

Installation and User's Guide

DIFFUSE

REFLECTANCE

ACCESSORY

December 1992

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Address Comments to:

PIKE Technologies, Inc.
2919 Commerce Park Drive
Madison, WI 53719

Introduction

The Pike Technologies Diffuse Reflection accessory is designed for use in FTIR spectrometers. The design utilizes four beam steering flat mirrors and a high collection angle ellipsoidal reflector to collect the maximum amount of energy from the sample. The PIKE Technologies Diffuse Reflection accessory has been created for high energy throughput and ease of use.

Two detented sampling positions are available in the accessory which are mounted on a precision slide. Sample loading is performed by pulling the specimen slide forward so that the sample cups are out of the accessory. The sample cups are removable for filling. Once the sample is in the analysis position the height of the sample may be adjusted by the use of a micrometer adjusted ball bearing slide.

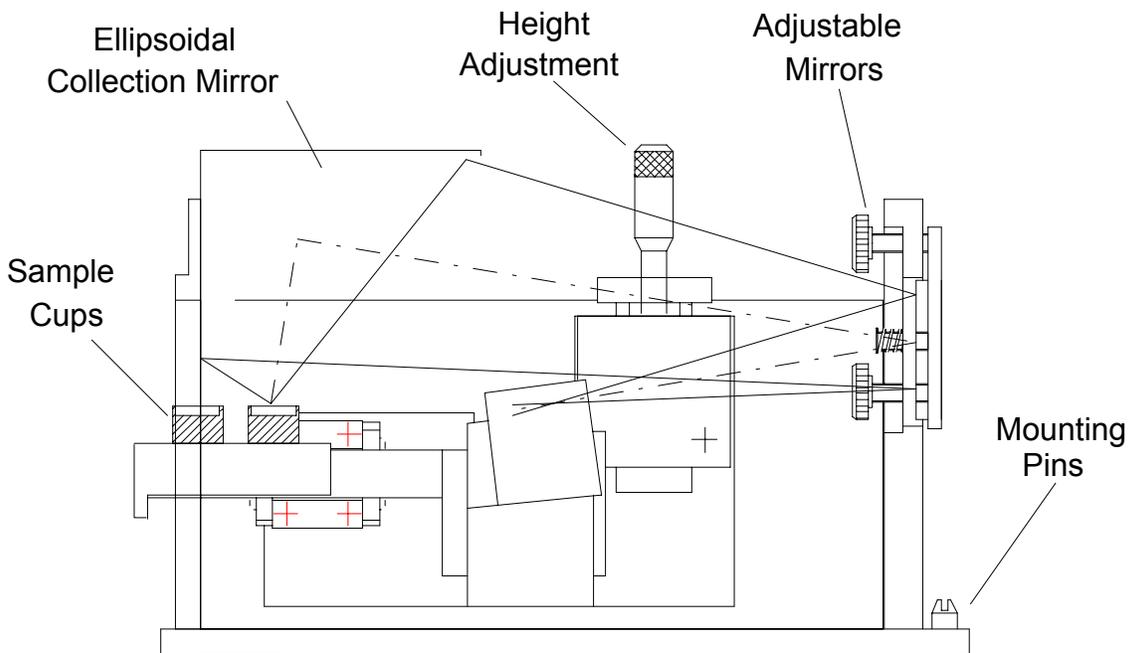


Figure 1. Diffuse Reflectance Accessory

Installing the Diffuse Reflectance Accessory

MIDAC INSTALLATION

To install the accessory in the Midac Spectrometer, two mounting pins are provided. These are located in "parking holes" at the rear of the accessory. To install the accessory please perform the following tasks:

- 1) Open the sample compartment of the instrument.
- 2) Remove the two mounting pins from the rear of the accessory.
- 3) Screw the mounting pins into the spectrometer sample compartment as shown in Figure 2.

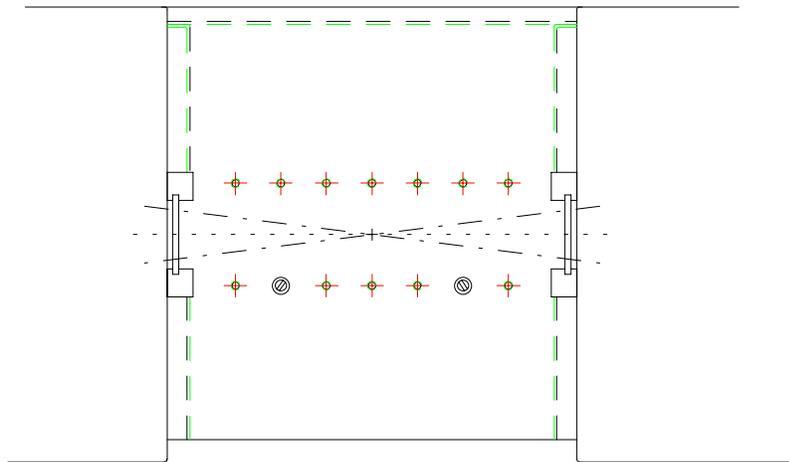


Figure 2. Midac Sample Compartment

- 4) Place the accessory over the two mounting pins.
- 5) If desired, the accessory may be secured further with the 10-32 screw provided.
- 6) The accessory is now ready for use.

When the accessory is removed from the spectrometer, remember to remove the two mounting pins and screw them into the parking holes provided in the rear of the accessory.

NICOLET INSTALLATION

To install the accessory in the Midac Spectrometer, two mounting pins are provided. These are located in "parking holes" at the rear of the accessory. To install the accessory please perform the following tasks:

- 1) Open the sample compartment of the instrument.
- 2) Remove the two mounting pins from the rear of the accessory.
- 3) Screw the mounting pins into the spectrometer sample compartment as shown in Figure 2.
- 4) Place the accessory over the two mounting pins.
- 5) If desired, the accessory may be secured further with the 10-32 screw provided.
- 6) The accessory is now ready for use.

When the accessory is removed from the spectrometer, remember to remove the two mounting pins and screw them into the parking holes provided in the rear of the accessory.

Alignment

The accessory has been aligned and tested to ensure that it performs to specification. There are two mirrors on the accessory that may be aligned to maximize performance in your spectrometer. In order to optimize the signal throughput in your instrument only small adjustments of these two mirrors will be required.

The alignment procedure is as follows

- 1) Compare your accessory with the drawing below and locate the left and right adjustable mirrors.
- 2) Peak up the energy of your spectrometer by adjusting the interferometer. This should be performed by following the manufacturers instructions.
- 3) Mount the accessory into the sample compartment.
- 4) Place the alignment mirror in one of the sample positions and move the mirror into the appropriate position in the beam.

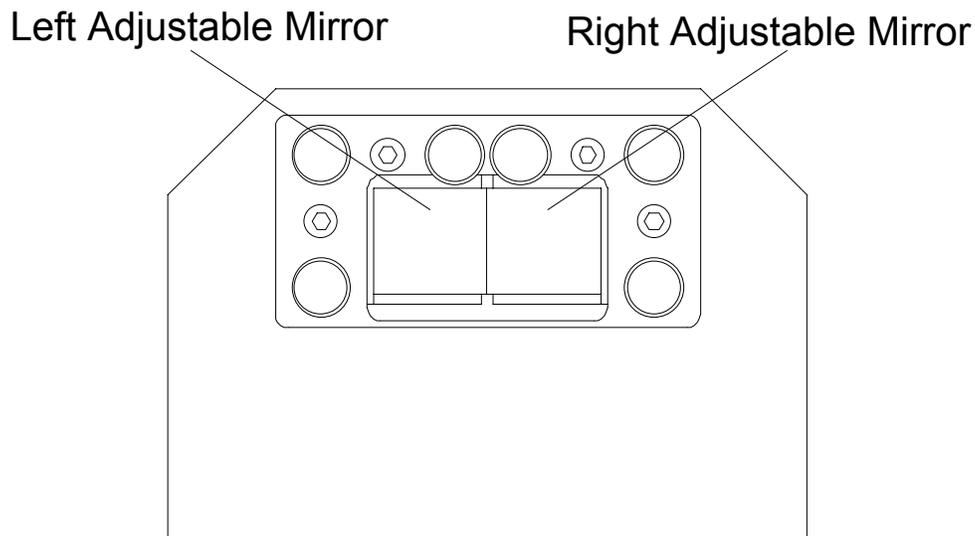


Figure 3. Accessory Back Plate

Alignment (Continued)

- 5) In the alignment mode, check the signal throughput of the spectrometer with the accessory in place.
- 6) Adjust the vertical height of the alignment mirror using the micrometer adjustment to maximize the infrared signal.
- 7) Adjust the tilt and rotation of the left adjustable flat mirror to maximize the infrared signal.
- 8) Adjust vertical height of the alignment mirror using the micrometer adjustment to maximize the infrared signal.
- 9) Adjust the tilt and rotation of the right adjustable flat mirror to maximize the infrared signal.
- 10) Adjust vertical height of the alignment mirror using the micrometer adjustment to maximize the infrared signal.
- 11) Repeat steps 7 through 10 until there is no further increase in infrared signal.

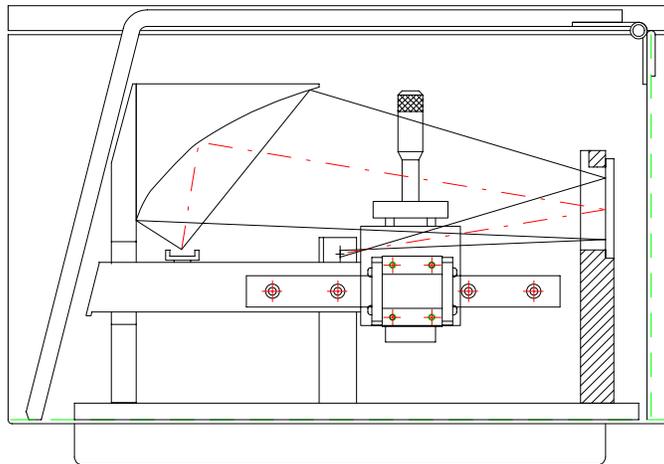


Figure 4, Diffuse Accessory in Sample Compartment

Sampling

Two macro cups and two micro cups are provided for specimen samples. The macro cups are 11mm in diameter and spectra using samples prepared in these cups will have the highest signal to noise ratio since the cups are larger than the beam size. The two micro cups are used when only a limited amount of the sample is available.

The sample cups should be filled carefully to make sure that the powder is level with the surface of the cup and is smooth. If the sample is suspended in KBr matrix then a suitable background may be obtained by using neat KBr powder as a sample.

Diffuse reflectance spectra should be displayed with the ordinate axis set to Kubelka-Munk units. The reflectance of the sample is related to concentration by the Kubelka-Munk equation:

$$f(R)=(1-R)^2/2R$$
$$=2.3aC/s$$

where \underline{s} is the scattering coefficient and \underline{a} is the absorptivity. The scattering coefficient depends on both the particle size and the degree of sample packing, so that the Kubelka-Munk function can only be used for quantitative work when the sample preparation method is accurately controlled. For good diffuse reflectors, the spectra displayed in Kubelka-Munk units is analogous to absorbance plots of transmission spectra.

Your PIKE Technologies Diffuse Reflection accessory should contain the following articles:

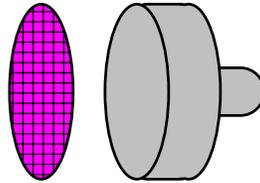
- Diffuse Reflection Accessory
- Manual
- One alignment mirror
- Two large sample cups
- Two small sample cups
- Two spatulas
- Mortar and pestle
- Vial of KBr powder
- Preparation Block
- Two Filling Funnels

Abrasion Sampler Kit (ASK)

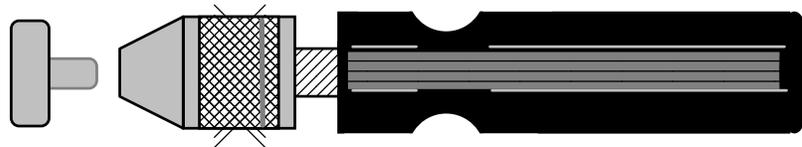
The Abrasion Sampler Kit provides a quick and reliable method for obtaining IR samples from solids that are normally difficult to sample. Any solid sample that can be transferred by abrasion is now available for processing. One hundred adhesive backed abrasion disks are included in each kit.

Obtaining a Sample

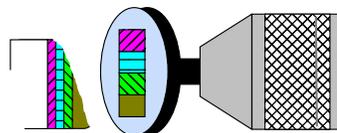
- 1) Select one of the .500 inch diameter disks and one of the sample holder.



- 2) Remove the protective coating on the rear of the disk and center the disk on the holder.
- 3) Open the jaws of the Pin Vise by turning the chuck clockwise. Insert the sample holder into the chuck and tighten the jaws by turning the chuck counter-clockwise until snug.



- 4) Samples can now be obtained by grasping the pin vise and rubbing the abrasive disk on the material you wish to investigate.



- 5) Remove the sample holder from the pin vise and slip the shaft of the holder into the accessory for diffuse reflection analysis.

Sample Analysis

The PIKE Diffuse Reflectance Accessory has two aligned positions to accommodate two separate samples. When using the Abrasion Sampling Kit it will become very useful to have a blank disk and sample holder placed in the rear specimen hole. This will conveniently allow a background spectra to be taken for the blank disk.

The background can now be collected using the blank disk. The resulting spectrum will contain the bands from the abrasive and can be ratioed out of the corresponding sample spectra.

Moving to the other sample positive, collect a spectrum of the sample and abrasive.

By ratioing this spectrum to the background spectrum the sample spectrum may be obtained.

The Abrasion Sampler Kit contains:

- Two Sample Holders
- One Pin Vise
- 100 Self-Adhesive Abrasive Disks