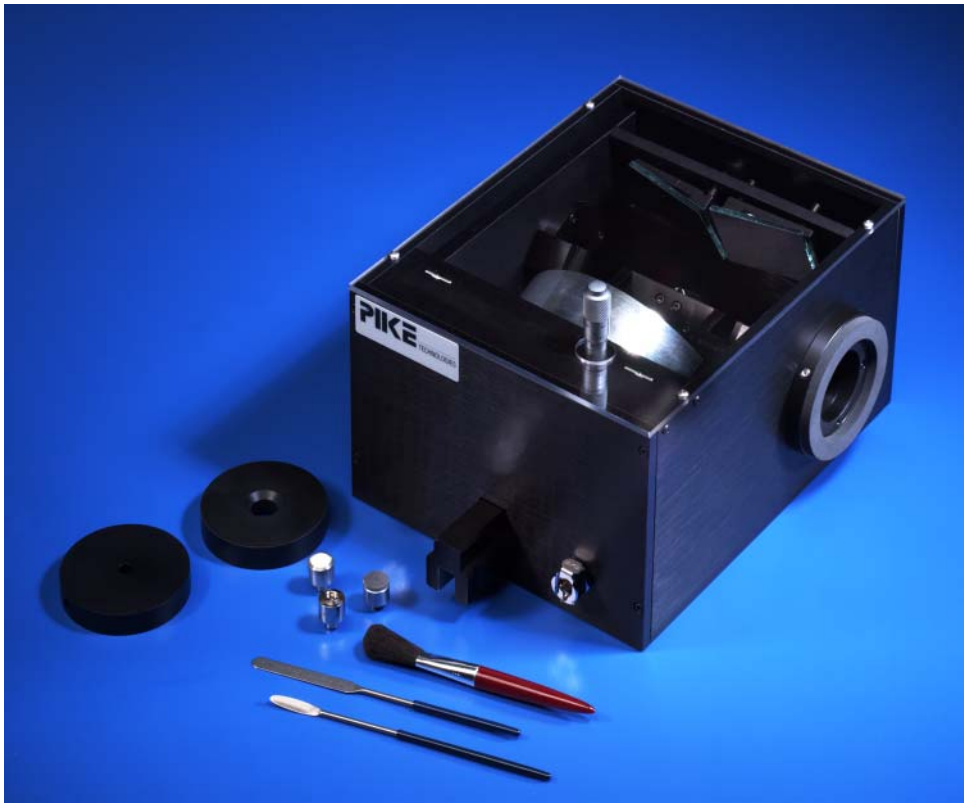
Installation and User Guide

PIKE TECHNOLOGIES
Spectroscopic Creativity



DiffusIR™

Diffuse Reflectance Accessory

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INTRODUCTION

The DiffusIR™ is an easy to use, diffuse reflectance accessory. The accessory features downward-looking optics. Its advantages include high throughput, constant optical path with all mirrors positioned above the sampling area. This is a significant design feature which protects all the optical components of the accessory against spills and corrosion often caused by KBr/sample mixes. It also makes cleanup much easier, as accidental spills the can be simply removed out of the bottom of the accessory.

The DiffusIR is equipped with a cover and purge tubes for the elimination of CO_2 and water interferences from mid-IR spectra. The accessory's optical design is optimized to efficiently collect diffuse radiation generated by the sample and minimize the effects of the specular radiation component. The heart of the DiffusIR is a large, monolithic ellipsoidal reflector.

Sample introduction is performed using an integral precision slide. The sample is repeatably brought to the optimum position for analysis using a micrometer screw. The DiffusIR comes equipped with sample preparation tools and it can be used with an optional abrasion kit.

INSTALLATION

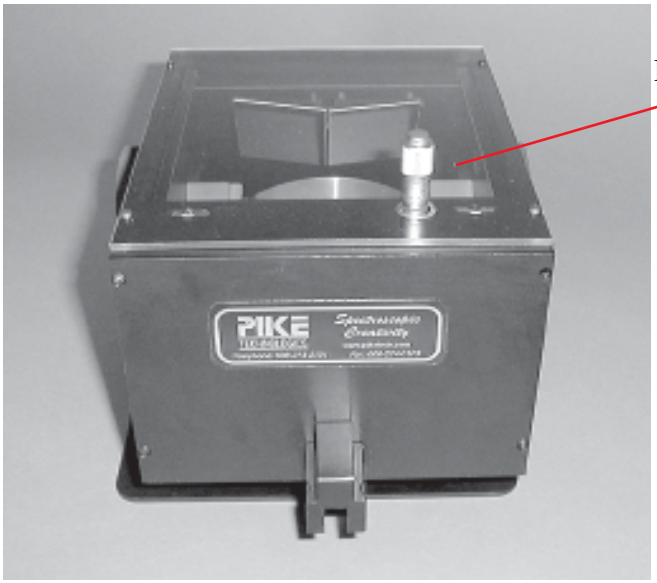
Place the accessory in the sample compartment of the spectrometer, and secure it to the baseplate with a captive thumbscrew.

ACCESSORY ADJUSTMENT

The accessory has been aligned and tested to ensure that it performs to specifications and no additional alignment is necessary. In order to optimize the signal throughput in your instrument only small adjustments will be required.

The alignment procedure is as follows

1. Place the alignment mirror in one of the sample positions and move the mirror into the appropriate position in the beam.
2. In the alignment mode, check the signal throughput of the spectrometer with the accessory in place.
3. Adjust the vertical height of the mirror using the Micrometer screw on top of the accessory.
4. Repeat step 3 until there is no further increase in infrared signal.

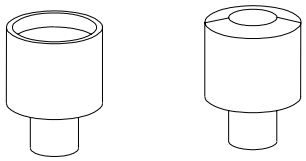


SAMPLE PREPARATION

The DiffusIR 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

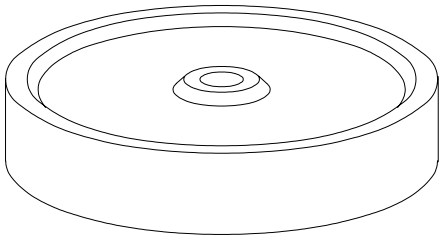
The sample to be analyzed must be diluted in a transmitting matrix. Place the sample in the mortar and grind finely using the pestle provided. Add KBr powder to the sample. With the pestle, mix the sample with the KBr powder so that the sample particles are small and evenly dispersed. The sample should 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, re-dilute the sample.

LOADING THE SAMPLE

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

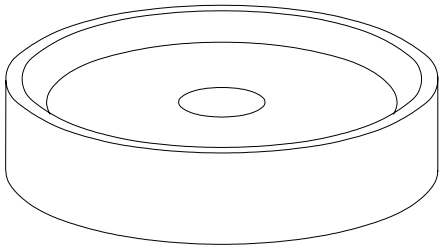
DiffusIR Sample Preparation Base

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



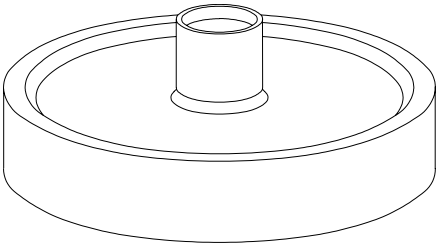
DiffusIR Sample Funnel

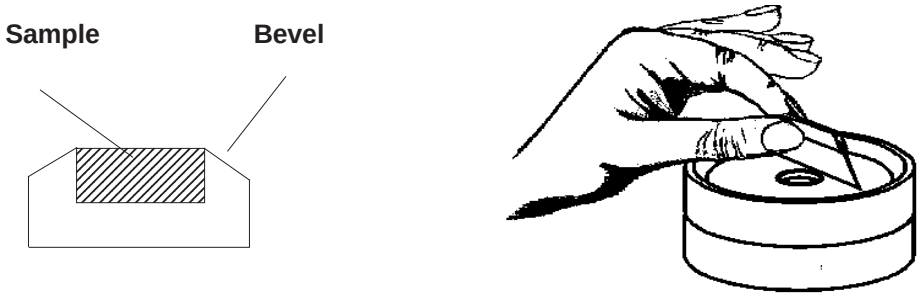
The sample funnel has a 0.5 inch hole in its center.



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 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.

COLLECTING SPECTRA

With the two sample cups filled, place them into the holder. 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 type 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 thumb wheel 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.

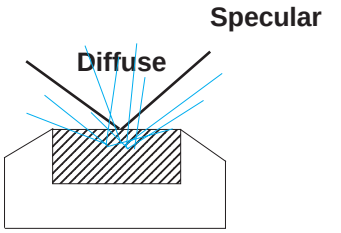
Once the signal has been maximized, collect a background spectrum of the neat KBr sample. Move the sample holder to the other position and collect a spectrum of the sample. The ratio of these two spectra will produce a spectrum of the sample.

THEORY

Diffuse reflectance spectroscopy is a widely used technique in FTIR 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.



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 is about 1.5 mm, 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.

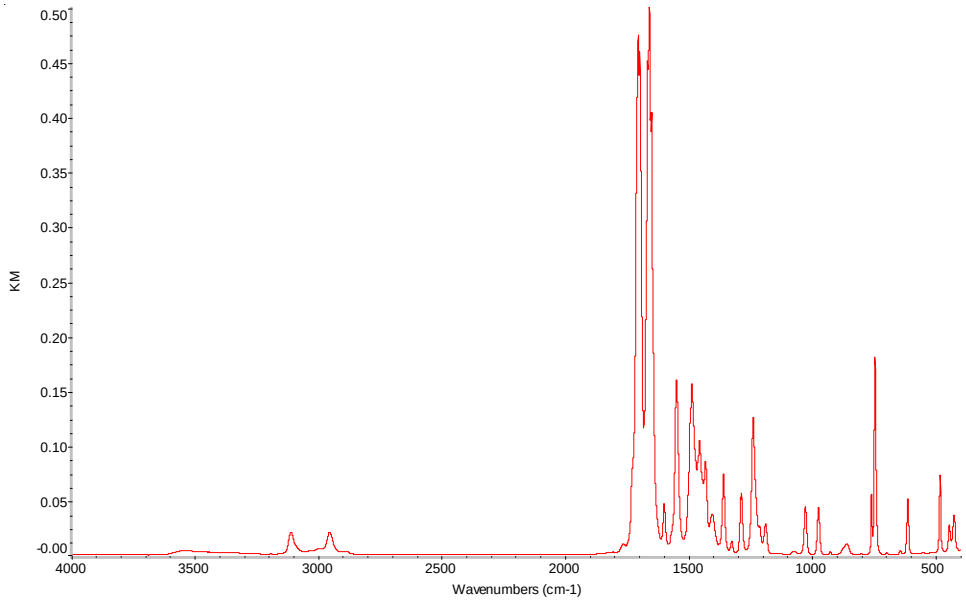
The spectra that are obtained by the diffuse reflection technique appear different from standard transmission spectra. The peak intensities at high wavenumbers are weak and the peak line shapes are rounded. The spectra can be transformed into Kubelka Munk units, compensating for these differences. The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

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

$$= 2.3\ ac/s$$

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. Most commercial spectrometers are equipped with software to perform this operation.

Raw Diffuse Reflectance Spectrum, Caffeine



Kubelka-Munk Transformed Spectrum, Caffeine

PRECAUTIONS

MIRRORS

In order to provide the maximum transmission in the infrared, with the minimum spectral interference, the mirrors used in this device are uncoated (bare) aluminum on glass substrate. 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.

PACKING LIST

The DiffusIR accessory kit contains the following:

DiffusIR Accessory	1
DiffusIR Sample Cup Holder	1
KBr Powder	1
Mortar	1
Pestle	1
Funnel Base	1
Funnel	1
Large Sample Cup	2
Small Sample Cup	2
DiffusIR Alignment Mirror	1
Spatula	2
Camel Hair Brush	1
Single Edge Razor Blades	5
User’s Guide	1