INSIDER

FTIR EMISSION ACCESSORY

User's Guide

Revised October 2003

CIC Photonics

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
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CIC Photonics, Inc.

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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.

INTRODUCTION

The CIC Photonics, Inc. emission accessory is different from most other emission accessories in several important respects. It is, therefore, important to read these instructions before installing or using the accessory.

All materials at temperatures above absolute zero emit energy. This emitted energy can be detected any time that the sample is at a different temperature than the detector. This emitted energy contains spectroscopic information similar to that which is obtained by absorption or reflection spectroscopy. No new information is obtained with emission spectroscopy, but it gives us an alternative method of obtaining spectral information. For some samples, emission spectroscopy is the method of first choice, giving better spectra than other more traditional sampling techniques.

At temperatures only slightly above ambient, 50deg C, there is sufficient emitted energy in the infrared region such that an infrared spectrum may be obtained. The sensitivity of the technique is governed by the temperature and the emissivity of the sample.

Since in emission spectroscopy the sample acts as the heat source, the normal infrared source is not used. It must be turned off. Also, since the heat energy from the sample must be modulated by the interferometer, the emitting sample must normally be placed before the interferometer. This would normally require that the experiment take place outside of the spectrometer sample compartment. The CIC Photonics, Inc. emission accessory uses a novel beam splitting method to allow the emission experiment to be performed in the sample compartment. This makes the measurement much more convenient.

The Insider Emission Accessory works only with non-corner-cube FTIRs.

INSTALLATION

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

Unpacking

Remove the emission accessory from it's case and place it on a bench. Carefully remove the cotton lined bags from the mirror mounts.

Mirror Mounts

The CIC mirror mounts are kinematically adjustable. They have been locked and taped for shipment. Remove the strips of black tape from each mount, being carefull not to touch the mirror surfaces. Unlock the mirrors by turning the black knurled lock screws on each mount.

Install

The emission accessory installs by bolting down to the spectrometer baseplate throught the two 8-32 tapped holes. Line up the accessory with these holes and bolt it in place. Refer to the sketch on the cover of this manual for proper orientation.

Heated Cell

Unpack the heated cell and insert it into the cell slide of the accessory with the wires leading up. Find a convenient way to route the wires of this unit out of the sample compartment. The cell slide can move forward and back to focus the sample. It is shipped in the proper postion for thin samples. The blackbody reference disk is installed in the heated cell. This is used both for alignment and as the reference material. This will provide the largest possible signal for alignment.

Alignment

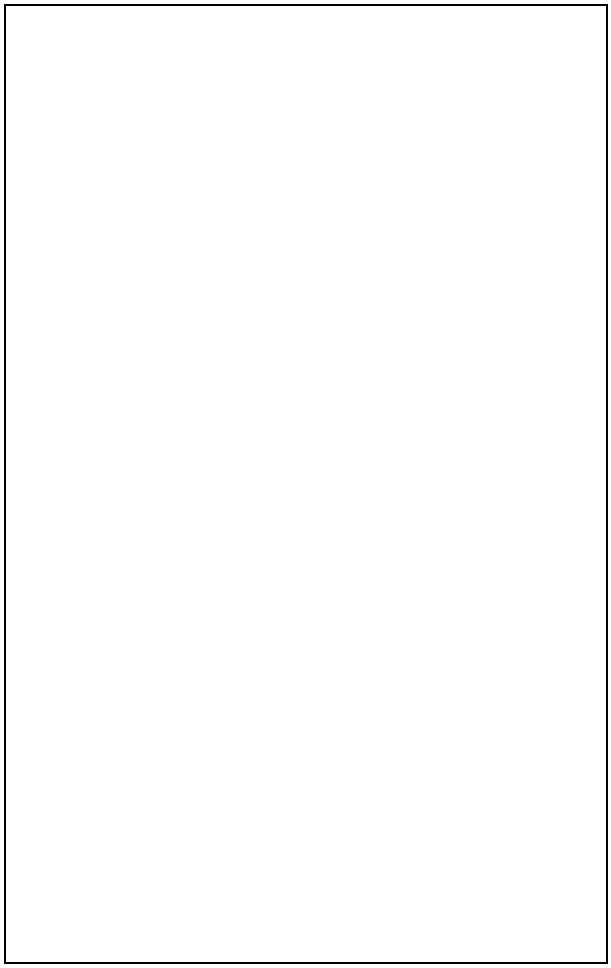
It should be possible to see the spectrometer laser beam impinging on the splitter mirror from the right or left, depending on orientation for your spectrometer (see page___). Use the adjusters on the splitter mirror to image this laser light onto the center of the relay mirror. Then use the adjusters on the relay mirror to image the laser energy onto the sample in the heated cell.

First, use **coarse** adjusters to align with spectrometer laser beam, then use **fine** adjusters. Adjust mirror-holding brackets and mirrors so that laser beam enters the

sample cell well and strikes the “blackbody” graphite sample disk. This will provide coarse alignment.

Turn on the temperature controller and allow the cell to heat up to 100deg C. Open the spectrometer and unplug the infrared source. The energy should slowly decrease as the source colls down. Wait about 10 minutes before using the emission accessory while the spectrometer source colols off. As the emission sample heats up, the energy should start returning. when the sample has reached 100deg C, the two mirrors may be adjusted slightly to peak up the energy. Do not overadjust the mirrors so that the laser light is no longer hitting the sample. This would cause the spectrometer to be seeing some other emitting object. It is important that the spectrometer “sees” the sample and not the surrounding mount.

Detector Interferometer



Flip diagram 180 deg if detector and interferometer positions are interchanged with your spectrometer.

OPERATION

Operation

The operation of the emission accessory involves placing a sample in the heated cell, heating it up to the desired temperature, and collecting a spectrum. This single beam emission spectrum may be ratioed to the spectrum of a black body at the same temperature to obtain an emittance (double beam) spectrum. The supplied graphite (black body) disk which was used for alignment is an appropriate pseudo-black body reference. Remember to let the heated cell cool down before handling, or alternatively, handle it with temperature resistant implements and gloves.

In order to fit in the heated cell, samples must be smaller than 3/4" diameter. The area of the sample that is seen by the field stop of the spectrometer is a half circle with a diameter of 1/2".

The heated cell is capable of being moved front to back in the spectrometer. For thim samples, leave the cell in the position in which it was received. For thicker samples, the cell can be moved back to focus the energy onto the field stop and thereby increase the performance.

Loading a Sample

Open the heated cell by removing the three 6-32 screws. Remove the ceramic cover and all of the installed spacers. Place the sample in the cell. If the sample is not free standing, it may be placed on one of the sample supports provided, or your own sample mount which must be 3/4" diameter or smaller. Also, a shiny penny makes an excellent sample support. Next, insert enough bored-out spacers to fill up the bore of the cell. Replace the ceramic cover plate and tighten the three screws. Do not overtighten the screws or the ceramic will break.

Temperature Control

If you are using the CIC temperature controller, please refer to the separate controller instruction manual. This controller is a self-tuning three-mode controller for accurate temperature control. The temperature of the heated cell can be varied from room temperature to 400deg C in 1deg increments. The CIC controller has an accuracy of 1deg C.

Auxiliary Alignment and Operation Procedures

Alignment Procedure:

In order to function correctly, the Insider accessory must be properly located in the FTIR spectrometer sample compartment. The beveled mirror must be located at the center of the incoming IR beam from the interferometer, positioned as close to the interferometer as possible. At the factory, CIC centered and aligned the mirrors using a He-Ne laser. However, fine adjustments may need to be made by the end user, as follows:

1. Prior to installation of the Insider, note the energy throughput of the spectrometer with no obstructions to the IR beam.

2. Install the Insider into the compartment per the mounting procedures.

3. With the Insider installed and assuming perfect alignment, the energy throughput should be ½ of the recorded throughput without obstructions. If this is true, skip to the Operation Procedures below.

4. If not, then fine adjustments can be made to the beveled mirror using two different methods. Both methods require the beveled mirror to be centered in the IR beam coming from the interferometer. The center of the IR beam can be found using a card with a thin liquid crystal which changes color when exposed to the beam.

Method A: Adjust the beveled mirror using the thumb screws until the energy throughput is ½ of the original throughput recorded without obstructions. The beveled mirror is now centered on the IR beam.

Method B: This requires use of the laser beam from the spectrometer and a piece of white paper; first be sure to confirm that the laser is centered with the IR beam. In the following, **BE CAREFUL NOT TO SCRATCH THE MIRROR SURFACE.**

(1) Locate the laser spot with the white paper coming from the interferometer window.

(2) Follow the laser to the beveled mirror and adjust the beveled mirror using the thumb screws until the beam is split into two beams: one going to the detector and the other to the concave mirror.

(3) Adjust the second beam using the thumb screws on the flat mirror so it hits the center of the concave mirror.

(4) Adjust the concave mirror so that the second laser beam is hitting the center of the installed black body in the sample cell.

(5) Check the energy throughput of the spectrometer; it should be ½ of the original recorded value.

Operation Procedures:

1. The IR source must be turned off and disconnected in the spectrometer; some spectrometers have a switch while others must be disconnected.

2. The energy throughput will diminish slowly as the IR source cools down.

3. The spectrometer settings may need to be adjusted (i.e. gain increased) in order to record the IR emission from the sample.

4. To obtain emission curves, heat the black body sample to 50 deg C; energy throughput should start to increase. The black body sample is the reference (background) for other samples.

REFERENCES

Interpretation of Emission Spectra

Infrared emission spectra are useful in the determination of molecular structure; however, they may exhibit some important differences from transmission or absorption spectra. Most importantly, an emission spectrum is easiest to obtain when the sample is thin. As the optical density of a sample increases, the emitted energy from that sample at a given temperature approaches the black body curve for that temperature. When this happens, spectral features disappear, precluding identification of the material. There are several ways to overcome this limitation. First, if the surface of the sample is at a higher or a lower temperature than the bulk of the sample, then spectral bands will reappear. The differential of the temperatures determines the band intensity. If the surface is cooler than the bulk, the spectral features will point towards lower intensity; and if hotter, the bands will point towards higher intensity. This effect can be used to advantage. Second, if the sample is a natural layered structure, then a good emission spectrum may be obtained even when the bulk of the sample is strongly re-absorbing. This again can be used to advantage with a class of samples which is classically very difficult to analyze by infrared spectroscopy—namely, thin coatings on optically opaque samples. An example of this is sizing on graphite fibers. For further information on the technique of emission spectroscopy, the interested reader is referred to the references listed below.

References

1) R.P. Eischens and W.A. Pliskin, Adv. Catal. **9**,1 (1958)

2) G. Fabbri and P. Baraldi, Appl. Spec. **26**, 593 (1972).

3) P.R. Griffiths, Appl. Spec. **26**, 73 (1973).

4) Y. Nagasawa and A. Ishitani, Appl. Spec. **38**, 168 (1984).

5) M.A. Ford and R.A. Spragg, Appl. Spec. **40**, 715 (1986).

6) B. Johnson, B. Rebenstorf, R. Larsson, and M. Primet, Appl. Spec. **40**, 798 (1986).

7) R.T. Rewick and R.G. Messerschmidt, Appl. Spec. **45**, 297 (1991)

8) S.V. Compton, D.A.C. Compton, and R.G. Messerschmidt, Spectroscopy, p. 35, July/August 1991.

PACKING LIST

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

Emission Accessory	Quantity	Part Number	Size/Type
Baseplate	1	09-00-01	
Mating Baseplate	1	09-00-01	
Standoffs	4		
Mirror Mounts	2	22224	
Relay Mirror	1	09-02-01	
Field Splitting Mirror	1	09-50-01	
Cell Rail	1	09-00-02	
Cell Mount	1	09-00-04	
Screws	2		8-32x1/2”
Instruction Manual	1		
Ball Driver	1		3/32”
Ball Driver	1		7/64”
Heated Cell			
Slide Plate	1	09-06-09	
Spacers	3	09-06-08	
Back Insulator	1	09-06-05	
Front Insulator	1	09-06-06	
Radial Insulator	1	09-06-07	
Cell Body	1	09-06-04	
Connector	1		
Heater Cartridges	3	E1A52-HR34	
Thermocouple	1	KMTSS-062G	Type K
Screws	3		6-32x1/2”
Blackbody Reference Disk,Graphite	1	09-06-10B	
Blackbody Ref Disk, Hi-Temp Black Painted Disk	1		
Gold-coated Disk	1		
Pyrex Disk	1		
Thin Spacer Disks	2	09-06-01	
Medium Spacer Disks	1	09-06-02	
Thick Spacer Disks	1	09-06-03	
Temperature Controller (Optional)			
Controller	1	25B600W	
Instruction Manual	1		

Instructions for tuning the CIC Photonics, Inc. PACESETTER temperature controller for the CIC Photonics, Inc. Insider Emission accessory

Be sure the Pacesetter is plugged in and the power switch is in the "on" position. Press and hold both arrows simultaneously. Press button in lower left corner. You may now review the operating parameters. Advance through the parameters by repeatedly pressing BLUE button in lower left corner.

Parameter	Value	Description
SEN	TC	Thermocouple
C-F	C	Degrees Celsius
SP.LO	0.0	Low temp set
SP.HI	400.0	Hi temp limit
Ot1	Heat	Heating action
rP	on	ramping on
rt	200	ramping rate, degrees per hour
PL1	100	power limiting default value
dSP	nor	normal display

Press middle button (infinity sign) to return to RUN mode.

Autotune Feature

Press button in lower left corner 3 times to access Autotune Mode. Note display in lower display "AUT." Push "UP" or Increment button to enable Autotune. Press Infinity button to enter RUN mode with Autotune. The Controller will now run to SETPOINT and derive the optimal P.I.D. values. The Autotune mode will self-terminate.

Technical Papers