

Diffuse Reflection



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

EasiDiff

AT A GLANCE

- ▶ 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 EasiDiff is an economical, high-quality diffuse reflection accessory designed to analyze a wide variety of solid samples. It is often used for 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.

FAST AND EFFICIENT DESIGN

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.

SAMPLING CUPS

Two standard sampling cups offer 0.18 and 0.03 cm³ capacities. The required approximate weight of the sample/KBr mixture is 450 mg for the large cup and 80 mg for the small one.

ABRASION KIT

The Abrasion Sampling Kit (optional) consists of a sample collector tool and a set of silicon carbide 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 reflection spectrum of the material is collected.

The abrasion technique is advantageous when the sample is intractable, too large to bring into the lab, or its shape may prevent the sample from fitting firmly onto the sampling platform of a traditional IR accessory.



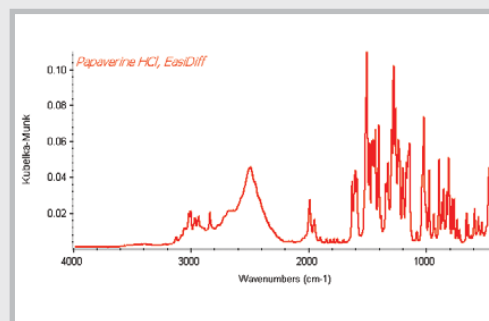
Abrasion Sampling Kit.

SPECIFICATIONS

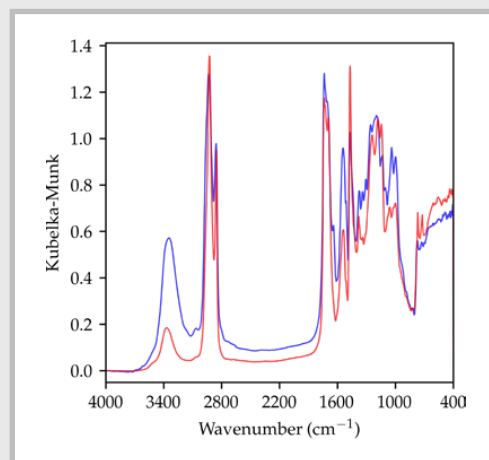
Optical Design	All reflection
Dimensions (W X D X H)	96 x 110 x 154 mm (excluding baseplate)
Sample Focus	Micrometer
Sample Positions	2 positions, slide stops for background and sample
Sample Cup Dimensions	
Micro	6 mm ID x 1.6 mm deep
Macro	10 mm ID x 2.3 mm deep
Sample Cup Volume	
Micro	0.03 cm ³
Macro	0.18 cm ³

APPLICATION

For powders and diffusely scattering solids, diffuse reflection is a convenient infrared sampling technique, and offers high sensitivity. For intractable samples, the abrasion method provides a simple solution for analyzing solids and coatings—example shown below.



Papaverine hydrochloride diluted to 1% in KBr powder.



Polymer spectra of the outer cover of two golf balls brands using the abrasion method.

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 listed in the back of the catalog.

Option

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

Replacement Parts and Supplies

042-2010	Micro Sample Cup (2 ea.)
042-2020	Macro Sample Cup (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, specular
042-3082	Alignment Mirror, gold, specular
042-3084	Alignment Mirror, gold, diffuse
042-3060	Flat Sample Post

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

DiffusIR

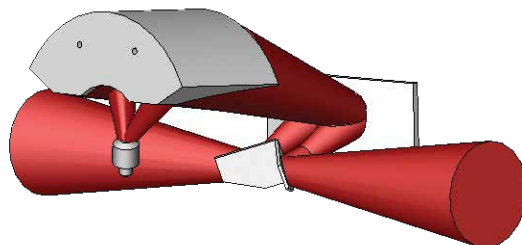
AT A GLANCE

- ▶ Large and efficient 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
- ▶ Sealed and purgeable optical design to eliminate water vapor and carbon dioxide interference

The DiffusIR™ is a research-grade diffuse reflection accessory with an efficient optical design and is often configured with optional 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.

DESIGN

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. The optical components are enclosed and equipped with purge tubes for the elimination of water and CO₂ interferences.



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.

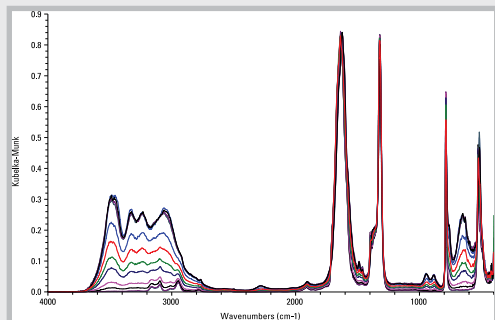


Low temperature chamber shown with connections.



APPLICATION

The DiffusIR offers a highly sensitive, research-grade accessory for the analysis of powders and other diffusely scattering samples. When equipped with an environmental chamber, influence of gas flow and thermal conditions on the sample may be investigated.



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

TEMPERATURE/GAS FLOW STUDIES

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 chambers allow for gas flow to monitor the effects of gas on catalysts and other powdered samples. The optional chambers are easily inserted into the DiffusIR and secured using push-lock pins.

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

OPTIONAL GOLD-COATED OPTICS

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

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 mm ID x 1.6 mm deep Macro: 10 mm ID x 2.3 mm deep
Purge	Standard purge tubes and purge connection

ENVIRONMENTAL CHAMBER SPECIFICATIONS

Temperature Range, HTV	Ambient to 1000 °C
Temperature Range, LTV	-150 to 500 °C
Accuracy	+/- 0.5° up to 100 °C +/- 0.5% of set point > 100 °C
Heating Rate, Maximum	20°C/min
Temperature Control	Digital, Touch-panel display
Kinetic Setup	
Requires Digital Temperature Control Module And PIKE TempPRO Software	Unlimited temperature ramps Individual ramp rate and hold time settings Graphical display of experiment settings Trigger data collection at specified USB interface
Sensor	K Type RTD Type, Pt100 (for LTV)
Input	100–240 VAC (for both versions)
Output	28VDC/84 W max. (HTV version) 54 VDC/84 W max. (LTV version)
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)
Leak Rate	< 6.0 x 10 ⁻¹¹ Pa m ³ /s
Pressure Maximum	1500 psi, with High-Pressure Adapter (available in HTV versions only) 14.7 psi (1 atmosphere) using KBr window
Sample Cup Size	5 mm ID x 2 mm deep
Sample Cup Design	Porous ceramic compatible with powders and gas flow
Cooling Ports	Quick-Fit, 6 mm ID
Gas/Vacuum Ports	1/8" Swagelok®

Environmental chamber for the DiffusIR.



PART NUMBER	DESCRIPTION
041-10XX	DiffusIR Accessory Includes Sample Preparation Kit with 2 micro and 2 macro sample cups, sample loading tools, alignment mirror, 35-mm mortar with pestle and KBr powder (100 g)
041-60XX	DiffusIR Accessory with Gold-Coated Optics Includes Sample Preparation Kit with 2 micro and 2 macro sample cups, sample loading tools, alignment mirror, 35-mm mortar with pestle and KBr powder (100 g)

Note: Replace XX with your spectrometer's Instrument Code listed in the back of the catalog.

DiffusIR Options	
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

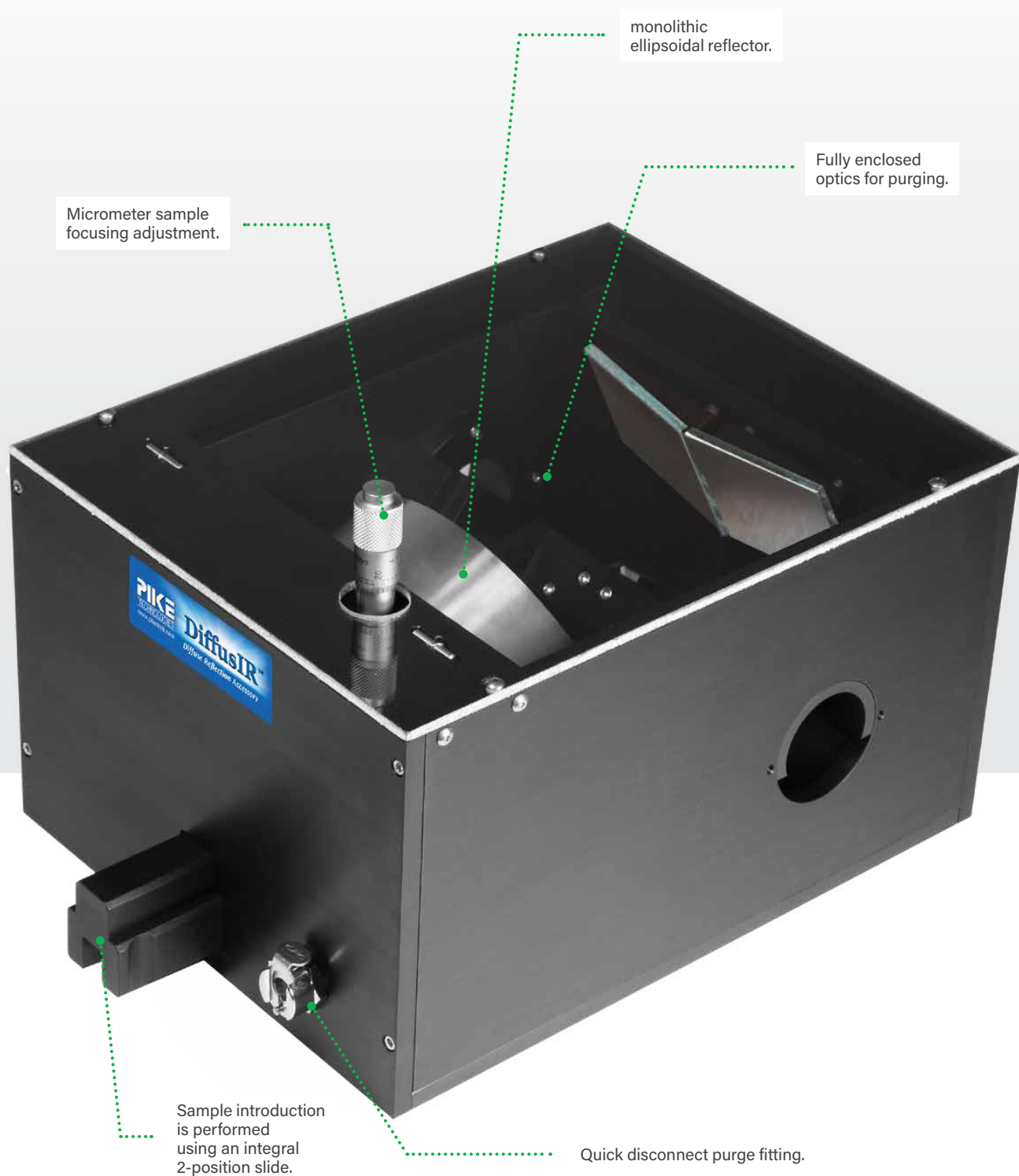
Notes: HTV and LTV chambers require the selection of a temperature control module. DiffusIR Chambers include front plate accommodating environmental chamber (easily changeable with standard DiffusIR front plate), Pin-Loc chamber insertion for easy sample exchange, KBr window, ceramic sampling cups compatible with vacuum and reaction formats, ports and 2 shut-off valves for vacuum operation and ports for connection of water cooling. The 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 162-4165 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.

PART NUMBER	DESCRIPTION
Temperature Control Modules	
076-2550	Digital Temperature Control Module
007-0207	PIKE TempPRO Software
162-4165	Liquid Nitrogen-Cooled System and Temperature Control Module DiffusIR Environmental Chamber, LTV, -150 to 500 °C, 115 V
162-4166	Liquid Nitrogen-Cooled System and Temperature Control Module DiffusIR Environmental Chamber, LTV, -150 to 500 °C, 220 V

Notes: Digital Temperature Control Module with TempPRO software (sold separately) 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	
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
042-3082	Alignment Mirror, gold
162-4298	Rotary Pump for vacuum insulation, 115V
162-4299	Rotary Pump for vacuum insulation, 230V
160-1132	Disk, KBr, 32 x 3 mm
160-1113	Disk, ZnSe, 32 x 3 mm
160-1229	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

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



UpIR

AT A GLANCE

- ▶ 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
- ▶ Analysis of powders, ground solid samples and coatings on metallic surfaces
- ▶ High optical throughput and exceptional signal-to-noise ratio
- ▶ Pre-aligned, fixed-position optical components.
- ▶ 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 reflection applications.

MID-IR ANALYSIS FOR BOTH SMALL AND LARGE SAMPLES

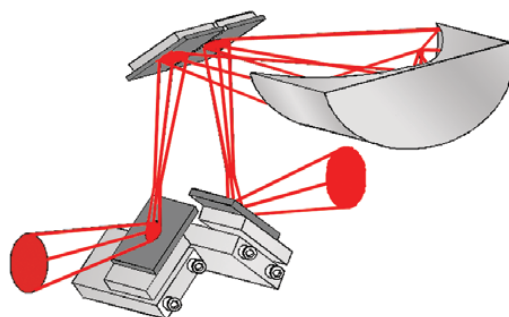
To make measurements, simply place large, solid samples face down onto the top plate of the accessory. Powders can be placed into a suitable sampling cup at the top of the UpIR. A mask set is included for the analysis of small solids such as gems and precious stones.

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 purge tubes for the elimination of CO₂ and water interferences from infrared spectra.

THE OPTICAL GEOMETRY OF THE UPIR

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



Optical geometry of 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.

OPTIONAL GOLD-COATED OPTICS

The gold-coated optics version of the UpIR provides the highest throughput in the mid-IR spectral region and is recommended for NIR sampling.



SPECIFICATIONS

Optical Design	All reflection
Dimensions (W X D X H)	154 x 197 x 228 mm (excluding purge tubes and baseplate)
Sample Focus	Micrometer
Purge	Standard purge tubes and purge connection

SAMPLING INSERTS

The UpIR accessory includes a solids sampling plate for flat samples, a ZnSe-windowed sampling cup for powders or other particulate samples, and a 4-piece mask set (aperture diameters of 10, 7, 5, and 3 mm) suitable for measuring small solids such as gemstones.



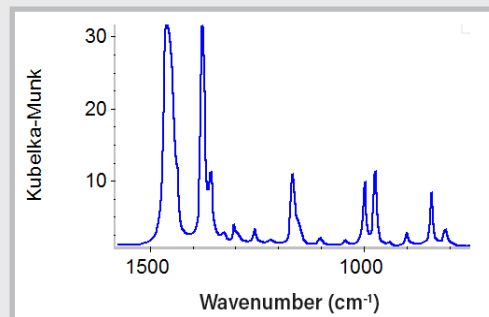
For NIR sampling of solids, powders or tablets, the sapphire-windowed sampling cup is recommended. In the NIR spectral region samples can be analyzed while contained in a glass vial; the optional 21-mm glass vial holder is recommended.



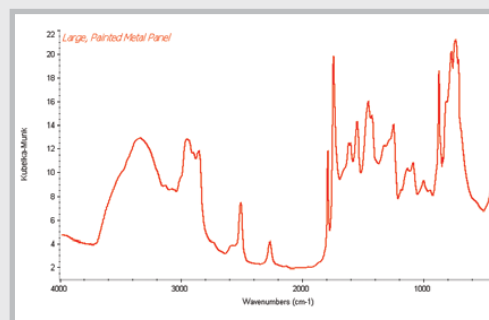
Glass vial holder and ZnSe powder cup.

APPLICATION

The UpIR is ideal for large samples because it is not limited to the confines of the sample compartment. With the mask set the UpIR can also accommodate smaller samples.



3-Ply face mask spectrum, non-destructive IR measurement using the UpIR.



Large painted metal panel spectrum collected using the UpIR.

PART NUMBER	DESCRIPTION
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044-10XX	UpIR
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044-60XX	UpIR with Gold-Coated Optics
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Notes: Includes solids sampling insert and powders sampling insert with ZnSe window, mask set, gold mirror, purge tubes, purge kit and spectrometer baseplate. Replace XX with your spectrometer's Instrument Code listed in the back of the catalog.

UpIR Options

044-3030	Solids Sampling Insert
044-3010	Glass Vial Holder, 21 mm
044-3020	Sample Vials, 21 x 70 mm (200 ea.)
044-3050	UpIR Mask Set, 3, 5, 7 and 10 mm
044-3040	Powders Sampling Cup (order window separately)
160-1155	Window, ZnSe, 25 x 2 mm
160-1307	Window, Ge, 25 x 2 mm
160-5000	Window, Sapphire, 25 x 2 mm
048-3000	Diffuse Gold Reference

AutoDiff II

AT A GLANCE

- ▶ Automated diffuse reflection accessory for unattended analysis of up to 60 samples
- ▶ High-performance optical design collects maximum amount of energy and provides high-quality spectra
- ▶ Easily programmable for automated sample collection
- ▶ Removable sample tray to speed sample loading and unloading



FULLY AUTOMATED
WITH AUTOPRO SOFTWARE



The AutoDiff II is a high-performance, automated diffuse reflection accessory developed to analyze multiple samples with minimal user intervention. This programmable accessory can be fully automated to save time and increase productivity.

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. Spectral quality and reproducibility are excellent with the AutoDiff II.

DESIGN

The design employs an automated R-theta sampling stage providing diffuse reflection analysis with greatly reduced operator intervention and increased sample throughput. The optical design of the AutoDiff II 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.

The AutoDiff II fully automates diffuse reflection FTIR spectroscopy. The sample tray 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.

Gold-coated optics version of the AutoDiff II is available for highest performance mid-IR analysis and for automated NIR diffuse reflection sampling.

AUTOPRO SOFTWARE

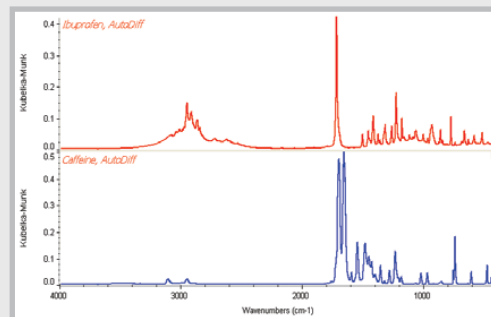
The AutoDiff II is controlled by PIKE AutoPRO software which incorporates multi-operator sample submission. The system is extremely flexible and the graphical user interface is intuitive and simple. Additional features include integrating sample information into the spectral data file and file name, and several background collection options such as collecting a background between intervals of sample data collection to minimize atmospheric contributions in the IR spectra. The AutoPRO software integrates easily with most commercially-available FTIR software packages.

SPECIFICATIONS

Optical Design	3X beam demagnification
Angle Of Incidence	30°
Accuracy	+/- 25 µm
Mechanical Specifications	
Repeatability	+/- 5 µm
Resolution	1 µm
Minimum Run Time	60 samples per min (without data collection)
Computer Interface	USB
Samples Focus	Z-direction adjustment
Sample Positions	60 cup positions per sampling tray
Sample Cups	Macro cups, 10 mm ID x 2.3 mm deep (micro cups optional, 6 mm ID x 1.6 mm deep)
Purge	Standard purge tubes and purge connections
Power Requirements	100-240 Volts AC; 50/60 Hz
Weight	8.2 kg
Dimensions (W X D X H)	219 x 290 x 333 mm (excluding purge tubes and baseplate)
Stage Motion	R-Theta

APPLICATION

To increase productivity and laboratory throughput, the diffuse reflection sampling is easily adapted to automated sampling. Popular applications include pharmaceutical, soil, and kidney stone analysis.



Ibuprofen and Caffeine spectra collected using the AutoDiff II accessory.

PART NUMBER	DESCRIPTION
043-29XX	AutoDiff II – Automated Diffuse reflection System Includes motion control unit (100-240 VAC), AutoPRO software 60-position sample mounting tray, 60 macro sample cups and Sample Preparation Kit
043-79XX	AutoDiff II – Automated Diffuse reflection System with Gold-Coated Optics Includes motion control unit (100-240 VAC), AutoPRO software, 60-position sample mounting tray, 60 macro sample cups and Sample Preparation Kit

Note: Replace XX with your spectrometer's Instrument Code listed in the back of the catalog.

Replacement Parts and Options

043-3090	AutoDiff II Sampling Cups, macro (60 ea.)
043-3085	AutoDiff II Sampling Cups, micro (60 ea.)
043-0902	AutoDiff II 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

AT A GLANCE

- ▶ Automated analysis with standard 24-, 48-, or 96-well plates. Special configurations available.
- ▶ Diffuse reflection of powdered samples or specular reflection 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 front-loading tray
- ▶ In-compartment mounting, compatible with most FTIR spectrometers



FULLY AUTOMATED
WITH AUTOPRO SOFTWARE

The XY Autosampler is designed around standard 24-, 48- or 96-well microplate architectures – ideal for high-efficiency sample loading and FTIR reflection analysis. Through automation, you will save time and improve consistency across measurements.

Applications include high throughput analysis of liquid residues and chemical reactions, powdered samples, and automated diffuse reflection analysis. Also, when configured with a custom sample tray, the XY may be easily converted into a precision mapping stage for IR measurements.

DESIGN

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.

XY AUTOSAMPLER FEATURES

The XY Autosampler features an X, Y stage with both axes driven by high-precision motors for speed and reproducibility. USB and DC power are the only external connections required for this accessory. The transmission option requires a spectrometer 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. With AutoPRO, custom mapping profiles to suit needs are easily generated, and data collection protocols can be saved and imported for later use.

OPTICAL DESIGN

The optical layout of the XY Autosampler is based upon a precision ellipsoidal reflector, which condenses the beam onto the sample with a power of approximately 3 times with an Angle of Incidence (AOI) striking the sample at 30°. 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. 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.



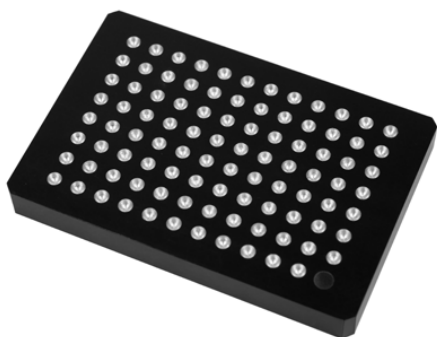
SPECIFICATIONS

Optics	Elliptical – 3X beam demagnification
Angle Of Incidence	30°
Mechanical Specifications	
Repeatability	+/- 5 µm
Accuracy	+/- 25 µm
Resolution	1 µm
Minimum Run Time	Time is spectrometer and application dependent
Computer Interface	USB
Purge	Standard purge tubes and purge connections
Power Requirements	100–240 Volts AC; 50/60 Hz
Dimensions (W X D X H)	159 x 336 x 141 mm (including micrometer)
Weight	4.5 kg

SAMPLING PLATES

The 96-well diffuse reflection plate features 96 polished cavities for placement of powder samples and small solids. For sampling convenience, an option 24-well plate for disposable cups is available for reflection measurements.

Please contact PIKE Technologies if you require specialized sampling plate configurations or a custom plate for mapping the sample



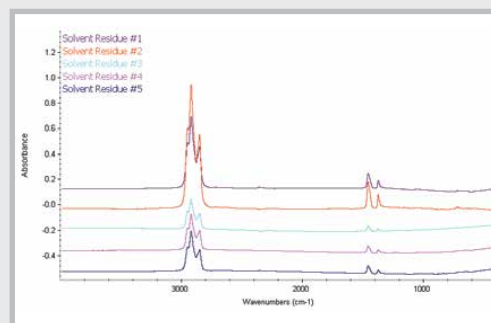
96-well diffuse reflection sampling plate.



96-well silicon transmission sampling plate.

APPLICATION

The XY Autosampler offers a flexible platform to automated diffuse/specular IR measurements. It may be configured to increase lab productivity by running several samples within a run, and also may be used as a precision mapping stage.



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

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

Notes: Replace **XX** with your spectrometer's Instrument Code listed in the back of the catalog. All XY Autosamplers include PIKE AutoPRO software and a 96-well diffuse reflection sampling plate. Diffuse reflection/Transmission versions include a 96-well plate for diffuse reflection 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	
073-9110	96-Well Diffuse reflection Sampling Plate
073-9130	96-Well Si Transmission Sampling Plate
073-9160	24-Well Diffuse reflection Sampling Plate for Disposable Cups
162-1920	Disposable Cups (50 ea.)

Sample Prep Kit

AT A GLANCE

- Convenient sample preparation tools for diffuse reflection measurements
- Micro and macro sampling cups
- Sampling cup holder to enhance loading reproducibility

The Sample Preparation and Loading Kit makes the handling of powder samples for diffuse reflection sampling easy.

This kit 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 cm³ 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.

PART NUMBER	DESCRIPTION
042-3040	Sample Preparation and Loading Kit Includes 2 micro and 2 macro cups, alignment mirror, sampling cup holder and base, 2 spatulas, brush
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, specular
042-3082	Alignment Mirror, gold, specular
042-3084	Alignment Mirror, gold, diffuse
042-3070	Camel Hair Brush
042-3035	Spatula, spoon
042-3050	Spatula, flat
160-8010	KBr Powder (100 g)
161-5050	Agate Mortar & Pestle 50mm
Note: The Sample Preparation and Loading Kit is included with EasiDiff, DiffusIR and AutoDiff diffuse reflection accessories.	



Abrasion Sampling Kit

AT A GLANCE

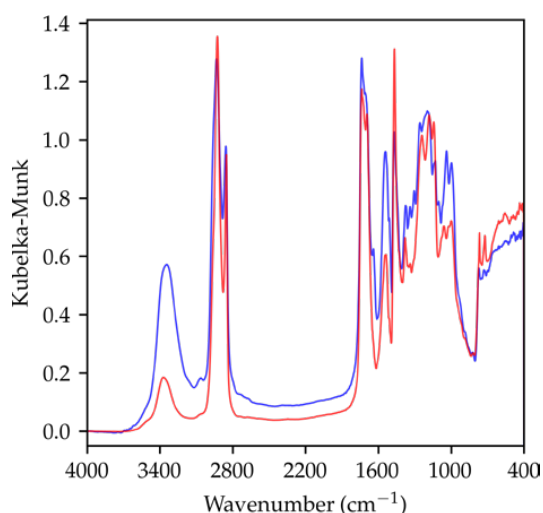
- Convenient set of tools for collection of difficult solid samples
- Rigid construction, diamond and silicon carbide disks
- Sampling post fits the EasiDiff, DiffusIR and AutoDiff accessories

The abrasion method for diffuse reflection sampling is particularly useful for the analysis of large painted surfaces (e.g. car panels) and other awkward objects, which makes measuring by traditional techniques difficult.

The Abrasion Sampling Kit consists of a sample collector tool with a set of silicon carbide 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 reflection spectrum of the material is collected.

The abrasion kit is compatible with most PIKE diffuse accessories.

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



Spectra of polymer outer cover of two brands of golf balls.



Brushing a silicon carbide (SiC) disk against a golf ball.



Diffuse Reflection

Theory and Applications

Diffuse reflection is an excellent sampling technique 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 in the mid-infrared, often samples to be analyzed by diffuse reflection are generally ground and mixed with an IR transparent salt such as potassium bromide (KBr) prior to sampling.

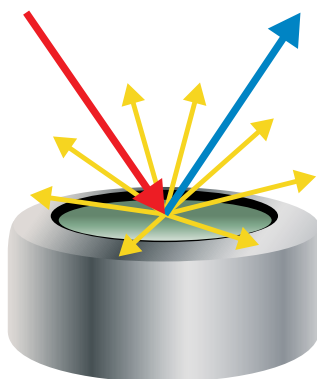
Diffuse reflection sampling offers an excellent alternative to transmission of KBr pellets as it eliminates the time-consuming process of pressing pellets. This technique can also be used to study the effects of temperature and gas flow for the analysis of catalysts and other diffusely scattering samples by configuring the accessory with a heating or cooling environmental chamber.

One of the greatest additional benefits of diffuse reflection 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 reflection 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 REFLECTION WORKS

Diffuse reflection relies upon the focused projection of the spectrometer beam into the sample where it is reflected, scattered and transmitted through the sample material. 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 diffuse reflection.

Incident Radiation
Specular Reflection
Diffuse Reflection

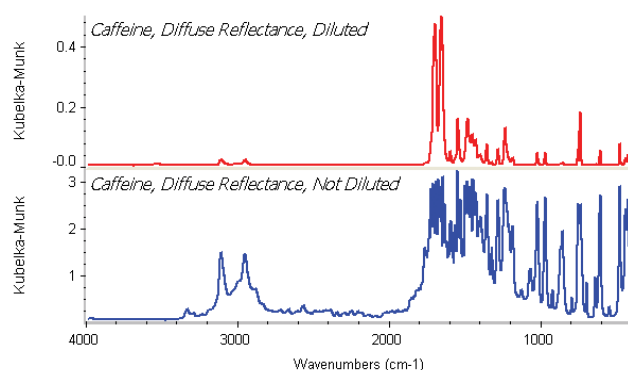


FACTORS THAT INFLUENCE SPECTRAL QUALITY

Some powders may be analyzed by diffuse reflection 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 reflection component in diffuse reflection 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 reflection 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 reflection spectra showing greatly improved results from sample dilution.

Other factors related to high spectral quality for diffuse reflection 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 reflection contributions (spectra of highly reflecting samples will be more distorted by the specular reflection component). This effect can be significantly reduced by sample dilution.
- ▶ **Homogeneity** – samples prepared for diffuse reflection 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 reflection 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.

THE KUBELKA-MUNK EQUATION

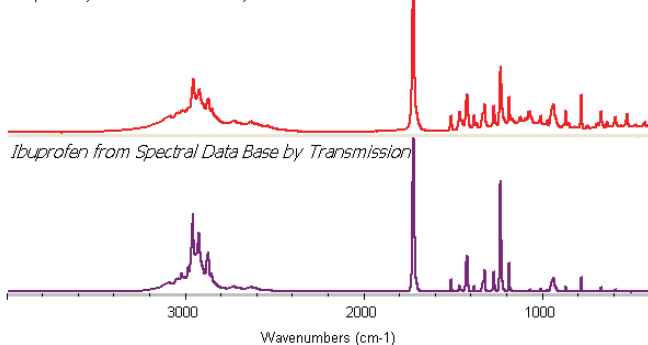
Even with all these sample preparation practices, the raw diffuse reflection spectra will appear different from its transmission equivalent (stronger than expected absorption from weak IR bands). A Kubelka-Munk conversion can be applied to a diffuse reflection spectrum to compensate for these differences.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

Ibuprofen, Diffuse Reflectance, Kubelka-Munk Converted



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

The spectra shown above demonstrate this spectral conversion for ibuprofen collected by diffuse reflection. 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 reflection spectroscopy can provide ppm sensitivity and high-quality results.

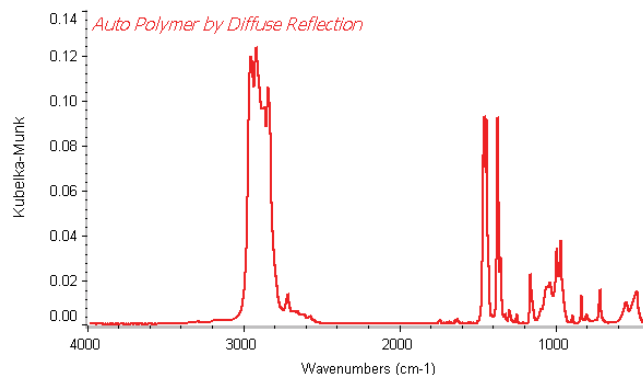
PLASTIC BUMPERS AND TOUGH SAMPLES

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 reflection 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 reflection accessory.

The figure below shows the diffuse reflection 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 reflection 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 reflection spectrum using diamond abrasion disk.

POWDERS

For the analysis of powders the following procedure is recommended:

- ▶ Place about 200–400 mg of KBr into the ShakIR ball mill 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 reflection 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 reflection accessories make the analysis of a wide range of solid samples easier, faster and more efficient. Advanced options for diffuse reflection 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.