should allow the slider to fit easily into the Slide-On ATR.



### 4. Install the nosepiece.

Hold the nosepiece by the ring on the top, and slide it onto the microscope mount. Use the 3/32 inch hex wrench or ball driver to tighten the set screw in the access hole on the right side of the microscope base.

## Aligning the Hemispherical ATR crystal

The Hemispherical ATR crystal is held in the crystal holder by three set screws. (Three .05 inch hex wrenches are provided to adjust the set screws.) The crystal holder is threaded into the crystal slider for height adjustment and is held by a locking set screw, which you access by removing the slider thumbscrew.

If you receive your Hemispherical ATR installed on a new microscope, you do not need to perform an alignment. If your crystal has been replaced or is out of alignment, perform the following procedure.



**Important** Do not loosen any set screw more than one-half turn at a time. If you do not keep the crystal engaged, it could fall out of the crystal holder and damage the tip of the crystal. ▲

**Important** To help avoid a misalignment, do not touch the crystal slider, the Contact Alert plate, or the stage during the alignment procedure. ▲

**1. Use the set screws to adjust for centration.**

“Centration” is the accuracy with which the optical axis coincides with the mechanical axis of the mounting. Use the hex wrenches to simultaneously adjust the set screws. Look at the back of the crystal and try to match the outer radius of the crystal to the inner radius of the mount. Repeat this process until you have achieved centration. After this adjustment is complete, tighten the set screws to make sure the crystal is secure in the crystal holder.



**2. Set the microscope to reflection mode.**

Select the desired detector and set the reflex aperture to 100 x 100 microns.

**3. Insert the crystal slider into the Slide-On ATR.**

Rotate and pull out the spring plunger handle of the ring mount and insert the crystal slider into the center position (ATR mode) of the Slide-On ATR. Release the spring plunger handle to lock the slider in place.



**4. Adjust the height of the crystal in the crystal holder for maximum energy.**

Remove the crystal slider thumbscrew to gain access to the height adjustment set screw. Loosen the set screw until the crystal holder is free to rotate. Open the Bench tab in the Experiment Setup dialog box, and watch the peak-to-peak values of the interferogram in the live display. Rotate the crystal holder in the slider clockwise or counter-clockwise until you achieve the maximum peak-to-peak value, and then tighten the set screw and replace the thumbscrew.



## 5. Check spatial alignment of the crystal.

Hold a glass slide just above the flame of a utility candle until a small area is covered by a uniform layer of soot. Install the slide on the sample stage. Monitor the interferogram energy through the crystal, and gently raise the sample stage until the green contact alert LED lights. You should see a loss of energy when contact is made.

Lower the sample stage so that there is no contact between the sample and the crystal tip. Next, set the Slide-On ATR to view mode. Focus the sample stage and inspect the crystal's impression on the slide. It should be centered in the field of view (reticle). If not, adjust the crystal centration as in step one, but set the crystal to ATR mode until maximum energy is obtained. Repeat the soot test until the crystal's impression is centered in the reticle.

**6. Compare the throughput of the crystal compared to the throughput of a reflective gold slide.**

Place the crystal in view mode, and then place a gold-coated reflective slide on the stage and focus. Collect a background with the following parameters:

- Number of scans: 10
- Spectral resolution: 8 cm<sup>-1</sup>
- Final format: %R
- Preview data collection: On

Next, place the crystal in ATR mode with no surface contact. Collect a sample spectrum with the same parameters as above. The %R at 2000 cm<sup>-1</sup> should be no less than the specification shown in the following table:

Crystal Material	Throughput
Ge ATR	30%
Si ATR	35%
Ge Tip ATR	50%

**Note** The table shown above gives minimum throughput values for three types of ATR crystals as compared to the 15X objective measured at 2000 cm<sup>-1</sup> using a gold reference mirror in reflection mode with the reflex aperture set to 100 x 100µm. ▲

## Aligning the Tip ATR crystal

The Ge Tip ATR crystal is held in the crystal holder by two set screws and a spring-loaded plunger. (A flat blade screwdriver and two .05 inch hex wrenches are provided to adjust the plunger and the set screws.) The crystal holder is threaded into the crystal slider for height adjustment and is held by a locking set screw, which you access by removing the slider thumbscrew.

If you receive your Tip ATR installed on a new microscope, you do not need to perform an alignment. If your crystal has been replaced or is out of alignment, perform the following procedure.



- Important** Do not loosen the plunger more than one-half turn at a time. If spring tension is not maintained, the crystal could fall out of the crystal holder and damage the tip of the crystal. ▲
- Important** To help avoid a misalignment, do not touch the crystal slider, the Contact Alert plate, or the stage during the alignment procedure. ▲

**1. Unlock the crystal from the crystal holder.**

Look at the backside of the crystal and slide, and loosen the ball plunger by turning the flat blade screwdriver one-half turn.



**2. Use the .05 inch hex wrenches to adjust the set screws for centration.**

“Centration” is the accuracy with which the optical axis coincides with the mechanical axis of the mounting. Use the hex wrenches to simultaneously adjust the set screws. Look through the cover on the back of the crystal and try to match the outer radius of the crystal to the inner radius of the mount. Repeat this process until you have achieved centration. After this adjustment is complete, use the flat blade screwdriver to tighten the ball plunger. This locks the crystal in the crystal holder.



**3. Set the microscope to reflection mode.**

Select the desired detector and set the reflex aperture to 100 x 100 microns.

**4. Insert the crystal slider into the Tip ATR.**

Rotate and pull out the spring plunger handle of the ring mount and insert the crystal slider into the center position (ATR mode) of the Slide-On ATR. Release the spring plunger handle to lock the slider in place.



**5. Adjust the height of the crystal in the crystal holder for maximum energy.**

Remove the crystal slider thumbscrew to gain access to the height adjustment set screw. Loosen the set screw until the crystal holder is free to rotate. Open the Bench tab in the Experiment Setup dialog box, and watch the peak-to-peak values of the interferogram in the live display. Rotate the crystal holder in the slider clockwise or counter-clockwise until you achieve the maximum peak-to-peak value. When this is done, tighten the set screw and replace the thumbscrew.



## **6. Check spatial alignment of the crystal.**

Hold a glass slide just above the flame of a utility candle until a small area is covered by a uniform layer of soot. Install the slide on the sample stage. Monitor the interferogram energy through the crystal, and gently raise the sample stage until the green contact alert LED lights. You should see a loss of energy when contact is made.

Lower the sample stage so that there is no contact between the sample and the crystal tip. Next, set the Slide-On ATR to view mode. Focus the sample stage and inspect the crystal's impression on the slide. It should be centered in the field of view (reticle). If not, adjust the crystal centration as in steps one and two, but set the crystal to ATR mode until maximum energy is obtained. Repeat the soot test until the crystal's impression is centered in the reticle.

## 7. Compare the throughput of the crystal compared to the throughput of a reflective gold slide.

Place the crystal in view mode, and then place a gold-coated reflective slide on the stage and focus. Collect a background with the following parameters:

- Number of scans: 10
- Spectral resolution: 8 cm<sup>-1</sup>
- Final format: %R
- Preview data collection: On

Next, place the crystal in ATR mode with no surface contact. Collect a sample spectrum with the same parameters as above. The %R at 2000 cm<sup>-1</sup> should be no less than the specification shown in the following table:

Crystal Material	Throughput
Ge ATR	30%
Si ATR	35%
Ge Tip ATR	50%

**Note** The table shown above gives minimum throughput values for three types of ATR crystals as compared to the 15X objective measured at 2000 cm<sup>-1</sup> using a gold reference mirror in reflection mode with the reflex aperture set to 100 x 100µm. ▲



# Using the Slide-On ATR

This chapter provides instructions for using and maintaining the Slide-On ATR and the ATR crystal. The topics that are covered include:

- Analyzing samples using internal Contact Alert
- Analyzing samples using external Quantitative Contact Alert
- Cleaning the ATR crystal

## Using internal Contact Alert

The Contact Alert is a pressure sensing system which is used with Thermo Electron's ATR objectives and Slide-On ATR to indicate and monitor contact force between the sample and ATR crystal. When either the internal Contact Alert or Quantitative Contact Alert system is activated, it indicates and monitors contact force between the sample and ATR crystal when performing ATR experiments.

### 1. Install the Contact Alert plate in the stage slide holder.



### 2. Connect the sensor plate to the microscope.

Connect the sensor plate cable to the nine-pin cable port labeled INTERNAL at the front of the microscope.

**Important** Use a glass microscope slide with cellulose tape covering the bottom surface as a mounting platform for samples. This will act as a safety device in case excessive pressure is applied to the crystal. If this happens, the glass will break instead of the crystal, and the tape will prevent broken glass from dropping into the condenser. Always place the sample in the center of the slide so that there is uniform distribution of pressure. ▲

**Important** Samples should be smaller than the glass slide, but if the sample is larger than the slide, place a taped glass slide on the sensor plate first, and then place the sample on the glass slide. Make sure the green and red LEDs will light properly by applying gentle pressure to the center of the slide with your finger. ▲

- 3. Set the microscope to reflection mode, and then place the sample slide on the Contact Alert sensor plate.**
- 4. Place the crystal slider in View mode, and then focus on and center the desired data collection area.**
- 5. Place the slider in ATR mode, and then collect a background spectrum with the ATR out of contact with the sample.**

If the sample has uneven topology, it may be necessary to lower the stage before sliding the crystal in place in order to clear any obstructions.

**Note** If you have Thermo Electron's OMNIC™ Atμs™ software, use the Auto-Contact feature to automatically get optimal contact between the crystal and the sample. ▲

**Important** Excessive pressure can damage the sample or the ATR crystal. ▲

**6. Use the Z-focus adjustment knob to raise the sample stage.**

Stop when the green LED lights. This tells you that the sample area has made contact with the ATR crystal.

**7. Raise the sample stage slowly.**

Use one of the following techniques:

- Slowly raise the stage and stop immediately when the red LED lights. This indicates that the contact pressure of the sample area to ATR crystal is optimal for infrared analysis.
- Collect the sample in Spectral Preview mode and watch the live display. Slowly raise the stage until it contacts the ATR crystal and the red LED lights. Select the best spectrum for the current analysis. (This may occur before the red LED lights.)

**8. Collect the sample spectrum.**

## Using external Quantitative Contact Alert

The Quantitative Contact Alert system features LED contact indication, as well as an adjustable audible alarm and analog meter deflection to measure and program highly reproducible contact pressure to  $\pm 1\%$  variation. Enabling this system will deactivate the internal Contact Alert LEDs located on the front panel of the microscope and must be used manually, not in Auto ATR Contact mode.

1. **Install the Contact Alert plate in the stage slide holder.**



2. **Connect the sensor plate cable to the microscope cable connector.**

The microscope cable connector is a nine-pin cable connector labeled “EXTERNAL” and is located on the front of the microscope.

- 3. Connect the communications cable from the Quantitative Contact Alert controller to the Contact Alert port on the side panel of the microscope.**
  
- 4. Connect the power cable of the Quantitative Contact Alert controller to a wall outlet or power strip, and then turn the controller power switch to the ON position.**
  
- 5. Adjust Low Alarm offset.**

The weight of the sample is sometimes sufficient to trigger the low alarm. (When this happens, the Alarm LED lights, a buzzer sounds, and the meter deflects.) If the low alarm is triggered when the sample is mounted on the sensor plate, turn the Offset adjustment counter-clockwise until the alarm no longer triggers. This offset circuit can correct for samples weighing up to 50 grams. Adjust the pressure between 5-9 for hard materials and 2-4 for elastomeric materials. (See the Quantitative Contact Alert User's Manual for more information.)

# Maintenance and Service

This chapter provides the following maintenance and service procedures:

- Cleaning the ATR crystal
- Replacing the Hemispherical ATR crystal
- Replacing the Tip ATR crystal

## **Cleaning the ATR crystal**

To clean the ATR crystal, rotate the nosepiece so that the crystal is accessible, or remove the slide-on mount. Use lens tissue and either water, methanol, ethanol, or isopropanol to clean the crystal.

**Important** Do not use acetone or chlorinated or strong solvents to clean the ATR crystal. These solvents will dissolve the adhesive used to attach the crystal to the slider and can damage the anti-reflective coating on the dome-shaped top of the Ge Tip ATR crystal. ▲

**Important** The conical surface of the Ge Tip ATR crystal is coated with aluminum to protect the germanium surface from contamination. Do not use abrasive materials to clean the Ge Tip ATR crystal. Using abrasive materials may cause the aluminum to peel off the surface of the crystal. ▲

## Replacing the Hemispherical ATR crystal

Use the following procedure to replace the Hemispherical ATR crystal:

1. Use the .05 inch hex wrenches to loosen the three set screws.

Look at the back side of the crystal as you loosen the set screws.



2. Remove the old crystal from the slider, and then install the new crystal.

**3. Use the .05 inch hex wrench to tighten the three set screws.**

Adjust and tighten the hex screws until the crystal is visually centered in the crystal holder.

**4. Proceed to the first step of “Aligning the Hemispherical ATR crystal” in the “Installing the Slide-On ATR” chapter.**

## Replacing the Tip ATR crystal

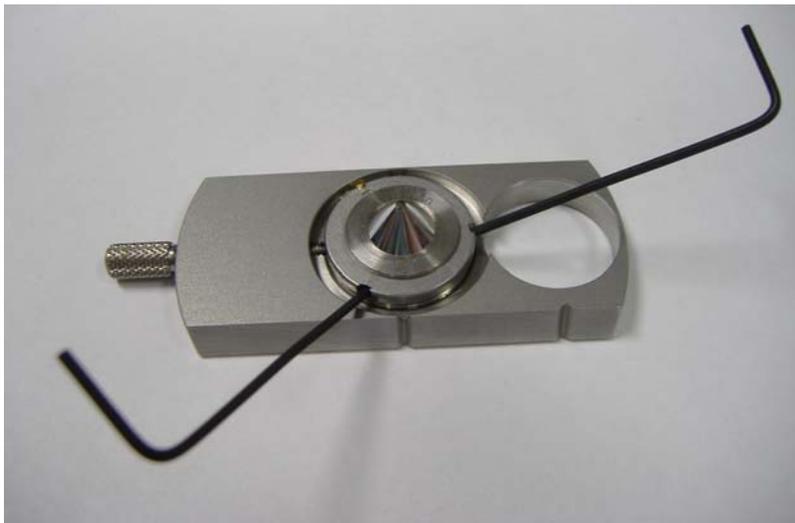
Use the following procedure replace the Tip ATR crystal:

**1. Unlock the crystal from the crystal holder.**

Look at the backside of the crystal and slide, and loosen the ball plunger by turning the flat blade screwdriver one-half turn.



2. Use the .05 inch hex wrenches to loosen the two set screws.



3. Remove the old crystal from the slider, and then install the new crystal.

4. Use the .05 inch hex wrench to tighten the two set screws.

Adjust and tighten the hex screws until the crystal is visually centered in the crystal holder.

5. Proceed to the first step of “Aligning the Tip ATR crystal” in the “Installing the Slide-On ATR” chapter.