

ATR Objective Users Manual

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Thermo Spectra-Tech

P/N 700-0004

Version 2.9

A Thermo Electron business

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General Information

The Manual

This manual is designed as a tutorial to guide you through the installation of the ATR objective and through a typical ATR microscopy analysis. It is recommended, however, that you familiarize yourself with the operation of your FT-IR microscope before using the ATR objective.

Packing & Unpacking

The ATR objective is shipped in a protective foam filled box. Upon 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 shipment of the kits.

Technical Support Center

Technical materials describing the use and theory of attenuated total reflectance, diffuse reflectance and specular reflectance are available from Thermo Spectra-Tech. Additionally, a team of scientists is available at Thermo Spectra-Tech to answer any of your questions. If you encounter any problems or difficulties, or desire additional information please contact the Technical Support Center at 800-THE-FTIR.

Copyrights & Trademarks

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The ATR objective is a trademark of Thermo Spectra-Tech.

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General Information

Technical Specifications

Magnification:	Dual 15X/25X
Numerical Aperture:	0.26/0.87
Range of Incidence:	45° ($\pm 5^\circ$) in ATR mode
Working Distance:	3 mm in survey mode
Tube Length:	142 mm
Objective Type:	Tri-mode Objective
Optics:	<i>Survey Mode:</i> Refracting lenses combined with ATR crystal. <i>Contact Mode:</i> Schwarzschild design. Combination of reflecting optics and high-refractive index crystal. <i>ATR Mode:</i> Schwarzschild design. Combination of reflecting optics and high-refractive index crystal.
Crystal Material:	Zinc Selenide (ZnSe)

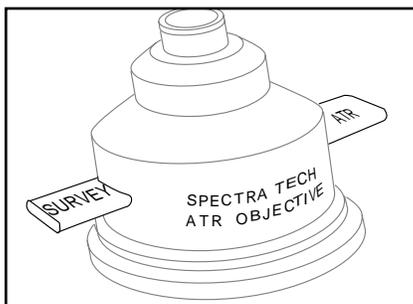
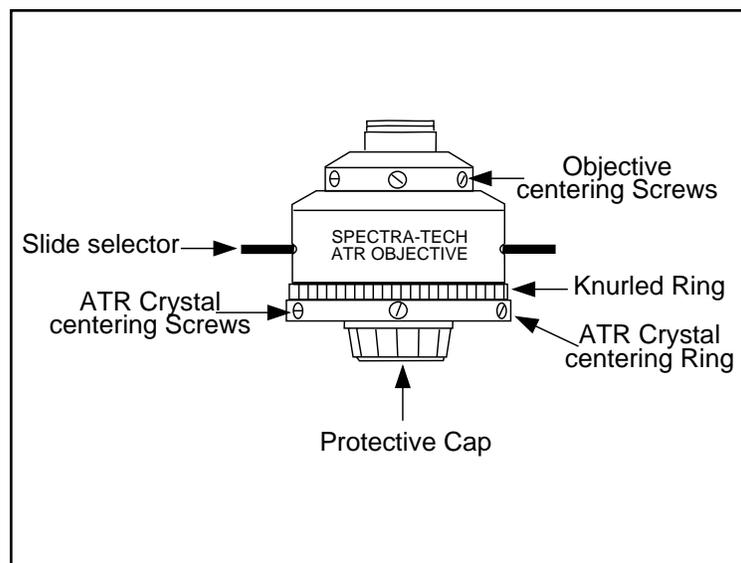
Introduction to ATR Microscopy

Internal reflection spectral measurements, commonly called ATR, is an established method for infrared analysis. The Attenuated Total Reflectance Objective (ATR) has been developed to allow the high- sensitivity FT-IR analysis of difficult, but not uncommon samples on the microscopic scale. Routine analysis of highly absorbing materials is practical with this objective due to the reproducible, short pathlength of ATR. Additionally, since the technique is noninvasive many materials can be analyzed without sample preparation or sample destruction. Plus, ATR often provides unique information about the structure of the material being analyzed. Some applications of this technique include:

- analysis of glass-filled fibers
- analysis of biomaterials
- surface degradation of polymer studies
- quantitative analysis

Getting Started

Product Description



Understand the slide selector- Contact Mode

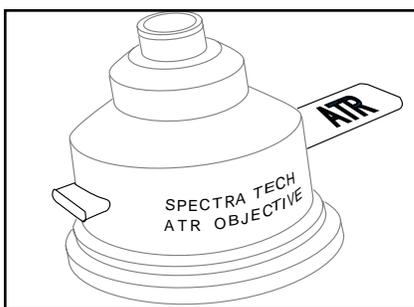
Examine the ATR objective with the words "ATR Objective" facing you.

Position the survey/contact/ATR (mode) selector so that the word "ATR" is visible on the right and the word "Survey" is visible on the left.

The mode selector is now in the contact mode.

Getting Started

Product Description

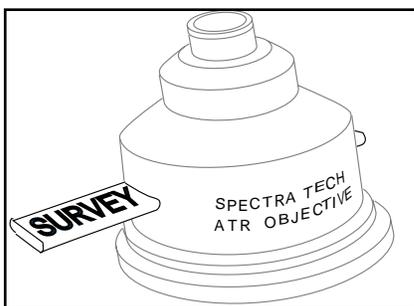


Understand the slide selector- ATR Mode

Examine the ATR objective with the words "ATR Objective" facing you.

Position the mode selector (survey/contact/ATR) so that only the word "ATR" is visible.

The mode selector is now in the ATR mode.



Understand the slide selector- Survey Mode

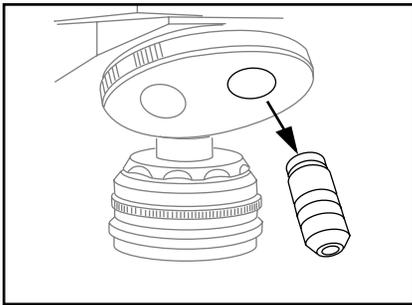
Examine the ATR objective with the words "ATR Objective" facing you.

Position the mode selector (survey/contact/ATR) so that only the word "Survey" is visible.

The mode selector is now in the Survey mode.

Getting Started

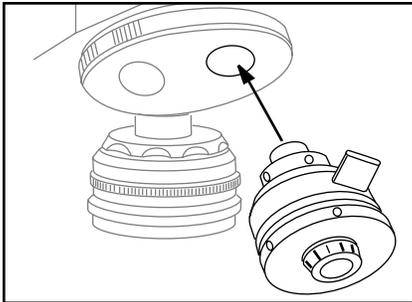
Installation & Alignment



Prepare microscope for ATR Objective

Because the IR objectives are too large to fit in adjacent positions, the ATR Objective must be installed on the 4-place nosepiece *opposite* to any other infrared objective.

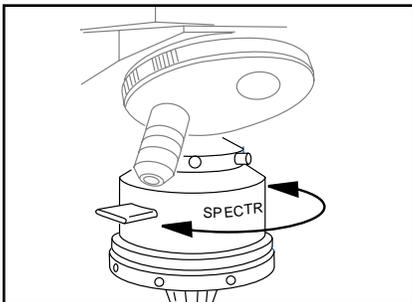
NOTE: If more than one IR objective is presently mounted on the nosepiece, you must remove one to allow room for the ATR objective. We suggest you leave either the 15X or 32X objective.



Install ATR on nosepiece

Rotate the 4-place nosepiece as shown. Firmly screw the ATR objective into the empty position (opposite the 15X or 32X objective).

NOTE: The mode selector should be in the Contact mode.



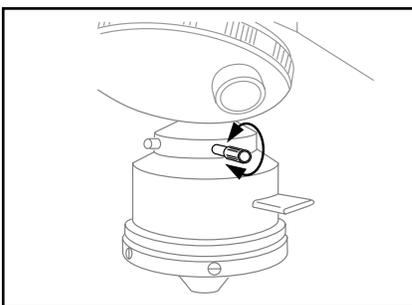
Proper Orientation of the Objective

The recommended orientation is to have the Thermo Spectra-Tech label facing the user and the mode selector should be oriented east-west with "survey" to the left and "ATR" to the right.

Note: Proper positioning of the objective is necessary to prevent the slide selector from striking other objectives on the nosepiece.

Getting Started

Installation & Alignment



Orient Objective

If the objective is not oriented properly, the following procedure must be followed:

Loosen the objective centering screws on the objective.

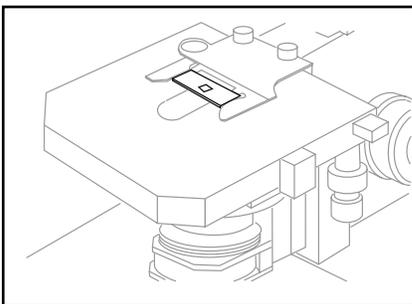
Rotate the objective to the proper position (so that the "Spectra-Tech ATR Objective" faces out..

Tighten the screw.

Prepare microscope for viewing

Rotate the 15X or 32X objective into position.

Put the microscope into the *Reflectance View* mode.



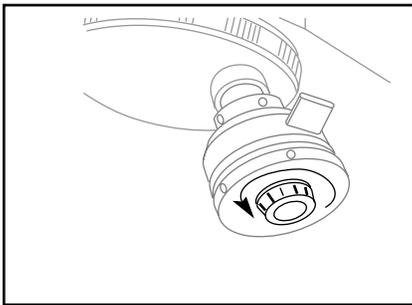
Establish the Reference

Place the 100 μ pinhole mount on the stage and fasten it securely using the stage clips.

View the pinhole with either the 15 or 32X objective and center the pinhole in the crosshair using the x,y stage controls.

Getting Started

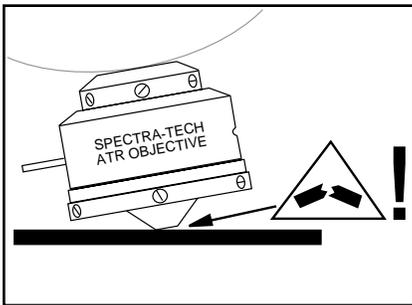
Installation & Alignment



Remove cap

Unscrew the cap which covers the ATR crystal.

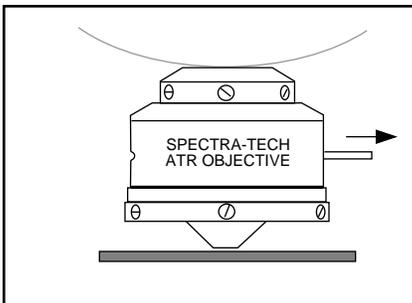
Note: Before carrying out the next step, note which way to move the microscope controls to LOWER the stage.



Adjust ATR objective

Lower the stage and rotate the ATR objective into position.

CAUTION: Before swinging the ATR objective into position, always lower the stage sufficiently to provide clearance for the ATR Objective. Lowering the stage as far down as possible is recommended.



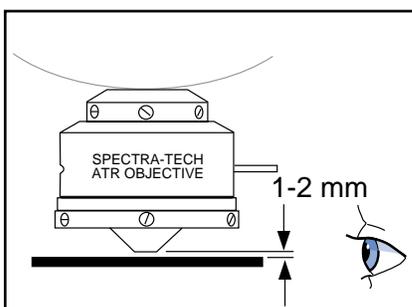
Select the survey mode

Slide the Survey/Contact/ATR Selector (on the ATR objective) into the *survey* position.

NOTE: There should *not* be an aperture in the upper aperture position.

Getting Started

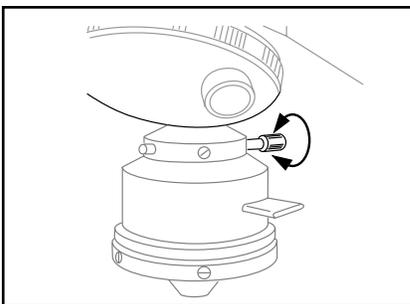
Installation & Alignment



Refocus on the pinhole

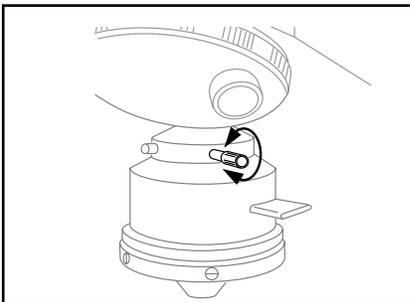
Re-focus on the pinhole

Note: While viewing from the side, raise the sample so that it is nearly touching the ATR crystal. While viewing through the eyepieces, lower the stage (using the Fine Focus Adjustment knob.) until the pinhole is in focus.



Loosen ATR Objective centering screws

Loosen every other screw on the top ring of the objective (3 full turns) using one of the centering tools provided.



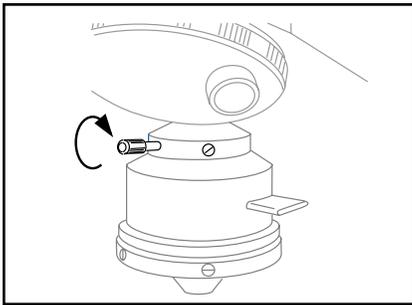
Adjust ATR Objective centering screws

Insert the three centering tools into the screw holes which were not loosened in the preceding step.

Center the pinhole on the crosshairs by adjusting these 3 screws. (This is done by first loosening one screw, then tightening another. All three screws should be snug at the end of each such adjustment.)

Getting Started

Installation & Alignment



Lock position

Re-tighten all six screws to lock the position (while viewing the pinhole to be certain that it remains centered).

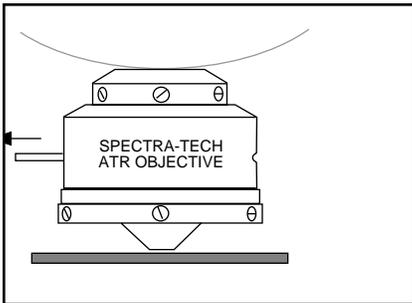
Repeat the centering steps to confirm that the ATR and standard 15X or 32X objectives are concentric.

Ensure that the ATR crystal is aligned properly.

Slide the Survey/Contact/ATR Selector (on the ATR objective) into the ATR position.

IR μ s/Nic-Plan Microscopes: A well-defined image of the circular aperture should be visible. If the image is not sharp, turn the lower knurled ring to focus the aperture image on the ATR crystal.

IR-Plan Microscopes: The ATR Crystal Focus Ring may be turned to produce a brighter image.



Measure signal throughput

Slide the mode (Survey/Contact/ATR) selector (on the ATR objective) into the *ATR* position.

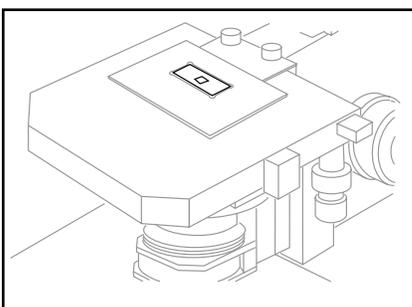
Insert the 2.5mm aperture into the upper aperture position.

With the microscope in the *IR Reflectance* mode, check the energy throughput.

Note through-put for later reference. (It should be 20-30% of that measured using the 15X objective in the reflectance mode, using the 1.5 mm fixed aperture and a gold mirror in the sample position).

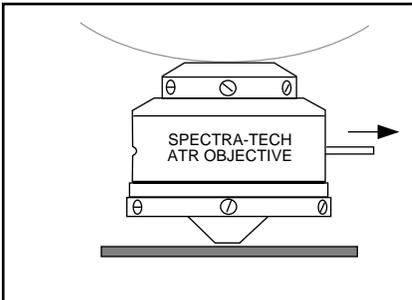
Operation

Performing an ATR Experiment



Place sample on stage

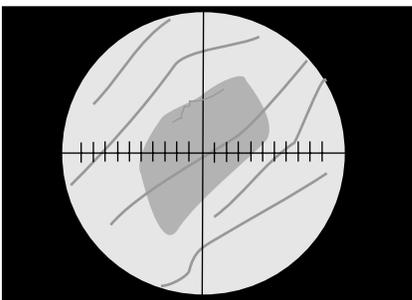
Place the sample securely on the stage. Fasten with the stage clips.



Adjust microscope controls

Put the microscope into the *Reflectance Viewing mode*.

Slide the mode (Survey/Contact/ATR) selector (on the ATR objective) into the *Survey* position.



Locate area of sample for IR analysis*

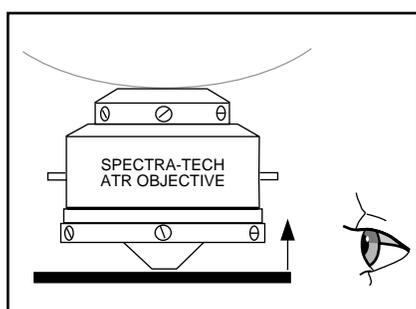
Position the area for IR analysis in the center of the field of view.

Note: The illumination intensity should be at its maximum.

* For low reflecting samples, the 15 or 32X Reflachromat objective may be used instead of the survey mode since both reflachromats are coaxial with the ATR Objective..

Operation

Performing an ATR Experiment



Establish contact

Slide the Survey/Contact/ATR Selector (on the ATR objective) into the *Contact* (center) position and insert the 2.5mm upper aperture.

While viewing from the side, use the coarse focus adjustment knob to raise the sample up so that it is just below the ATR crystal.

While looking through the eyepieces, use the fine focus adjustment knob to raise the sample so that it contacts the ATR crystal .

Note: If sample is not flat, it might be necessary to tilt the sample.

NOTE: While excellent spectra of hard materials (such as paint on metal) can be acquired with the ATR objective, these samples naturally create a much greater risk of scratching or breaking the ATR crystal.

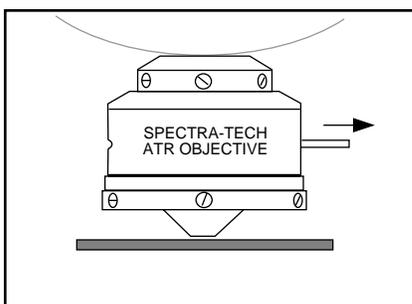
Adjust apertures

Install the 2.5 mm (100 μ equivalent) Circular Sampling Aperture into the upper aperture mount.

If the sample does not fill the 2.5 mm Circular Sampling Aperture, remove the circular aperture and insert the variable rectangular aperture. Adjust the blades to conform with the sample's geometry and dimensions.

Operation

Performing an ATR Experiment



Set ATR objective for IR spectral measurement

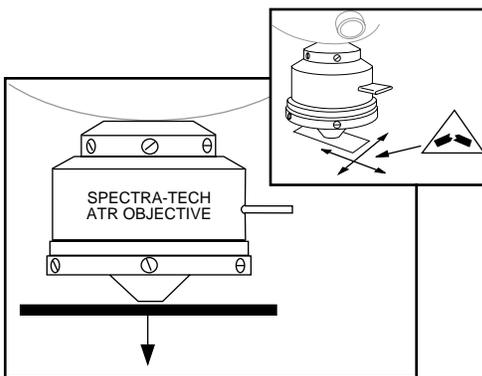
Move the Survey/Contact/ATR Selector Slide (on the ATR objective) into the *ATR* position.

Put the microscope into the *IR Reflectance* mode.

Collect a Sample Single-Beam Spectrum

Collect a sample single-beam spectrum.

- a. Check energy.
- b. Set gain.
- c. Collect long enough to achieve adequate signal-to-noise (typically 1-2 minutes).
- d. 8 cm^{-1} resolution is suggested for initial ATR experiments.

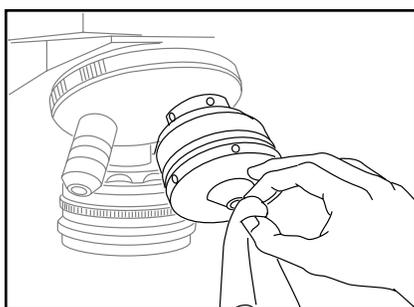


Break contact

Without adjusting or changing the aperture, use the Coarse Focus Adjustment to **lower** the sample stage.

Operation

Performing an ATR Experiment

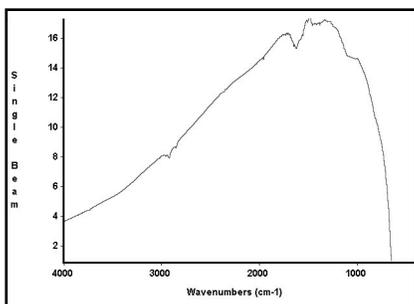


Clean crystal

Rotate the nosepiece so the ATR crystal is accessible.

Wipe the ZnSe crystal with a soft tissue (lens or facial tissue).

If necessary, moisten the tissue with an appropriate solvent (e.g. distilled water or isopropanol).



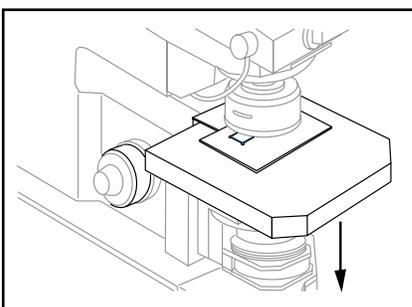
Collect a Background Spectrum & Ratio

Collect a Background single-beam spectrum.

Ratio the background single-beam spectrum to the previously acquired sample single-beam spectrum.

Appendix A

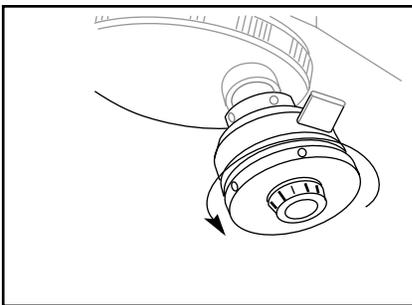
Replacing the ATR Crystal



Lower stage

Use the Coarse Focus Adjustment knob to lower the stage.

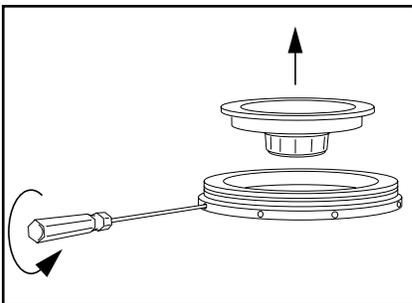
Replace the protective cap (covering the crystal).



Remove the crystal nose cone

With one hand, turn the bottom-most ring on the ATR Objective counter-clockwise while holding the ring just above it with the other hand to remove the crystal nose cone. (If the ring is difficult to turn, loosen the ATR crystal centering screws.

Note: Leave the protective cap covering the crystal in place.



Remove the crystal

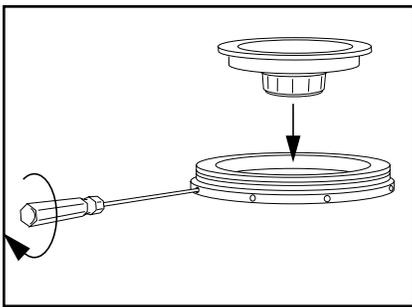
Using one of the ball drivers provided (.050), back every other screw (on the crystal nose cone) three full turns.

Loosen one of the *remaining* screws until the cone holding the crystal drops out.

Note: While only 3 screws are needed for centering, the 3 additional screws are provided to lock the position.

Appendix A

Replacing the ATR Crystal

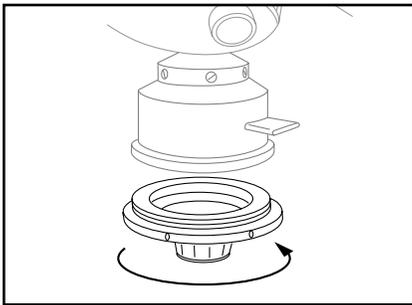


Replace the crystal

Place the new crystal in the nose cone assembly into the centering ring..

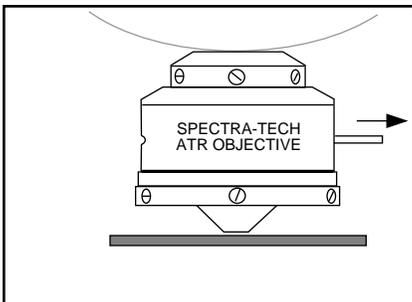
Tighten three of the six screws to hold the crystal in place, leaving three screws loose..

Note: Leave the protective cap over the crystal.



Install the nose cone

Carefully thread the crystal nose cone assembly in the objective.



Adjust microscope controls

Put the microscope into the *Reflectance Viewing mode*.

Slide the Survey/Contact/ATR Selector (on the ATR objective) into the *ATR* position.

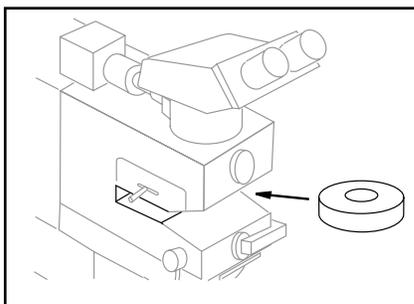
Appendix A

Replacing the ATR Crystal

Install aperture (Nic-Plan™ & IR μ s only)

Place the 2.5mm fixed circular aperture in the upper aperture position.

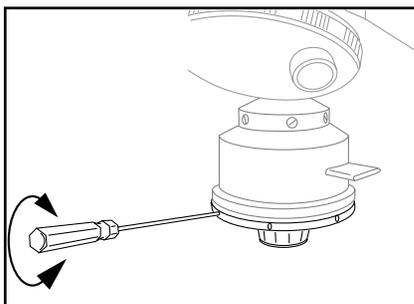
Turn the ATR crystal focus knurled knob on the ATR nose cone 4-6 turns until the image of the upper aperture is in sharp focus. If this image is distorted it is necessary to adjust the three centering screws alternately until the image becomes sharp.



Install aperture (IR-Plan® only)

Place the 2.5mm fixed circular aperture in the upper aperture position.

Turn the knurled knob on the ATR nose cone 4-6 turns until the aperture is filled with a yellow glow (from the ZnSe crystal). If this image is distorted it is necessary to adjust the three centering screws alternately until the image becomes uniformly bright

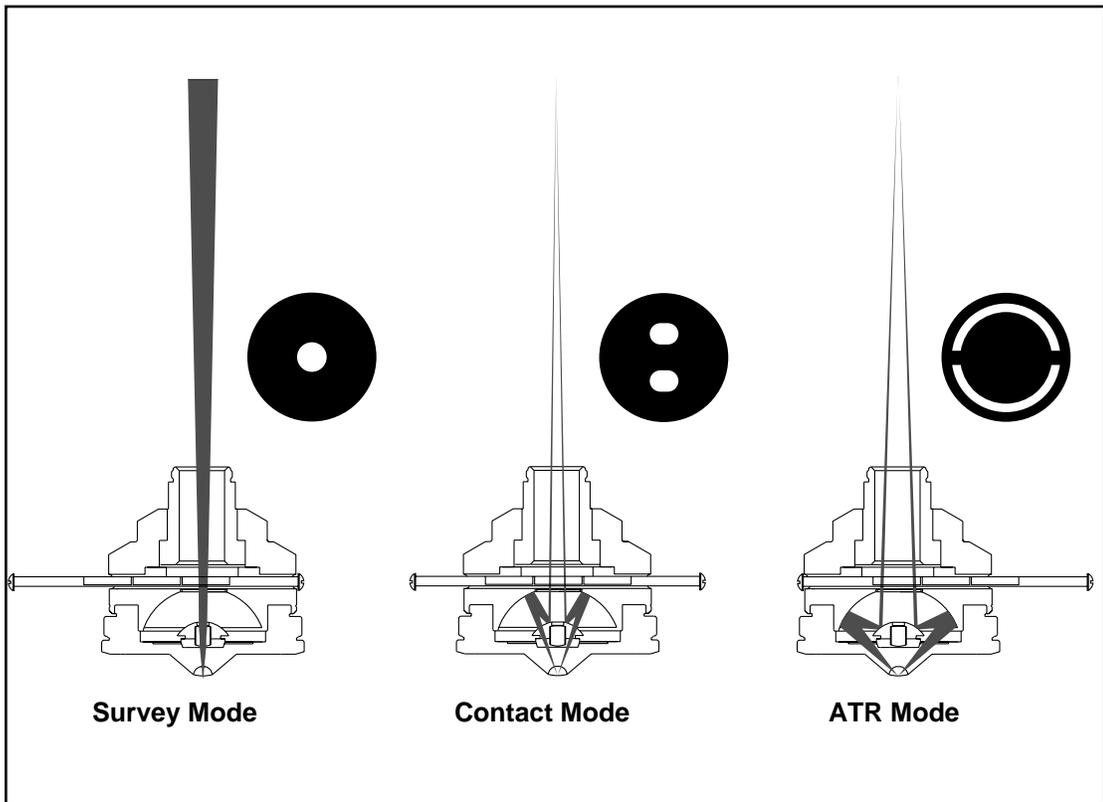


Tighten centering screws

Alternately tighten the three *other* crystal centering screws (to lock in the centration) while ensuring that the image remains in sharp focus (Nic-Plan & IR μ s), or continues to fill the aperture with a yellow glow (IR-Plan).

Note: It might be necessary to readjust the previously loosened centering screws during the tightening process to maintain the sharp image.

Appendix B



Thermo Spectra-Tech

Empowering Your FT-IR

A Thermo Electron business

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