

DIFFUSE REFLECTANCE

Diffuse reflectance is a highly sensitive technique for the analysis of powdered and solid samples. Typically, a sample is ground with KBr into a fine powder and run without making pellets. Some samples can be run directly without dilution, especially if one is looking for minor components.

Diffuse reflectance sampling is ideally suited for automation and PIKE offers configurations for high-capacity sampling.

EasiDiff™
Diffuse reflectance accessory
Analyze a wide variety of solid and powder samples

DiffusIR™
Research-grade diffuse reflectance accessory
With environmental chambers for heating/cooling

UpIR™
Upward-looking diffuse reflectance
Out-of-compartment design for large sample analysis

AutoDiff™
Automated diffuse reflectance
Analyze multiple samples with minimal intervention

XY Autosampler
Transmission and reflection in microplate format
Ideal for speed and reproducibility

Sampling Kits
Sample collection, preparation and loading
For analysis of powders or solids

**THEORY AND
APPLICATIONS
INCLUDED**

EasiDiff – Workhorse Diffuse Reflectance Accessory



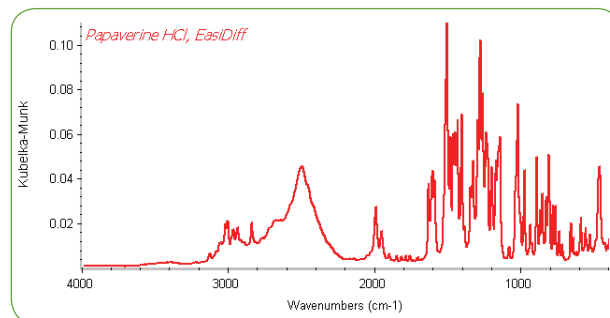
FEATURES

- Pre-aligned optical components for reproducible, high-quality data
- Micrometer-controlled sample positioning and focusing
- High-energy throughput providing nanogram sensitivity
- Precision slide for repeatable sample introduction and efficient collection of background and sample spectra
- Unique Sample Preparation and Loading Kit included

The PIKE Technologies EasiDiff is an economical, high-quality diffuse reflectance accessory designed to analyze a wide variety of solid samples. It is most often used in the analysis of pharmaceuticals, illicit drugs, inorganic solids and minerals, and powdered chemicals. The EasiDiff reduces the time required to produce an infrared spectrum compared to KBr pellet techniques. Typically, a small amount of sample (about 1%) is mixed with KBr powder and the spectrum is collected.

The EasiDiff employs an elegant, high-performance optical design for maximum energy throughput and ease of operation. Optical components critical to achieving this performance are permanently aligned. Focusing is achieved by bringing the sample (not the collection mirror) to the optimum position with a micrometer. A dual-position sample holder permits background and sample collection in a simple, two-step process.

A special version of the EasiDiff with gold-coated optics for NIR measurements is also available.



A spectrum of papaverine hydrochloride (1% in KBr powder) collected using the PIKE Technologies EasiDiff diffuse reflectance accessory.

ORDERING INFORMATION

PART NUMBER DESCRIPTION

042-10XX EasiDiff Accessory with Sample Preparation Kit
Includes 2 micro sample cups, 2 macro sample cups, EasiPrep Sample Preparation Kit, alignment mirror, 35-mm mortar with pestle and KBr powder (100 g)



042-50XX EasiDiff Accessory, NIR Version with Gold-Coated Optics
Includes 2 micro sample cups, 2 macro sample cups, EasiPrep Sample Preparation Kit, alignment mirror, 35-mm mortar with pestle and KBr powder (100 g)

Note: Replace **XX** with your spectrometer's Instrument Code. [Click for List >](#)

OPTION

PART NUMBER DESCRIPTION

042-3010 Abrasion Sampling Kit
Includes sample collector tool and stainless steel sample post, 25 diamond abrasive disks and 75 silicon carbide abrasive disks



Notes: The abrasion sampling kit is used to measure intractable solids. Disks are disposable. Ordering information for replacements may be found below.

REPLACEMENT PARTS AND SUPPLIES

PART NUMBER DESCRIPTION

042-2010	Micro Sample Cup, 6.0 mm diameter, 1.6 mm deep (2 ea.)
042-2020	Macro Sample Cup, 10 mm diameter, 2.3 mm deep (2 ea.)
042-2025	EasiDiff Sample Slide
160-8010	KBr Powder (100 g)
042-3020	Abrasion Disks, silicon carbide (100 ea.)
042-3025	Abrasion Disks, diamond (50 ea.)
042-3030	Sample Cup Holder and Base
042-3040	Sample Preparation Kit
042-3080	Alignment Mirror, aluminum
042-3082	Alignment Mirror, gold
042-3060	Flat Sample Post

Note: Please contact PIKE Technologies for items not described in this list.

DiffusIR – Research Grade Diffuse Reflectance Accessory



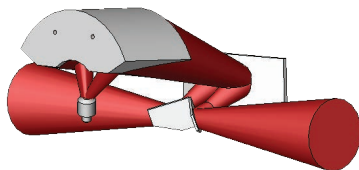
FEATURES

- Large, highly efficient collection optics for maximum sensitivity and detection limits
- Micrometer-controlled sample focus to optimize results for every sample
- Optional environmental chambers for heating, cooling, high-vacuum and high-pressure applications
- Quick release feature of environmental chambers for easy insertion and removal of sealed chambers
- Digital PC controller option for macro control of data collection at user specified temperatures or times
- Sealed and purgeable optical design to eliminate water vapor and carbon dioxide interference

The PIKE Technologies DiffusIR™ is a research-grade diffuse reflectance accessory with an efficient optical design accommodating the optional PIKE Technologies environmental chambers. These specialized chambers can be used to study thermodynamic properties of materials, to determine reaction mechanisms, to perform catalytic studies and much more.

The heart of the DiffusIR is a unique monolithic ellipsoidal reflector permanently fixed in place – eliminating the need for repositioning the focus optics for sample placement. The DiffusIR optical design is optimized to efficiently collect diffuse radiation generated from the sample and minimize the effects of the specular radiation component.

Optical geometry of the DiffusIR accessory



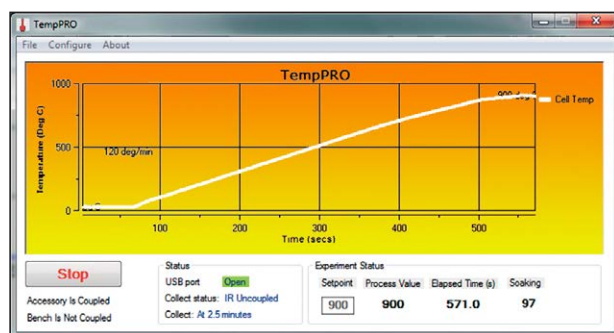
With the DiffusIR, sample introduction is performed using an integral 2-position slide – enabling background and sample spectra to be collected without loss of purge. The sample height can be optimized by using the micrometer sample focusing adjustment. In this manner the sensitivity of the accessory is maximized without sacrificing precision. The DiffusIR comes equipped with a Sample Preparation and Loading Kit and a Sample Abrasion Kit for the analysis of intractable samples. The DiffusIR optics are enclosed and equipped with purge tubes for the elimination of atmospheric interferences.

Advanced temperature studies of materials in controlled environments can be done using the PIKE environmental chambers. Chambers for the DiffusIR can be operated at temperatures ranging from -150 to 1000 °C and at pressures up to 1500 psi. The optional chambers are easily inserted into the DiffusIR and secured using push-lock pins.

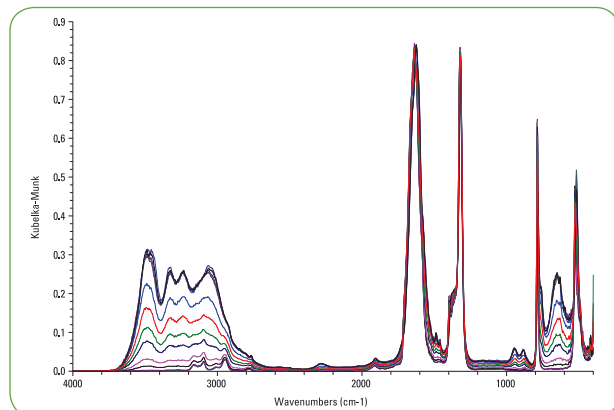


Coupling the environmental chambers with the PIKE PC-Controlled Temperature Module and TempPRO™ software provides the ability to graphically set up the experiment with up to 20 ramps and initiate data collection at specified time or temperature intervals when used with most FTIR instruments.

A special version of the DiffusIR with gold-coated optics is available for maximum mid-IR performance and for NIR diffuse reflectance sampling. The DiffusIR and its options are compatible with most FTIR spectrometers.



PIKE Technologies TempPRO software provides a graphical interface for temperature control and kinetic measurements.



Thermal transformation of hydrated inorganic compound measured using the DiffusIR with environmental chamber. Spectra automatically collected between 80 and 160 °C at 5° increments using PIKE TempPRO software.



Liquid nitrogen cooled system and temperature control module

ORDERING INFORMATION

DIFFUSIR ACCESSORY (must select one)

PART NUMBER	DESCRIPTION
041-10XX	DiffusIR Accessory <i>Includes Sample Preparation Kit with 2 micro and 2 macro sample cups, sample loading tools, Abrasion Sampling Kit, SiC and diamond sampling disks, alignment mirror, 35-mm mortar with pestle and KBr powder (100 g)</i>
041-60XX	DiffusIR Accessory with Gold-Coated Optics <i>Includes Sample Preparation Kit with 2 micro and 2 macro sample cups, sample loading tools, Abrasion Sampling Kit, SiC and diamond sampling disks, alignment mirror, 35-mm mortar with pestle and KBr powder (100 g)</i>

Note: Replace XX with your spectrometer's Instrument Code. [Click for List >](#)

DIFFUSIR OPTIONS

PART NUMBER	DESCRIPTION
162-4150	DiffusIR Environmental Chamber, HTV, ambient to 500 °C
162-4200	DiffusIR Environmental Chamber, HTV, ambient to 1000 °C
162-4180	High-Pressure Adapter Dome for Chambers, HTV
162-4140	DiffusIR Environmental Chamber, LTV, -150 to 500 °C

Notes: HTV and LTV chambers require the selection of a temperature control module. DiffusIR Chambers include front plate accommodating environmental chamber (easily changeable with standard DiffusIR front plate), Pin-Loc chamber insertion for easy sample exchange, KBr window, ceramic sampling cups compatible with vacuum and reaction formats, ports and 2 shut-off valves for vacuum operation and ports for connection of water cooling. The 500 °C and 1000 °C HTV chambers may be fitted with the high-pressure adapter and are easily switchable from standard vacuum to high-pressure operation. The LTV chamber is not compatible with simultaneous pressurization and low temperature operation. Operation of the LTV at sub-ambient temperatures requires part number 162-4160 Liquid Nitrogen-Cooled System and Temperature Control Module and rotary pump for vacuum insulation. All chambers require a liquid circulator to reduce heat transfer to the outer housing and to preserve the life of the chamber heaters.

TEMPERATURE CONTROL MODULES

PART NUMBER	DESCRIPTION
076-2450	PC Controlled Temperature Module, HTV Chambers <i>Includes Digital Temperature Selection and TempPRO software</i>
076-2250	Digital Temperature Control Module, HTV Chambers
162-4160	Liquid Nitrogen-Cooled System and Temperature Control Module DiffusIR Environmental Chamber, LTV, -150 to 500 °C

Notes: PC Controlled Temperature Module with TempPRO software provides a graphical user interface for setting experiment parameters and data collection. Please contact PIKE for PC compatibility. The Temperature Control Modules for the HTV and LTV chambers are not interchangeable.

REPLACEMENT PARTS AND SUPPLIES

PART NUMBER	DESCRIPTION
170-1100	Liquid Recirculator
042-2010	Sample Cup, micro, 6 mm diameter, 1.6 mm deep (2 ea.)
042-2020	Sample Cup, macro, 10 mm diameter, 2.3 mm deep (2 ea.)
042-3030	Sample Cup Holder and Base
160-8010	KBr Powder (100 g)
042-3040	Sample Preparation Kit
042-3010	Abrasion Sampling Kit
042-3020	Abrasion Disks, silicon carbide (100 ea.)
042-3025	Abrasion Disks, diamond (50 ea.)
042-3060	Flat Sample Post
042-3080	Alignment Mirror, aluminum
162-4303	Rotary Pump for vacuum insulation

REPLACEMENT PARTS AND SUPPLIES (cont.)

PART NUMBER	DESCRIPTION
160-1132	Disk, KBr, 32 x 3 mm
160-1113	Disk, ZnSe, 32 x 3 mm
160-1231	Disk, ZnSe, 32 x 3 mm, with anti-reflective coating
160-5049	Disk, SiO ₂ , 32 x 3 mm
160-5125	Disk, SiO ₂ , 32 x 3 mm, low OH
160-1159	Disk, Si, 32 x 3 mm
162-4210	O-Ring for DiffusIR Chamber (10 ea.)
162-4215	O-Ring for DiffusIR Chamber cooling line (10 ea.)
162-4251	Ceramic Cup for DiffusIR Chamber, porous
162-4270	Alignment Mirror for DiffusIR Chamber
042-3082	Alignment Mirror, gold

Notes: Please contact PIKE Technologies for items not described in this list.

DIFFUSIR SPECIFICATIONS

Optical Design	3X ellipsoidal
Angle of Incidence	30 degrees, nominal
Dimensions (W x D x H)	180 x 230 x 130 mm (excluding purge tubes and baseplate)
Sample Focus	Micrometer
Sample Positions	2 positions, slide stops for background and sample with no purge loss
Sample Cups	Micro: 6 x 1.6 mm deep Macro: 10 x 2.3 mm deep
Purge	Standard purge tubes and purge connection

ENVIRONMENTAL CHAMBER SPECIFICATIONS

Temperature Range, HTV	Ambient to 500 or 1000 °C
Temperature Range, LTV	-150 to 500 °C
Accuracy	+/- 0.5%
Input Voltage	100–240 VAC (HTV version) 110/220 V switchable (LTV version)
Operating Voltage	28 VDC/84 W (HTV and LTV versions)
Temperature Control	Digital or Digital PC
Heating Rate, Maximum	120 °C/minute
Kinetic Setup (requires Digital PC Controller, includes PIKE TempPRO software)	<ul style="list-style-type: none"> Up to 20 temperature ramps Individual ramp rate and hold time settings Graphical display of experiment settings Trigger data collection at specified times or temperatures USB interface
Sensor	K Type (for HTV) RTD Type, Pt100 (for LTV)
Vacuum Achievable	1 x 10 ⁻⁶ Torr (13 x 10 ⁻⁴ Pa)
Window Size	32 x 3 mm disk (vacuum) 32 mm ZnSe dome (pressure)
Leaking Volume	< 6.0 x 10 ⁻¹¹ Pa m ³ /sec
Pressure Maximum	<ul style="list-style-type: none"> 1500 psi, with High-Pressure Adapter (available in HTV versions only) 14.7 psi (1 atmosphere) using KBr window
Sample Cup Size	Macro: 6.0 mm OD, 4.0 mm height Micro: 4.7 mm ID, 2.0 mm depth
Sample Cup Design	Porous ceramic compatible with powders and gas flow
Cooling Ports	Quick-Fit, 6 mm ID
Gas/Vacuum Ports	1/8" Swagelok®

UpIR – Upward Looking Diffuse Reflectance Accessory



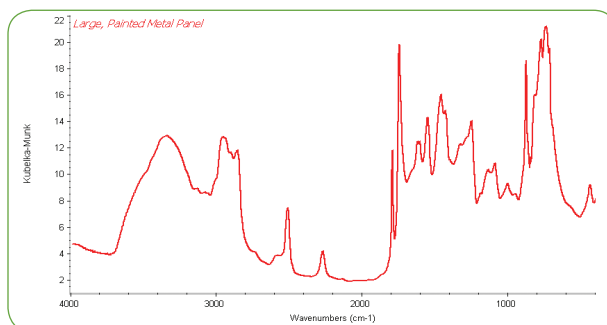
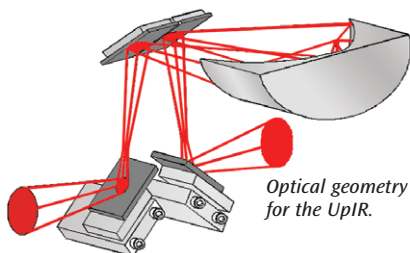
FEATURES

- Upward-looking optics provide fast and easy analysis of samples placed face down on the sample port
- Out-of-compartment design for analysis of large samples
- High optical throughput and exceptional signal-to-noise ratio
- Analysis of powders, ground solid samples and coatings on metallic surfaces
- Pre-aligned, fixed-position optical components for reproducible, high-quality data
- Micrometer-controlled sample stage positioning and focusing
- Optional gold-coated optics version for highest performance mid-IR and NIR applications

The UpIR is an innovative FTIR accessory developed to support a wide range of diffuse reflectance applications. To make measurements, simply place large, solid samples face down onto the top plate of the accessory. Powders can be placed into a suitable sampling cup at the top of the UpIR. A mask set is included for the analysis of small solids such as gems and precious stones.

This design is uniquely suitable for mid-IR analysis of coatings on metallic surfaces of large or small samples. For this application, analysis is rapid and easy because no sample preparation or cleanup is required. Since the sampling area of the UpIR is above the plane of the FTIR instrument, even large samples that do not fit into the sample compartment can be analyzed with this accessory.

The accessory is equipped with an upward-looking, high-performance ellipsoidal mirror. The sampling stage provides a sampling port with inserts for diffuse reflectance or specular reflectance measurements.



Analysis of a large painted metal panel using the UpIR accessory.

All mirrors, including the ellipsoidal collection mirror, are permanently mounted. The position of the sampling stage is controlled with an adjustable micrometer to achieve the best possible throughput. Spectral analysis involves collecting a background spectrum with the reference mirror in the sampling position. After this step, the sample is simply placed face down onto the sampling port and data collection is initiated.

The gold-coated optics version of the UpIR provides the highest throughput in the mid-IR spectral region and is recommended for NIR sampling. The UpIR accessory includes a solids sampling plate for flat samples, a ZnSe-windowed sampling cup for powders or small solids analysis and a 4-piece mask set (aperture diameters of 10, 7, 5 and 3 mm). The accessory is equipped with purge tubes for elimination of CO₂ and water interferences from infrared spectra.

For NIR sampling of solids, powders or tablets, the sapphire-windowed sampling cup is recommended. In the NIR spectral region samples can be analyzed while contained in a glass vial; the optional 21-mm glass vial holder is recommended.

ORDERING INFORMATION

PART NUMBER	DESCRIPTION
044-10XX	UpIR – Out-of-Compartment Diffuse Reflectance Accessory <i>Includes solids sampling insert or powders sampling insert with ZnSe window, mask set, gold mirror, purge tubes, purge kit and spectrometer baseplate</i>
044-60XX	UpIR – Out-of-Compartment Diffuse Reflectance Accessory with Gold-Coated Optics <i>Includes solids sampling insert or powders sampling insert with ZnSe window, mask set, gold mirror, purge tubes, purge kit and spectrometer baseplate</i>

Note: Replace XX with your spectrometer's Instrument Code. [Click for List >](#)

UPIR OPTIONS

PART NUMBER	DESCRIPTION
044-3030	Solids Sampling Insert
044-3040	Powders Sampling Insert (order window separately)
044-3010	Glass Vial Holder, 21 mm
044-3020	Sample Vials with Threaded Caps, 21 mm x 70 mm (200 ea.)
044-3050	UpIR Mask Set
160-1155	Window, ZnSe, 25 x 2 mm
160-1307	Window, Ge, 25 x 2 mm
160-1201	Window, AMTIR, 25 x 2 mm
160-5000	Window, Sapphire, 25 x 2 mm
048-3000	Diffuse Gold Reference

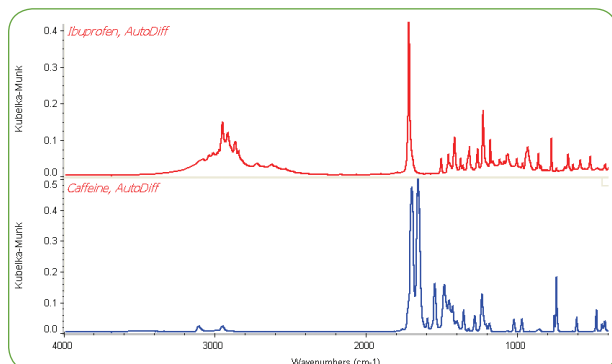
AutoDiff – Automated Diffuse Reflectance Sampling



FEATURES

- Complete automated diffuse reflectance accessory and software package for unattended analysis of up to 60 samples
- High-performance optical design collects maximum amount of diffusely reflected energy and provides high-quality spectra in a short time period
- Flexible sample sequencing and background collection to provide maximum sampling efficiency and greatly minimize atmospheric contributions to sample spectra
- Easily programmable AutoPRO software delivers automated sample collection
- Easily removable sample tray to speed sample loading and unloading

The AutoDiff is a high-performance, automated diffuse reflectance accessory developed to analyze multiple samples with minimal user intervention. Typical applications include powdered pharmaceutical samples, high-throughput forensic sampling, kidney stone analysis, soils analysis and analysis of many other powdered samples where speed and efficiency are important. The design employs an automated R-theta sampling stage providing diffuse reflectance analysis with greatly reduced operator intervention and increased sample throughput.



Spectra of pharmaceuticals using the AutoDiff accessory.

The optical design of the AutoDiff utilizes a high-efficiency fixed ellipsoidal reflector to collect the maximum amount of diffusely reflected energy from the sample. Other optical components important to achieving this high performance are aligned and permanently located. The accessory is baseplate-mounted in the FTIR spectrometer sample compartment and can be purged independently or it can use the spectrometer's purge.

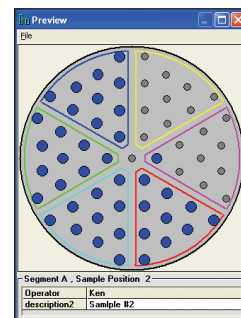
Spectral quality and reproducibility are excellent with the AutoDiff. By programming the collection of spectra at precise time periods and alternating sample and background collection, any effects of atmosphere are greatly reduced.

The PIKE AutoDiff fully automates diffuse reflectance FTIR spectroscopy. The sample holder contains positions for 60 samples, plus a center position for a background sample, which usually consists of pure KBr powder.

The sample plate is marked into six areas, labeled from A to F. Each area has ten sample positions marked from 1 through 10. This sample position numbering scheme is also used within the software for describing and positioning the samples.

The AutoDiff is controlled by PIKE AutoPRO software which incorporates multi-operator sample submission. The system is extremely flexible and the graphical user interface is intuitive and simple. Multiple operators may independently log samples onto the system. The AutoPRO software integrates easily with most commercially-available FTIR software packages.

The AutoDiff is also available with gold-coated optics for highest performance mid-IR analysis and for automated NIR diffuse reflectance sampling.



ORDERING INFORMATION

PART NUMBER	DESCRIPTION
043-28XX	AutoDiff – Automated Diffuse Reflectance System <i>Includes motion control unit (85/265 VAC), AutoPRO software, 60-position sample mounting tray, 60 macro sample cups and Sample Preparation Kit</i>
043-78XX	AutoDiff – Automated Diffuse Reflectance System with Gold-Coated Optics <i>Includes motion control unit (85/265 VAC), AutoPRO software, 60-position sample mounting tray, 60 macro sample cups and Sample Preparation Kit</i>

Note: Replace XX with your spectrometer's Instrument Code. [Click for List >](#)

REPLACEMENT PARTS AND OPTIONS

PART NUMBER	DESCRIPTION
043-3090	AutoDiff Sampling Cups, macro (60 ea.)
043-3085	AutoDiff Sampling Cups, micro (60 ea.)
043-0900	AutoDiff 60-Position Sampling Tray
042-2010	Sample Cup, micro, 6.0 mm diameter, 1.6 mm deep (2 ea.)
042-2020	Sample Cup, macro, 10 mm diameter, 2.3 mm deep (2 ea.)
042-3010	Abrasion Sampling Kit
042-3020	Abrasion Disks, silicon carbide (100 ea.)
042-3025	Abrasion Disks, diamond (50 ea.)
042-3080	Alignment Mirror, aluminum
042-3082	Alignment Mirror, gold

XY Autosampler – Transmission and Reflection, Automated Sampling in Microplate Format



FEATURES

- Complete hardware and software package for automated analysis with standard 24-, 48-, or 96-well plates. Special configurations available.
- Diffuse reflectance of powdered samples or specular reflectance sampling for reaction residues
- Gold-coated optics version for highest performance mid-IR and NIR sampling
- Optional transmission sampling with integrated DTGS or InGaAs detector and transmission sampling plate
- Fully enclosed, purgeable design with CD-style loading tray
- In-compartment mounting, compatible with most FTIR spectrometers

The PIKE Technologies XY Autosampler is designed around standard 24-, 48- or 96-well microplate architectures – ideal for high-efficiency sample loading and FTIR analysis. The loading tray moves to a position outside of the accessory for easy loading and unloading of samples while conserving the purge. This also permits interface to a robot/autoloader.

Applications include high throughput analysis of liquid residues and chemical reactions, powdered samples, and automated diffuse reflection analysis. The XY Autosampler is available with standard aluminum optics or with gold-coated optical components for highest performance in mid-IR and optimized NIR sampling.

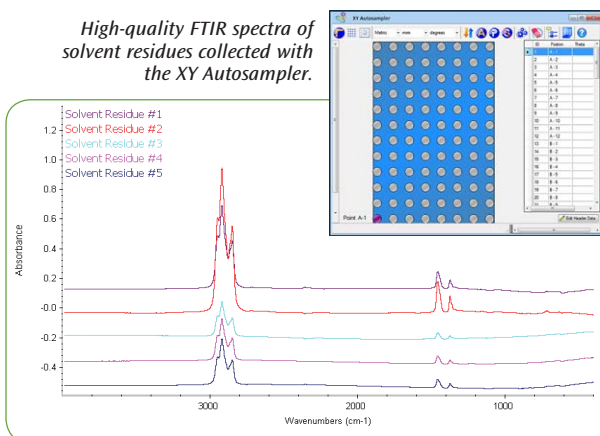
The XY Autosampler features an X, Y stage with both axes driven by high-precision servo motors with optical encoders for speed and reproducibility. USB and DC power are the only external connections required for this accessory. The transmission option requires an external IR detector port.

Programming and control of the XY Autosampler is done through PIKE Technologies' AutoPRO software, which can be integrated easily with most FTIR software packages.

The optical design of the XY Autosampler is based upon a precision ellipsoidal reflector. The size of the spot illuminated at the sample is approximately 2 mm which is ideal for up to 96-well configurations. The accessory is compatible with most FTIR spectrometers.

A special silicon well plate is available for mid-IR sample analysis by transmission. This unique 96-well plate allows collection of transmission spectra in mid-IR range. For diffuse reflection measurements a dedicated plate is available. The plate features 96 polished cavities for placement of powder samples. Please contact PIKE Technologies if you require specialized sampling plate configurations.

High-quality FTIR spectra of solvent residues collected with the XY Autosampler.



SPECIFICATIONS

Optics	Elliptical – 3X beam demagnification
Accuracy	+/- 25 μ m
Mechanical Specifications	
Repeatability	+/- 5 μ m
Resolution	1 μ m
Minimum Run Time	56 seconds for 96-well plate (actual time is spectrometer and application dependent)
Computer Interface	USB
Dimensions (W x D x H)	6.25 x 13.2 x 5.55" (including micrometer)
Weight	10 lbs

ORDERING INFORMATION

PART NUMBER	DESCRIPTION
047-21XX	XY Autosampler – Diffuse Reflectance
047-61XX	XY Autosampler – Diffuse Reflectance with Gold-Coated Optics
047-22XX	XY Autosampler – Diffuse Reflectance/Transmission with integrated DTGS detector
047-62XX	XY Autosampler – Diffuse Reflectance/Transmission with Gold-Coated Optics with integrated DTGS detector
047-23XX	XY Autosampler – Diffuse Reflectance/Transmission with integrated InGaAs detector
047-63XX	XY Autosampler – Diffuse Reflectance/Transmission with Gold-Coated Optics with integrated InGaAs detector

Notes: Replace **XX** with your spectrometer's Instrument Code. [Click for List >](#)
 All XY Autosamplers include PIKE AutoPRO software and a 96-well sampling plate. Diffuse Reflectance/Transmission versions include a 96-well plate for diffuse reflectance and a 96-well plate for transmission. For other options please contact PIKE Technologies. For transmission option, your spectrometer must be capable of interfacing with an external detector. A glass-bottom well plate is recommended for NIR transmission measurements.

OPTIONS

PART NUMBER	DESCRIPTION
073-9110	96-Well Diffuse Reflectance Sampling Plate
073-9130	96-Well Si Transmission Sampling Plate
073-9160	24-Well Diffuse Reflectance Sampling Plate for Disposable Cups
162-1920	Disposable Cups (50 ea.)

Sample Preparation and Loading Kit – The Easiest Way to Work With Powder Samples



The Sample Preparation and Loading Kit makes the handling of powder samples for diffuse reflectance sampling easy. It includes a round mounting base and a matching tray with an opening which accommodates sampling cups. The cup is placed in the assembly and overfilled with sample, and then excess powder is leveled-off with a spatula. The overflow is retained on the tray and can be easily returned to the sample container or disposed.

Two standard sampling cups offer 0.18 and 0.03 cubic centimeter capacities (10-mm diameter, 2.3-mm deep and 6.0-mm diameter, 1.6-mm deep). The required approximate weight of the sample/KBr mixture is 450 mg for the large cup and 80 mg for the small one.

ORDERING INFORMATION

PART NUMBER	DESCRIPTION
042-3040	Sample Preparation and Loading Kit <i>Includes 2 micro and 2 macro cups, alignment mirror, sampling cup holder and base, 2 spatulas, brush</i>
042-2010	Sample Cup, micro, 6-mm diameter, 1.6-mm deep (2 ea.)
042-2020	Sample Cup, macro, 10-mm diameter, 2.3-mm deep (2 ea.)
042-3030	Delrin® Sampling Cup Holder and Base
042-3080	Alignment Mirror, aluminum
042-3082	Alignment Mirror, gold
042-3070	Camel Hair Brush
042-3035	Spatula, spoon
042-3050	Spatula, flat
160-8010	KBr Powder (100 g)

Note: The Sample Preparation and Loading Kit is included with EasiDiff, DiffusIR and AutoDiff diffuse reflectance accessories.

Abrasion Sampling Kit – The Plastic Bumper Sampler



The Abrasion Sampling Kit consists of a sample collector tool and a set of silicone carbide (SiC) and diamond disks. Sampling is performed by abrading the surface of the investigated substance with a selected disk. The disk is placed in the accessory and a diffuse reflectance spectrum of the material is collected. This method is particularly useful for the analysis of large painted surfaces (e.g. car panels) and other awkward objects.

- Convenient set of tools for collection of difficult solid samples
- Rigid construction, diamond and silicon carbide disks

ORDERING INFORMATION

PART NUMBER	DESCRIPTION
042-3010	Abrasion Sampling Kit <i>Includes sample collector tool with stainless steel flat sample post, 75 SiC disks and 25 diamond abrasion disks</i>
042-3020	Abrasion Disks, silicon carbide (100 ea.)
042-3025	Abrasion Disks, diamond (50 ea.)
042-3060	Flat Sample Post

Diffuse Reflectance – Theory and Applications

Diffuse Reflectance – Ideal for Powdered Samples and Intractable Solids

Diffuse reflectance is an excellent sampling tool for powdered or crystalline materials in the mid-IR and NIR spectral ranges. It can also be used for analysis of intractable solid samples. As with transmission analysis, samples to be run by diffuse reflectance are generally ground and mixed with an IR transparent salt such as potassium bromide (KBr) prior to sampling. Diffuse reflectance is an excellent sampling technique as it eliminates the time-consuming process of pressing pellets for transmission measurements. Diffuse reflectance can also be used to study the effects of temperature and catalysis by configuring the accessory with a heating or cooling environmental chamber.

Perhaps one of the greatest additional benefits of diffuse reflectance sampling is that it is ideally amenable to automation. Methods can be developed with a manual version diffuse reflection accessory and then moved to automation to increase sample throughput. PIKE Technologies offers several diffuse reflectance accessory configurations – basic, advanced with heat chamber capabilities, upward directed IR beam for easy sampling access and fully automated for maximum sampling efficiency.

How Diffuse Reflectance Works

Diffuse reflectance relies upon the focused projection of the spectrometer beam into the sample where it is reflected, scattered and transmitted through the sample material (shown below). The back reflected, diffusely scattered light (some of which is absorbed by the sample) is then collected by the accessory and directed to the detector optics.

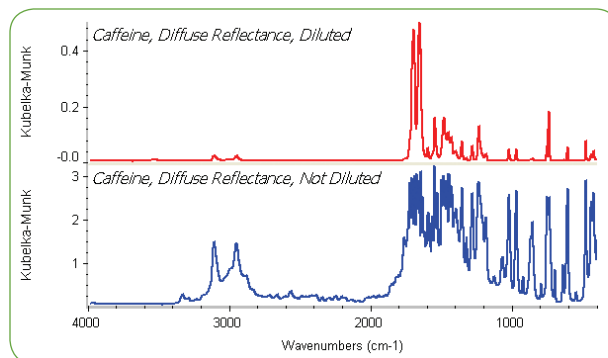


Only the part of the beam that is scattered within a sample and returned to the surface is considered to be diffuse reflection.

Some powders may be analyzed by diffuse reflectance as neat samples (coal samples, soil samples, diffuse coatings on a reflective base). Usually, the sample must be ground and mixed with a non-absorbing matrix such as KBr. The sample to matrix ratio is generally between 1 to 5% (by weight). Diluting ensures a deeper penetration of the incident beam into the sample which increases the contribution of the scattered component in the spectrum and minimizes the specular reflection component.

The specular reflectance component in diffuse reflectance spectra causes changes in band shapes, their relative intensity, and, in some cases, it is responsible for complete band inversions (Reststrahlen bands). Dilution of the sample with a non-absorbing matrix minimizes these effects (particle size and sample loading mechanics also play an important role).

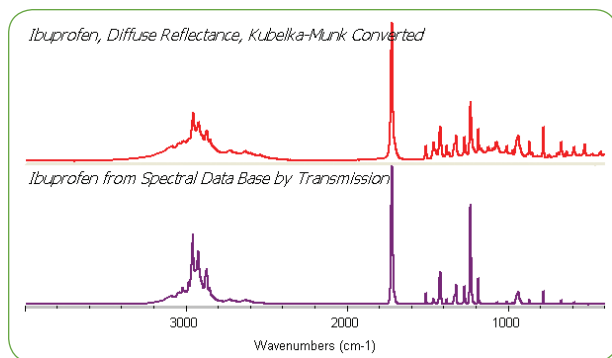
This is shown below in the spectral data for caffeine, where the upper spectrum is diluted to about 2% by weight in KBr and demonstrates very high quality with sharp, well-defined absorbance bands. The lower spectrum is of undiluted caffeine measured by diffuse reflectance and shows derivative-shaped bands in the 1700 cm^{-1} and 1500 cm^{-1} region of the data. The upper spectrum of diluted caffeine is clearly of higher spectral quality than that of the undiluted caffeine.



Diffuse reflective spectra showing greatly improved results from sample dilution.

Other factors related to high spectral quality for diffuse reflectance sampling are listed below.

- **Particle Size** – reducing the size of the sample particles reduces the contribution of reflection from the surface. Smaller particles improve the quality of spectra (narrow bandwidths and better relative intensity). The recommended size of the sample/matrix particles is 50 microns or less (comparable to the consistency of the finely ground flour). This fine powder is easily achieved by using the PIKE Technologies ShakIR ball mill.
- **Refractive Index** – effects result in specular reflectance contributions (spectra of highly reflecting samples will be more distorted by the specular reflectance component). This effect can be significantly reduced by sample dilution.
- **Homogeneity** – samples prepared for diffuse reflectance measurements should be uniformly and well mixed. Non-homogenous samples will lack reproducibility and will be difficult to quantify. An ideal way to mix samples for diffuse reflectance is by using the PIKE Technologies ShakIR.
- **Packing** – the required sample depth is governed by the amount of sample scattering. The minimum necessary depth is about 1.5 mm. The sample should be loosely but evenly packed in the cup to maximize IR beam penetration and minimize spectral distortions.



Diffuse reflectance spectra of ibuprofen with Kubelka-Munk conversion compared to a transmission spectrum.

Even with all these sample preparation practices, the raw diffuse reflectance spectra will appear different from its transmission equivalent (stronger than expected absorption from weak IR bands). A Kubelka-Munk conversion can be applied to a diffuse reflectance spectrum to compensate for these differences. This conversion is available in most FTIR software packages.

The Kubelka-Munk equation is expressed as

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

where R is the absolute reflectance of the sampled layer, k is the molar absorption coefficient and s is the scattering coefficient.

The spectra shown above demonstrate this spectral conversion for ibuprofen collected by diffuse reflectance. The sample was diluted to about 1% by weight in KBr and mixed using the ShakIR. The Kubelka-Munk converted spectrum for ibuprofen shows excellent comparison with the transmission spectrum and is easily identified using library search of a transmission spectral database.

The Kubelka-Munk equation creates a linear relationship for spectral intensity relative to sample concentration (it assumes infinite sample dilution in a non-absorbing matrix, a constant scattering coefficient and an “infinitely thick” sample layer). These conditions can be achieved for highly diluted, small particle samples (the scattering coefficient is a function of sample size and packing) and a sample layer of at least 1.5 mm. With proper sample preparation, diffuse reflectance spectroscopy can provide ppm sensitivity and high-quality results.

Plastic Bumpers and Tough Samples

Sometimes it is necessary to analyze a sample which simply does not fit in a spectrometer's sample compartment – the analysis of polymer-based automotive components or painted panels are typical examples.

A special diffuse reflectance technique allows quick and simple analysis of such samples in a relatively non-destructive manner. A small amount of the sample can be collected by abrasion on a diamond or silicon carbide abrasion disk and analyzed immediately with the help of a diffuse reflectance accessory.

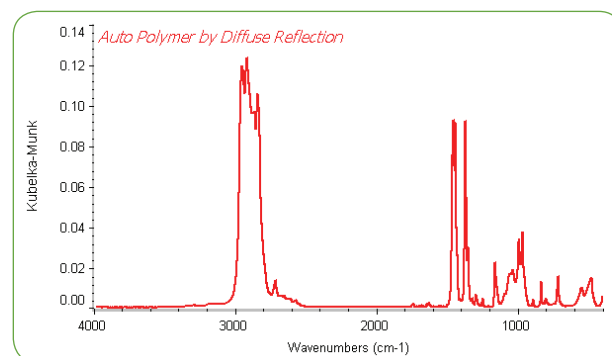


Heavy-duty sample collecting tool with diamond abrasion disk on removable sample post



The figure below shows the diffuse reflectance spectrum of an automotive body component. The PIKE Abrasion Sampling Kit with diamond sampling disk was rubbed across the large automotive component which collects some of the polymer material into the web of the sampling disk. Spectra were co-added for 1 minute and ratioed to the diamond disk background spectrum. The resulting spectrum is of excellent quality and is identified as a polypropylene copolymer.

Diffuse reflectance can also be used for the analysis of liquid samples. In this application a small amount of the sample is dispensed directly onto the KBr powder and analyzed.



Diffuse reflectance spectrum using diamond abrasion disk.

For the analysis of powders the following procedure is recommended;

- Place about 200–400 mg of KBr into the ShakIR vial with a stainless steel ball and grind for 30 seconds
- Fill the background diffuse cup with this KBr
- Remove excess KBr with a flat edge – the KBr should be loosely packed
- Add 1 to 5 mg of the sample to the remaining KBr in the ShakIR vial and mix for 30 seconds
- Fill the sample diffuse cup with this mixed sample/KBr
- Remove excess sample with a flat edge – the sample should be loosely packed
- Place the background and sample diffuse cups into the sample holder
- Slide the sample holder into the accessory
- Position the KBr cup in the beam and collect a background
- Move the holder to the sample position and collect a sample spectrum (the ratio of these two spectra will produce a spectrum of the sample)
- Convert the raw diffuse reflectance spectrum to Kubelka-Munk

Under ideal conditions the transmission of the strongest band in the spectrum should be in the 50% range. If the resulting bands are too intense or distorted, further dilute the sample and make sure that all other measurement affecting factors (particle size, homogeneity and packing) are within required limits.

Summary

Diffuse reflectance accessories make the analysis of a wide range of solid samples easier, faster and more efficient. Advanced options for diffuse reflectance provide the ability to heat and cool the sample and monitor a reaction process. Automation versions of diffuse reflectance accessories provide the ability to greatly increase sample throughput.

ORDERING TERMS, CONTACT INFORMATION AND GUARANTEE

PART NUMBERS AND PRICE

The PIKE price list includes accessories that may be used with a variety of makes and models of spectrometers. Please specify the part number and description when ordering, including your instrument type and model number. [Click here](#) for a list of spectrometer and spectrophotometer instrument codes. When placing an order, substitute these codes for the final two digits (XX) in the accessory part number.

PIKE Technologies is continually extending the accessory product range. If you are unable to find a required item, please contact us to discuss your needs. We will be glad to assist.

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Please include the following information when placing an order: your name, phone number, product part number, quantity, ship to address, bill to address, purchase order number and spectrometer model on which the accessory will be used.

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Madison, WI 53719
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(608) 274-0103 (FAX)
orders@piketech.com (E-MAIL)
www.piketech.com

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When ordering a PIKE accessory, replace the **XX** or **XXX** portion of the product's part number with your spectrometer's instrument code below. For assistance, please contact a PIKE customer service representative at (608) 274-2721 or sales@piketech.com.

FTIR INSTRUMENT CODES (XX)

ABB Bomem

FTLA2000-100 (Arid Zone)	80
Michelson 100, MB Series	81
MB 3000	82

Agilent

Excalibur™, Scimitar™, FTS, 600-IR Series	10
Excalibur™, Scimitar™, 600-IR Series with recognition	13

Analect (See Hamilton Sundstrand)

Bio-Rad (See Agilent)

Bruker Optics

IFS™, Vector™, Equinox™ Series.	50
Tensor™, Vertex™ with recognition (Quick-Lock)	51

Buck Scientific

M500	65
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Digilab (See Agilent)

Hamilton Sundstrand AIT

Diamond 20	60
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Horiba

7000 Series	35
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Interspectrum

Interspec 200-X	90
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Jasco

300/600 Series	56
400	57
4000/6000 Series	58

JEOL

Winspec™ Series	46
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Lambda Scientific

Lambda FTIR 7600	66
Lambda FTIR 8600	64

Lumex

INFRALUM FT-02, FT-08	67
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Mattson (See Thermo Electron)

Midac

M Series	30
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Nicolet (See Thermo Electron)

Oriel	95
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Optical Table

	99
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PerkinElmer

1700 Series	70
Spectrum™ GX, 2000	71
Spectrum BX / RX, 1600, Paragon 1000	73
Frontier, Spectrum One, 65, 100, 400 with recognition	74
Spectrum Two with recognition	75

Shimadzu

8300, 8400 Series, IRPrestige™-21, IRAffinity-1s	15
IRPrestige™-21, IRAffinity-1s with recognition (QuickStart)	16
IRTracer™-100	18
IRTracer™-100 with recognition	19

Thermo Electron / Nicolet / Mattson

Infinity, Galaxy, RS Series	20
Genesis™, Satellite, IR 300	21
Impact™ 400, Magna, Protege™, 500 / 700 Series	40
Avatar™, Nexus™, Nicolet™, iS™10, iS™50	40
Model 205/210	41
Nicolet iS™5	42
Avatar, Nexus, Nicolet Series with recognition (Smart)	47

Varian (see Agilent)

UV-VIS INSTRUMENT CODES (XXX)

Agilent/Varian

Cary 50	100
Cary 60	111
Cary 100, 300	110
Cary 4000, 5000, 6000i	120

Jasco

600 Series	600
Optical Table	999

PerkinElmer

Lambda 650, 750, 850, 950 and 1050	700
Lambda 25, 35, 45	730

Shimadzu

1600 and 1700	200
1800 Series	210
2600	240
3600	220

Thermo Fisher Scientific

Evolution 300/600	400
Evolution 200	410

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