

# DiaMaxATR

## High-Throughput Single Reflection Diamond ATR

USER'S MANUAL







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**UNPACKING**

Before proceeding to installation of the accessory, make sure that all of the parts on the included Checklist are present. See Appendix E. If any parts are missing or damaged, contact Harrick Scientific immediately.

**TECHNICAL SUPPORT**

For additional information please contact our Technical Support Center at 800-248-3847 between 9 a.m. and 5 p.m. EST; or e-mail your questions to: [techsupport@harricksci.com](mailto:techsupport@harricksci.com)

**FEEDBACK**

Your comments and suggestions are welcome. Please send them to:

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Web: [www.harricksci.com](http://www.harricksci.com)

The DiaMaxATR is a high performance single reflection diamond ATR, compatible with most FTIR spectrometers. The accessory has a fixed 45° incident angle and the supplied high performance diamond is suitable for use from 4000  $\text{cm}^{-1}$  to 550  $\text{cm}^{-1}$ , with limited signal-to-noise in the region of the diamond lattice bands around 2000  $\text{cm}^{-1}$ . The DiaMaxATR features a convenient horizontal sampling surface and a user removable sampling plate for ease of cleaning. For solid sampling, it includes a built-in pressure applicator with slip-clutch to provide good contact between the sample and the ATR crystal. The pressure applicator swings out of the way to enable unobstructed access to the sampling surface. The DiaMaxATR is enclosed and includes a fitting for purge line. Available options include a force sensor with digital display for precise control of the force applied to compress solids against the ATR crystal in addition to flow and temperature controlled liquid cells.

Liquids, pastes and slurries are analyzed by simply covering the sampling area with the sample. Liquids can be placed on the crystal with an eyedropper or syringe. Pastes can be simply smeared over the sampling surface. Powders and other solids are analyzed by placing a small amount of the sample on the sampling surface and applying pressure.

### OPEN BEAM SPECTRUM

Prior to installation, collect an open beam background spectrum (no accessory in the sample compartment). This spectrum should be used later to verify the throughput.

### GETTING READY

Before installing the DiaMaxATR, familiarize yourself with the accessory and its various components by referring to the drawing found below. The accessory includes a purgeable chassis with a high performance diamond ATR sampling plate installed.

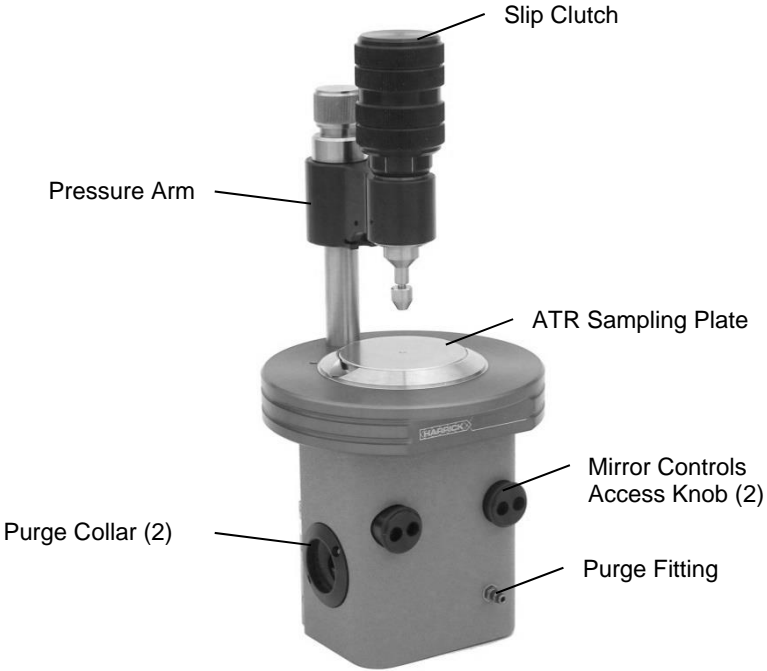


Figure 1. The DiaMaxATR

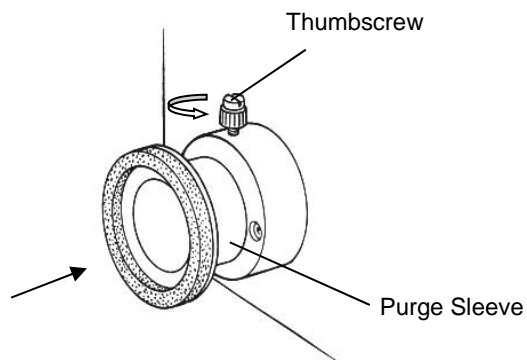
## INSTALLATION AND ALIGNMENT

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### PURGE INSTALLATION

#### ***PURGE SLEEVES***

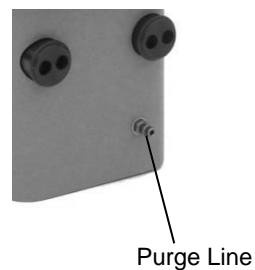
Loosen the thumbscrews and push the purge sleeves in to retract them (Figure 2).



**Figure 2 • Purge Sleeve**

#### ***PURGE LINE***

For quicker purging or if the spectrometer has windows on the beam ports, connect an additional purge line to the fitting on the front of the DiaMaxATR, shown in Figure 3.



**Figure 3 • Purge Line**



## INSTALLATION AND ALIGNMENT

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### INSTALLING THE DiaMaxATR

#### **RAIL MOUNTING**

If your spectrometer is equipped with rails:

- Install the accessory onto the rails.

**NOTE:** *If you received the Harrick rail plate with your DiaMaxATR, first mount the supplied rails directly onto the floor of the sample compartment (see Appendix A).*

- Move the DiaMaxATR along the rails until the spectrometer focal point is in the center of the accessory.
- Lock the DiaMaxATR in place by tightening the locking mechanism knob.
- Extend the purge sleeves until they firmly contact the sides of the sample compartment.
- Lock the purge sleeves in place with the thumbscrews.

#### **FIXED FLOOR MOUNTING**

- Install the DiaMaxATR onto the floor of the sample compartment using the base plate provided.
- Extend the purge sleeves to the walls of the spectrometer and tighten the thumbscrews on the purge collars.

## INSTALLATION AND ALIGNMENT

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### ALIGNMENT

This accessory has been pre-aligned prior to shipment. Only minor adjustments should be required to optimize the performance to your spectrometer:

- Once the accessory is installed, set the spectrometer to measure the “energy” on the detector.
- Rotate the mirror control access knobs a quarter of a turn, so the holes are vertical instead of horizontal.

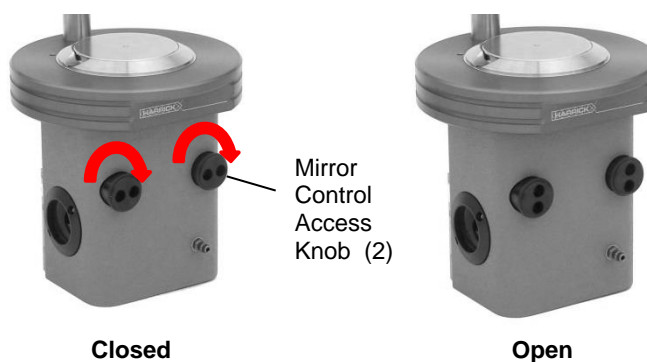


Figure 4 • Accessing the mirror adjustments

*NOTE: Make sure that the ATR Sampling Plate has been correctly installed. The plate should be pushed down as far as it will go.*

- Feed the supplied 3/32” ball driver through the lower hole in the left knob and engage it on the screw inside.
- Make minute adjustments to maximize the energy on the detector.
- Remove the ball driver and insert it in the upper hole in the left knob. Adjust to maximize the energy.
- Repeat this procedure with the two adjustments on the right.
- Continue adjusting both sets until no significant gain in energy is achieved.



**CAUTION:**

*The two turn adjustments are continuous, without stops. Stop adjusting if resistance is felt or damage may occur.*

## INSTALLATION AND ALIGNMENT

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- If the accessory has been locked in place, it must be unlocked before adjusting the height of the accessory. Feed the supplied 3/32" ball driver through the locking screw access hole on the left side of the accessory (see Figure 5) and engage it in the screw below. Turn the screw counterclockwise to release the locking mechanism.



**Figure 5 • Unlocking**

- To adjust the height of the accessory, feed the supplied 3/32" ball driver through the height adjustment control access hole on the right side of the accessory (see Figure 6) and engage it in the screw below.



**Figure 6 • Height adjustment**

## INSTALLATION AND ALIGNMENT

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- Then turn the ball driver to make minute height adjustments to maximize the energy on the detector. Turning the driver clockwise will elevate the accessory. Counter-clockwise turning will lower it. Be sure to apply slight downward pressure on the accessory when lowering it.
- Confirm that the signal is still maximized with regard to the mirror adjustments.
- Repeat as necessary until no significant gain in energy is achieved.
- Rotate the mirror control access knobs counter-clockwise, a quarter of a turn, so the holes are horizontal instead of vertical, closing the accessory for purge

### LOCKING

Once the accessory has been installed and aligned, it should be secured in place.

- To lock the accessory, feed the supplied 3/32" ball driver through the height adjustment control access hole on the left side of the accessory (see Figure 7) and engage it in the screw below.
- Rotate the ball driver clockwise to tighten the screw and lock the accessory in position.



Figure 7 • Locking



**CAUTION:**

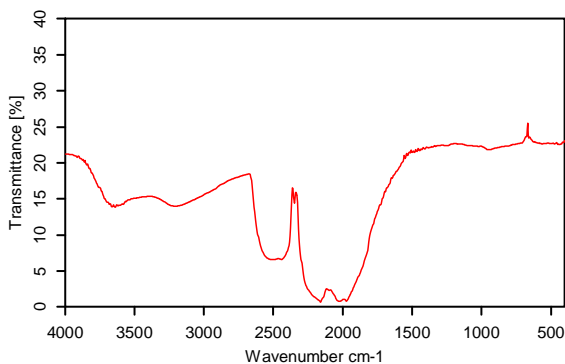
*The accessory must be unlocked before removal or height adjustment.*

To confirm the performance of your accessory, it should be tested before first use and at regular intervals thereafter.

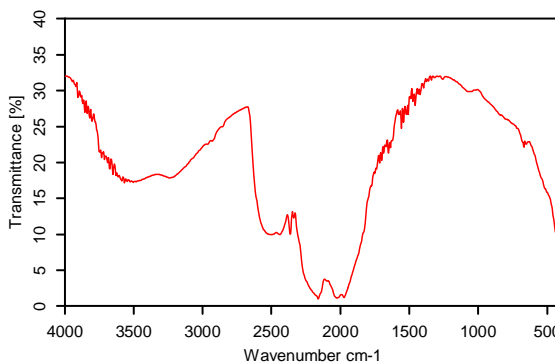
### VERIFYING THE THROUGHPUT

- Make sure that the specified background spectrum is the previously collected open beam background spectrum. This background spectrum should be obtained with the accessory removed from the dovetail and temporarily set aside.
- Clean the ATR element. See the Operation section for details.
- With the accessory and its sampling plate installed, collect a transmittance spectrum.

*NOTE: Make sure that the ATR sampling plate has been correctly installed. The plate should be pushed down as far as it will go.*



FIR Diamond



High Performance Diamond

**Figure 8 • Throughput.**

- Record the percent transmittance at  $2700\text{ cm}^{-1}$ .
- The throughput should be at least 15% with the high performance diamond installed. The spectra should resemble those shown above in Figure 8.

*NOTE: The sampling surface of the ATR element is the small rectangle located in the center of the top of the sampling plate.*

### CLEANING THE CRYSTAL

The sampling surface of the ATR crystal should be cleaned before each use.

- To clean the sampling surface of the crystal, wipe with a cotton swab or lens tissue dampened with a low residue solvent. Use very light pressure while wiping. Methyl ethyl ketone (MEK), isopropyl alcohol or acetone is recommended.

### BACKGROUND SPECTRUM

- Collect the background single beam spectrum with the accessory in the sample compartment.

### SOLID SAMPLES

- Raise the pressure head by turning the pressure applicator knob counterclockwise.
- Place the sample face down on the center of the ATR crystal.

### APPLYING PRESSURE

- Turn the built-in slip clutch (Figure 9) clockwise until adequate pressure is applied. Pressure is sufficient when the absorption band intensity no longer significantly changes with increased pressure or when the slip clutch begins to 'slip' and the guide lines on the slip clutch base no longer move.

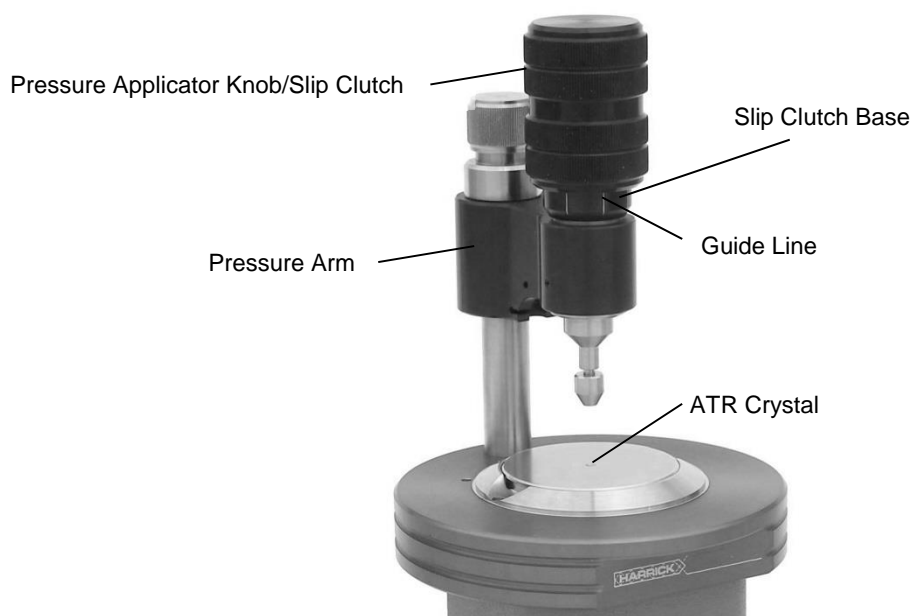


Figure 9 • Pressure Applicator Knob

### SAMPLE SPECTRUM

- Record the sample spectrum.
- Loosen the pressure applicator.
- Remove the sample.
- Clean the crystal prior to running the next sample.

### POWDERS

- Place a small amount (covering at least a 2 mm diameter area) of the sample on the center of the crystal.
- Apply pressure carefully so the powder is not displaced from the center of the crystal.
- Record the sample spectrum.

**NOTE:** *To avoid spilling powder in the spectrometer, carefully remove the solid sampling plate and clean it outside the spectrometer.*

- Wipe the sample off the crystal with a damp cloth.
- Clean the crystal.

### LIQUIDS AND PASTES

- Raise the pressure applicator about 1/8" away from the surface of the sampling plate.
- Swing the pressure applicator out of the way.
- Place a small amount (covering at least a 2 mm diameter area) of the sample on the center of the crystal.



**CAUTION:**

*If the liquid sample is volatile, the use of the optional liquid cell is recommended. The liquid cell can be filled with a Luer-Lok syringe in a glove box or other enclosed environment.*

**NOTE:** *For liquid and paste analysis, no pressure is needed.*

- Record the sample spectrum.
- Wipe the sample off the crystal.
- Clean the crystal.

### USING THE VOLATILES COVER

- Place a few drops of the volatile sample on the center of the crystal.
- Place the cover slide over the sample.
- Record the sample spectrum.
- Clean the crystal and the cover slide.



Figure 10 • Volatiles Cover

### TEMPERATURE CONTROLLED LIQUID CELLS

The DiaMaxATR™ has optional heated and flow cells for operation up to 200°C. The liquid cells are equipped with a cooling conduit for near-ambient or low temperature operation and are assembled with either Luer or Swagelok fittings.

### INSTALLATION

- Clean the ATR crystal prior to installation of the cell.
- Turn the pressure applicator knob counterclockwise to raise the pressure applicator well above the sampling plate.
- Loosen the arm locking knob and swing the pressure applicator out of the way, either to the left or right

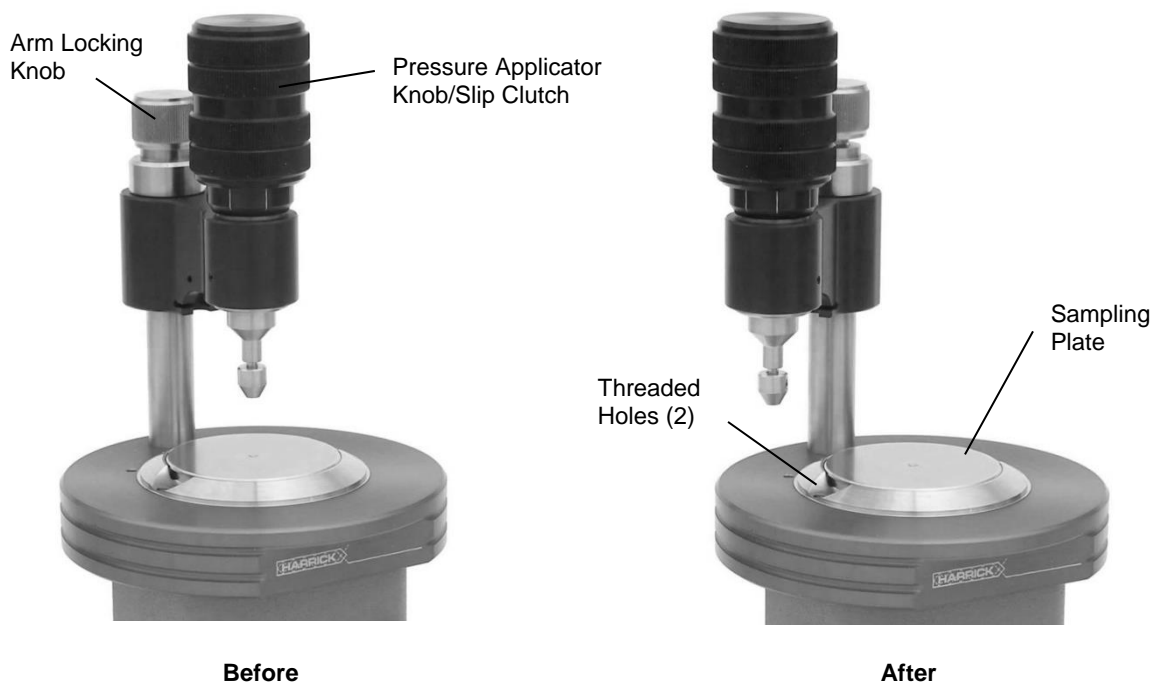


Figure 11 • Swinging the pressure applicator to the side



## USING THE OPTIONAL EQUIPMENT

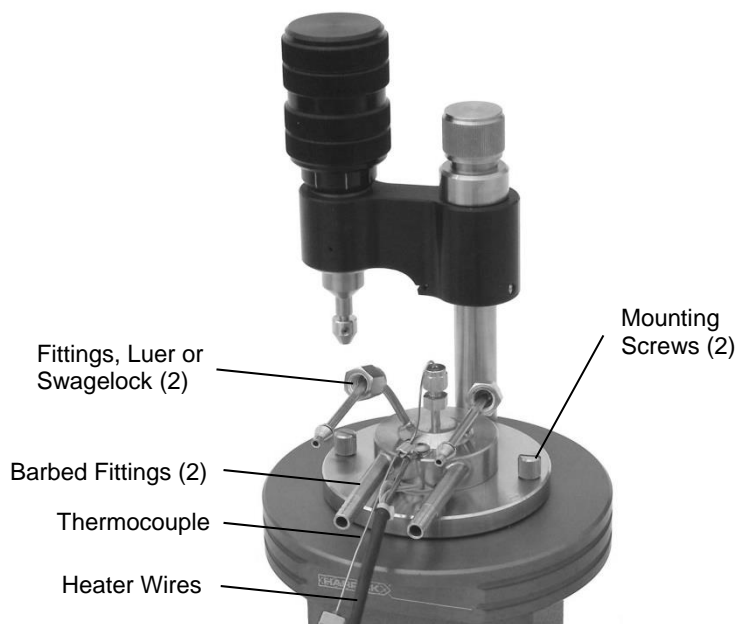
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- Make sure the o-ring and o-ring groove is clean and free of dust.
- Insert the o-ring into the recess in the bottom of the cell.
- Locate the cell over the sampling plate, so the two screws align with the threaded holes and the barbed fittings face forward.



**Figure 12. O-ring installation**

- Thread and tighten the screws to secure the cell to the accessory.



**Figure 13 • Liquid cell with Swagelok Fittings affixed to the DiaMaxATR.**

## USING THE OPTIONAL EQUIPMENT

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### **FLUID / ELECTRICAL CONNECTIONS**

- If heating is desired, connect the thermocouple and the heater to a suitable temperature controller.
- If cooling or operation near-ambient is desired, use 1/4" tubing to connect the two cooling ports to a thermostated chiller. Use a hose clamp to secure the tubing to the barbed fittings.
- To use the cell equipped with Luer fittings, remove the Luer plugs if they are installed. Fill a Luer Lock syringe with ~0.3 mL of liquid. Engage the Luer Lock on the fitting on the cell and inject the liquid. For volatile samples, reinstall the Luer plugs to prevent evaporation.



**CAUTION:**

*Do not install the Luer plugs unless the cell is being operated at near-ambient temperature.*

*Do not touch the hot surfaces during above-ambient operation. Do not operate above 200°C.*

- To use the cell equipped with Swagelok fittings, connect the supplied 1/8" Swagelok nuts to appropriate adapters and tubing to flow liquid through the cell.

**NOTES:** *Purging is recommended during operation at elevated temperatures.*

*Be careful when removing the heated liquid cells, since the o-ring may stick to the sampling plate.*

## USING THE OPTIONAL EQUIPMENT

### REPLACING THE HEATERS

- To replace the heaters, use a 0.050" hex driver to loosen the two set screws.
- Using a 1/16" hex driver, loosen the clamping screw just enough to unclamp the ground wire and slide it out.
- Then slide the heaters out. If a heater sticks, insert a rod into the knockout hole on the opposite side of the cell from the heater and push the heater out.
- Reverse this procedure to install the new heater, taking care to route the thermocouple wire under the clamp when securing the clamping screw as needed.

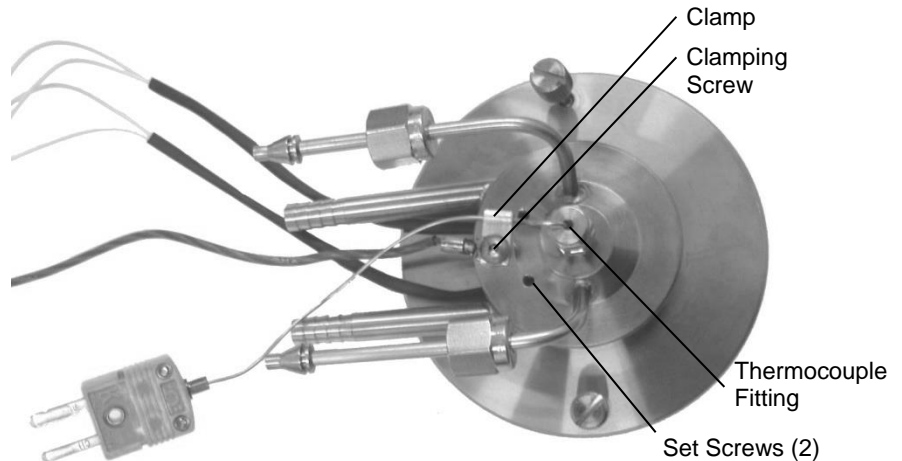


Figure 14 • Replacing the Temperature Controllable Liquid Cell heaters and thermocouple.

### REPLACING THE THERMOCOUPLE

- Use a 1/16" hex driver to loosen the clamping screw and remove the thermocouple wire from under the clamp.
- Then unscrew the thermocouple fitting.
- Remove the two ferrules and nut from the thermocouple.
- Slide the nut onto the replacement thermocouple, followed by the two ferrules in the order shown below.
- Then feed the end of the thermocouple into its hole, as far as it will go.
- Slide the nut down to engage the thread and tighten.
- Then route the thermocouple wire through the clamp and tighten the clamping screw.

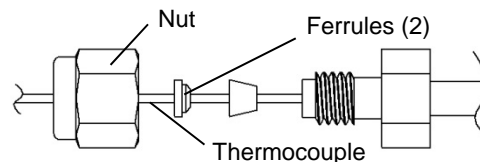


Figure 14 • Ferrule Orientation

## USING THE OPTIONAL EQUIPMENT

### HEATED STAGE

The DiaMaxATR™ has an optional heated stage for operation up to 200°C. The heated stage is equipped with a cooling conduit for near-ambient or low temperature operation.

### INSTALLATION

- Clean the ATR crystal prior to installation of the cell.
- Turn the pressure applicator knob counterclockwise to raise the pressure applicator above the sampling plate.
- Loosen the arm locking knob and swing the pressure applicator out of the way, either to the left or right.
- Locate the heated stage mounting over the sampling plate as shown below and tighten the thumbscrews to secure it in place.

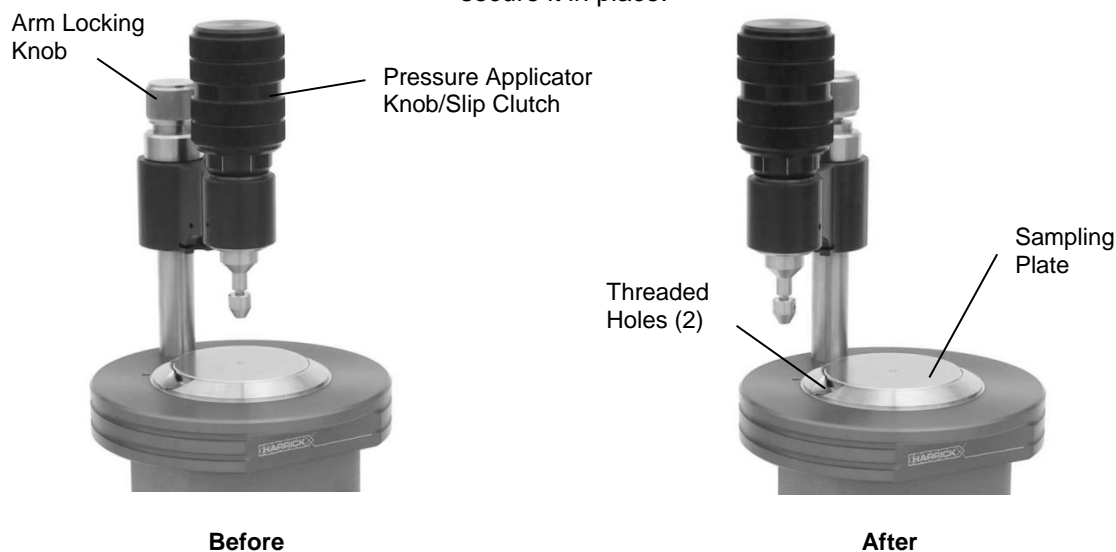


Figure 15 • Swinging the pressure applicator to the side

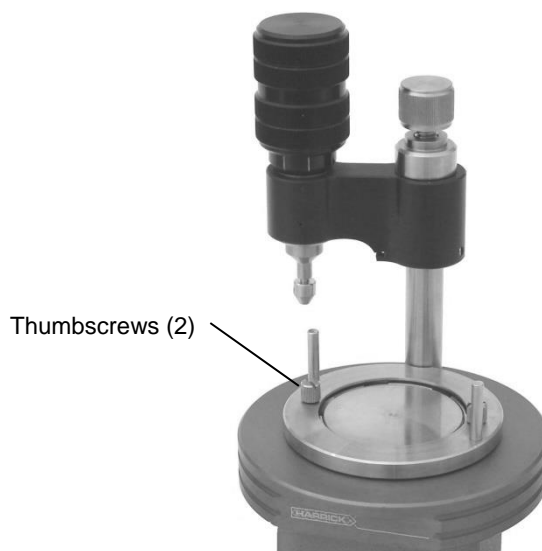


Figure 16 • Affixing the heated stage mounting bracket

## USING THE OPTIONAL EQUIPMENT

### INSTALLATION

- If an ambient temperature single beam background spectrum is desired, collect it.
- Clean the underside of the pressure pad on the heated stage.
- Place the sample on top of the ATR crystal.
- Align the two holes in the heated stage over the pins and lower the heated stage into position.
- Swing the pressure applicator back into position.

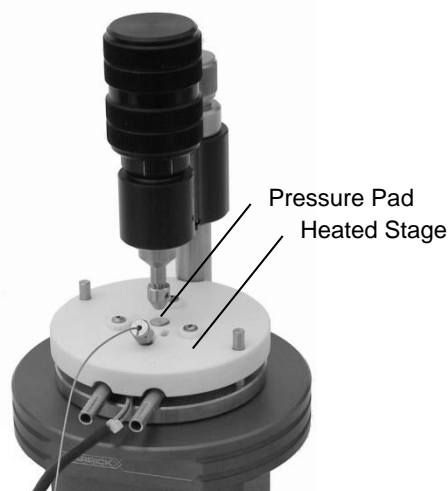


Figure 17 • Affixing the heated stage mounting bracket

### FLUID / ELECTRICAL CONNECTIONS

- If heating is desired, connect the thermocouple and the heater to a suitable temperature controller.
- If cooling or operation near-ambient is desired, use 1/4" tubing to connect the two cooling ports to a thermostated chiller. Use a hose clamp to secure the tubing to the barbed fittings.



#### **CAUTION:**

*Do not use the force sensor to measure the force applied when operating at elevated temperatures. The force sensor can withstand the specified maximum operating temperature but does not display the correct applied force.*

*Do not touch the hot surfaces during above-ambient operation.*

*Do not operate above 200 °C.*

*NOTE: Purging is recommended during operation at elevated temperatures.*

## USING THE OPTIONAL EQUIPMENT

### REPLACING THE HEATERS

- To replace the heaters, use a 1/16" hex driver to unscrew the three screws that secure the PTFE insulator to the heated block.
- Separate the PTFE insulator from the heated block.
- To replace the heaters, use a 0.050" hex driver to loosen the two set screws that secure the heaters in place.
- Then slide the heaters out.
- Reverse this procedure to install the new heaters and to reinstall the PTFE insulator.

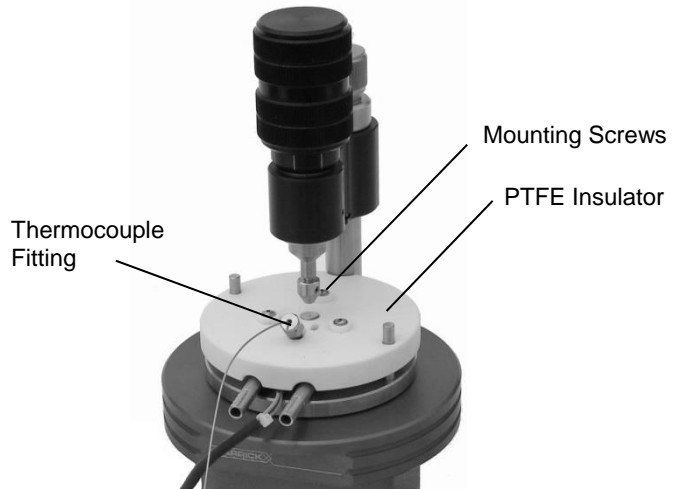


Figure 18 • Replacing the Heated Stage heaters and thermocouple.

### REPLACING THE THERMOCOUPLE

- Unscrew the thermocouple fitting.
- Then remove the two ferrules and nut from the thermocouple.
- Slide the nut onto the replacement thermocouple, followed by the two ferrules.
- Then feed the end of the thermocouple into its hole, as far as it will go.
- Slide the nut down to engage the thread and tighten.

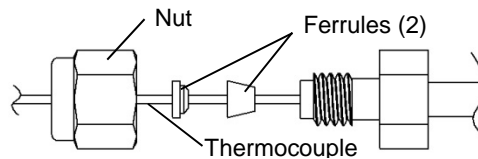


Figure 14 • Ferrule Orientation

### MOUNTING THE HARRICK RAIL PLATE

To install the supplied rail plate onto the floor of the sample compartment of your spectrometer:

- Remove any existing sample holders from the floor of the spectrometer.
- Install the rail plate in the orientation indicated in Figure 12. See Table 1 for the appropriate screws/holes for your spectrometer.

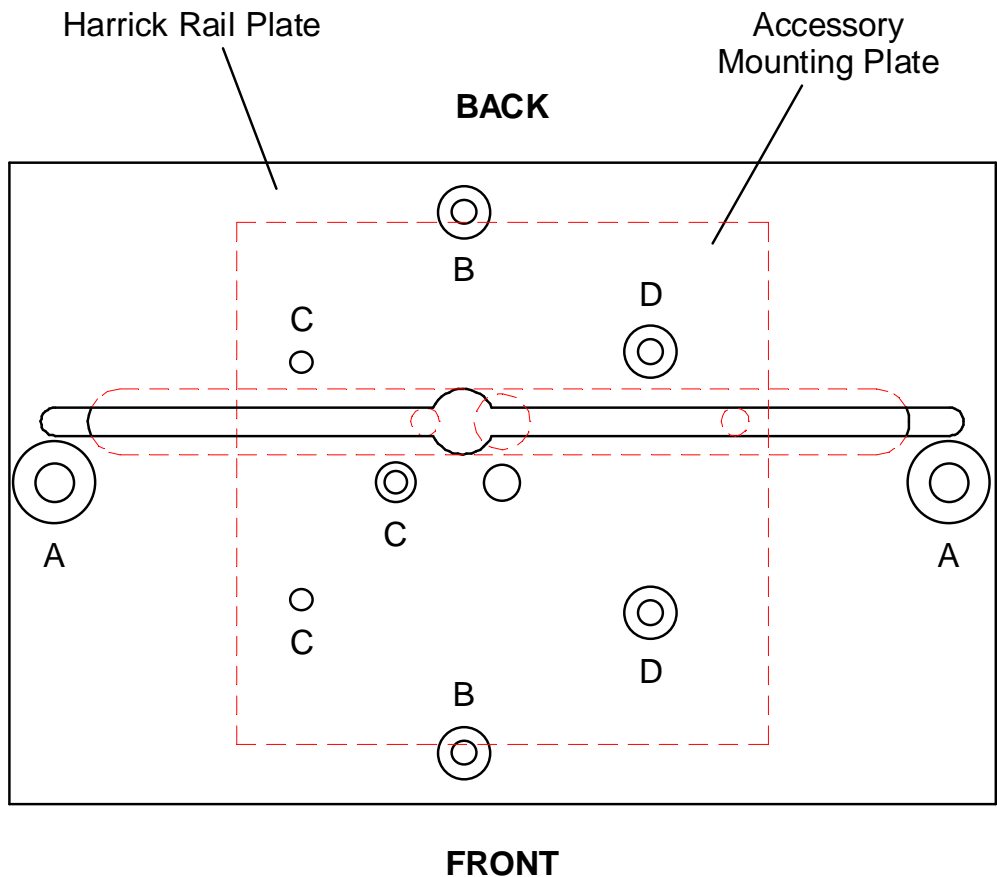


Figure 19 • Screw and Pin Positions for Rail Plate

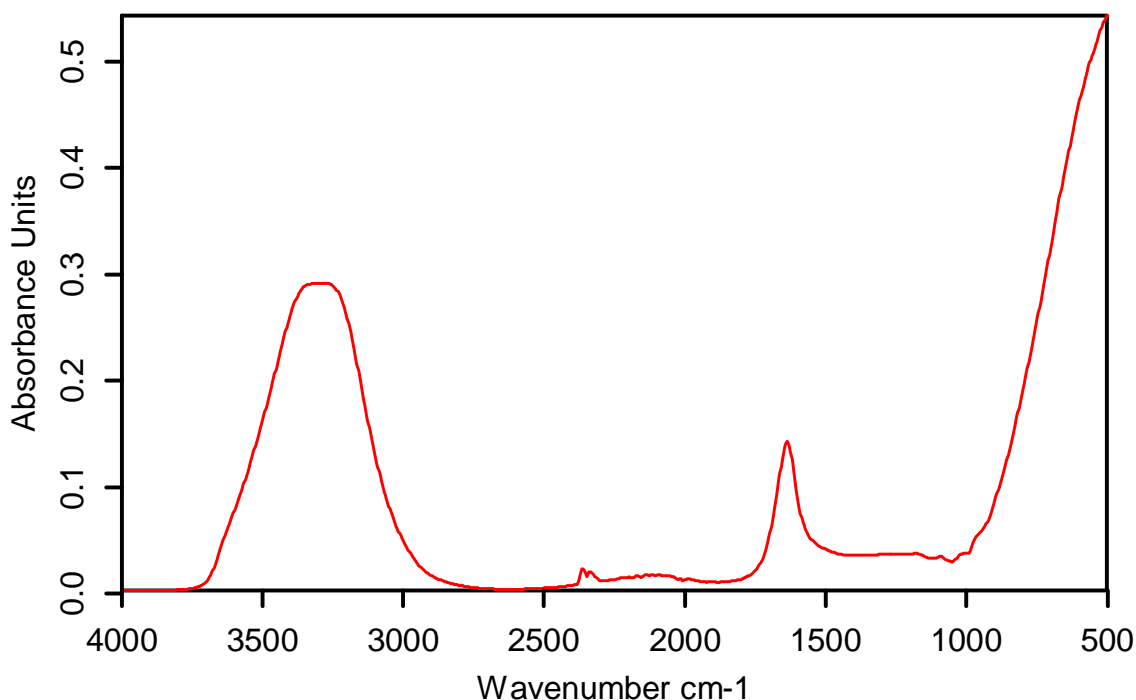
FTIR Manufacturer	Model	Holes
AGILENT/VARIAN/DIGILAB	All models(DI4, DI6, DI8)	A
THERMO-MATTSON	Polaris, Galaxy, RS Infinity, Genesis, Satellite (MA2, MA3)	B
PERKIN ELMER	Spectrum 1, 65, 100, 400, Frontier, Optica (P10, P11)	C
THERMO-NICOLET	Nexus, Avatar, Magna, Impact, 380, 4700, 5700, 6700, iS50, 8700 (NI6, NI8)	D

Table 1 • Screw and Pin Positions for Rail Plate

**VALIDATION**

For applications requiring higher photometric accuracy, a validation procedure on a specific liquid sample is recommended. An example, using water, is shown here.

- Collect a background spectrum with the accessory in the sample compartment.
- Place a few drops of a liquid sample to cover most of the ATR crystal.
- Collect a sample spectrum.
- Convert the spectrum into absorbance.



**Figure 20 • Absorbance of Water.**

- Choose two peaks at different ends of the spectrum. Write down the wavenumbers and absorbance values at these peaks.
- Every time the validation procedure is performed, the data should be within the noise level of those recorded the first time. On the next page, a table is provided to record these values.



## USING THE OPTIONAL EQUIPMENT

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<b>LIQUID SAMPLE USED</b>				
<b>WAVENUMBER (cm<sup>-1</sup>)</b>				
<b>ABSORBANCE VALUE</b>				

### REPLACING THE SAMPLING PLATE

#### REMOVING THE SAMPLING PLATE

To remove the sampling plate for cleaning or replacement:

- Turn the pressure applicator knob counterclockwise to raise the pressure applicator well above the sampling plate.
- Loosen the arm locking knob and swing the pressure applicator out of the way, either to the left or right.
- Then use the access slots to grab the sampling plate from underneath and lift it up off the accessory.

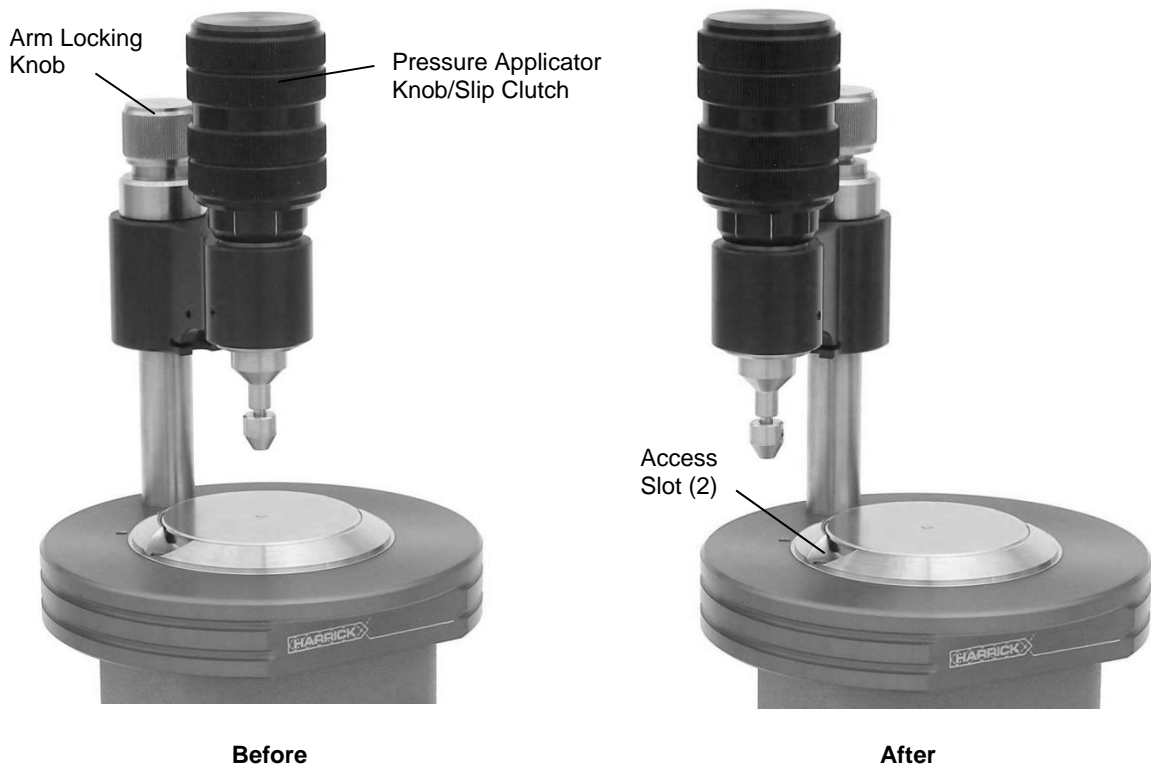


Figure 21 • Swinging the pressure applicator to the side

### ***INSTALLING A SAMPLING PLATE***

To reinstall the sampling plate after cleaning or install a replacement:

- Make sure the pressure applicator is raised well above the sampling plate and that it has been swung out of the way, either to the left or right.
- Orient the sampling plate so its slot faces the front of the accessory.
- Lower the plate so it locates on the pin in the recess for the plate.
- Push down on the plate to seat it firmly in place.



**Figure 22 • Orienting the sampling plate**

**OPTIONAL AND REPLACEMENT PARTS**

Replacement High Performance Diamond sampling plate.....	MVD-ATR-WC
FIR Diamond sampling plate .....	MVD-ATR-W
Volatiles cover.....	FAS-XCS
Temperature controllable liquid cell with Luer fittings.....	MVD-FSL-3
Temperature controllable liquid cell with Swagelok fittings .....	MVD-FSS-3
Viton o-ring for the liquid cell .....	ORV-010
Kalrez o-ring for the liquid cell .....	ORK-010
Temperature controllable solid sampling adapter.....	MVD-SSP-3
K-type thermocouple.....	008-144
24 V Heater Assembly for the temperature controlled cells and heated stage .....	UQA-HTR
Temperature Controller Kit (110V).....	ATK-024-3
Temperature Controller Kit (220/240V .....	ATK-024-4





*Manual Part No. MVD-M-01*

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