Installation and User Guide

PIKE TECHNOLOGIES
Spectroscopic Creativity

EasiDiff

Diffuse Reflectance Accessory

For FTIR Spectrometers

Address Comments to:

PIKE Technologies, Inc.
2901 Commerce Park Drive
Madison, WI 53719

Phone	(608) 274-2721
Fax	(608) 274-0103
E-mail	Sales@piketech.com
Web Site	www.piketech.com

Part Number **350-042003**

CONTENTS

INTRODUCTION

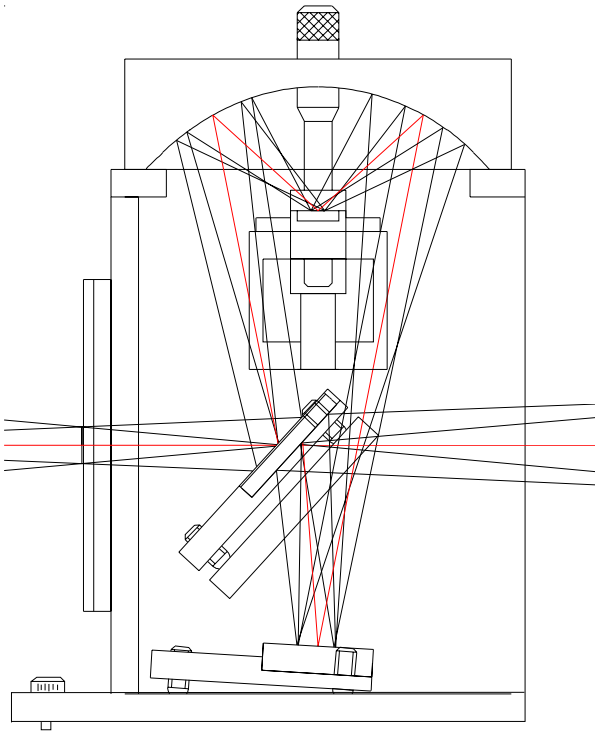
The EasiDiff™ is an easy to use, inexpensive, diffuse reflectance accessory. The design employs a high efficiency fixed ellipsoidal reflector to collect the maximum amount of diffusely reflected energy from the sample. Alignment is performed using a precision micrometer for quick, simple and repeatable adjustment.

With this accessory, you will be able to quickly obtain the infrared spectra of a wide range of powders.

ACCESSORY DESCRIPTION

The infrared beam enters the accessory, strikes the double mirror assembly and is sent to the ellipsoidal reflector. The infrared beam is then focused on the sample.

The reflected energy from the sample is collected by the ellipsoidal reflector, which then passes to the lower mirror and on to the back side of the double mirror to the instrument detector.



EasiDiff Optical Path

Two sample positions are provided, one for a neat sample of KBr for use as a background and the other to hold the sample to be measured.

The KBr powder used for sampling is corrosive. For this reason, the front panel of the accessory is removable for cleaning any spilled powder from the mirrors. In order to remove the front cover, the screws holding the cover in place are fitted with thumb turn knobs.

Normally, infrared accessories are built with mirrors that are aluminized, and have no overcoating. This bare aluminum surface is very soft and it is difficult to clean the mirrors without producing scratches. The alignment mirror in this accessory is overcoated with silicon monoxide, to provide a hard durable layer. This allows the mirrors to be cleaned if a KBr spill occurs. Cleaning should be performed using the small brush provided. This silicon monoxide film produces a small absorption band in the infrared at about 1200 wavenumbers. This band will not affect the spectral performance of the accessory.

UNPACKING YOUR ACCESSORY

In order for you to quickly verify receipt of your accessory, we have included a packing list. Please inspect the package carefully. Call PIKE immediately if any discrepancies are found. A spectrum of the accessory throughput is included. This data was collected on a brand of spectrometer the accessory is configured for, as test data prior to shipping. However, due to differences in spectrometers, spectra collected with this accessory on our spectrometer during the alignment and test procedure may differ from the spectra that you collect. If you have any questions regarding this spectrum, please contact PIKE.

Packing List

The EasiDiff accessory kit contains the following

EasiDiff Accessory
Quantity 1
042-10XX

Sample Cup Holder
Quantity 1
042-3030

Alignment Mirror
Quantity 1
042-0027

Small Sample Cup
Quantity 2
042-2010

Large Sample Cup
Quantity 2
042-2020

KBr Powder, 100 g
Quantity 1
160-8010

Funnel Base
Quantity 1
042-0024

Spoon Spatula, Quantity 1
042-0010
Flat Spatula, Quantity 1
042-0011

Mortar and Pestle
Quantity 1
161-5035

Funnel
Quantity 1
042-0022

Camel Hair Brush
Quantity 1
162-4015

INSTALLATION

In order to align your accessory, perform the following steps.

1. Ensure that the spectrometer is correctly aligned. Refer to your spectrometer manual to perform this operation.

2. Place the accessory into the sample compartment of the instrument. Fix down the accessory with the captive baseplate screw.

3. Remove the alignment mirror from the sampling kit. This mirror is provided with a protective polymer coating, which should be removed prior to use. Instructions for removal are provided in the sampling kit.

4. Insert the mirror into the front position in the sample holder and place the holder into the accessory. Slide the holder all the way into the accessory.

5. Place the instrument into alignment mode to monitor the infrared signal level.

6. Slowly turn the micrometer adjustment on the top of the accessory to maximize the size of the signal.

7. The accessory is now aligned.

PERFORMANCE VERIFICATION

Perform the following steps:

- With the accessory removed from the sample compartment of the instrument, collect a background spectrum

- Place the EasiDiff accessory into the sample compartment.

- Place the alignment mirror into the front position of the sample holder and place the holder into the accessory. Slide the holder all the way into the accessory.

- Slowly turn the micrometer adjustment on the top of the accessory to maximize the size of the signal.

- Collect a transmission spectrum using the same collection parameters as used to collect the background spectrum.

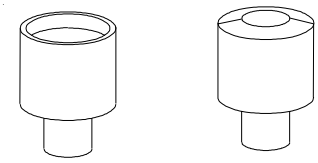
The minimum transmission value should be 30% at 5000 cm^{-1} against the alignment mirror. If your accessory does not meet this minimum value, contact PIKE Technologies. On contacting PIKE, please have the serial number of the accessory. This number is found on the rear of the accessory**.**

SAMPLE PREPARATION WITH EASIDIFF

The EasiDiff is provided with a sample preparation system to aid in the accurate and repeatable preparation of samples. With this system, the sample cups may be filled precisely with minimum sample spillage and inconvenience.

SAMPLE CUPS

Two small and two large sample cups are provided. The large sample cup has a 10 mm diameter by 2.3 mm deep rebate to hold the sample while the small cups are 4.7 mm diameter by 1.6 mm deep.



Large and Small Sample Cups

PREPARING THE SAMPLE

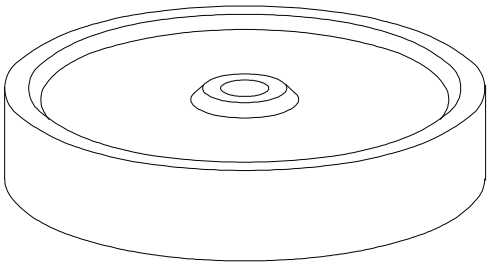
The sample to be analyzed must be diluted in a transmitting matrix. Place the sample in the mortar and grind finely using the pestle provided. Add KBr powder to the sample. With the pestle, mix the sample with the KBr powder so that the sample particles are small and evenly dispersed. The sample should be diluted to 1 to 5%. The optimum amount of dilution will depend on the sample to be analyzed but the percent transmission of the strongest band in the resulting spectrum should ideally be in the range from 10 to 50%. If possible start with a 5% dilution and if the resulting bands are too intense then redilute the sample.

CAUTION

KBr readily absorbs moisture. To reduce the effect of moisture in the sample spectrum the following guidelines should be used. Always keep the cap on the KBr bottle when not in use. If possible store the KBr in a dry place. Fill the reference cup (neat KBr) and the sample cup (sample diluted in KBr) at the same time. Any water that has been absorbed in the reference and sample will tend to ratio out in the final spectrum.

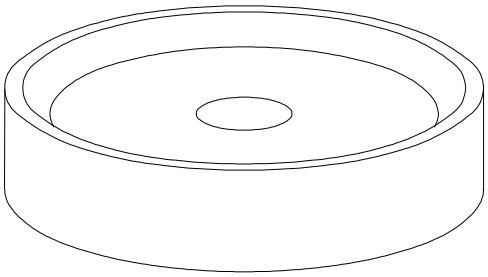
LOADING THE SAMPLE

Two sets of parts are provided. Identify the parts with reference to the drawings below.



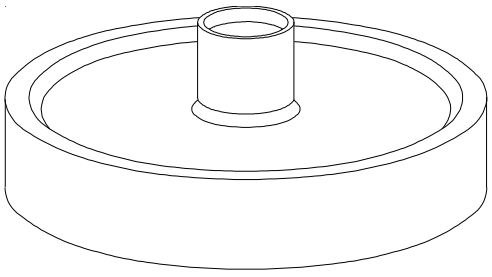
EasiDiff Sample Preparation Base

The base has a 0.25 inch hole in the center and a raised lip around this hole.

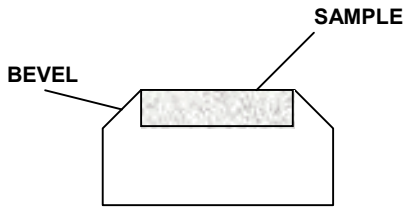


EasiDiff Sample Funnel

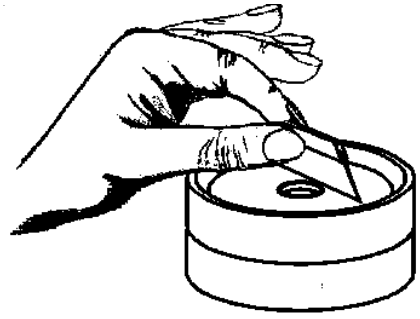
The sample funnel has a 0.5 inch hole in its center. With the sample prepared in the mortar, place the sample cup in the sample preparation base**.**



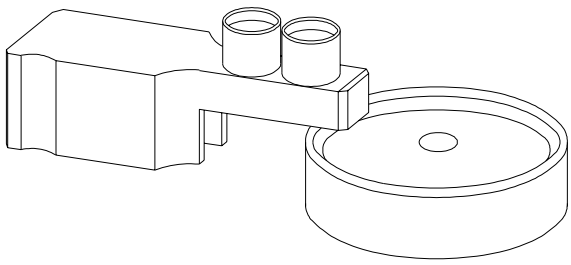
Place the funnel over the sample cup. The sample cup will appear to be slightly proud of the surface of the cup. The sample cups have a small bevel on the top surface. This enables a high quality sample surface to be prepared.



Pour the sample from the mortar into the sample funnel. The cup may now be filled using the spatula and plastic razor blade provided. With practice, a perfect repeatable sample surface may be prepared.



When the sample cup is filled, remove the funnel and place the sample cup into the cup holder. The cup holder may be placed on the preparation base as shown in the drawing below to prevent the holder from tipping forwards.



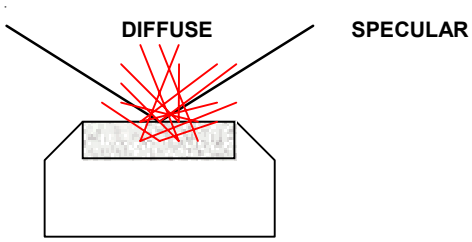
USING YOUR ACCESSORY

With the two sample cups filled and inserted into the sample holder, place the sample holder into the accessory. By sliding the sample holder all the way in, the rear position is brought into the beam. By sliding the sample holder all the way out, until the sample holder strikes the dowel pin stop, the front position of the sample holder is brought into the beam.

Note that both sample positions must use the same sized cups. There is a height difference between the two types of cups and in changing from one size of cup to the other, a focus adjustment of the micrometer will be required.

Slide the sample holder to bring the neat KBr sample into the beam. Adjust the micrometer screw to maximize the signal throughput through the accessory. Note that this will not be the same position as the alignment mirror, since the IR beam penetrates into the sample a short distance.

Once the signal has been maximized, collect a background spectrum of the neat KBr sample. Move the sample holder to the other position and collect a spectrum of the sample. The ratio of these two spectra will produce a spectrum of the sample. You may wish to covert your spectrum to an absorbance-like spectrum by performing a Kubelka-Munk transform on the data.



The optics of a diffuse reflectance accessory are designed to do two things:

- Focus all of the infrared energy from the spectrometer onto the sample.
- Collect as much diffusely reflected energy from the sample as possible.

This energy is scattered into a complete hemisphere, and it is important that the optics of the accessory collect this energy efficiently and direct it to the instrument detector. An important point in the use of a diffuse reflectance accessory is sample preparation. The sample is usually ground and mixed with a material such as potassium bromide, which acts as an infrared transmitting matrix. The sample is diluted in this matrix to give a 1 to 5 percent mixture. In this way the infrared beam penetrates into the sample cup and maximizes the detected signal. The depth of the sample that is required is governed by the amount of scattering in the sample. The minimum depth of sample required be about 1.5mm, and this is known as the “infinite depth” of the sample.
In order to produce a diffuse reflectance spectrum, a background spectrum must first be collected. The sample used for this background spectrum is the pure matrix material (i.e. KBr). The background sample is placed in one position on the sample holder. The prepared sample to be analyzed is placed in the other sample position. The resulting spectrum is produced by ratioing the sample spectrum to the background spectrum.

Samples may also be analyzed without dilution. For samples that are not powders, the sample may be abraded with a piece of silicon carbide. The Abrasive Sampling Kit (ASK) is available from PIKE Technologies. For this technique a background spectrum is taken of the clean silicon carbide paper prior to abrading the sample.

The spectra that are obtained by the diffuse reflection technique appear different from standard transmission spectra. The peak intensities at high wavenumbers are weak and the peak line shapes are rounded. The spectra can be transformed into Kubelka Munk units, compensating for these differences. The reflectance of a sample is related to concentration by the Kubelka-Munk equation:

$$\frac{(1-R)^2}{2R}$$

$$=2.3\ ac/s$$

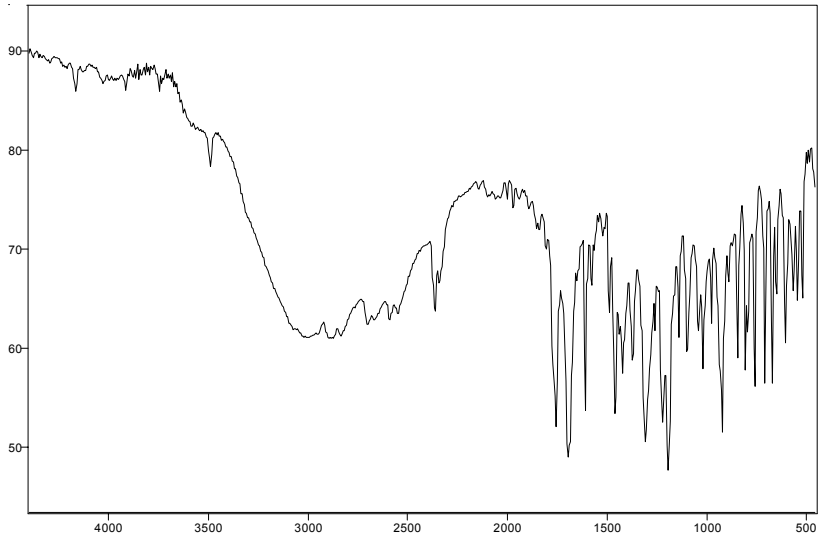
where **s** is the scattering coefficient and **a** is the absorptivity. The scattering coefficient depends on both the particle size and sample packing, which explains why sample preparation is important for accurate results. In order to perform a Kubelka Munk Transform, select Kubelka Munk from the menu of your FTIR software. There are no parameters to set for this routine.

THEORY

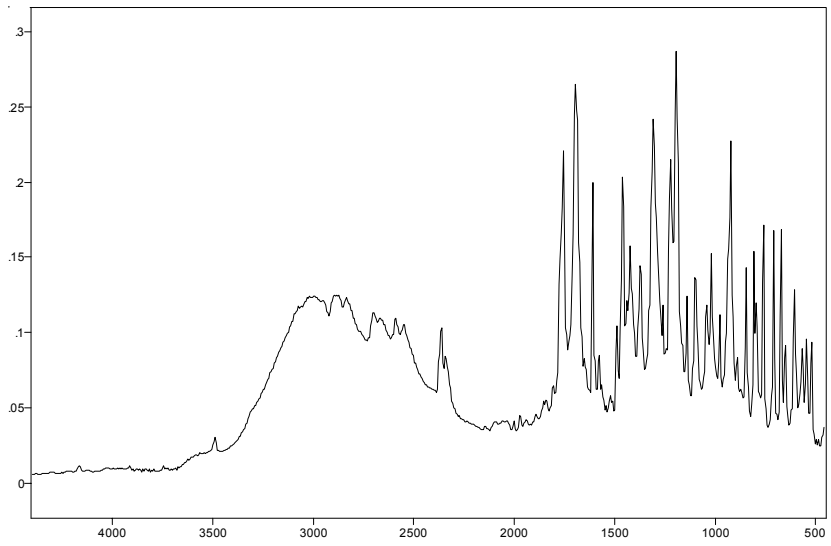
Diffuse reflectance spectroscopy is a widely used technique in FT-IR analysis. The primary application is in the analysis of powders, although it has been used for investigating rough surfaces and even fibers.

In diffuse reflectance spectroscopy, the sample is mixed with an infrared transmitting powder. When the beam strikes the sample, three things happen.

- Some of the beam is specularly reflected. The amount of energy reflected is governed by the Fresnel equations, which state that the reflectivity of a sample is dependent on the refractive index of the sample. At an absorption band, the refractive index changes widely, an effect known as anomalous dispersion, and this gives rise to a reflection spectrum.
- Some of the beam is absorbed in the sample and is lost.
- The remainder of the beam is transmitted into the sample. Only that part of the beam that is scattered within the sample and returned to the surface may be collected. This energy is diffusely reflected energy and is collected by the accessory energy and is collected by the accessory.



Raw Transmittance Data, Aspirin



Kubelka-Munk Transformed Data, Aspirin

PRECAUTIONS

MIRRORS

In order to provide the maximum transmission in the infrared, with the minimum spectral interference, the flat mirrors and the reflector mirrors used in this device are uncoated (bare) aluminum. Since the coatings are soft, care must be taken to avoid damage. Normally, these mirrors will not need cleaning, since they are contained within the housing of the accessory. If they do need cleaning, they may be gently wiped with a lint free, abrasive free cloth, such as lens tissue, or with a camel hair brush. Under no circumstances must the mirrors be rubbed with paper products such as “Kleenex” since this will produce scratching of the mirror coating.

MAINTENANCE PARTS LIST

The following spare parts are available

Description	Part Number
Sample Cup Holder	042-3030
KBr Powder	160-8010
Mortar and Pestle	161-5035
Funnel Base	042-0024
Funnel	042-0022
Large Sample Cup (quantity 2)	042-2020
Small Sample Cup (quantity 2)	042-2010
Alignment Mirror	042-0027
Spoon Spatula	042-0010
Flat Spatula	042-0011
Camel Hair Brush	162-4015
Razor Blades (quantity 5)	410 0013