INFINITY SERIES
ATR OBJECTIVE
P/N 0045-4XX
USER'S MANUAL

Version 1.0

P/N 700-0133

SPECTRA-TECH

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General Information

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 non-evasive, many materials can be analyzed without sample preparation or sample destruction. ATR also 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

For more information to select the most appropriate crystal material for your type of samples, please refer to Spectra-Tech's Product Data Sheet #8: *ATR Microscopy.*

Theory

For a complete description of the theory of Attenuated Total Reflectance please refer to Spectra-Tech's FT-IR Technical Note #1: *Introduction to Attenuated Total Internal Reflectance (ATR) Spectroscopy.*

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 experiment. However, you should be familiar with the operation of your FT-IR microscope before using the ATR objective.

Note! Your ATR Objective should be installed by a qualified Service Engineer to ensure optimum performance.

Technical Specifications

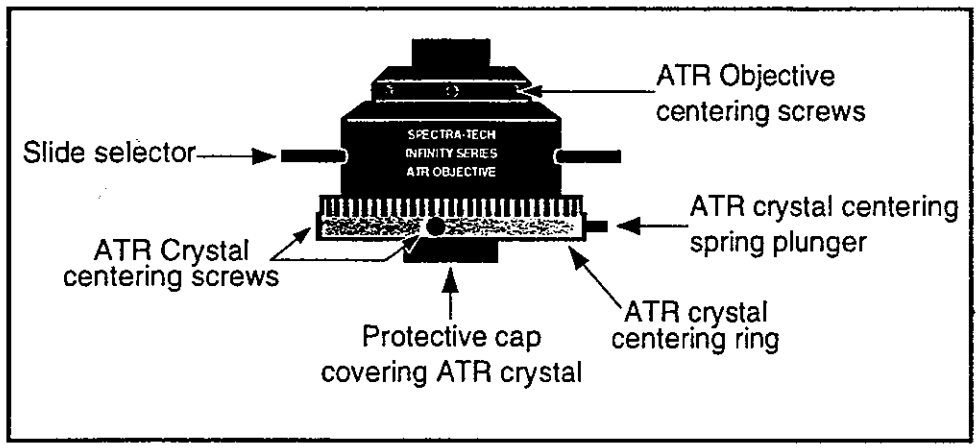
Magnification:	Dual 15X/25X
Numerical Aperture:	0.26/0.87
Range of Incidence:	45° (±5°) in ATR mode
Working Distance:	3 mm in survey mode
Tube Length:	Infinity
Objective Type:	Tri-mode Objective
Optics:	*Survey Mode*: Refracting lenses combined with ATR crystal.
	Contact Mode: Schwarzschild design. Combination of reflecting optics and high-refractive index crystal.
	ATR Mode: Schwarzschild design. Combination of reflecting optics and high-refractive index crystal.
Crystal Materials:	Zinc Selenide (ZnSe)
	Diamond
	Silicon (Si)
	Germanium (Ge)

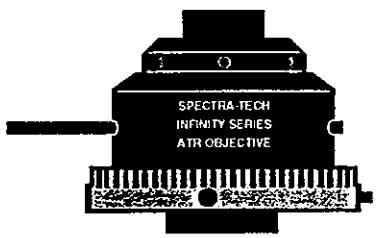
Technical Support

Technical materials describing the use and theory of ATR Spectroscopy are available from Spectra-Tech. Additionally, a team of scientists is available at Spectra-Tech to answer any questions that you may have concerning ATR Microscopy and the use of the ATR Objective. If you have any questions regarding the ATR Objective, please contact a technical representative at **1-800-243-9186**.

Product Description

The ATR Objective is a unique and dedicated accessory specially designed to optimize ATR microscopy experiments. Preparing the three modes of operation (CONTACT, SURVEY, and ATR) are easily achieved by moving the Slide selector to the desired position.
The Contact Alert System; a pressure sensing device ensures repeatable sample/crystal contact force and also protects the crystal from inadvertent damage. The *internal* Contact Alert System (which comes standard with all ATR objectives) uses an LED display on the microscope's front panel to signify contact has been established and appropriate pressure has been applied. The optional, *external* Quantitative Contact Alert System has a programmable controller to measure contact pressure and an audible alarm to enhance crystal protection.





Survey Mode

The SURVEY mode is composed of a lens assembly which provides the ability to view the sample through Zinc Selenide and Diamond ATR crystals. Germanium and Silicon ATR crystals are too opaque to view through.

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

Position the Slide selector so that only the word SURVEY is visible.

The slide selector is now in the ***SURVEY*** mode.

Product Description

Contact Mode

The CONTACT mode allows direct viewing of contact between the sample and the ATR crystal. The image should be bright yellow and will go dark when contact is made.

It is also possible to use the single beam in the spectrometer software to monitor sample/crystal contact. The presence of sample bands will be observed when contact is made. For opaque crystals such as Germanium and Silicon, this method is especially useful when the slide selector is in the ATR mode

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

Position the Slide selector so that the word ATR is visible on the right and the word SURVEY is visible on the left.

ATR Mode

The ATR mode allows the selection of light optimal for ATR infrared data collection. Use this mode only when collecting data or aligning a Germanium or Silicon crystal.

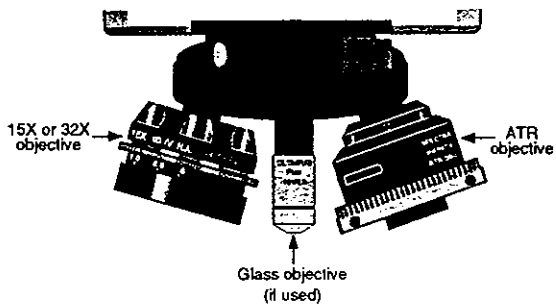
For opaque crystals such as Germanium and Silicon, it is also possible to use the single beam in the spectrometer software to monitor sample/crystal contact. The presence of sample bands will be observed when contact is made.

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

Position the Slide selector so that only the word ATR is visible.

The Slide selector is now in the ***ATR*** mode.

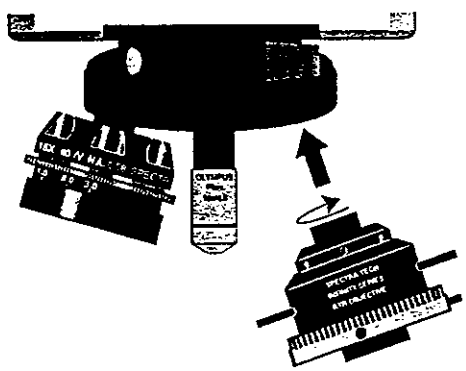
Zinc Selenide and Diamond ATR Objectives



Prepare microscope for ATR Objective

Due to their size, IR objectives must be placed in *opposite* ports on the 4-place nosepiece. Glass objectives (if used) can be placed in adjacent ports.

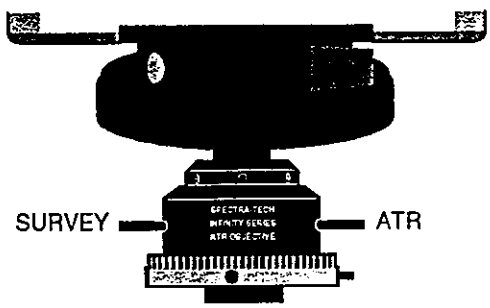
NOTE: If two IR objectives are presently mounted on the nosepiece, you must remove one to allow room for the ATR objective. We strongly recommend you leave either the 15X or 32X objective for centering the ATR Objective.



Install ATR on nosepiece

Carefully screw on the ATR objective in a clockwise direction.

Note: The slide selector should be in the *Contact* mode and the ATR crystal protective cap should be on to avoid damage to the crystal.

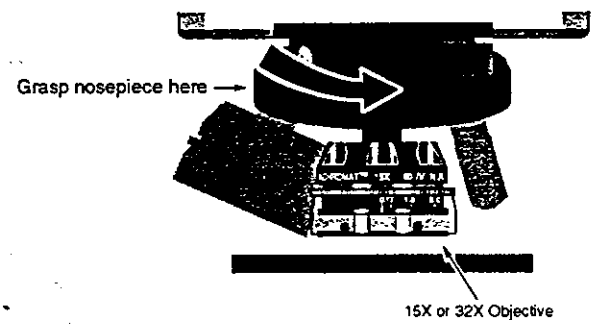


Proper Orientation of the ATR Objective

The recommended orientation is to have the Spectra-Tech label facing the user and.the slide selector should be oriented side to side 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.

Zinc Selenide and Diamond ATR Objectives



Orient ATR Objective

If the ATR objective is **not** oriented properly (facing forward), the following procedure must be followed:

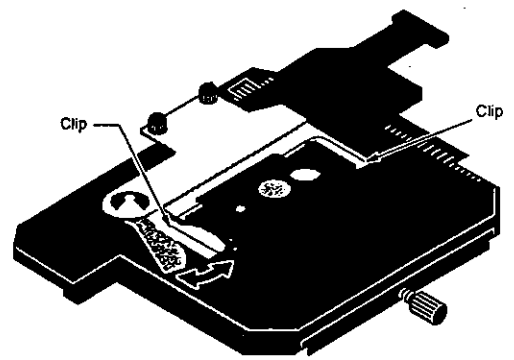
Prepare microscope for viewing

Grasp the black knurled section of the nosepiece and rotate the 15X or 32X objective into position.

Caution: Do not rotate by holding the objectives.

Put the microscope into the *Reflection* mode.

Adjust the reflection illumination to a comfortable viewing level.

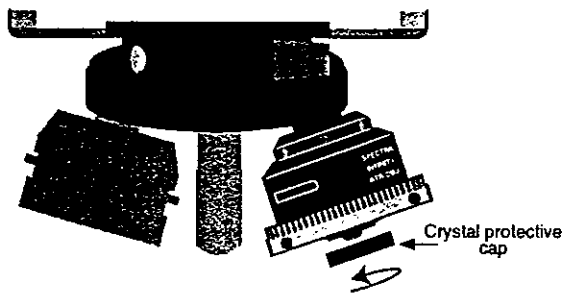


Establish the Reference

Place the100µm 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.

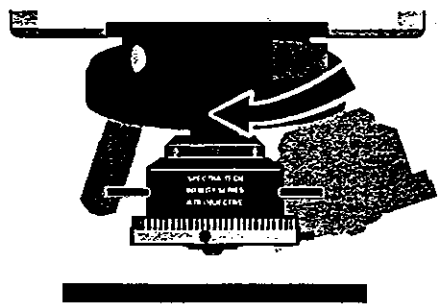
NOTE: Verify the Reflex aperture is open to its maximum position.

Zinc Selenide and Diamond ATR Objectives



Remove Crystal protective cap

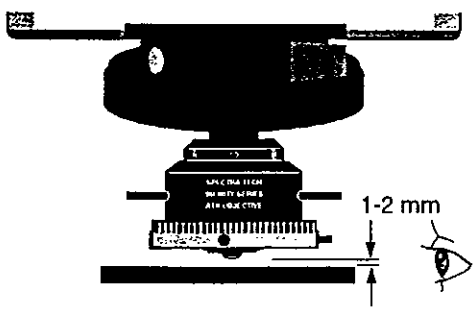
Unscrew the cap (counter clockwise) which covers the ATR crystal.



Select ATR objective

Grasp the black knurled section of the nosepiece and rotate the ATR Objective into position.

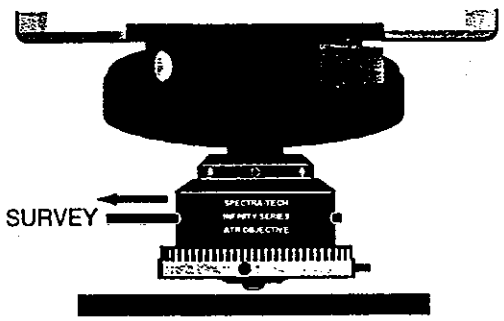
CAUTION! Be sure there is clearance above the stage before selecting the ATR Objective.



Adjust stage

While viewing from the side, bring the stage (with pinhole) 1-2mm from the crystal.

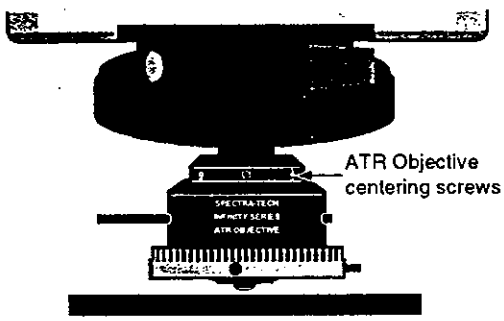
Zinc Selenide and Diamond ATR Objectives



Select survey mode and focus on pinhole

Place the slide selector into the *Survey* mode and refocus on the pinhole.

NOTE: The focus of the crystal is approximately 2mm with Zinc Selenide and 1mm with Diamond from the sample surface. One method to ensure proper focus is to raise the stage 1mm beyond focus of the sample then lower the stage to obtain a sharp image.

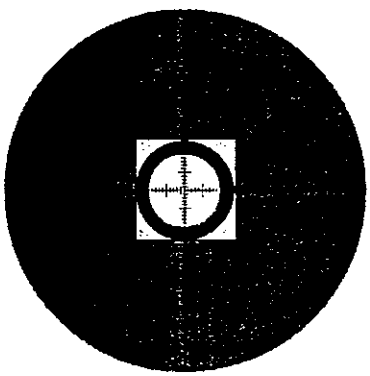


Loosen ATR Objective centering screws

Using a 5/64 balldriver, loosen every other screw on the top section of the objective (3 full turns).

Now loosen only one of the remaining setscrews and rotate the objective to the proper position (so that the engraved "SPECTRA-TECH - INFINITY SERIES - ATR OBJECTIVE" faces forward.

Tighten the screw.



Center pinhole by adjusting ATR objective centering screws

Insert three 5/64 balldrivers into the screw holes which were **not** loosened in the preceding step.

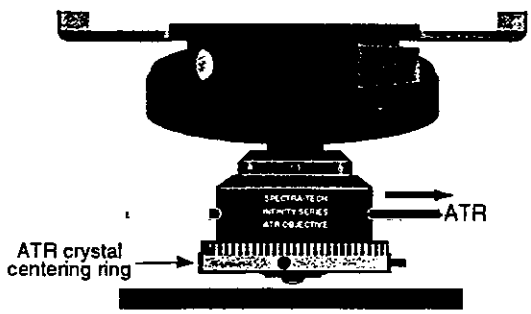
Center the pinhole on the crosshairs by adjusting these 3 screws. This is done by first loosening one screw, then tightening another. All three screws should be snug at the end of each such adjustment.

Zinc Selenide and Diamond ATR Objectives

Lock position

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

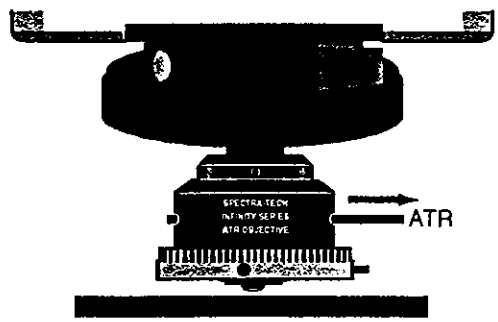
Repeat the centering steps to confirm that the centered pinhole as viewed through the ATR Objective and the standard 15X or 32X objectives are concentric.



Ensure that the ATR crystal is aligned properly.

Place the slide selector into the *ATR* mode.

An image of the circular aperture should be visible. If the image is not sharp, slightly turn the ATR crystal centering ring (the lower knurled ring) in either direction to focus the aperture image on the ATR crystal.



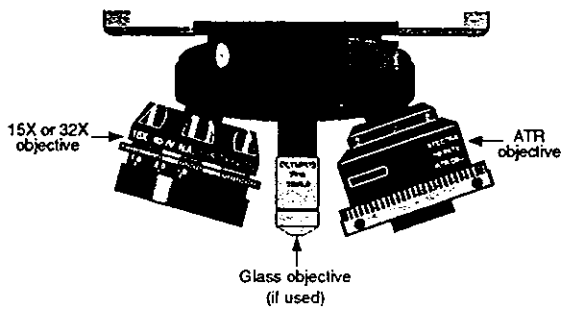
Measure signal throughput

With the slide selector in the *ATR* mode and the microscope in the *Reflection* mode, check the energy throughput.

Note throughput for later reference.

Refer to *Appendix B*; the Crystal Throughput Table for minimal specifications.

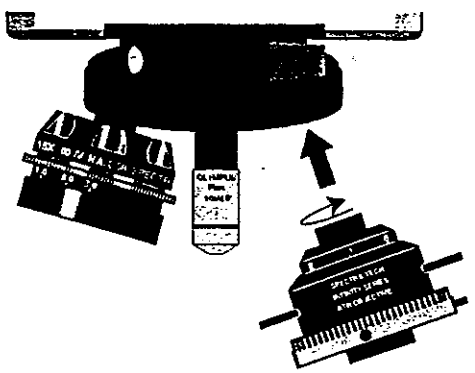
Germanium and Silicon ATR Objectives



Prepare microscope for ATR Objective

Due to their size, IR objectives must be placed in *opposite* ports on the 4-place nosepiece. Glass objectives (if used) can be placed in adjacent ports.

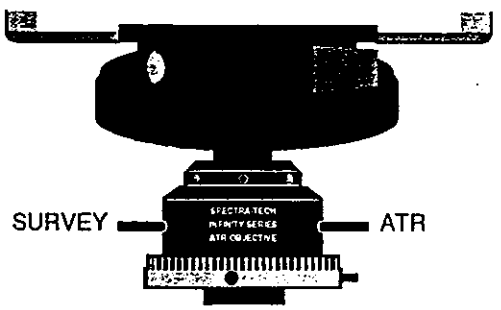
NOTE: If two IR objectives are presently mounted on the nosepiece, you must remove one to allow room for the ATR objective. We strongly recommend you leave either the 15X or 32X objective for centering the ATR Objective.



Install ATR on nosepiece

Carefully screw on the ATR objective in a clockwise direction.

Note: The slide selector should be in the *Contact* mode and the ATR crystal protective cap should be on to avoid damage to the crystal.



Proper Orientation of the ATR Objective

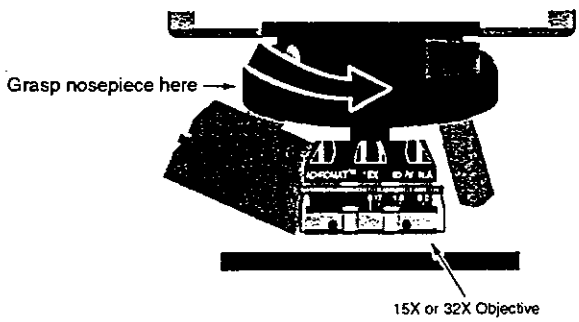
The recommended orientation is to have the Spectra-Tech label facing the user and.the slide selector should be oriented side to side with "Survey" to the left and "ATR" to the right.

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

Germanium and Silicon ATR Objectives

Orient ATR Objective

If the ATR objective is **not** oriented properly (facing forward), the following procedure must be followed:



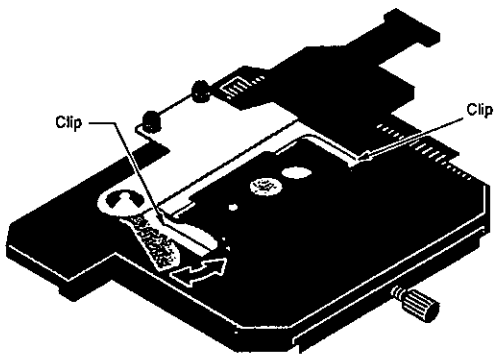
Prepare microscope for viewing

Grasp the black knurled section of the nosepiece and rotate the 15X or 32X objective into position.

Caution: Do not rotate by holding the objectives.

Put the microscope into the *Reflection* mode.

Adjust the reflection illumination to a comfortable viewing level.

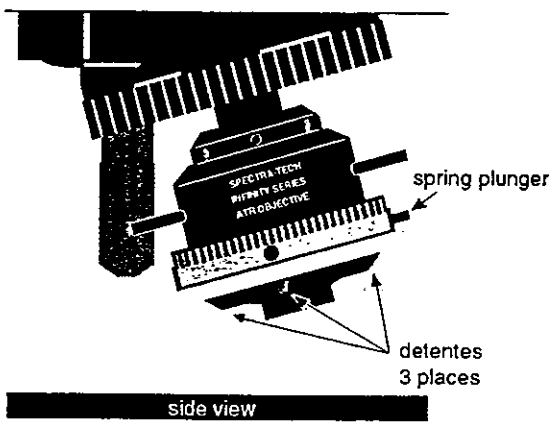


Establish the Reference

Place the100µm 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.

NOTE: Verify the Reflex aperture is open to its maximum position.

Germanium and Silicon ATR Objectives



side view

Lower stage and position ATR Objective

Using only the Z-focus control knob, lower the stage.

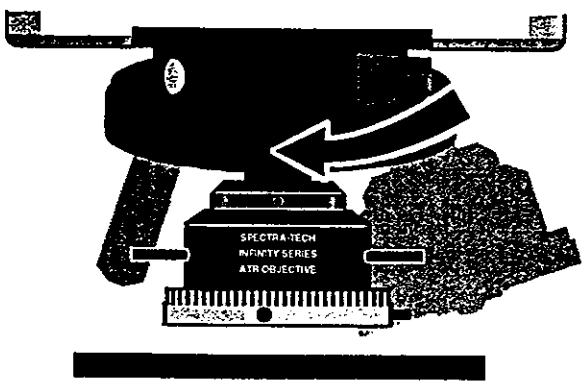
Grasp the black knurled section of the nosepiece and rotate the ATR Objective to the side position for easy access.

Remove the crystal housing

Hold the crystal housing by the protective cap and loosen only the protruding spring plunger just enough to remove the crystal housing.

Rotate the crystal housing until the detentes are aligned to the setscrews and spring plunger, then lower the crystal assembly.

Note: Do **not** loosen the two centering setscrews.



Select ATR objective and raise stage

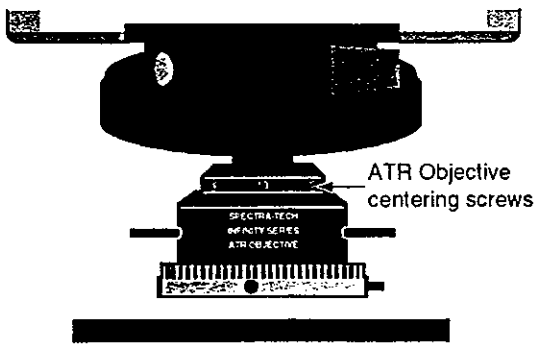
Grasp the black knurled section of the nosepiece and rotate the ATR Objective (now with no crystal in place) into position.

Be sure the slide selector is in the *Contact* mode.

Using only the Z-focus control knob, raise the stage to view the 100µm pinhole through the ATR Objective.

Note: You may want to check that the pinhole is still centered in the crosshairs with the 15X or 32X Objective before centering the ATR Objective.

Germanium and Silicon ATR Objectives

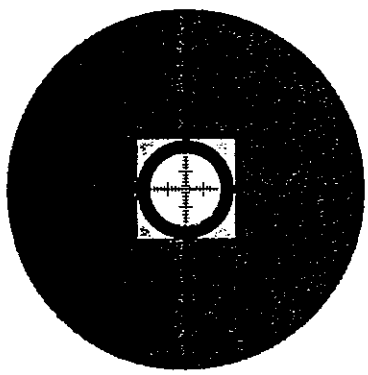


Loosen ATR Objective centering screws

Using a 5/64 balldriver, loosen every other screw on the top section of the objective (3 full turns).

Now loosen only one of the remaining setscrews and rotate the objective to the proper position (so that the engraved "SPECTRA-TECH - INFINITY SERIES - ATR OBJECTIVE" faces forward.

Tighten the screw.



Center pinhole by adjusting ATR objective centering screws

Insert three 5/64 balldrivers into the screw holes which were **not** loosened in the preceding step.

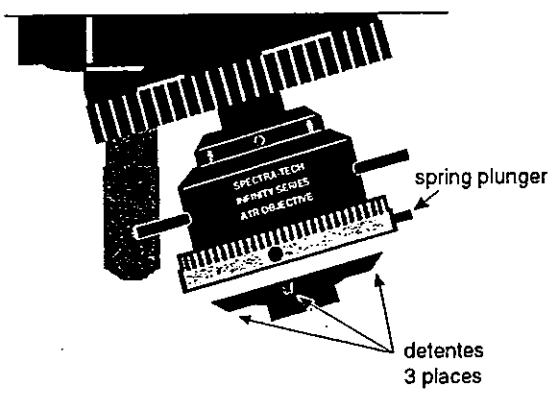
Center the pinhole on the crosshairs by adjusting these 3 screws. This is done by first loosening one screw, then tightening another. All three screws should be snug at the end of each such adjustment.

Lock position

Retighten 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 centered pinhole as viewed through the ATR Objective and the standard 15X or 32X objectives are concentric.

Germanium and Silicon ATR Objectives



side view

Lower stage and position ATR Objective

Lower the stage.

Grasp the black knurled section of the nosepiece and rotate the ATR Objective to the side position for easy access.

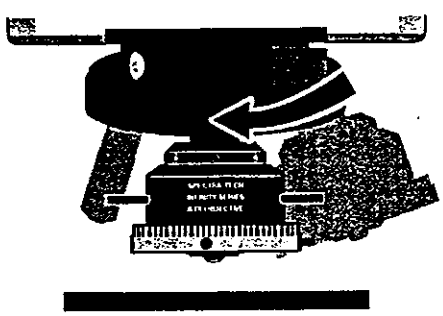
Reinstall crystal housing

Orient the crystal housing so that the three detentes are aligned to the setscrews and spring plunger, then raise the housing into the centering ring.

Rotate the housing until the setscrews and spring plunger are away from the detentes and are now making direct contact to the beveled edge.

Tighten the setscrew just enough to create tension against the spring plunger and hold the housing in place.

Note: Do not over tighten the spring plunger. Some travel is necessary when centering the crystal.

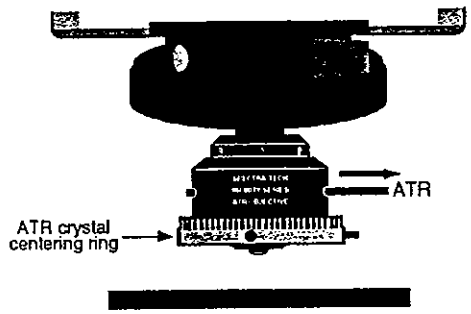


Remove crystal protective cap and select ATR objective

Remove crystal protective cap.

Grasp the black knurled section of the nosepiece and rotate the ATR Objective into position.

Germanium and Silicon ATR Objectives

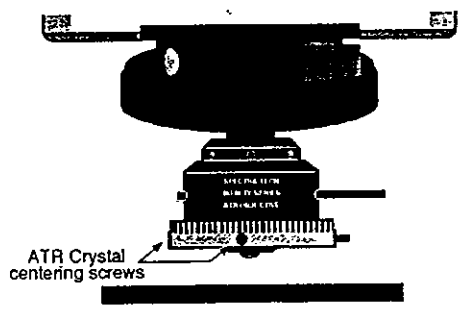


Select ATR mode and monitor energy throughput

Place the slide selector into the *ATR* mode.

Be sure the microscope is in the *Reflection* mode.

While viewing the infrared signal (interferogram; peak to peak), slightly turn the ATR crystal centering ring (the lower knurled ring) in either direction to maximize energy throughput.



Maximize energy throughput

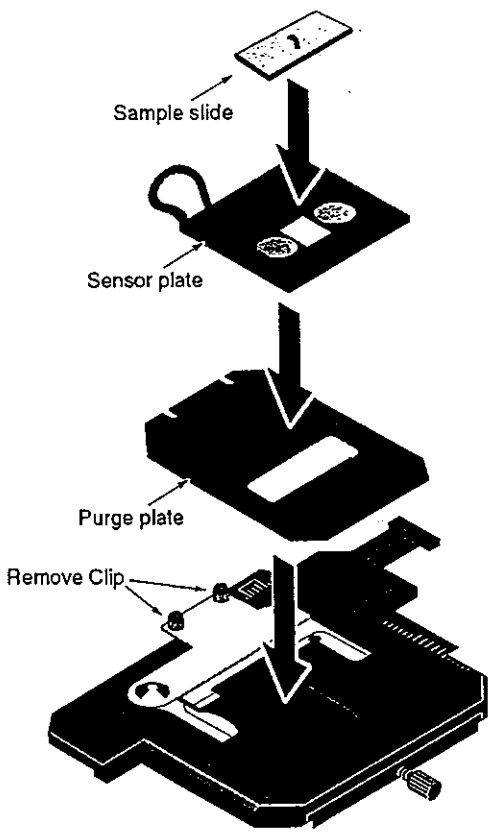
While still viewing the infrared signal, use two 5/64 balldrivers to maximize energy throughput.

Repeat these last two steps to ensure maximum energy throughput.

Note throughput for later reference.

Refer to *Appendix B*; the Crystal Throughput Table for minimum specifications.

Using the *Internal* Contact Alert System



Internal Contact Alert

The Contact Alert is a pressure sensing system which is used with Spectra-Tech's ATR Objectives and Slide-On ATR attachments to indicate and monitor contact force between the sample and ATR crystal.

When either the internal Contact Alert or the external Quantitative Contact Alert system is activated, it indicates and monitors contact force between the sample and ATR crystal when performing ATR experiments.

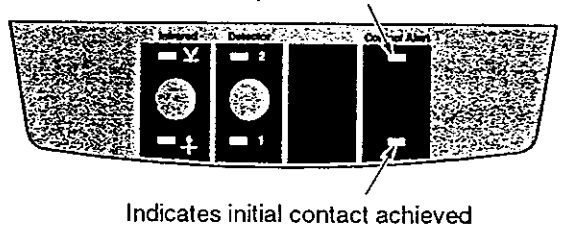
1. To use the internal Contact Alert System, remove the stage clip by the two silver knurled thumbscrews, then remove the thumbscrews from the clip. Attach the Purge plate (at the two slot positions) to the stage with the thumbscrews, then place the Contact Alert Sensor Plate into the Purge plate.

Note: The Purge plate is not used with microscopes equipped with the Glide Stage or Motorized Stage. The Sensor Plate fits directly into those stages.

Caution! Before connecting the Contact Alert Sensor Plate or the external Quantitative Contact Alert System, place the AC power switch of the microscope to the **off** position.

2. Plug the Sensor plate cable into the outside, upper nine pin cable port labeled INTERNAL located at the front of the microscope.

Using the *Internal* Contact Alert System



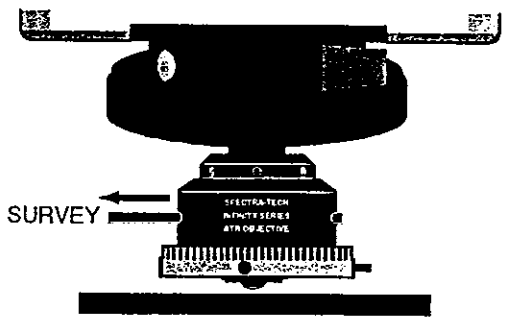
3. Place the AC power switch of the microscope to the on position.

   The internal Contact Alert LED's located at the front panel of the microscope will be activated.

4. Put the microscope in the *Reflection* mode.

5. Place the sample slide onto the Contact Alert Sensor Plate.

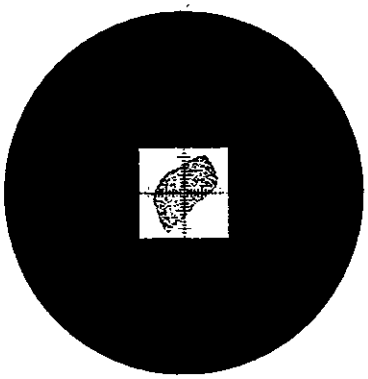
Caution! If the sample is larger than the 1x3 inch glass slide, place the glass slide onto the Sensor Plate first and then place the sample onto the glass slide.



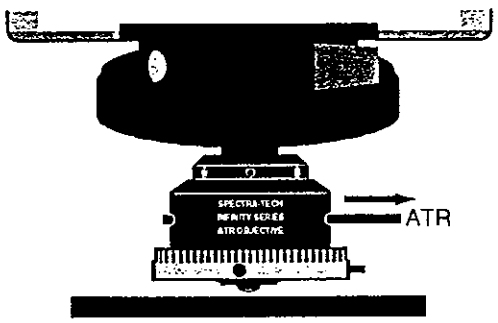
6. Place the slide selector into the *Survey* mode and focus on a clean section of the sample slide or substrate.

7. Collect background spectrum.

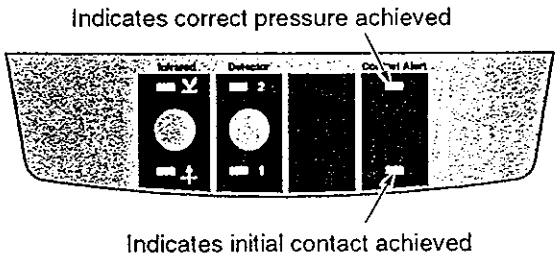
Using the *Internal* Contact Alert System



8. Still in *Survey* mode, focus and center the sample area of interest in the field of view using the x and y adjustment knobs for the Sample Stage.



9. Put the slide selector into the *ATR* mode and raise the Sample Stage using the Z-focus adjustment knob.



10. Stop when the lower LED illuminates green. This indicates that initial contact of the sample area to ATR crystal has been established.

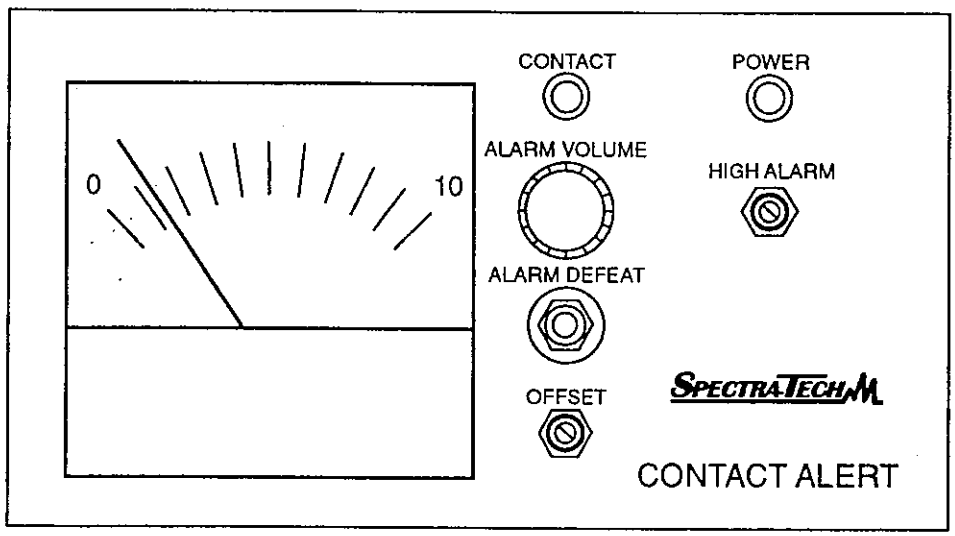
11. Now slowly raise the stage and immediately stop when the upper LED illuminates red. This indicates that contact pressure of the sample area to ATR crystal is optimal for infrared analysis.

Caution! Excessive pressure can damage the ATR crystal and/or sample.

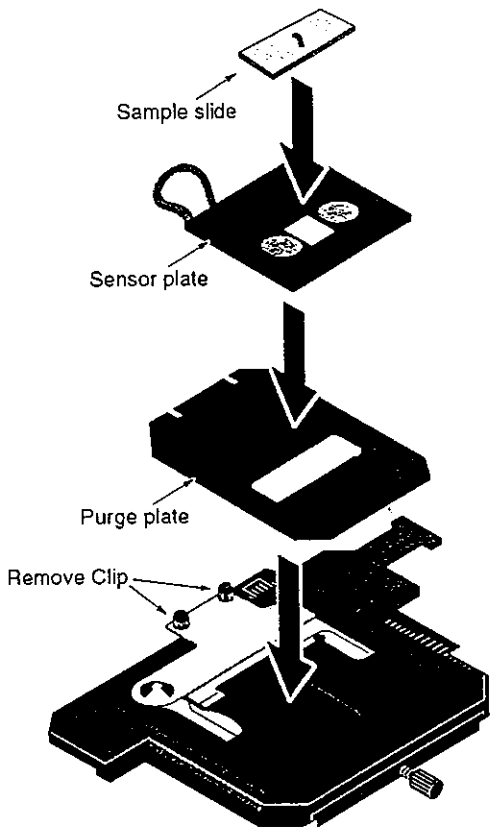
12. Collect sample spectrum.

Using the *Quantitative* Contact Alert System

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 +1% height variation. Enabling this system will de-activate the internal Contact Alert LED's located on the front panel of the microscope.



Using the *Quantitative* Contact Alert System

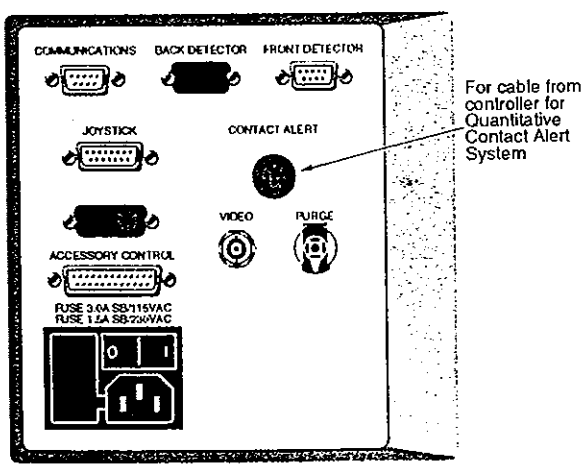


1. To use the external Quantitative Contact Alert System, remove the stage clip by the two silver knurled thumbscrews, then remove the thumbscrews from the clip. Attach the Purge plate (at the two slot positions) to the stage with the thumbscrews, then place the Contact Alert Sensor Plate into the Purge plate.

Note: The Purge plate is not used with microscopes equipped with the Glide Stage or Motorized Stage. The Sensor Plate fits directly into those stages.

Caution! Before connecting the Contact Alert Sensor Plate or the external Quantitative Contact Alert System, place the AC power switch of the microscope to the **off** position.

2. Plug the Sensor plate cable into the inside, upper nine pin cable port labeled EXTERNAL located at the front of the microscope.



3. Connect the communications cable of the Quantitative Contact Alert Controller into the Contact Alert labeled port of the I/O panel located on the back, right side of the microscope.

4. Connect the AC power cable of the Quantitative Contact Alert Controller into an AC outlet and place the power switch of the Controller to the on position.

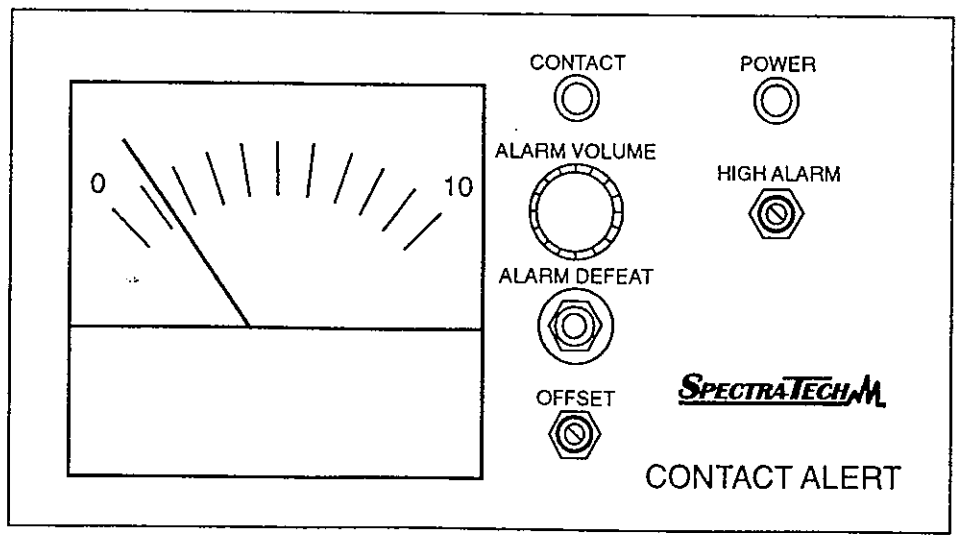
Using the *Quantitative* Contact Alert System

Adjust Low Alarm Offset

The weight of the sample is sometimes sufficient to trigger the Low Alarm (Alarm LED lights, buzzer sounds and meter deflects). If the Low Alarm is triggered when the sample is mounted on the Sensor Plate, turn the Low Alarm Offset adjustment counter-clockwise until the alarm no longer triggers. This offset circuit can correct for samples weighing up to 50 grams.

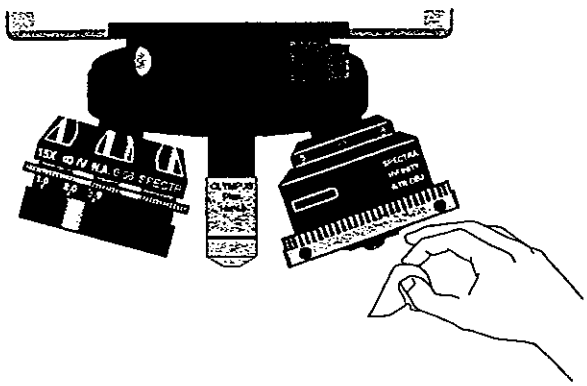
Adjust the pressure between 5-9 for hard materials and 2-4 for elastomeric materials.

Note: Refer to the Quantitative Contact Alert User's Manual (P/N 700-0035) for more detailed procedures.



MAINTENANCE

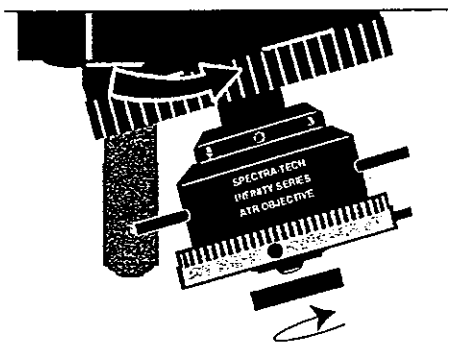
Cleaning the Crystal



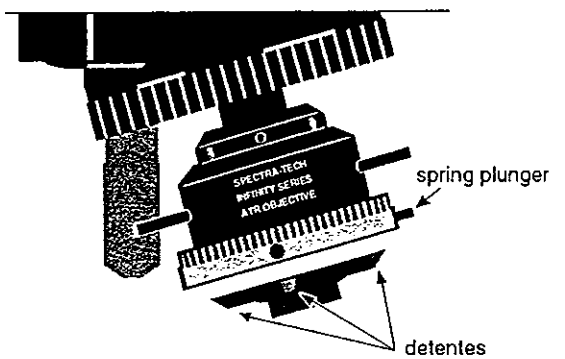
Rotate the nosepiece so that the ATR crystal is accessible. Wipe the crystal with lens tissue. Water or isopropanol may be used to clean the crystal.

Caution: Avoid acetone and chlorinated solvents as these will attack the adhesive used to secure the crystal to the slide.

Replacing the ATR Crystal



side view



side view

Lower stage and position ATR Objective

Lower the stage and replace the protective cap (covering the crystal).

Grasp the black knurled section of the nosepiece and rotate the ATR Objective to the side position for easy access.

Remove the crystal housing

Hold the crystal housing by the protective cap and loosen only the protruding spring plunger just enough to remove the crystal housing.

> **Note:** Do **not** loosen the two centering setscrews.

Rotate the crystal housing until the detentes are aligned to the setscrews and spring plunger, then lower the crystal assembly.

Install new crystal housing

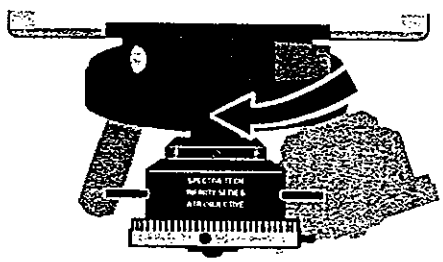
Orient the new crystal housing so that the three detentes are aligned to the setscrews and spring plunger, then raise the housing into the centering ring.

Rotate the housing until the setscrews and spring plunger are away from the detentes and are now making direct contact to the beveled edge.

Tighten the setscrew just enough to create tension against the spring plunger and hold the housing in place.

> **Note:** Do not over tighten the spring plunger. Some travel is necessary when centering the crystal.

Aligning a ZnSe or Diamond Crystal

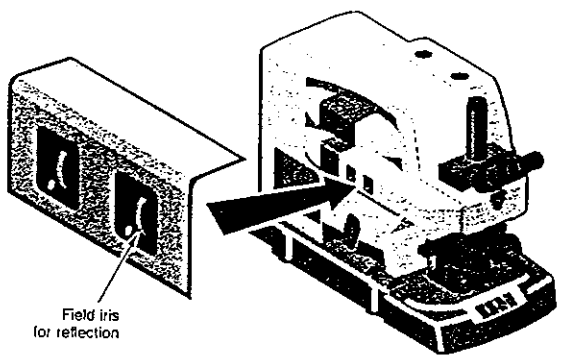


Select ATR Objective

Remove the protective cap.

Grasp the black knurled section of the nosepiece and rotate the ATR Objective into position.

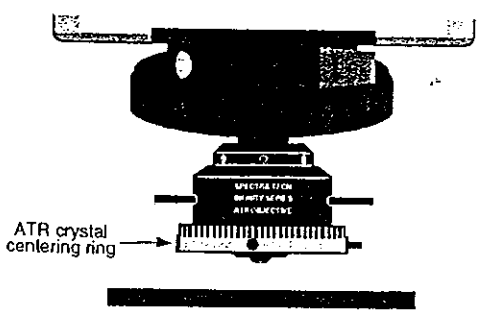
Ensure the Slide selector is in the *Contact* mode.



Adjust illumination

Adjust the reflection illumination to a comfortable viewing level.

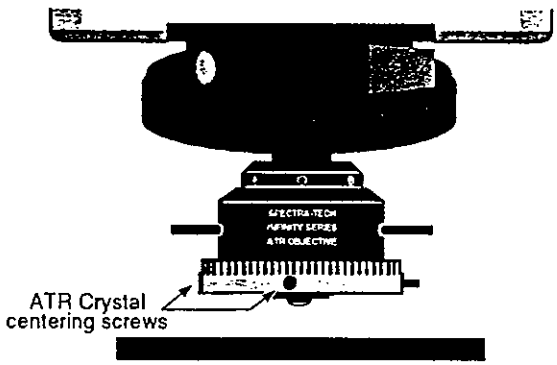
Close the reflection field iris to approximately 300µm.



Focus Contact image

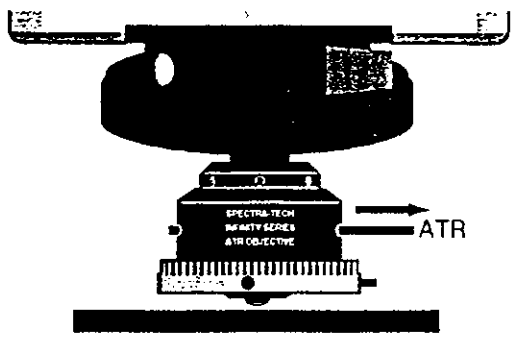
Turn the ATR crystal centering ring (the lower, silver knurled section) in either direction to focus the contact image.

Aligning a ZnSe or Diamond Crystal



Center crystal

Using two 5/64 balldrivers, center the Contact image .



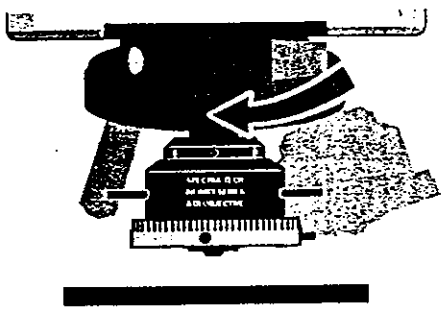
Measure signal throughput

Put the slide selector in the *ATR* mode and check the energy throughput.

Note throughput for later reference.

Refer to *Appendix B*; the Crystal Throughput Table for minimal specifications.

Aligning a Ge or Si Crystal

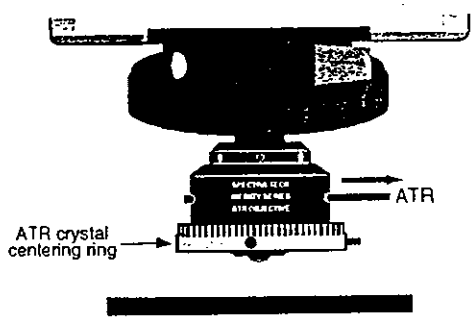


Remove crystal protective cap and select ATR objective

Remove crystal protective cap.

Grasp the black knurled section of the nosepiece and rotate the ATR Objective into position.

> **CAUTION!** Be sure there is clearance above the stage before selecting the ATR Objective.

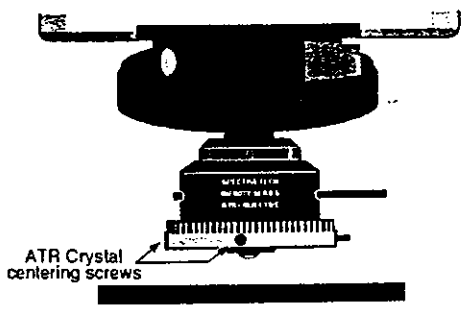


Select ATR mode and monitor energy throughput

Place the slide selector into the *ATR* mode.

Be sure the microscope is in the *Reflection* mode.

While viewing the infrared signal (interferogram; peak to peak), slightly turn the ATR crystal centering ring (the lower knurled ring) in either direction to maximize energy throughput.



Maximize energy throughput

While still viewing the infrared signal, use two 5/64 balldrivers to maximize energy throughput.

Repeat these last two steps to ensure maximum energy throughput.

Note throughput for later reference.

Refer to *Appendix B*; the Crystal Throughput Table for minimum specifications.

APPENDIX A

Cross-Section of the ATR Objective