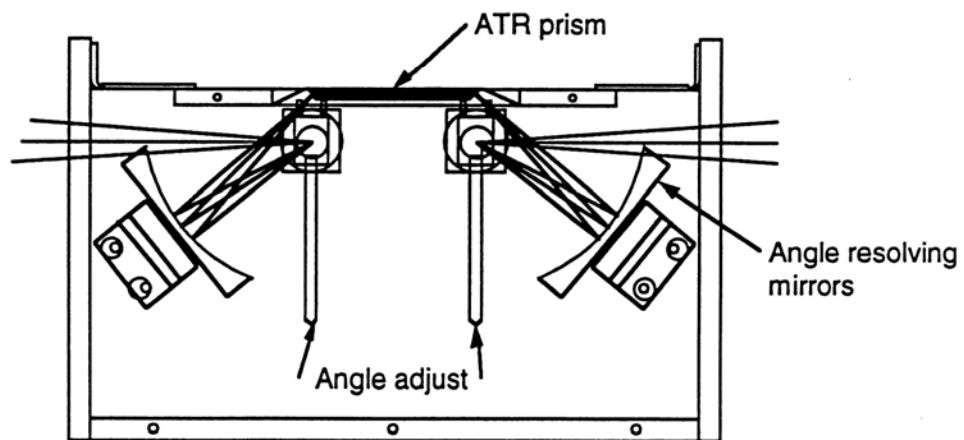

EXPLORER

Horizontal ATR

User's Guide

 ***CIC Photonics***

Explorer™ Horizontal MIR/MER Reflectance Accessory



FOREWORD

Thank you for purchasing a CIC Photonics sampling accessory. We strive to build the best sampling accessories available and believe that you will be pleased with the performance of this horizontal ATR long path gas cell. Should you have any difficulties at all please call 505-343-1489 for technical assistance. We should be able to help you immediately.

If you have any comments on this or any of our other products we would like to hear from you. We can be reached at the address, telephone numbers or E-mail address as given below. Thank you again for your business.

Sincerely,

Richard T. Meyer
CEO & President
CIC Photonics, Inc.

CIC Photonics, Inc.
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509-479-2980 fax
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OUR WARRANTY

- I. Since CIC Photonics builds its products to last, we warrant them that way. If you have a problem with our accessory, within the first year of ownership, that is a result of a defect in workmanship or the wearing out of a component that should not wear out, we shall fix it.

- II. Parts that normally wear out or are consumed or can be damaged in the normal operation of the accessory, such as fragile optical elements (lenses, windows, crystals, mirrors, filters, etc.) are warranted against defect in manufacture for a period of 30 days after original delivery to the purchaser.

Packing List

The items listed below (except optional equipment) should be found shipped with this accessory. If you think that any of these items are missing or damaged, please contact CIC right away.

	<u>Quantity</u>	<u>Part Number</u>	<u>Size/Type</u>
<u>Explorer Accessory</u>	1	42B100	
<u>ATR Prism Assembly</u>			
45 Zinc Selenide	1	42B500	
<u>Tools</u>			
Ball Driver	1		1/16"
<u>Hardware</u>			
Hold-down Screws	2		8-32 x 3/4"
<u>Instruction Manual</u>	1		

Optional Equipment

Available Optional Equipment

White Nalgene™ Sample Pads (20)	28B180
Torque Wrench	28B110
Heated Pressure Plate (110V)	42B400
Heated Pressure Plate (220V)	42B401
Liquid Flow Cell	42B402
Heated Liquid Flow Cell (110V)	42B403
Heated Liquid Flow Cell (220V)	42B404
Temperature Controller (110V)	09B600
Temperature controller (220V)	09B601
45deg Zinc Selenide Prism Assembly (Flush)	42B500
60deg Zinc Selenide Prism Assembly (Flush)	42B501
45deg KRS-5 Prism Assembly (Flush)	42B510
60deg KRS-5 Prism Assembly (Flush)	42B511
30deg Germanium Prism Assembly (Flush)	42B520
45deg Germanium Prism Assembly (Flush)	42B521
60deg Germanium Prism Assembly (Flush)	42B522
45deg Zinc Sulfide Prism Assembly (Flush)	42B530
60deg Zinc Sulfide Prism Assembly (Flush)	42B531
45deg Zinc Selenide Prism Assembly (Saucer)	42B600
60deg Zinc Selenide Prism Assembly (Saucer)	42B601
45deg KRS-5 Prism Assembly (Saucer)	42B610
60deg KRS-5 Prism Assembly (Saucer)	42B611
30deg Germanium Prism Assembly (Saucer)	42B620
45deg Germanium Prism Assembly (Saucer)	42B621
60deg Germanium Prism Assembly (Saucer)	42B622
45deg Zinc Sulfide Prism Assembly (Saucer)	42B630
60deg Zinc Sulfide Prism Assembly (Saucer)	42B631
Wire Grid Polarizer	42B800

Introduction

Attenuated Total Reflectance or ATR Spectroscopy is one of the most useful and well established sampling techniques in infrared spectroscopy. When infrared energy is made to propagate through a high refractive index, infrared transmissive crystal, such as zinc selenide or KRS-5, at the proper angles of incidence, the phenomenon of total internal reflection occurs. As the energy propagates through the crystal, it encounters the faces of the crystal. If a sample is placed against the face of the crystal, the infrared spectrum of the sample can be measured.

Over the years, the multiple reflection sampling crystal has become the standard. With this type of crystal, infrared energy is focused onto the crystal bevel and travels through the crystal by multiple internal reflection. Typically, 5 to 20 reflections against the face of the crystal are achieved before the energy exits through a bevel at the other end. At each of these encounters with the face of the crystal, an evanescent or standing wave is set up. If an absorbing material is placed in contact with the crystal face, some of this evanescent wave will be conducted into the material in the region of absorption bands in the material. In this manner, the infrared spectrum of the material is measured.

The technique of attenuated total reflectance spectroscopy was developed well before the introduction of FT-IR spectrometers. The traditional multiple sampling ATR crystals were matched in geometry to the slits of the dispersive spectrometers. After the introduction of FT-IR, these designs were modified to more accurately match the round field stop image of the FT-IR spectrometer. This modification consisted of decreasing the height of the crystal from 20mm to 10mm and increasing the width of the crystal from 2mm to 3mm. These modifications still left the throughput of the ATR accessory lower than that which could be achieved with dispersive spectrometers. Since FT-IR instruments have inherently high sensitivity, it was not deemed important to really optimize the accessory for the spectrometer. Many if not all of the ATR accessories currently on the market are derivative from these early dispersive units.

Recently, the horizontal configuration for ATR accessories has become popular, and the Explorer falls into this category. Horizontal ATRs also initially followed the optical geometry's appropriate for dispersive spectrometers. The horizontal geometry allows the sample to be placed on the top of the accessory. In the case of liquids or pastes, no pressure is required; the liquid is simply poured in a trough surrounding the sample. In the case of solids, a clamp creates contact with the crystal. In either case, sample throughput is improved because it becomes much easier to load and remove the sample.

The Explorer™ was conceived of as a clean slate approach to the design of a no-compromise ATR unit. Every part has been re-thought with the beam characteristics of an FT-IR spectrometer in mind for high sensitivity. In addition, the mechanical design has been geared towards ease of use, versatility, and repeatability. The sampling crystal is larger than other ATR units, measuring 50x10x6mm for the 45deg crystal. This is to allow the full beam of the FT-IR to get through, and to maintain a sufficient number of multiple reflections. High quality mirror mounts are used which provide precise adjustment and stability. Angle change is accomplished through a simple dial mechanism.

Installation

In preparing to install the ATR accessory, first remove existing accessories and sample holders from the sample compartment. All necessary tools are provided with the accessory.

Unpacking

Remove the Explorer accessory from its case and place it on a bench. Remove all packing material from the unit. Inspect the unit for shipping damage, and notify CIC immediately of any problems.

Mirror Mounts

The C.I.C. Photonics mirror mounts are kinematically adjustable. The Explorer has been pre-aligned. Please install the accessory as described below before adjusting any of the mirrors.

Install

The Explorer accessory installs directly into the spectrometer, by bolting to the baseplate. Remove any existing accessories from the spectrometer. Position the Explorer in the sample compartment and place down over any dowel pins in the compartment. The accessory should be positioned so that it is centered left/right and so that the beam openings on the sides are even with the beam openings of the spectrometer. Lock the accessory down with the supplied screws and ball driver. Refer to the sketch on the cover of this manual for proper orientation.

ATR Sample Holder

The standard ATR holder supplied with the Explorer is a solids holder for films sheets and solids. It includes a 45deg Zinc Selenide crystal. This holder is shipped already mounted in the unit. This assembly should be used for the following alignment procedure.

Angle Adjustment

The angle of incidence is variable from 30deg to 90deg. The large angles will be most useful with external reflectance measurements. For a 45deg crystal, the angle should nominally be set at 45deg. Changing the angle away from the cut angle of the crystal will cause refraction at the entrance and exit of the crystal. When this occurs, Snell's law can be used to determine the actual angle of incidence for the crystal:

$$n \sin u = n' \sin u'$$

Where n' is the refractive index of the crystal (about 2.45 for zinc selenide) and n is the refractive index of the surrounding medium (in this case air, $n = 1$). u and u' are the angle of incidence before and after refraction, with respect to the cut of the crystal bevel. When the crystal is cut at 45deg and the angle is set at 45deg, $u=0$ and therefore $u'=0$. There is no refraction. If the angle is set to 50, then there is a $u=5$ deg angle with respect to the cut of the crystal. After refraction, $u'=2.04$, so the angle of incidence is 45.04deg.

To change the angle on the Explorer, simply turn the dial on the input side to the desired angle, then rotate the output dial until an energy maximum is obtained. Set the angle to 45deg for purposes of alignment.

Alignment

Alignment

This Explorer accessory has been pre-aligned prior to shipment. Because of normal variations from bench to bench, you may need to slightly peak up the energy. Again, after changing crystals, you will need to re-align slightly. Never re-align or change angles between running a sample and a background spectrum. The alignment process is simply a matter of adjusting each of the four mirror adjuster screws until maximum energy is reached while the spectrometer is in the tune mode. These adjustments should be done slowly in order to allow the spectrometer detector time to respond to the change. Adjust all four screws successively, in any order, and go through a few times to make sure that a maximum is reached for each screw. After changing angles, it is a good idea to maximize energy throughput again by adjusting these 4 screws. These screws are accessible through the front plate of the accessory using the supplied 1/16" ball driver. Practice removing and replacing the solid sample holder and watch the energy to make sure you achieve the same alignment each time. This is important for the utmost photometric accuracy. A properly aligned Explorer will get higher percentage energy throughput than virtually any other ATR accessory. If you run a spectrum referenced to the open beam, the percent transmission should be between 12% and 15% from 4000cm⁻¹ to 700cm⁻¹ with the zinc selenide crystal*. If not, make sure the accessory and the solid sample holder are installed properly and go through the alignment procedure again. Collect a background spectrum using your chosen spectrophotometric parameters into the background file.

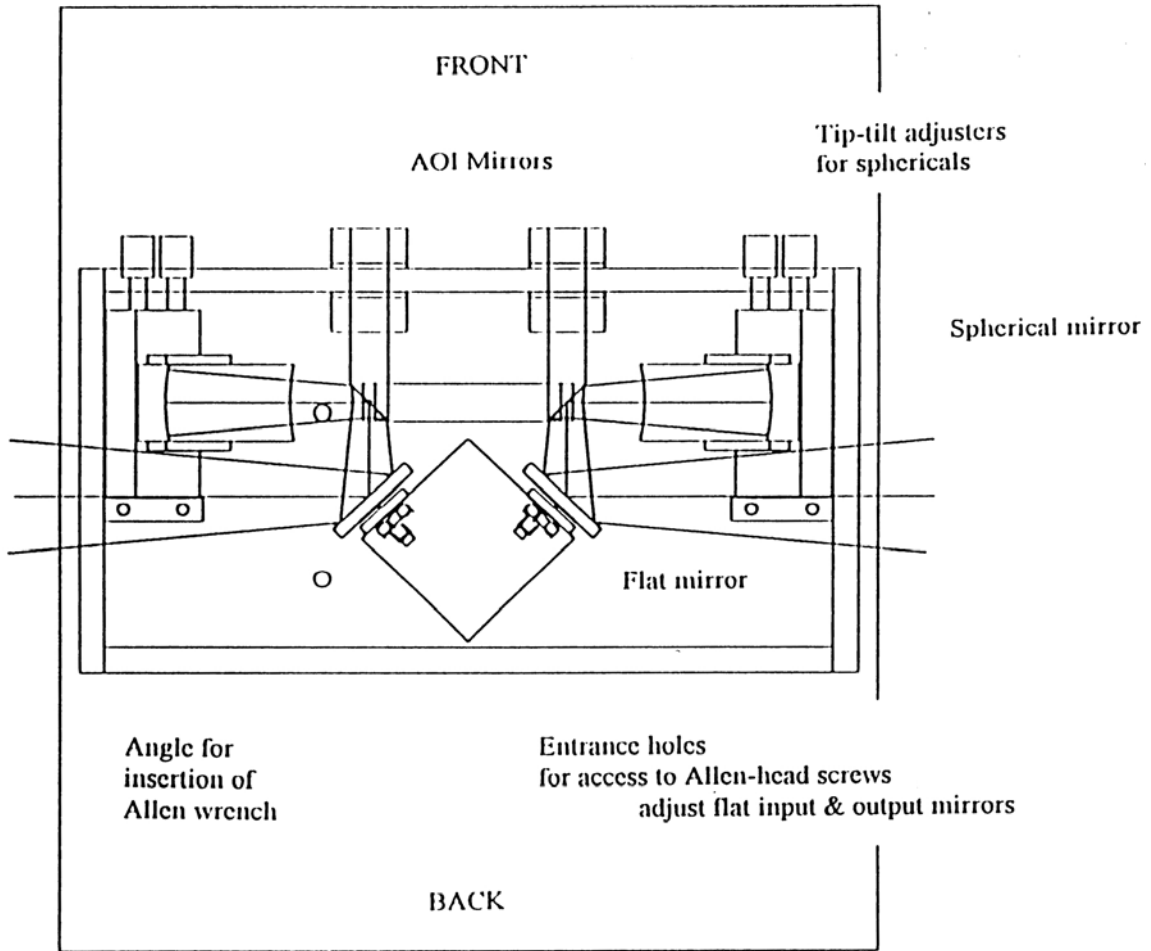
Major Realignment

Should it become necessary to perform a major realignment of the Explorer, refer to the drawing on the next page and apply the following procedures:

1. On the front panel, set the AOI for mirrors at 45 degrees.
2. From the backside of the accessory and using an Allen wrench through the 1/2" diameter holes, fine adjust the tip-tilt of the two flat mirrors, starting with the output mirror and then the input mirror; repeat the process on both mirrors to achieve maximum energy throughput. (Note: to reach the sockets of the Allen head screws, you have to point the Allen wrench at about a 45 deg angle through the 1/2" diameter holes on the backside of the instrument; there are two screws for each mirror that control the tip-tilt.)
3. Next fine adjust the two spherical mirrors from the front panel using the two knurled knobs for each mirror which project out through the front panel.
4. Then repeat the above steps 2 and 3 until the best throughput is achieved.

Since we aligned the Explorer on our Mattson Galaxy 5000 FTIR and since the IR beam characteristics vary from spectrometer to spectrometer, either a minor Alignment or a major realignment may be required on the user's FTIR.

*Note: Scratched or otherwise damaged crystals will have diminished throughput.



Top Down View

Operation

Loading a Sample

Collect a background spectrum before loading the sample. Place a sample (try a piece of polystyrene film) on top of the crystal, so that the entire crystal is covered. Place a white NalgenetTM pad on top of the sample. Unpack the pressure plate assembly. Locate the dowel pins of the pressure plate assembly into the keyholes in the top plate of the ATR. Once engaged, the screw of the pressure plate can be turned to apply pressure to the sample. Tighten the knurled thumb screw, making sure it engages the dimple in the top pressure plate. The amount of pressure applied is subjective and depends on the hardness of the crystal and the hardness and thickness of the sample. For starters, apply only a gentle thumb pressure onto the crystal. The crystal is very tough and will withstand a lot of pressure. The degree of pressure can be used as a variable in achieving the proper level of absorbance. A good sample to practice on is the NalgenetTM foam strips which were installed in the holder for shipment. Do not apply a lot of pressure at this point, e.g., do not completely squeeze the Nalgene. If the sample is smaller than the crystal, a piece of foil should be used between the sample and the Nalgene pad so that the crystal does not “see” the Nalgene. Tighten the knurled thumb screw, making sure it engages the dimple in the top pressure plate. The amount of pressure applied is subjective and depends on the hardness of the crystal and the hardness and thickness of the sample.

Changing Crystals

The crystals of the Explorer are changeable. Four flat head screws should be removed from the crystal plate. This will allow the crystal plate to be removed. Lever the crystal out of the top plate, being careful to not actually touch the crystal with any implements. Install a new crystal by reversing these steps. Again be careful to not touch the crystal, as it could break.

These crystal assemblies can be disassembled at the factory and reconditioned. Please call CIC for further information.

Reference

- 1) *Standard Practices for Internal Reflection Spectroscopy*, E573-81; American Society for Testing and Materials: Philadelphia, 1981.