
Installation and User Guide



AutoDiffusIR™

Automated Diffuse
Reflectance Accessory

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INTRODUCTION

The PIKE Technologies AutoDiffusIR™ is high performance automated diffuse reflection accessory which is available for most FT-IR Spectrometers. The design employs a high efficiency fixed ellipsoidal reflector to collect the maximum amount of diffusely reflected energy from the sample. Operating under program control from within the FTIR software, the data generated may be processed either sequentially after each scan or stored to disk for further analysis.

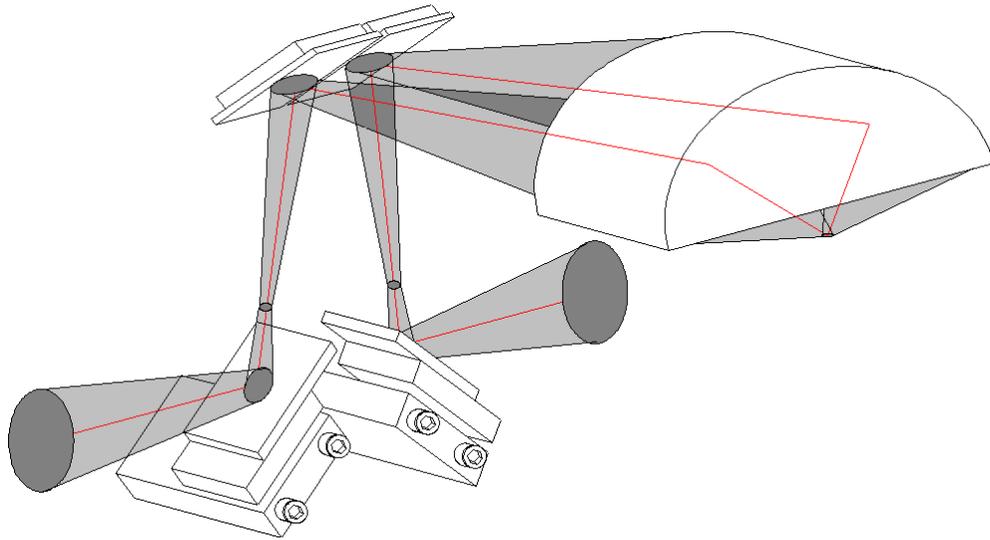
The sample holder contains positions for sixty samples. Also, in the center of the sample holder is a position for a background sample. This background sample usually consists of pure KBr powder. The sample holder is marked into six areas, labeled from A to F. Each area has ten sample positions marked from 1 through 10. This sample position numbering scheme is also used within the AutoPRO software for describing and positioning the samples.

Focusing of the sample to coincide with the infra red beam is by means of a focusing adjustment knob, located under the sample holder. This focussing adjustment need only be performed for the central background sample. All other samples will then be in focus.

The samples are moved automatically into position in the infrared beam by means of the Motion Control unit and AutoPRO software. An R-Theta motion is used by the accessory for positioning each sample in turn in the infrared beam.

OPTICAL DESCRIPTION

The optical system of the AutoDiffusIR is symmetrical with identical mirrors being used for the optical path from the spectrometer interferometer to the sample and the sample to the spectrometer detector. Two flat mirrors and an ellipsoidal mirror are used in each path. A drawing of the optical path is shown below.

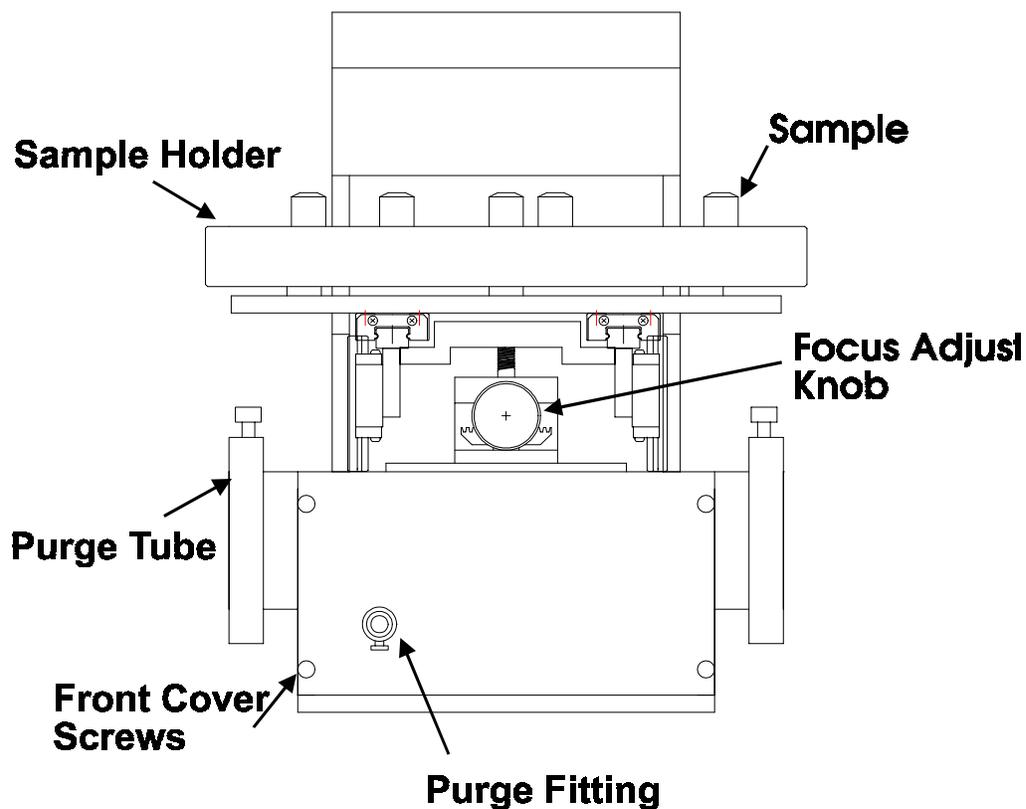


The two lower mirrors are adjustable and are used for aligning the optical system at installation. The upper flat mirrors and ellipsoid mirror are fixed in position. The ellipsoid is a large solid angle monolithic mirror which condenses the beam onto the sample with a power of 6 times. If the beam in your spectrometer sample compartment with no accessory in place is 9 mm in diameter, the size of the focussed spot at the sample in the AutodiffusIR is 1.5 mm.

Note that the size of the beam in the spectrometer is dependent on manufacturer. Refer to your spectrometer user guide for the correct size.

INSTALLATION AND ALIGNMENT

Inspect the drawing below and locate the indicated items on your accessory.



AutoDiffusIR Front View

To install the accessory in your sample compartment perform the following:

PREPARE THE SPECTROMETER

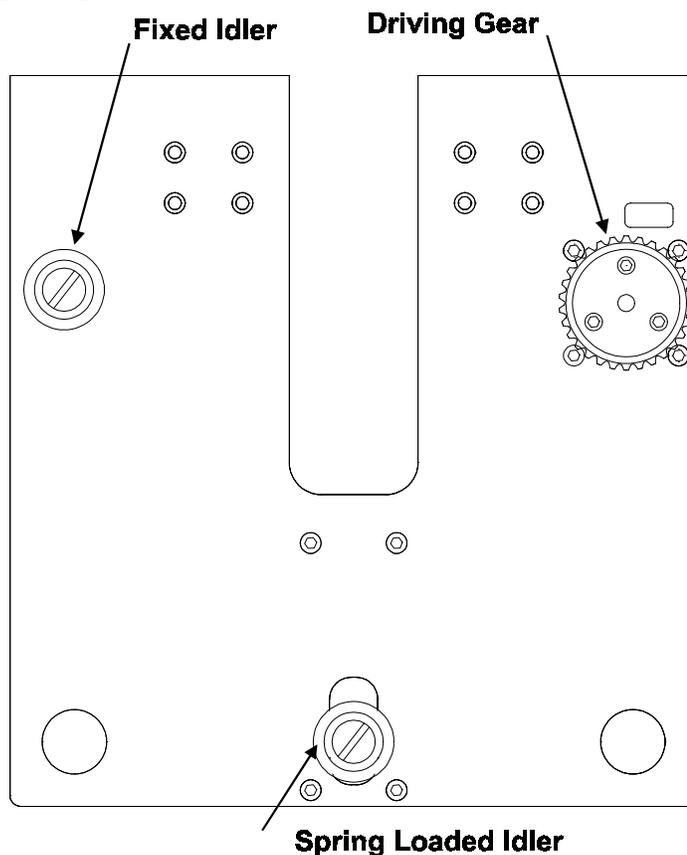
- Remove any sample holder present in the sample compartment.
- Scan a background with your FTIR software and save to disk. This single beam spectrum can be used to verify the performance of the system.

PREPARE THE ACCESSORY

- If your accessory is equipped with the instrument's manufacturer standard baseplate, remove the original sample compartment baseplate from the spectrometer. Place the accessory in the sample compartment and secure it with screws used for baseplate attachment.
- If the accessory uses a sub-plate that attaches to the instrument's original sample compartment baseplate, remove the lower front cover of the accessory first. Place the autosampler in the sample compartment of the spectrometer and position it on the baseplate pins. Find a captive thumbscrew located on the bottom plate of the accessory and tighten it securely. Reinstall the front cover.
- Using a 3/32" wrench, loosen the screws on the two purge seal tubes on the sides of the accessory. Push the tubes toward the side walls of the spectrometer. This will seal the accessory and ease future placement of the accessory in the sample compartment.

LOAD THE SAMPLE HOLDER

- Place the sample holder in the accessory. In order to do this refer to the drawing of the sample stage below.

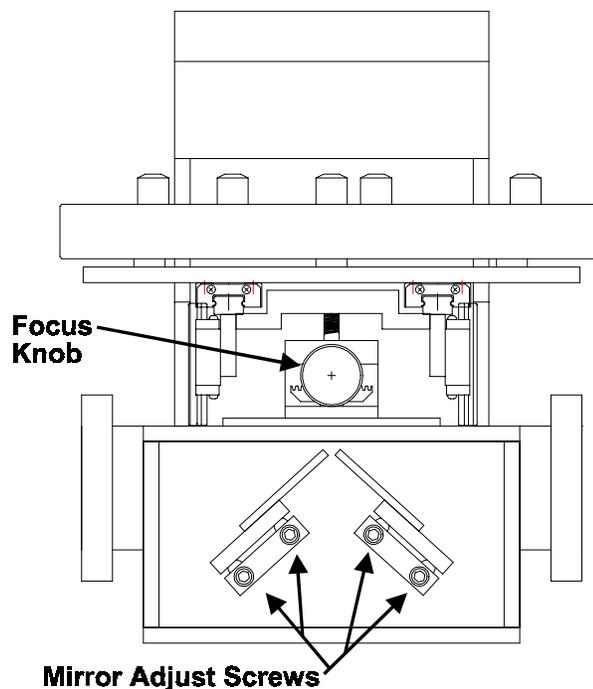


ACCESSORY SAMPLE STAGE

- The accessory sample stage contains:
 - A driving gear on the right hand rear of the sample table.
 - A fixed idler on the left hand rear of the sample table.
 - A spring loaded idler at the front center of the sample table.
- Holding the sample holder horizontally, slightly tilt the holder, so the left part of the holder is approximately one inch higher than the right part of the holder.
- Place the sample holder over both the spring loaded idler and driving gear. Make sure that the sample holder is completely over the driving gear, and the gear is located in the “V” shaped portion on the underside of the holder.
- Push backwards to compress the idler spring. You may need to hold the sample stage to prevent it moving backwards and two holes are provided in the front of the sample stage to help in doing this.
- Rotate the sample holder so that it is horizontal and fits over the fixed idler.
- Gently release the sample holder so that it is located on all three supports. The sample holder is now in place in the accessory.

ALIGN THE ACCESSORY

- Place the alignment mirror (contained in the Easi Prep Sample preparation kit) into the center position. Note that this alignment mirror is provided with a peelable protective coating. Follow the instructions in the sample preparation kit to remove this coating.
- Remove the four thumbscrews holding the lower front cover in place and remove the cover. Behind this cover, there are two adjustable mirrors which are used for alignment.



- Set up the FTIR software to alignment or monitor mode and note the size of the interferogram. Turn the focus adjust knob to raise and lower the sample holder to achieve maximum throughput.
- Adjust the output adjustable mirror. This is the mirror that is the closest to the instrument detector. Turn both screws on this mirror mount to maximize throughput.
- Adjust the focus knob to maximize throughput.
- Adjust the input adjustable mirror. This is the mirror that is the closest to the instrument interferometer. Turn both screws on this mirror mount to maximize throughput.
- Adjust the focus knob to maximize throughput.
- Repeat the last four steps until there is no further increase in signal. Typically, you should achieve a signal throughput which is at least thirty percent of the signal that was present before the accessory was placed into the sample compartment.

Replace the lower front cover and fix in place with the four thumb screws.

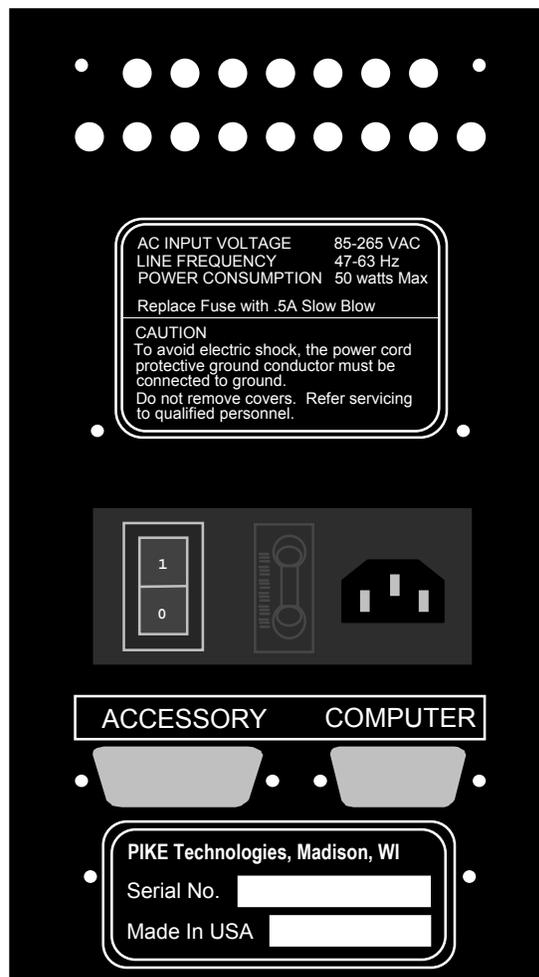
INSTALLING THE AUTOPRO MOTOR CONTROLLER

The motion control electronics interfaces AutoDiffusIR to your computer. Commands are sent to this electronics unit using RS 232. A 15 wire ribbon cable is used to connect this unit to the accessory.

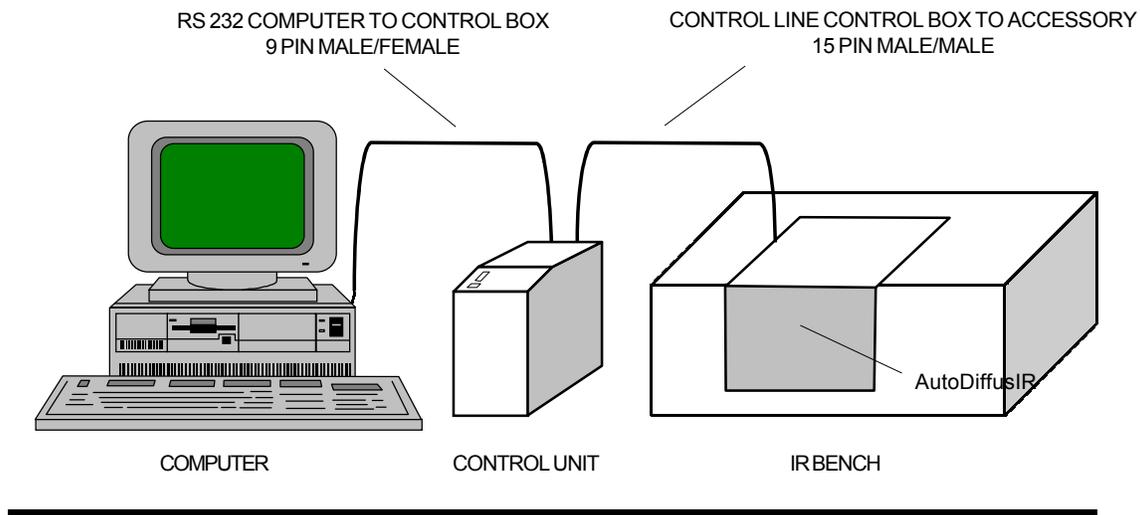
CONNECTING THE MOTOR CONTROLLER TO YOUR COMPUTER.

The power supply for the motor controller is self adjusting and can be used in most locations. Please read the labels on the rear of the motor controller before attempting to connect the system. The power for the motor controller should be turned off when attaching the cables.

Motor Controller Rear Panel



The correct cabling procedure for the accessory is shown below.



- The accessory port of the motor controller should be connected to the accessory through the six foot, male-to-male 15 pin ribbon cable provided.
- The serial RS 232 connection from the COMPUTER port is made with a female-to-male, molded 9 pin cable which is also provided. This cable should be connected to a serial port (COM1 or COM2) of your PC compatible computer.

INSTALLATION OF THE AUTOPRO SOFTWARE

SYSTEM REQUIREMENTS

AutoPRO is a Microsoft Windows compliant program. The program was designed to run within Windows 95 or higher. It is recommended for Pentium based computers with CD-ROM and VGA monitors. A Microsoft - compatible mouse is required to access all of AutoPRO's features.

LOADING FROM WINDOWS

- Step 1 Insert the program disk into the CD-ROM Drive of your computer.
- Step 2 From within the Program Manager, select **Run...** from the **File Menu**.
The **Run** dialog box will appear to enter a filename.

You may either enter: **x:setup.exe** or "browse" to the CD Disk directory and select **setup.exe**.



A setup dialog box will appear for a few moments while the installation program checks for available memory and configuration.

The AutoPRO dialog box will appear:

- | | |
|---|-----------|
| Choose to use the default path and select: | Enter, or |
| Choose to enter an alternative path and then select | Enter, or |
| Choose to exit Setup by selecting | EXIT |

The software will be copied. The source and destination files, and the percentage of the completed task is displayed. A dialog box will appear when the program has been loaded. Click on the OK box to complete installation.

FILES PLACED ON YOUR HARD DISK

During installation the following files are placed on your hard drive in the AutoPRO Directory

AP5.exe	AutoPRO Control
AP5.hlp	AutoPRO Control help file
APV.exe	AutoPRO/VeeMaxprogrammer
APV.hlp	AutoPRO/Veemax help file
APW.exe	AutoPRO/Wafer programmer
APW.hlp	AutoPRO/Wafer help file
APD.exe	AutoDiffusIR programmer
APD.hlp	AutoDiffusIR help file
AP5.wri	File containing latest information
Motor.exe	Remote Control Software
init.exe, newfile.exe unload.exe, load.exe next.exe, point.exe	} System Control executable files

In the Windows/System Directory

mscomm32.ocx	oleaut32.dll	comctl32.ocx
ccmouse.ocx	mfe40.dll	comdlg32.ocx
commdlg32.dll	ctl3d32.dll	roboex32.dll
comcat.dll	msflxgrid.ocx	
olepro32.dll	sysinfo.ocx	

ON LINE HELP

AutoPRO provides on-screen help for commands and functions. More information on the general attributes of the help screens may be found in the AutoPRO Manual.

How to use Help

Choose Help from the menu, or press F1 on the keyboard.

Index	Displays an index of Help topics including menus, commands, and shortcuts.
Using Help	Provides information on how to use Windows Help.
About...	Provides specific information on the version AutoPRO and current system information.

You can use the Help buttons to display related Help topics. Options are:

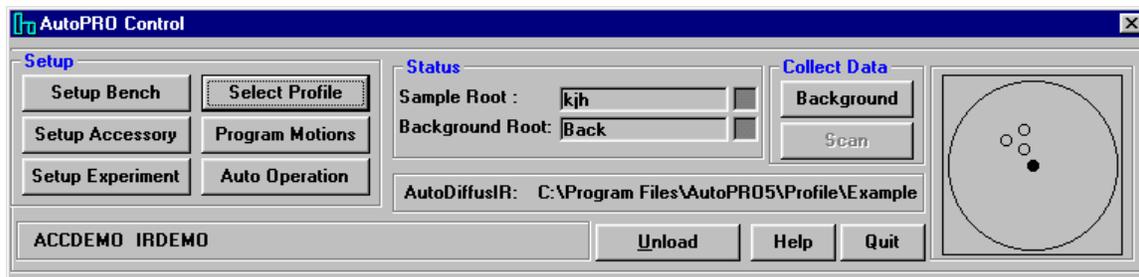
Contents	Displays a list of Help topics.
Search	Lists the keywords for AutoPRO. Enter a keyword or phrase in the Search For text box or select a keyword from the list box.
Back	Displays the last topic you displayed.
History	Displays a list of recent topics displayed.
Glossary	Displays a list of terms and parameters used in AutoPRO and their definitions.

AUTOPRO OVERVIEW

AutoPRO is a Windows based automation software program for use with PIKE automated accessories and the AutoPRO motion controller. With this software package a range of automated accessories may be programmed and operated in conjunction with most Windows based FTIR software packages. Several programs comprise the complete software package, but the following two programs are central to the function of the software and will be introduced briefly here.

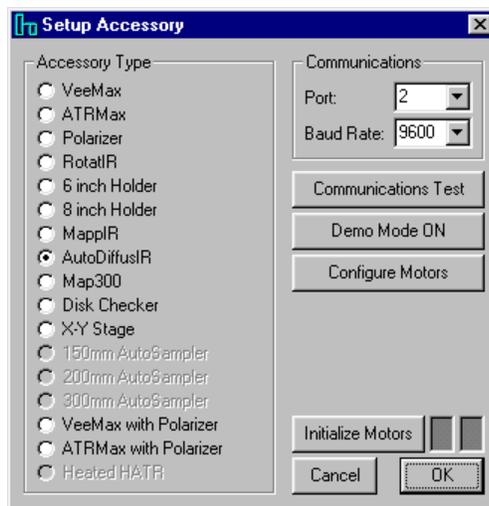
AUTOPRO CONTROL

This program contains the tools required to operate your automated accessory



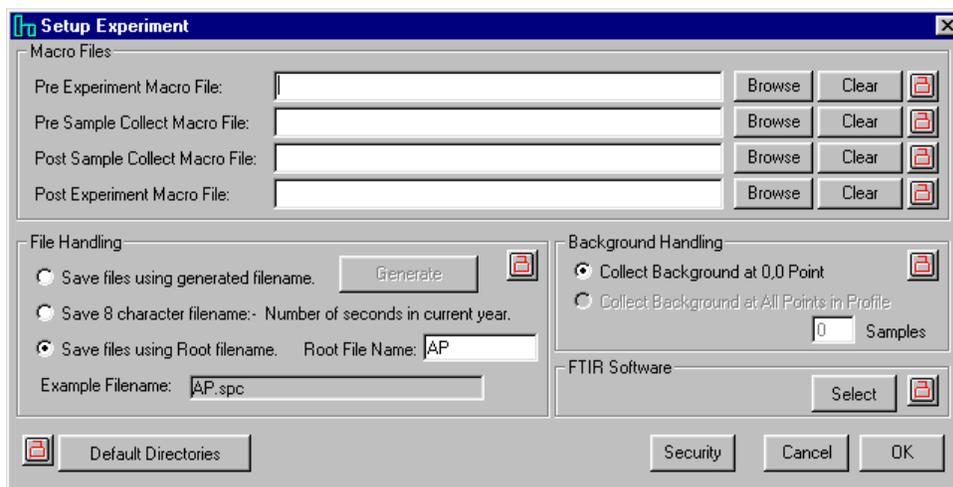
The AutoPRO control panel contains the following major functions:

- Setup Bench** A basic parameters for data collection (number of scans, resolution, etc.) may be defined and stored to a file.
- Setup Accessory** This function allows selection of the actual accessory used with the spectrometer, computer/accessory communication test and basic accessory setup.



Setup Experiment

Macros and executable files can be integrated into the autosampler routine. This function also allows special handling of multiple filenames and provides different security options.



Program Motions

A series of samples may be defined and stored to a file. This file may be subsequently used to move your accessory while collecting data from your spectrometer.

Load and unload

The AutoDiffusIR may be moved to an unload position to simplify the insertion of new samples.

Status

The status of the accessory at any time is displayed. This includes the position of the accessory, the current status of the motors and a thumbnail view of the file being used for programming the motion.

These and all other functions are described fully in the AutoPRO manual.

MOTOR . EXE

This is a program which may be inserted into an FTIR macro. With this program the basic functions required to run the accessory may be accessed from within the macro. While the software is running a small status box is displayed in the lower right hand corner of the display.

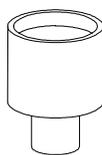
More details of how to use this and other .exe files available in the AutoPRO software are given in the User's Manual.

SAMPLE PREPARATION

The AutoDiffusIR 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

Sixty large sample cups are provided. The large sample cup has a 10 mm diameter by 2.3 mm deep rebate to hold the sample.



Sampling Cup

PREPARING THE SAMPLE

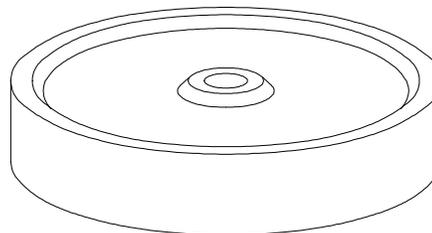
The sample to be analyzed must be diluted in a transmitting matrix. Place the sample in the mortar and grind finely using the pestle provided. Add KBr powder to the sample. With the pestle, mix the sample with the KBr powder so that the sample particles are small and evenly dispersed. The sample should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

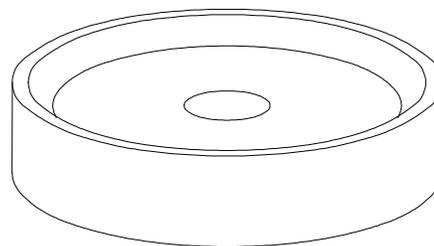
AutoDiffusIR Sample Preparation Base

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



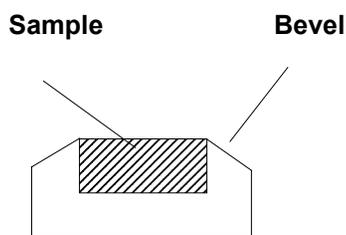
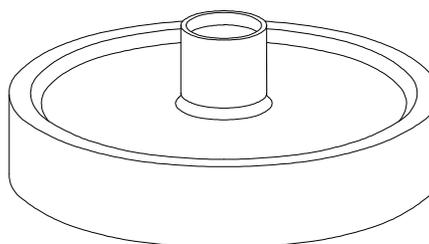
AutoDiffusIR Sample Funnel

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.

LOADING SAMPLING TRAY

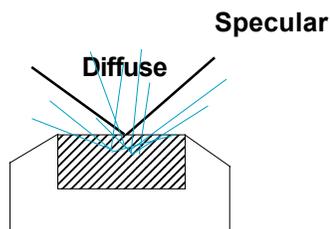
With the sample cup filled, place it into the holder. Repeat for all remaining samples to be analyzed.

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.

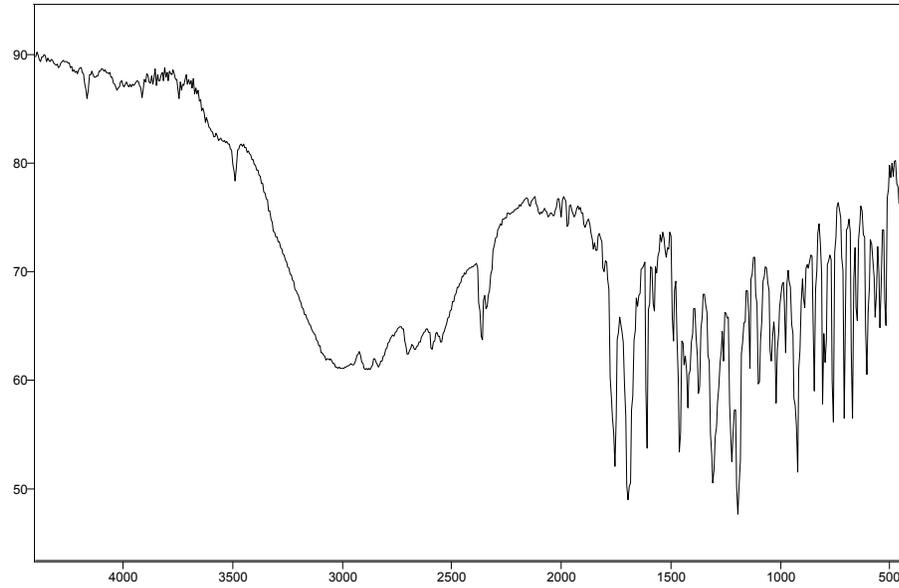
Samples may also be analyzed without dilution. For samples that are not powders, the sample may be abraded with a piece of silicon carbide. The Abrasive Sampling Kit (ASK) is available from PIKE Technologies. For this technique a background spectrum is taken of the clean silicon carbide paper prior to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

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

$$=2.3 \text{ ac/s}$$

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.



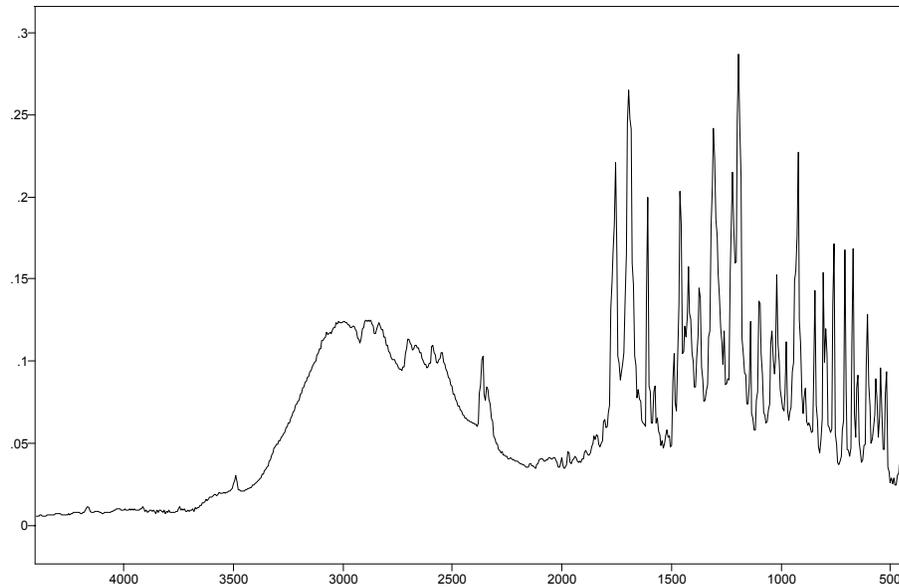
Transmittance / Wavenumber (cm-1)

Number of Scans= 4 Apodization= None

File # 2 = ASPRIN5

3/9/94 4:58 PM Res=4 cm-1

Raw Transmittance Data, Aspirin



Kubelka-Munk / Wavenumber (cm-1)

Number of Scans= 4 Apodization= None

File # 1 : ASPRIN5

3/9/94 4:58 PM Res=4 cm-1

Kubelka-Munk Transformed Data, Aspirin

USING YOUR ACCESSORY

In order to explain the operation of the accessory, an example experiment will be described. Three samples will be analyzed with the accessory. To perform the experiment, the following steps must be performed.

- Load the samples.
- Set up AutoPRO.
- Enter data on samples into AutoPRO and load sample into the sample holder.
- Collect the Data.

LOAD THE SAMPLES

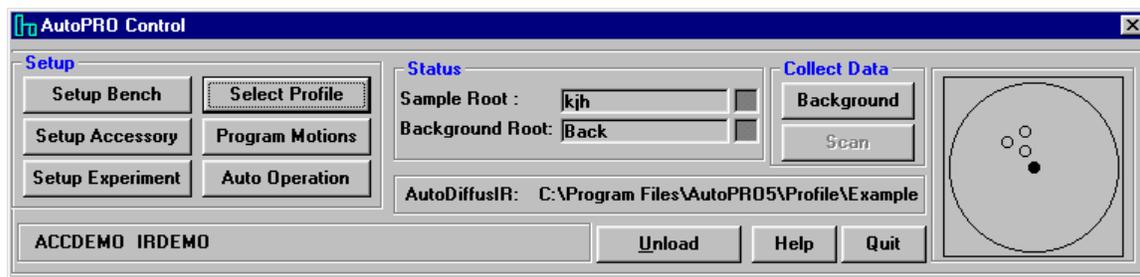
This step has already been described in the previous section of this manual. Besides loading the three samples, you should also prepare a sample of neat KBr to be used for the background.

SET UP AUTOPRO

Decisions need to be made prior to setting up AutoPRO. The descriptions of five fields needs to be decided upon. For this example we will use the following descriptions.

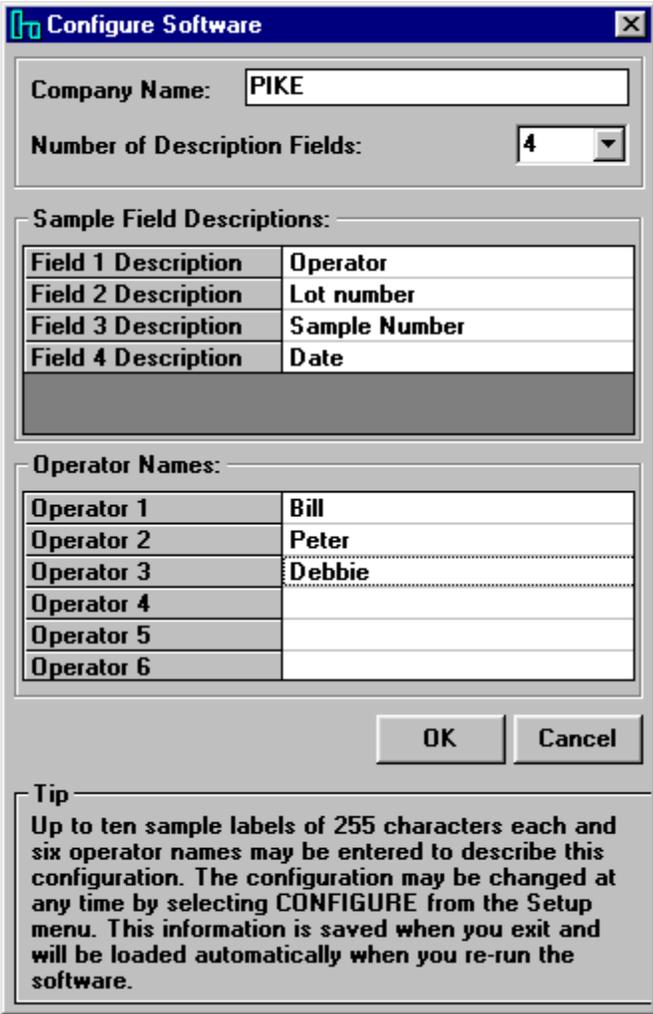
- Operator
- Lot Number
- Sample Number
- Date

Click on the AutoPRO program Icon. The following Screen will appear.

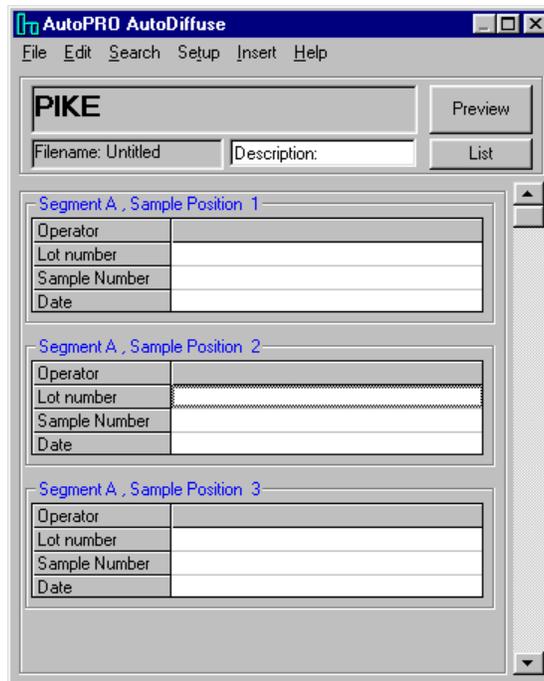


Click on the Program Motions button. The AutoDiffusIR Programmer will open. If you are opening this program for the first time, the configure screen will open. If you have previously configured AutoPRO and you wish to reconfigure, then click the Configure item in the Setup Menu.

The following screen will appear. In this view, the fields have already been entered.

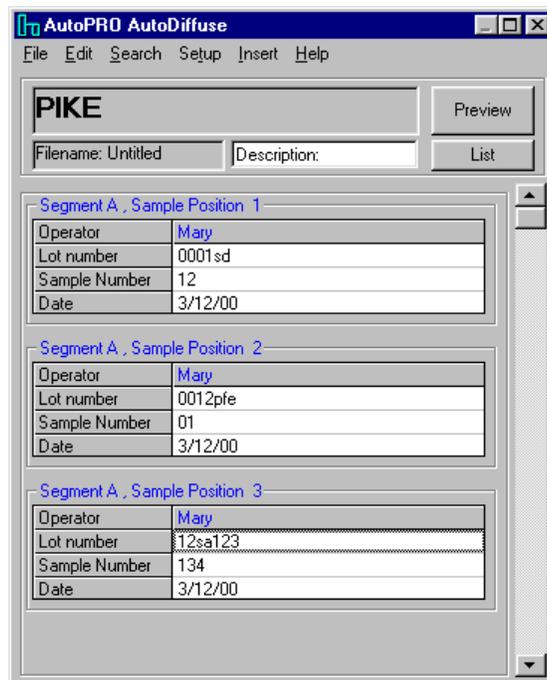


When the OK button is clicked, the AutoDiffusIR Programmer will be updated with the new descriptions.

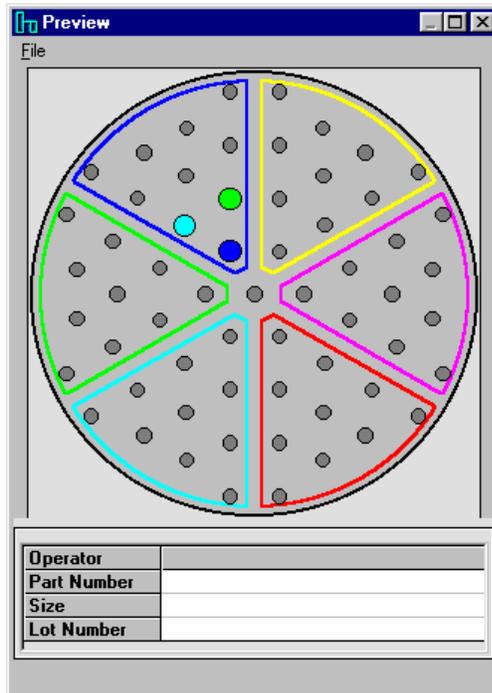


LOAD THE SAMPLE INFORMATION

The fields may now be filled in. In the following view, three fields have been entered with information about the samples.



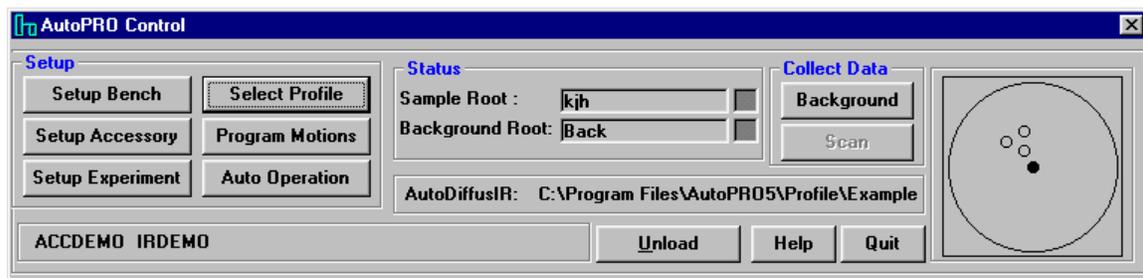
If preview is clicked, a pictorial view of the sample holder is shown, to aid in the placing of the samples.



Click on File and select exit, and then save the information to a file. In this case the filename chosen is **Example.APD**

The programming is complete and the Programmer may be exited.

Double click on the thumbnail sketch in AutoPRO control. You will be asked to select a filename. When this is done, the display will be:



This shows the three samples and the central background sample of neat KBr.

COLLECT THE DATA

To collect data on these samples, please consult the AutoPRO users manual Appendix for the instrument that you are using. The mode of operation varies from one instrument to another.

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

AutoDiffusIR - Automated Diffuse Reflectance System, Part Number 043-28XX:

AutoDiffusIR - Autosampler Unit	1
Autosampler 60 Position Tray	1
Sample Preparation Kit	1
Set of Sampling Cups	60
Motor Control Unit	1
9-pin Male/Female Computer Cable	1
15-pin Male/Male Accessory Cable	1
AutoPRO Software	1
User Manuals	2

AutoDiffusIR - Optional Components:

043-3090	AutoDiffusIR Set of 60 Sampling Cups (macro)
042-2020	Large Sample Cup (macro), two per package
042-3010	Abrasion Sampling Kit
042-3020	Abrasion Sampling Disks (Silicon Carbide pack of 100)
042-3025	Abrasion Sampling Disks (Diamond Grit pack of 50)
042-3080	Alignment Mirror, Aluminum Coated
042-3090	Alignment Mirror, Gold Coated