

SEAGULL[™]

USER'S MANUAL





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UNPACKING	Before installing the Seagull [™] make sure all the parts on the included check-off list are present. 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 or 914-762-0020 between 9 a.m. and 5 p.m. EST; or e-mail your questions to: techsupport@harricksci.com
FEEDBACK	Your comments and suggestions are welcome. Please send them to: Harrick Scientific Products, Inc. PO Box 277 141 Tompkins Ave, 2 nd floor Pleasantville, NY 10570 Phone: 800-248-3847; Fax: 914-747-7209 E-mail: info@harricksci.com Web: www.harricksci.com



The Seagull[™] is a variable angle reflection accessory. The optical configuration of the Seagull[™] allows three different reflection modes:

- External Reflection a flat-surfaced sample is placed at the focal point.
- Diffuse Reflection a rough-surfaced sample (for example, a powder) is placed in a sampling cup at the focal point.
- ATR a sample is placed under and in contact with a hemispherical internal reflection element.

Selection of a specific reflection mode requires only a simple exchange of a sample holder. In all three modes, the angle of incidence can be continuously changed from 5° to 85° without realigning the accessory. Note that in ATR mode there is a critical angle for total internal reflection. The angle of incidence should be greater than the critical angle. Spectra collected below the critical angle will qualitatively resemble specular reflection spectra.

The external reflection sample holder is used to analyze smooth, flat surfaced solids and liquids in the external reflection mode. It is also used to analyze powders and rough surfaces in the diffuse reflection mode. A trough liquid cell is provided for the analysis of liquids and a sample cup for the analysis of powders. The trough liquid cell and the sample cup readily mount onto the external reflection sample holder. The external reflection sample holder is adjustable to accept a range of sample sizes up to 2" L x 2" W x $\frac{1}{2}$ " thick.

For ATR studies, an ATR kit is available. This kit includes a ZnSe hemispherical ATR crystal mounted in a holder that slides into the SeagullTM in place of the external reflectance holder. The holder has a built-in pressure applicator and can accept samples up to 2' L x ³/₄" W x ¹/₂" thick . The kit also includes a compatible pressure plate. For ATR analysis of powders, a specially designed powder adapter is available. This adapter provides a spring-loaded removable cup that minimizes spillage of the sample while optimizing contact with the ATR crystal. For ATR studies of liquids, a flow-through liquid cell is available. The liquid cell is equipped with two luer ports and seals against the ATR crystal with an o-ring. The cell can be used for both flow-through measurements and for applications requiring a sealed liquid cell. A temperature-controlled version of the cell is also available for examining liquids from room temperature to 150°C. Purging is not possible when using the liquid cells.

For studying oriented polymers and determining ordering in systems with molecules on inorganic surfaces, the Ming-Sung[™] ATR Rotator is available. This device is a specially designed sample holder that permits a 360° rotation of the sample without affecting the contact with the ATR crystal.

For heated studies, a sample stage is available that can be heated to 150°. This stage can be used to support samples for either ATR or external reflectance measurements. An optional polarizer can be inserted into the Seagull[™] to set the polarization of the incident light.



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 of the Seagull[™].

GETTING READY

Before installing the Seagull^M, familiarize yourself with the accessory and its various components by referring to the drawing of the Seagull^M found below (Figure 1).

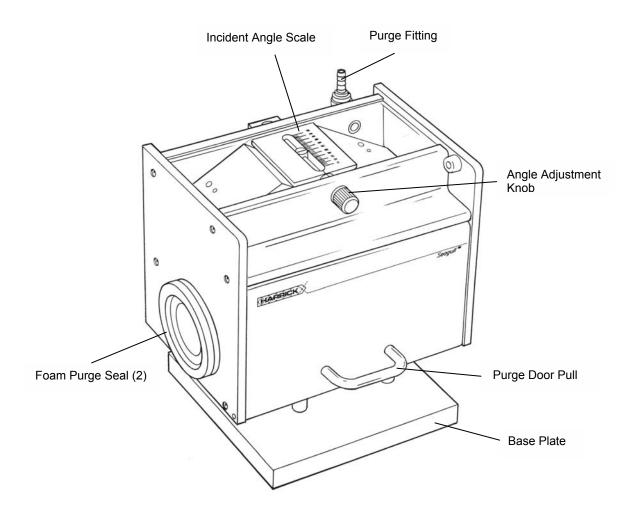


Figure 1 • Seagull™

PURGE INSTALLATION

PURGE SLEEVES

If your Seagull[™] is equipped with retractable purge sleeves (some of the purge sleeves are fixed), loosen the thumbscrews and push the purge sleeves in to retract them (Figure 2).

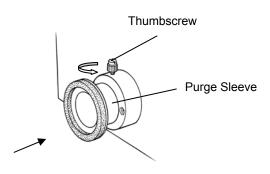


Figure 2 • Purge Sleeve

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 Seagull[™] as illustrated in Figure 3.

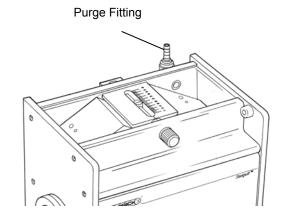


Figure 3 • Purge Fitting

Seagull™ INSTALLATION

RAIL MOUNTING

If your spectrometer is equipped with original manufacturer's rails:

- Install the Seagull[™] onto the rails.
- **NOTE:** If you received the **Harrick rail plate** with your Seagull[™], mount the supplied rails directly onto the floor of the sample compartment (see Appendix A).
 - Move the Seagull[™] along the rails until the spectrometer focal point is in the center of the attachment.
 - Secure the Seagull[™] in place by tightening the locking mechanism knob.

FIXED FLOOR MOUNTING

- Install the Seagull[™] 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.

Seagull[™] ALIGNMENT

The Seagull[™] is shipped pre-aligned and hence only minor adjustments should be required to fine-tune its alignment. Alignment is carried out with the accessory in the external reflection mode and 45° incident angle. Once this is performed, changing the angle of incidence will not misalign the system. Selection of the diffuse reflection or ATR mode will require only minor additional readjustments.

PREPARATION OF THE SAMPLE HOLDER

- Turn the slide knob on the external reflection sample holder clockwise (Figure 4) until the slide is free to move.
- Lower the height adjustment knob by rotating it clockwise.
- Locate the sample plate on the height adjustment knob.
- Place the mirror face up on the sample plate and move the slide so it lightly touches the mirror.
- Secure the slide by turning the slide knob clockwise.
- Elevate the mirror by rotating the height adjustment knob counterclockwise until the mirror is held in place against the retainer plate and slide.
- Open the purge door and slide the sample holder into the Seagull™.

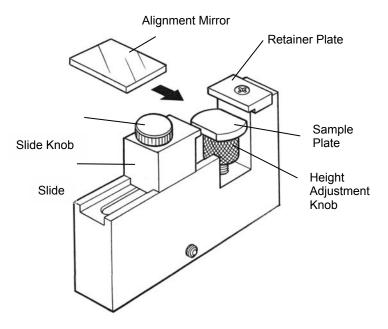


Figure 4 • Preparation of the External Reflection Sample Holder THE ANGLE OF INCIDENCE ADJUSTMENT The incident angle θ (Figure 5) is read from the scale on top of the Seagull™ (Figure 6).

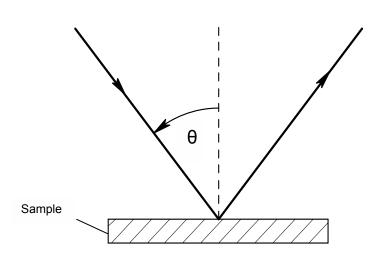


Figure 5 • Seagull™ (Top View)

To set the angle of incidence:

- Rotate the angle adjustment knob (Figure 6) until the pointer lines up with the desired angle on the scale (the scale is divided into 2° increments).
- For fine tuning, use the marks on the indexing ring (Figure 6). Rotating the angle adjustment knob from a long mark to a short mark will increase/ decrease the angle of incidence 0.5°. Further rotation to a long mark will increase/decrease the angle another 0.5°.
- **NOTE:** The accuracy of a setting is $\pm 0.5^{\circ}$ across the scale. The setting can be reproduced to better than $\pm 0.25^{\circ}$.

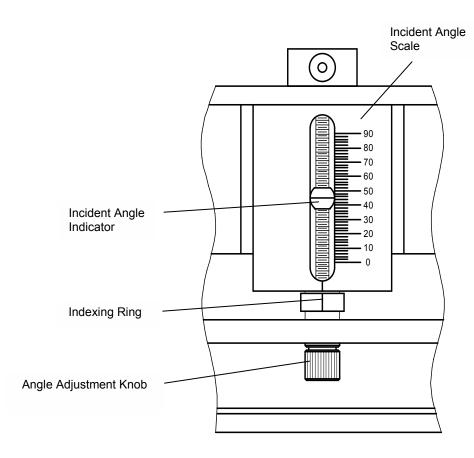


Figure 6 • Seagull™ (Top View)

MIRROR ADJUSTMENT

- Set the incident angle to 45°.
- Adjust the two knobs on mirror M1 first and then those on mirror M2 to maximize the energy striking the detector (Figure 7). These adjustments should be made in sequence. Only minor adjustments should be required.

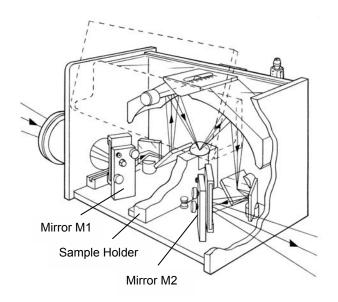


Figure 7 • Alignment

For rail mounting:

- Loosen the locking mechanism.
- Slide the Seagull[™] along the rails until the signal is maximized.
- Lock the locking mechanism.
- Extend the purge sleeves (Figure 2) until they firmly contact the sides of the sample compartment.
- Lock the purge sleeves in place with the thumbscrews.



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

SEAGULL THROUGHPUT, EXTERNAL REFLECTANCE

- Make sure that the specified background spectrum is the previously collected open beam background spectrum.
- Collect a sample spectrum with the Seagull[™] in the sample compartment.

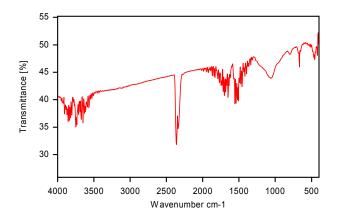


Figure 8 • Seagull™ Throughput, External Reflection

- Read the maximum value at 2500 cm⁻¹.
- The Seagull[™] throughput at that wavenumber should be at least 30% and the spectrum should resemble the Seagull[™] throughput spectrum shown in Figure 8.



EXTERNAL REFLECTION OPERATION

EXTERNAL REFLECTANCE OPERATION

To use the Seagull[™] to analyze solids in external reflectance mode install the external reflection sample holder (Figure 9):

- Set the incident angle.
- Place the alignment mirror or reference on the sample plate of the external reflection sample holder (as described in *Preparation of the Sample Holder*, page 6).
- Collect the background spectrum.
- Replace the reference with the sample.
- Collect the sample spectrum.

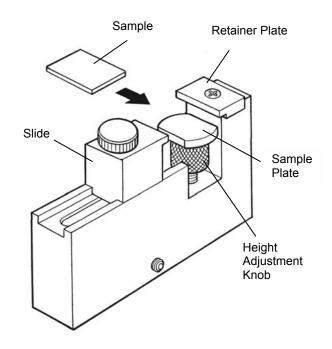


Figure 9 • The External Reflection Sample Holder



DIFFUSE REFLECTION OPERATION

To use the Seagull[™] to analyze powders and rough surfaces by diffuse reflection (both the diffusely and specularly reflected components are collected):

- Lower the height adjustment knob.
- Locate the sample cup (Figure 10) or sample plate on the height adjustment knob (as described in *Preparation of the Sample Holder*, page 6).

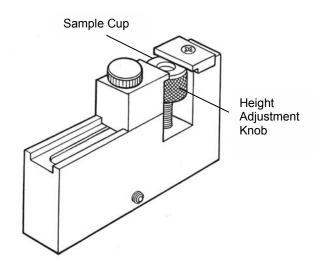


Figure 10 • The Sample Cup

To collect the background spectrum choose a reference with a reflectivity similar to your sample.

- **NOTE:** If your sample is a good reflector, a mirror would be a good reference. If it is a poor reflector, KBr powder would be a good choice.
 - Set the incident angle.
 - Place the reference on the sample plate of the external reflection sample holder (as described in *Preparation of the Sample Holder*, page 6), or fill the sample cup with the reference if using a powder as reference.
 - Collect the background spectrum.
 - Replace the reference with the sample.
 - Collect the sample spectrum.



TROUGH FOR ANALYZING LIQUIDS IN EXTERNAL REFLECTION MODE

To analyze liquids with the Seagull ${}^{\rm T\!M}$ in external reflectance mode:

- Set the incident angle.
- Place the alignment mirror or reference on the sample plate of the external reflection sample holder (as described in *Preparation of the Sample Holder*, page 6).

NOTE: If desired, a liquid could be used as a reference.

- Collect the background spectrum.
- Remove the mirror or reference.
- Replace the sample plate with the trough liquid cell (Figure 11).
- Fill the trough with approximately 3.75 ml of the liquid sample.
- **NOTE:** It is important to fill the trough exactly to the top. Otherwise the Seagull[™] is not aligned.
 - Collect the sample spectrum.

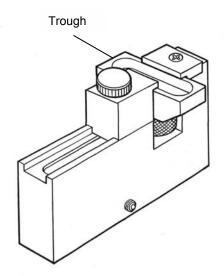


Figure 11 • The Trough Liquid Cell

ATR SAMPLE HOLDER	The ATR sample holder (Figure 13) is used for ATR operation.

Mount the ATR crystal into the ATR sample holder (Figure 12):

- Using a 7/64" ball driver undo the two screws on the crystal clamp of the ATR sample holder.
- Remove the crystal clamp.
- Carefully place the crystal on the ATR sample holder body as shown in Figure 12.

L CAUTION:

MOUNTING THE ATR CRYSTAL

The ATR crystal scratches easily so it should be handled with care. Wipe only with damp lens tissue or cotton swabs.

• Carefully replace the crystal clamp and tighten the screws evenly so the crystal clamp is seated with equal gaps on both ends (See detail in Figure 12).

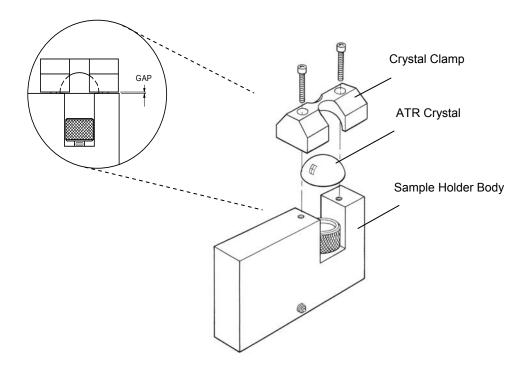


Figure 12 • Mounting the ATR Crystal

USING THE ATR SAMPLE HOLDER To use the ATR sample holder:

- Install the ATR sample holder into the Seagull[™].
- Set the Seagull for a 45° incident angle.
- Make sure the crystal is clean.
- Fine tune the alignment of the Seagull[™] by adjusting the two knobs on each mirror (Figure 7).
- Set the desired incident angle.
- Collect the background spectrum.
- Locate the pressure plate (Figure 13) on the height adjustment knob of the ATR sample holder.
- Place the sample on the pressure plate.
- Raise the pressure plate to make contact between the crystal and the sample.
- Collect the sample spectrum.
- Clean the crystal.
- **NOTE:** For reproducible pressure, use the optional torque screwdriver to tighten the hex screw located in the bottom of the ATR sample holder. **Do not exceed 20 oz-in of torque.**

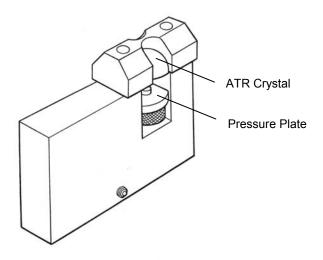


Figure 13 • ATR Sample Holder

ATR POWDER ADAPTER

For ATR powder analysis the powder adapter may be used:

- Lower the height adjustment knob all the way by rotating it clockwise.
- Locate the powder adapter (Figure 14) on the height adjustment knob.
- Set the incident angle.
- Collect the background spectrum.
- Fill the cup with the sample.
- Elevate the powder adapter by rotating the height adjustment knob counterclockwise until the powder adapter spring is fully compressed against the ATR crystal.
- **NOTE:** For reproducible pressure use the optional torque screwdriver to tighten the hex screw located in the bottom of the ATR sample holder. **Do not exceed 20 oz-in of torque.**
 - Collect the sample spectrum.
 - Clean the crystal.

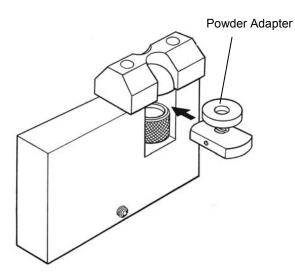


Figure 14 • Powder Adapter

FLOW-THROUGH LIQUID ATR CELL

For liquid analysis the flow-through liquid cell is used. To install the liquid cell:

- Using a 7/64" ball driver undo the two screws on the crystal clamp of the ATR sample holder.
- Remove the crystal clamp being careful to hold the crystal so it does not get damaged.

L CAUTION:

The ATR crystal is not attached to the crystal clamp and should be held while removing the clamp. The crystal scratches easily so it should be handled with care. Wipe only with damp lens tissue or cotton swabs.

- Make sure the liquid cell o-ring is clean, free of dust and in good condition.
- Locate the flow-through liquid cell on the height adjustment knob of the ATR sample holder (Figure 15).
- Replace the clamp and tighten the screws evenly.
- Clean the ATR crystal (see caution).
- Elevate the liquid cell by rotating the height adjustment knob counterclockwise until the liquid cell is secured against the crystal.

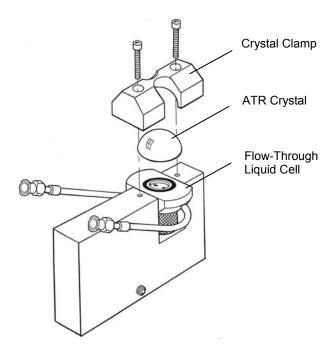


Figure 15 • The Flow-Through Liquid Cell

The liquid cell can be used for static (Figure 16) and flow (Figure 17) applications.

STATIC APPLICATIONS

If the liquid sample is volatile:

- Collect the background spectrum.
- Remove the plugs on the Luer fittings.
- **NOTE:** To fill the liquid cell in a glove box or other enclosed environment, remove the liquid cell/ATR sample holder assembly from the Seagull[™].
 - Use a Luer-Lok syringe to fill the cell with approximately 1 ml of the sample.
 - Reinstall the plugs to seal the cell.
 - Check for leaks before installing the liquid cell/ATR sample holder assembly into the Seagull™.
 - Collect the sample spectrum.
 - Clean the crystal.
 - To clean the cell, remove the cell from the sampling plate and rinse it with solvent.

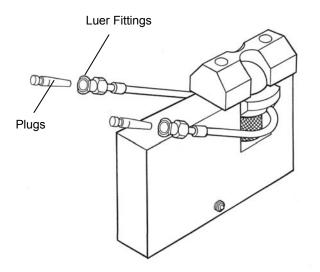


Figure 16 • Liquid Cell Setup for Static Applications

FLOW- THROUGH APPLICATIONS For flow-through applications (Figure 17):

- Collect the background spectrum.
- Remove the liquid cell/ATR sample holder assembly from the Seagull™.
- Connect the inlet and outlet, using appropriate adapters, to the Luer fittings on the cell.
- Turn the flow on and check for leaks. Fix any leaks before proceeding.
- Return the liquid cell/ATR sample holder assembly into the Seagull™.
- Collect the sample spectrum.
- Clean the crystal.
- To clean the cell, flow some of the new sample or some solvent through it. For more thorough cleaning, remove the cell from the sampling plate and rinse it with solvent.

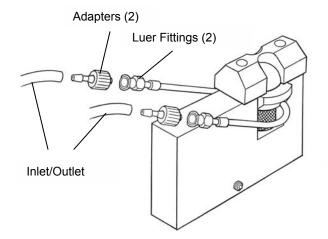


Figure 17 • Liquid Cell Setup for Flow-Through Applications

THE MING-SUNG™ ATR ROTATOR

INSTALLING THE ATR CRYSTAL

To install the ATR crystal in the Ming-Sung Rotator (Figure 18):

- Insert the supplied 3/32" ball driver into the clamping screw and turn counter-clockwise to retract the clamping piston.
- Place the ATR crystal on the crystal holder in the locating recess.
- Using the 3/32" ball driver gently turn lower the clamping screw until the ATR crystal is securely held in place.

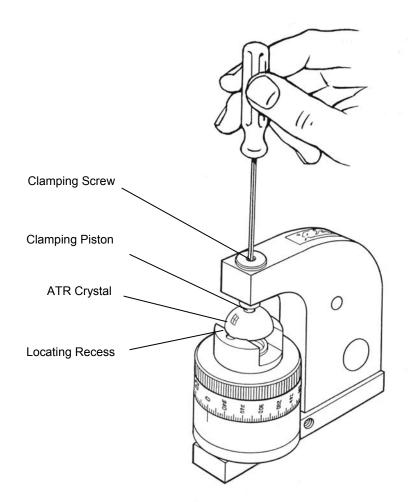


Figure 18 • Installing the ATR Crystal

USING THE MING-SUNG™ ATR ROTATOR

To analyze solid samples using the Ming-Sung[™] ATR Rotator:

- Set the incident angle (Figure 6).
- Collect the background spectrum. For best results collect a background at each incident angle that requires sample measurements.
- Insert the supplied 1/8" ball driver in the hole in the bottom of the Ming-Sung[™] ATR Rotator (Figure 19).
- Turn the ball driver to lower the height adjustment stage.

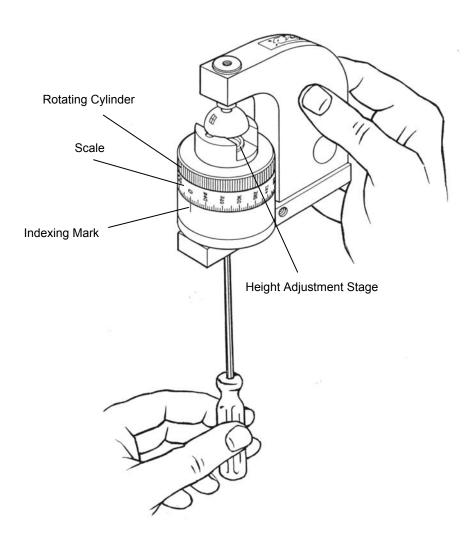


Figure 19 • The Ming-Sung™ ATR Rotator

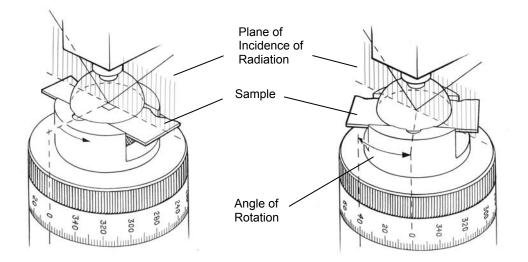


Figure 20 • Ming-Sung™ Operation

- Locate the pressure plate on the height adjustment stage.
- Place the sample on the pressure plate in such a way that the straight edge (reference edge) of the sample lines up with either the front or back edge of the sample slot (Figure 20).
- **NOTE:** The angle on the scale on the Ming-Sung[™] is the angle between the reference edge of the sample and the plane of incidence of radiation.
 - Elevate the pressure plate with the sample using the 1/8" ball driver until the sample is secured against the crystal.
 - Check that the sample is still aligned. If it is not, realign it.
 - Rotate the scale on the rotating cylinder to select the desired angle.
 - Collect the sample spectrum. Use the appropriate background spectrum.
- **NOTE:** For reproducible pressure use the optional torque screwdriver instead of the supplied 1/8" ball driver. **Do not exceed 20 oz-in of torque.**

SEAGULL[™] POLARIZER

The Seagull[™] polarizer is a wire grid polarizer on a KRS-5 substrate. It can be mounted in the Seagull[™] or on a standard sample slide plate for other applications.

SEAGULL™ MOUNTING

- Make sure that the slide plate is not attached to the polarizer mount (see page 24). Remove if installed.
- To mount the polarizer in the Seagull[™], slide it into its mount (Figure 21).
- To select the polarization, rotate the lever until it locks in place. The polarization is indicated by the icon (|| for parallel, ⊥ for perpendicular).

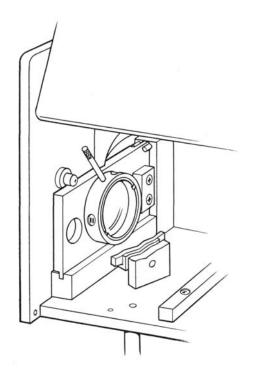


Figure 21 • Polarizer Mounted in the Seagull™

SAMPLE SLIDE MOUNTING

To mount the polarizer on a sample slide holder:

- Orient the slide plate so the countersunk holes face away from the flat side of the polarizer holder (Figure 22).
- Install the two screws provided.
- **NOTE:** The icons || for parallel and ⊥ for perpendicular refer to the polarizer's use in the Seagull. When the polarizer is mounted on a sample slide holder the electric field of transmitted light will be either vertical (if the polarizer is in the ⊥ setting) or horizontal (if the polarizer is in the || setting). For p-polarization, the electric field should be parallel to the plane that contains the incoming rays, reflected rays and the normal to the sample. S-polarization is perpendicular to p.

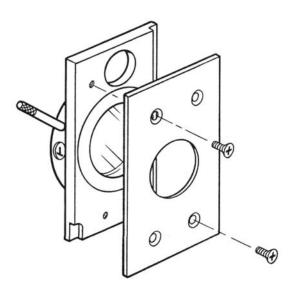


Figure 22 • Polarizer Mounted on Sample Slide Plate

HEATED SAMPLE STAGE

The heated sample stage (Figure 23) can be used with both the external (page 15) and ATR (page 20) sample holders. To control the temperature, connect the thermocouple and the heater to a suitable temperature controller.

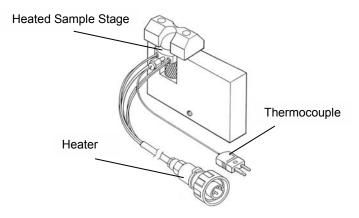


Figure 23 • Heated Sample Stage in the ATR Sample Holder

HEATABLE FLOW-THROUGH LIQUID CELL

The heatable flow-through liquid cell (Figure 24) is used in the same way as the unheatable cell (page 17). To control the temperature, connect the thermocouple and the heater to a suitable temperature controller.

NOTE: The heatable flow-through liquid cell is compatible with the Harrick Automatic Temperature Controller, but an adapter is provided for use with other temperature controllers.

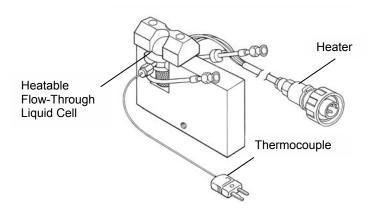


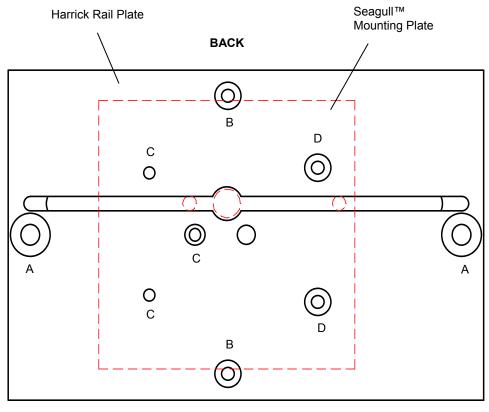
Figure 24 • Heatable Flow-Through Liquid Cell



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 25. See Table 1 on the next page for the appropriate screws/holes for your spectrometer.



FRONT

Figure 25 • Screw and Pin Positions for Rail Plate

SPECTROMETERS	HOLES
DIGILAB (All Models)	A
MATTSON (Polaris, Galaxy, RS Infinity, Genesis, Satellite)	В
PERKIN ELMER (Spectrum 1)	С
THERMO-NICOLET (Nexus, Avatar, Magna, Impact)	D

Table 1 • Screw and Pin Positions for Rail Plate



SEAGULL™ THROUGHPUT, ATR MODE

- Make sure that the specified background spectrum is the previously collected open beam background spectrum.
- Collect a transmittance spectrum with the Seagull™ in the sample compartment.

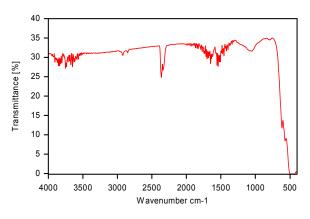


Figure 26 • Seagull™ Throughput, ZnSe

- Read the maximum value at 2500 cm⁻¹.
 - The Seagull[™] throughput for ZnSe ATR crystal at that wavenumber should be at least 10% and the spectrum should resemble the Seagull[™] throughput spectrum shown in Figure 26. If you are using other crystals (Ge or Si), compare the throughput with that supplied with the crystal.



OPTIONAL PARTS

Flow-Through Liquid Cell. Heatable Flow-Through Liquid Cell, 24V. Heated Sample Stage ATR Kit (includes ZnSe hemisphere and holder, pressure plate and clamp). KRS-5 Wire Grid Polarizer Ming-Sung ATR Rotator	SEA-FLC SEA-HLC-3 SEA-HOT-3 SEA-IRK PWG-SEA
(includes hemisphere holder, pressure plate and clamp) Powder Adapter SOS™ Software Package Torque Screwdriver Trough Liquid Cell Ge ATR Hemisphere Si ATR Hemisphere Kalrez O-ring for the Flow-Through Liquid Cell Automatic Temperature Controller (110V) Automatic Temperature Controller (220V)	SEA-MNP SEA-PSH SOS-102 PTW-SXX SEA-TLC EJ6xBB EE6xBB ORK-013 ATC-024-1 ATC-024-2

REPLACEMENT PARTS

Alignment Mirror	MOP-112
ZnSe ATR Hemisphere	EM6xBB
Viton O-ring for the Flow-Through Liquid Cell	ORV-013
K-type Thermocouple	008-148
24V Heater Assembly for the SEA-HOT heated stage	SEA-HOT-HTR
24V Heater Assembly for the SEA-HLC heated liquid cell.	SEA-HLC-HTR
1.00"x0.125" Cartridge Heater, 24V, 30W	HTRS-20



Manual Part No. SEA-M-LV

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