COLLECTOR™ *II*

User's Manual

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Thermo Spectra-Tech

A Thermo Electron business

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General Information

The Manual

This manual is designed as a tutorial to guide you through the installation of Collector II and through a typicalanalysis. If you have any questions, please contact a Thermo Spectra-Tech Technical Representative.

Packing & Unpacking

The Collector II is shipped in a protective foam filled cardboard box. Upon arrival please check the box to ensure that all pieces have been received and that no pieces are damaged. Save the box for storage and shipment of the kits.

Technical Support Center

Technical materials describing the use and theory of attenuated total reflectance, diffuse reflectance and specular reflectance are available from Thermo Spectra-Tech. Additionally, a team of scientists is available at Thermo Spectra-Tech to answer any of your questions. If you encounter any problems or difficulties, or desire additional information please contact the Technical Support Center at 800-THE-FTIR.

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Banner

Introduction to Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS)

Diffuse reflectance spectroscopy is a powerful technique for infrared analysis of fine particles and powders in concentrations that range from undiluted to parts per thousand (0.1%).

Analyses are fast and easy because little or no sample preparation is required. Powder samples can be measured “as is” or mixed with a diffusely scattering matrix such as potassium bromide (KBr). Solids can be ground and deposited on a particle matrix then measured as a powder.

The Si-Carb™ Sampler, a special tool can be used to analyze large, intractable solids by scratching the sample surface with an abrasive paper and then measuring the particles adhering to the paper.

The high throughput and signal-to-noise ratios of FT-IR spectrometers, in combination with on-axis diffuse reflectance accessories, have made diffuse reflectance a powerful infrared sampling technique. Currently, DRIFTS (Diffuse Reflectance Infrared Fourier Transform Spectroscopy) offers a number of advantages as a sampling technique, including:

- Minimal or no sample preparation.
- Very high sensitivity (down to low ppm levels).
- Ability to analyze most non-reflective materials, including highly opaque or weakly absorbing materials.
- Ability to analyze irregular surfaces or coatings, such as polymer coatings.
- Ability to analyze very large, intractable samples.

For a complete description of the theory of Diffuse Reflectance please refer to Spectra-Tech's FT-IR Technical Note #2: Introduction to Diffuse Reflectance Infrared Fourier Transform Spectroscopy.

COLLECTOR II Product Description

The Spectra-Tech Diffuse Reflectance (DRIFT) Accessory, “The COLLECTOR II” is designed for use in FT-IR spectrometers. The design employs 4 flat and 2 aspherical reflectors, plus an alignment mirror. The aspherics are off-axis ellipsoids which focus and collect infrared energy with a 6X condensation of the beam. A typical FT-IR beam has from a 3mm to 18mm spot size at the focus, so the spot size with the DRIFT accessory will be between 0.5 and 3.0mm. The collection angle is a full pi steridians, collecting 50% of the available diffuse energy. The Spectra-Tech COLLECTOR II has been designed for high energy throughput and ease of use. Access to the sample is from the top, by sliding the ellipsoids out of the way. In addition, a mechanism is provided to slide the sample cup forward to remove it without sliding open the ellipsoids for additional convenience. This can be useful for repetitive sampling situations. The sample cup is removable for filling, and the sample height is fully adjustable. Figure 1 is an optical layout of the COLLECTOR II, showing the beam path through the accessory.

In order to deal with the “specular component,” which can cause spectral distortions especially in neat, inorganic samples, a BLOCKER device is provided. This device is described fully on page 5.

CAUTION: Please note that all mirrors are uncoated (bare) aluminum. Care must be taken to avoid damaging or smudging these reflective surfaces, as they are relatively soft and difficult to clean without scratching.

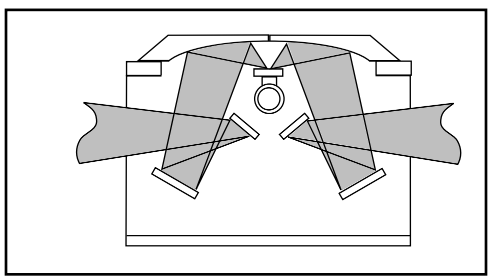


Figure 1 Optical Layout of the COLLECTOR *II*

BLOCKER Description

For certain samples, it might be desirable to eliminate the component of the reflected energy which is not diffused by scattering - commonly called the specular component (reflectance from a mirror is pure specular). While specular reflectance occurs over all angles, the COLLECTOR II has a provision for lowering it's contribution to the spectrum - the BLOCKER.

It is not always obvious when the specular component should be excluded. Inorganics or other samples of high index of refraction might be highly specular, and the BLOCKER would be used. But one might want to first try grinding the sample to a smaller particle size, or running the sample in a KBr matrix. It is rarely necessary to exclude the specular component with KBr diluted samples. In general, if the specular component is excluded, signal-to-noise will suffer and longer scan times will be required. An MCT detector might even be indicated to improve the signal-to-noise ratios. The high throughput advantage of the COLLECTOR II over other DRIFT designs results partly from the large collection angle which can include the specular energy. In general, don't remove this component unless you must.

Normally, the BLOCKER must be positioned so that the blade is contacting the surface of the powder but not embedded deeply into the powder (see Figure 2, below). The powder should be level with the surface of the cup. Overfill the cup with powder, then scrape a spatula across the top of the cup (see page 20).

After the cup is filled properly, the BLOCKER should be positioned so that it rests firmly on the sample cup. If it does not rest firmly on the cup, loosen or tighten the screw on top of the BLOCKER while pushing down on the blade.

Energy throughput is significantly reduced when using the BLOCKER. This is due to several factors. Most notably, samples which are suspected of containing specular distortions (Restrahlen bands) generally are poor diffuse reflectors. Although they may exhibit high energy throughput without the BLOCKER, most of this is specular energy. At best, this merely dilutes the spectrum. At worst, the aforementioned distortions result, potentially leading to spectral misinterpretation.

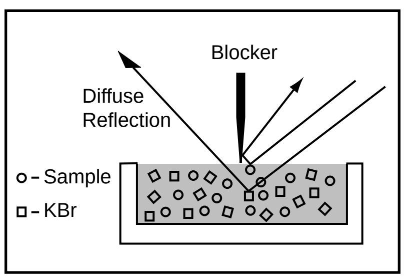


Figure 2 *BLOCKER*

Diffuse Reflectance Sampling Technique

Specular Reflection

When the infrared beam is focused on the surface of the particulate sample, it can interact with the sample two ways. First, it may simply reflect off the sample surface in the same way visible light reflects off a mirror. The phenomenon, called “true specular reflection,” is a function of the refractive index of the sample.

The diffuse reflectance accessory collects infrared radiation from true specular reflection along with other reflected light. However, the specular component of the infrared light reaching the detector is considered an interference, because it does not provided information about the absorption characteristics of the sample.

Diffuse Reflection

The second way the infrared beam may interact with a particulate sample is to penetrate a particle then scatter or “diffuse.” Depending on the angle at which it emerges from the particle, the diffused light may penetrate other particles as it moves through the sample material or it may simply reflect off their surfaces. Diffused light that travels through and is partially absorbed by particles of the sample material, contains information about the absorption characteristics of the sample.

Particles that are highly absorbing may produce totally absorbing peaks in the resulting spectrum because little energy is released from the sample. Highly absorbing material can be mixed with a diffusely scattering matrix such as KBr, which lowers absorption and improves throughput.

This explains the origin of the term “diffuse reflection” and the primary purpose of the diffuse reflectance accessory.

General Information

Diffuse Reflectance Sampling Technique

Diffuse Reflectance Spectrum

The goal of diffuse reflectance spectroscopy is to maximize the diffused component, because it contains information about the absorption characteristics of the sample, and to minimize the specular component, which causes distortions in the data.

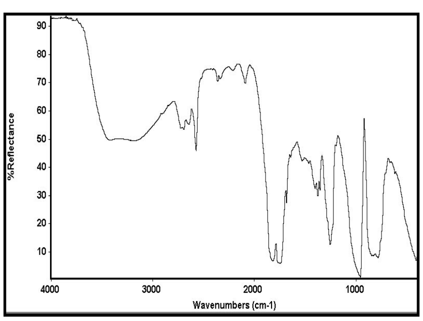
Since it is difficult to determine the absorption characteristics and the refractive index of a powdered sample just by looking at it, the recommended approach is to try running the pure powder using a KBr background. If distortions or totally absorbing bands appear in the data or if the sensitivity is low, you can mix the sample with KBr at the recommended ratio and collect the spectrum again.

Totally Absorbing Bands

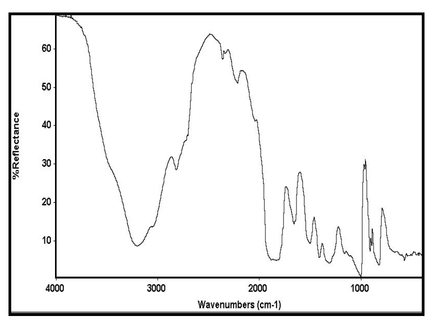
Bands with values below 1% R (above 49 Kubelka-Munk units) are considered totally absorbing and normally have a flattened or "squared off" end.

Derivative-shaped Bands

One relatively common effect in diffuse reflectance spectroscopy are bands that are inverted or derivative-like in shape. These distortions called Restrahlen bands are caused by specular reflection off the sample surface.



*Potassium permanganate undiluted, with **derivative-shaped bands** at 919 cm-1.*



*Potassium dichromate undiluted with **totally absorbing** bands.*

Sample Compartment Baseplate Removal

All Collector II's are pre-aligned and tested in a FT-IR spectrometer prior to shipment. Additionally, most versions of the Collector II are shipped with the Baseplate pre-attached to the accessory for rapid and reproducible installations (as illustrated below). This baseplate attaches to the floor of your spectrometer sample compartment and provides properly positioned mounting holes for the Collector II. Therefore, only minor adjustments to the mirrors should be required to maximize energy throughput. Read all installation and alignment instructions before proceeding with any adjustments to the optics of your Collector II.

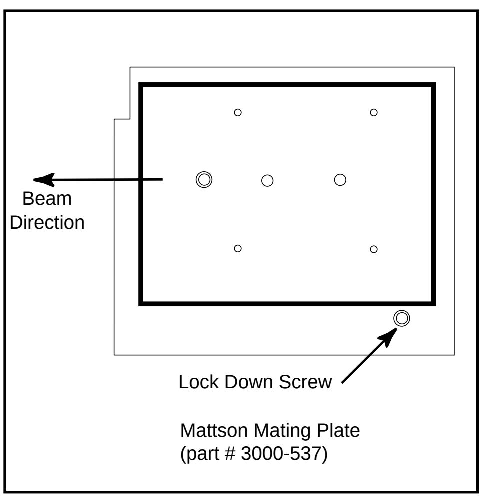
Remove the baseplate

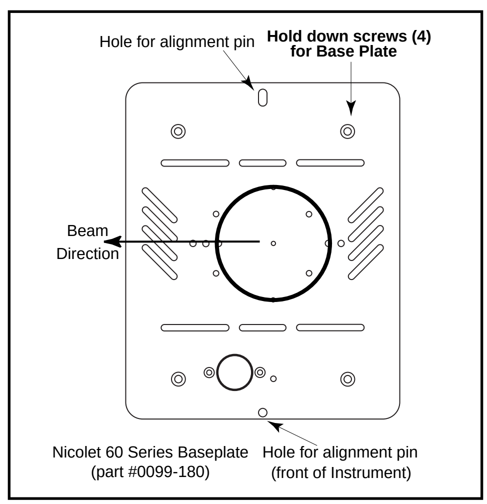
Locate the four (4) shipping hold down screws (8-32) on the Baseplate of your spectrometer (if not already removed).

Remove the 4 screws with a slotted head screwdriver.

Remove the Baseplate completely from the sample compartment.

Note: The accessory with pre-attached baseplate is oriented the same way as the sample compartment baseplate was before removal.



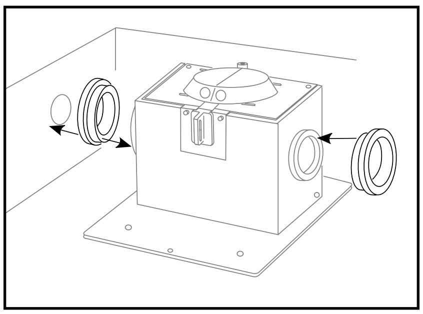


COLLECTOR II Installation

Record the open beam energy

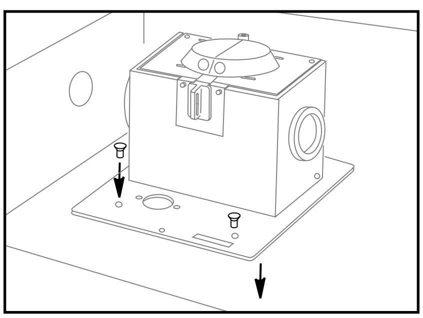
Record the open beam energy of your spectrometer by monitoring either the throughput energy number or the height of the centerburst of the interferogram.

Note this value for later use.



Attach foam purge rings

Attach purge rings to both ends of the Collector II.

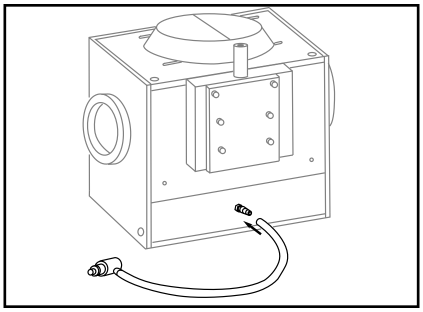


Place COLLECTOR II in Sample Compartment

Use the four hold down screws (8-32) that were removed from the sample compartment to attach the Collector II baseplate to your spectrometer*.

***Note:** See Appendix A for individual spectrometer mounting requirements and diagrams.

COLLECTOR II Installation

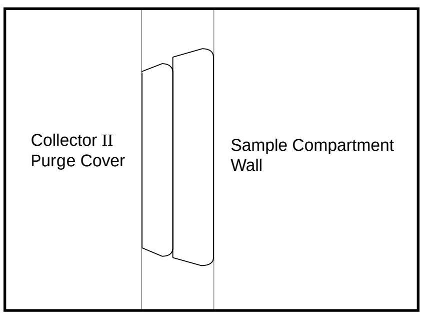


Attach hosing

Attach the open end of the plastic hosing (provided) to the purge nipple on the back of the Collector II.

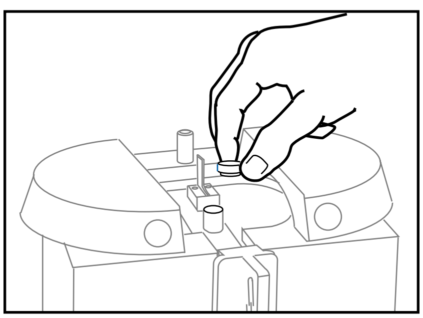
For Nicolet systems with purge capabilities: Attach the other end of the tubing to the socket in the sample compartment.

For all other instruments: Remove the fitting and attach the other end of the hose to an external dry, CO_2 free air source.



Seal Purge Rings

Once the unit is placed in the sample compartment, adjust the rings until they meet the the wall of the sample compartment and create a seal.



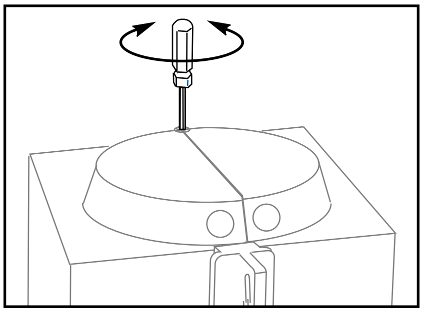
Install Alignment Mirror

Slide the ellipsoidal mirrors open.

Place the aluminum alignment mirror into the cup holder.

Slide the ellipsoidal mirrors closed.

COLLECTOR II Installation



Adjust Sample Height/Focus

While monitoring maximum throughput energy value or the height of the centerburst of the interferogram, use a 3/32 balldriver to adjust the screw that positions the alignment mirror for maximum throughput signal.

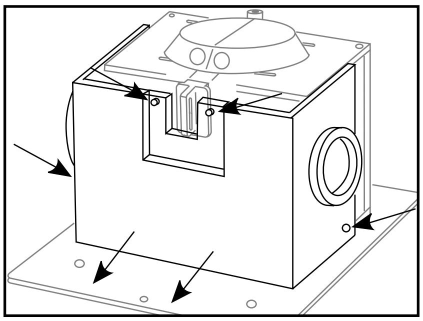
Energy Throughput Validation

When the Collector II is installed, observe the "throughput count" of the FT-IR. Throughput should be between 30% and 50% of the open beam throughput before the accessory was installed.

If you are getting this percentage, no further adjustments are necessary and the Collector II is ready for use. The accessory will provide sufficient energy to permit good signal-to-noise ratios using a DTGS detector and moderately short acquisition times.

If you are not getting this percentage, the Collector II must be removed from the sample compartment and the Purge Cover /Purge Insert needs to be detached from the accessory to access the mirrors for optimal alignment.

Purge Cover/Purge Insert Removal



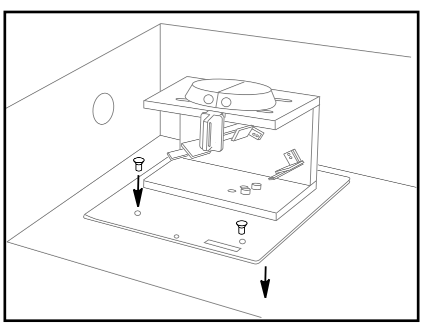
Detach Purge Cover/Purge Insert

The Collector *II* with its attached baseplate must be removed from the sample compartment to access the screws that hold the Purge Cover/Purge Insert.

Remove the four buttonhead screws that hold the Purge Cover/Purge Insert in place using a 1/16 balldriver. Two screws are located at the lower back corners of the accessory and the other two are located in the front.

The Purge Cover slides straight forward for removal and straight back for replacement.

Note: The Purge Cover is designed to fit snugly against the walls of the Collector *II* accessory. It may be necessary to gently pry the outer walls of the Purge Cover to remove it.

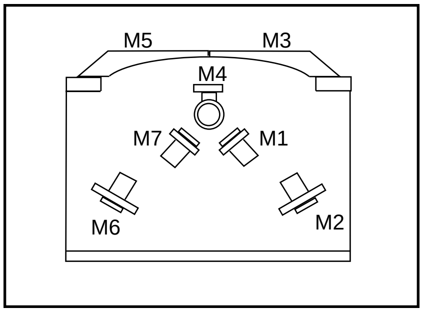


Install Collector *II* without Purge Cover/Purge Insert

Re-install the Collector *II* and baseplate back into the sample compartment for re-alignment.

It is not necessary to install the back two screws. The accessory will have to be removed to replace the Purge Cover/Purge Insert.

Mirror Alignment

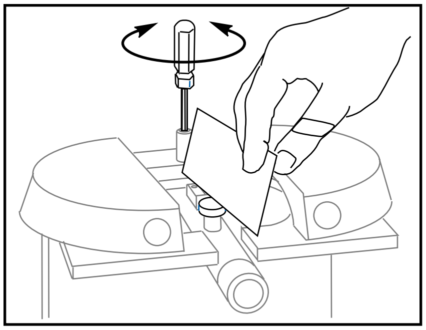


Adjustment Introduction

The COLLECTOR II is configured at the factory for your spectrometer.

The COLLECTOR II is also pre-aligned so that only minor adjustments (tilt and/rotation) of the mirrors may be necessary.

There are four mirrors to adjust M1, M2, M6, M7 and the sample position.

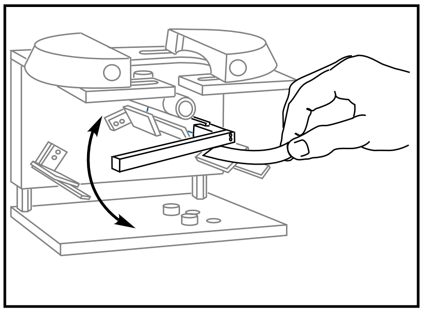


Adjust the Sampling Position

Open the ellipsoidal mirrors and place the alignment mirror (face up) on the sample stage.

Using a 3/32 balldriver, adjust the screw so that the mirror is approximately in the same plane as the top surface of the Collector *II*'s top plate.

Note: Place a piece of paper on edge across the top plate and mirror to help position the stage.



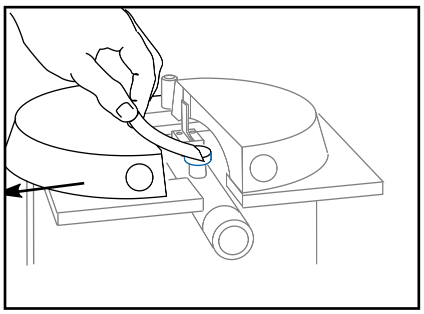
Adjust Mirror M1

Carefully place a piece of paper in front of Mirror M2.

Rotate Mirror M1 until the spectrometer's laser beam is visible in the center of M2.

Note: Be certain that the instrument's laser beam is coaxial with the IR beam.

Mirror Alignment



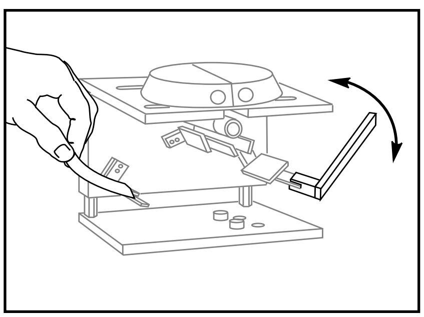
Check Beam on Alignment Mirror

Leave the OUTPUT ellipsoid mirror open. Close the INPUT ellipsoid mirror.

Note: The output is the left ellipsoid for a right to left beam and the right ellipsoid for a left to right beam.

Hold a small piece of paper on the surface of the alignment mirror. The laser beam should be visible in the center of the alignment mirror. If the beam is not in the center, rotate Mirror M2. Slide the OUTPUT ellipsoid closed.

Slide the ellipsoid closed.



Adjust Mirror M2

Carefully place a piece of paper in front of Mirror M6.

Rotate Mirror M2 until the laser beam is visible in the center of M6.

Adjust Mirror M6

Carefully place a piece of paper in front of Mirror M7.

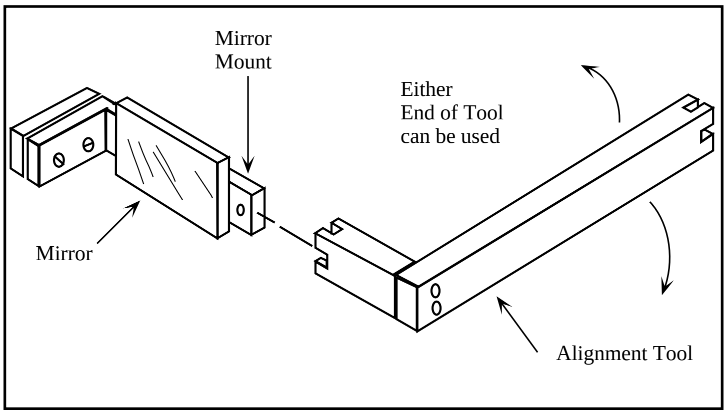
Rotate Mirror M6 until the laser beam is visible in the center of M7.

Adjust Mirror M7

Carefully place a piece of paper in front of the exit port to the detector.

Rotate Mirror M7 until the laser beam is visible in the center of the port.

Mirror Fine Adjustment



Fine-Tune Mirror Rotation

Put the spectrometer in align mode (throughput, tune etc.).

Peak the throughput energy to all the mirrors by rotating each mirror in the following order M7, M6, M2 and M1.

Repeat order a second time.

Mirror Fine Adjustment

Fine-Tune Tilt Adjustment

Test the tilt of the mirror by lightly pressing the mirror mount in each direction noting the energy count. The tilt of the mirrors is adjusted through the two screws located at the base of the mirrors. If the energy increases as the mirror is tilted in the direction shown in Figure 3A (below), loosen the front screw and tighten the back screw 1/8" of a turn . If the energy increases as the mirror is tilted in the direction shown in Figure 3B (below), loosen the back screw and tighten the front screw 1/8" of a turn (see 3B below). Retest the tilt. When no further adjustment is needed, carefully tighten the screws without moving the mirror.

When the COLLECTOR *II* is fully aligned, it may be removed to re-attach the Purge Cover and Purge Insert and then replaced in the FT-IR sample compartment.

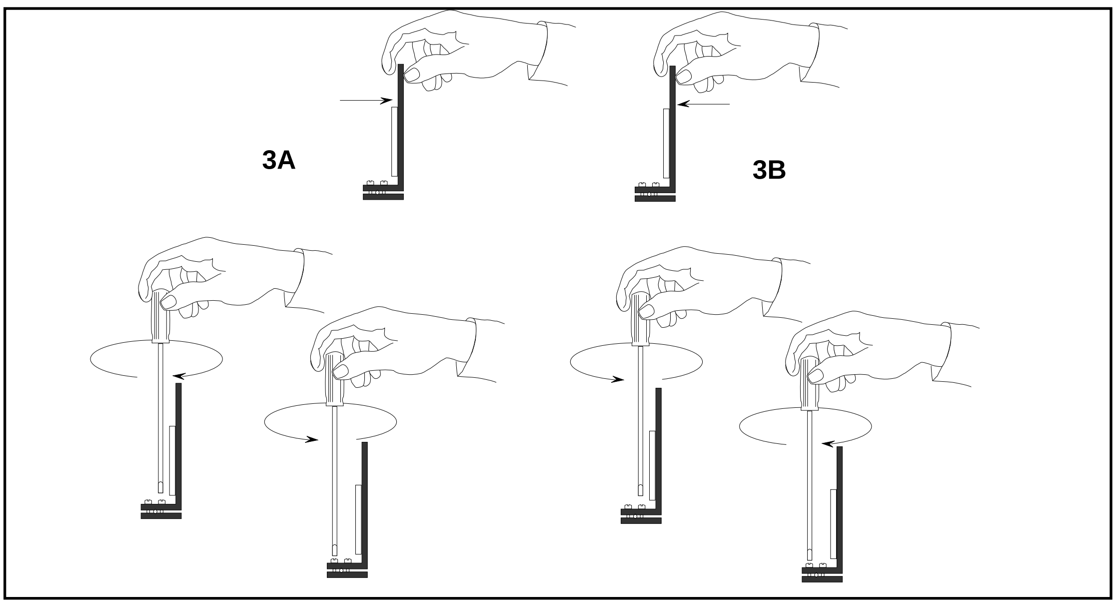
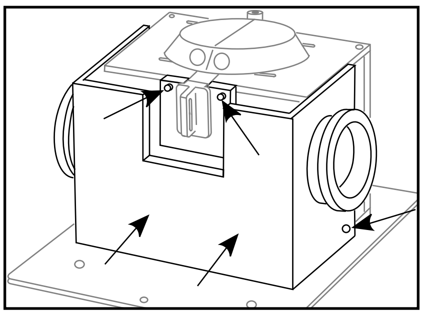


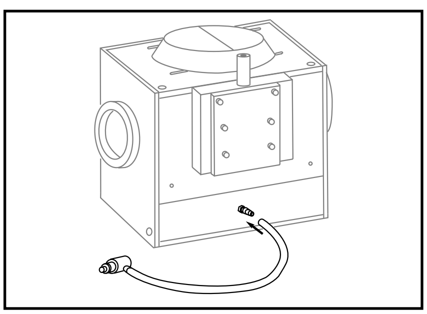
Figure 3 *Tilt Adjustment*

Purge Cover/Purge Insert Reinstallation



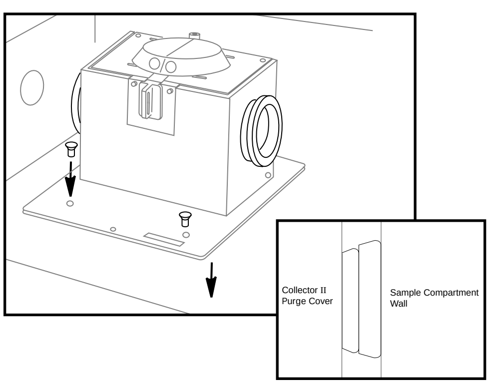
Attach Purge Cover/Purge Insert

After the Collector *II* is aligned for optimal energy throughput, remove the accessory from the compartment and attach the Purge Cover/Purge Insert.



Reattach Purge Hose

Reattach Purge Hose (see instructions on page 9 & 10).



Reinstall Collector *II* and Purge Rings

Replace the accessory into the sample compartment and reseal Purge Rings to the walls of the spectrometer's sample compartment (see instructions on page 9 & 10).

The Collector *II* is ready to use.

Operation

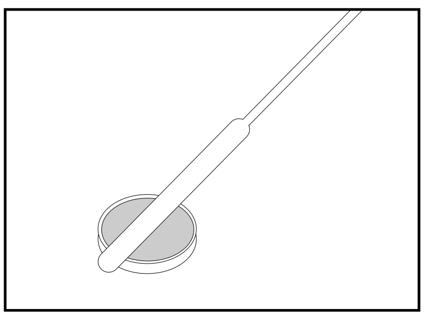
Using the COLLECTOR II

Select an Appropriate Background

A suitable background must be chosen. The background must match the sample matrix. Potassium Bromide (KBr) makes a good diffuse reflectance dilution matrix since it has no absorption peaks in the infrared. KBr is also a good background for many undiluted or neat samples. Other dilution matrices include: Potassium Chloride (KCl), Diamond Powder, HDPE Powder and Cesium Iodide (CsI).

Sampling Cups

Four sampling cups are provided. The two (2) macro cups are 13 mm in diameter, the two (2) micro cups are 3 mm in diameter. Generally, throughput with the micro cup will be substantially lower than with the macro cup due to vignetting of the diffuse energy off of the rim of the sample cup.



Fill the Sample & Background Cups

Overfill the cup with the background powder, then scrape a spatula across the top of the cup. The powder should be level with the surface of the cup.

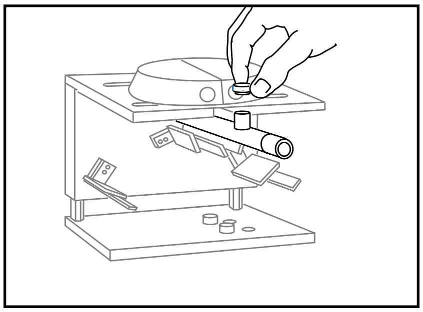
Repeat above for the sample powder.

A funnel is provided to aid in filling the micro cup.

Note: Care should be taken in filling the cups to make sure that the powder is level and smooth. The sample must not be packed down too hard into the cup. Hard-packed samples may cause specular reflectance which results in restrahlen bands. Good technique in loading the sample will result in more reproducible spectra.

Note: The COLLECTOR *II* can also be used to obtain high quality specular reflectance spectra of films and coatings at a fixed 50 degree mean angle of incidence.

Using the COLLECTOR II



Collect Background Spectrum

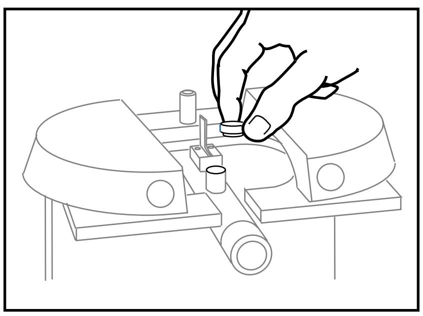
Pull the push/pull tube out.

Place the cup containing the background powder into the cup holder.

Push the push/pull tube firmly into the sampling position.

> **Note:** The ellipsoids should remain closed.

Collect the Background spectrum.



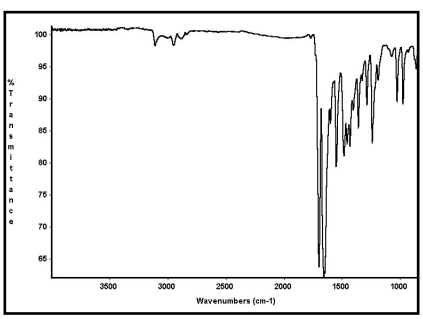
Collect Background Spectrum - Alternate Method

Slide the ellipsoids open.

Place the cup containing the background powder into the cup holder.

Slide the ellipsoids closed.

Collect the Background spectrum.



Collect the Sample Spectrum

Follow the steps above substituting the sample powder for the background powder.

Collect the Sample spectrum.

> **Note:** If the absorbance is too strong, proceed to the Sample Preparation Section.

Operation

Sample Preparation

Powders

The diffuse reflectance technique is ideal for measuring pure samples or mixtures that are available in powdered form, such as pharmaceuticals and other high molecular weight organic materials. Some samples may need to be ground for the analysis.

Powders are easier to run by diffuse reflectance than by infrared transmission because less preparation is required. When a diffuse reflectance accessory is used, the powder is placed directly in the sample cup or mixed with a premeasured amount of KBr and then poured into the sample cup. The sample cup is then placed onto the sample cup holder of the diffuse reflectance accessory and data collection can begin. Some powders can be analyzed neat (without dilution):

1. Grind the sample if necessary. A particle size of 2 to 5 micrometers is recommended. You can use a mortar and pestle or an electric grinder to grind the sample. One to two minutes of grinding is usually sufficient. Grind the sample slowly to avoid creating heat due to friction. Vigorous grinding may change the chemical structure of some samples.

Note: The WIG-L-BUG™ electric grinder available from Spectra-Tech is designed to grind samples to the correct particle size for diffuse reflectance measurements.

2. (Optional) If the sample is highly absorbing or has a high refractive index, dilute the sample with a non-absorbing matrix material such as KBr. Make sure you use a high quality (ground and dried) matrix. If the particles are bigger than five micrometers, use a clean mortar and pestle or an electric grinder to grind the matrix material.

Note: Do not grind the sample and matrix together. Premeasured packets of high quality KBr that has been thoroughly dried and ground to the correct particle size are available from Spectra-Tech.

Sample Preparation
Powders

The following is a general rule to follow when preparing samples and matrices for Diffuse Reflectance analysis:

Organic Samples:	10% Sample (by weight)	90% Matrix (by weight)
Inorganic Samples:	2-5% Sample (by weight)	95-98% Matrix (by weight)

Sample Cups Volume By Weight:

Micro Cup: 0.10 grams KBr

Macro Cup: 0.25 grams KBr

Example: Organic Sample, 10% by weight:

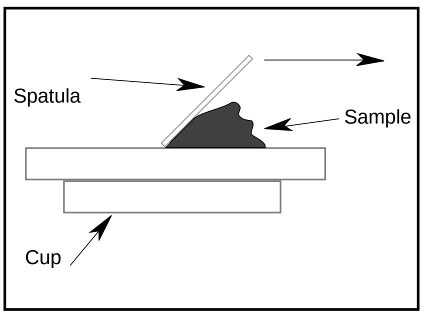
Micro Cup:	0.01 grams Sample	0.09 grams KBr
Macro Cup	0.025 grams Sample	0.225 grams KBr

3. Place the measured quantities of the sample and matrix material into a glass vial and shake for approximately one minute. Do not stir.

Note: A glass vial is necessary to avoid contamination.

4. Fill the sample cup to overflowing. Be careful not to tap down the sample cup when pouring the powder. Tamping packs the particles too closely and may reduce the sensitivity of the analysis.

Sample Preparation
Powders

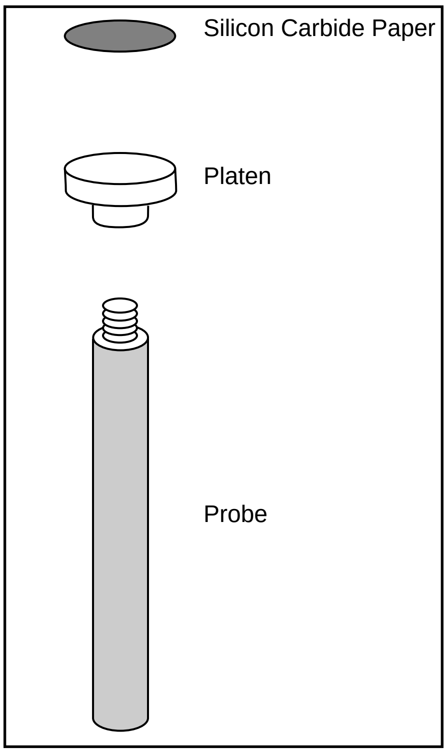


5. When the sample cup is filled, place it on a flat surface and level the sample surface with a spatula. This reduces reflections off the sample surface and helps ensure reproducible results.

Operation

Sample Preparation
Intractable Samples

Diffuse Reflectance is not limited to powders and small solid samples. A **Si-Carb™ Sampling Kit** can be used in conjunction with the COLLECTOR™ *II* to analyze many types of intractable samples without time consuming sample preparation. A small disk of silicon-carbide paper is used to abrade the surface to be analyzed. This technique transfers a small amount of sample to the disk. An infrared spectrum is obtained of the material adhering to the surface of the silicon carbide disk. For abrading extremely hard samples, diamond paper is available.



Si-Carb Sampler Operation

1. Screw the Platen onto the Probe.
2. Remove the protective backing from a piece of carbide paper.
3. Using the backing to protect the silicon carbide, press the paper firmly onto the Platen.
4. Unscrew the Platen from the Probe.
5. Place the Platen into the Collector *II*'s sample cup holder.
6. Adjust sample cup height for maximum energy.
7. Collect a background spectrum.
8. Remove the Platen from the sampling cup.
9. Screw the Platen onto the Probe.
10. Rub a small amount of the sample onto the Platen.
11. Unscrew the Platen from the Probe.
12. Place the Platen into the Collector *II*'s sample cup holder.
13. Adjust sample cup height for maximum energy.
14. Collect a sample spectrum.

Note: Resulting spectra may be above the 100% line. This is a common occurance due to the sample surface being more reflective than the silicon carbide paper alone.

Factors that Affect Diffuse Reflectance Spectra

The spectral information you get from a diffuse reflectance accessory is affected by a number of factors.

Refractive Index of Sample

The higher the refractive index of the sample, the more true specular (mirror) reflection will occur at the sample surface. When the specular component of a diffuse reflectance measurement is large, the bands in the resulting spectrum tend to broaden and the relationship between band intensity and sample concentration becomes nonlinear.

Materials that are particularly reflective, such as many inorganic substances, can produce bands that are completely inverted. These are called the Restrahlen bands (see “Diffuse Reflectance Sampling Technique”).

You can minimize specular reflection by mixing the sample with a non-absorbing matrix material such as KBr.

Particle Size of the Sample

The larger the refractive index of the sample, the more true specular (mirror) reflection will occur at the sample surface. When the specular component of a diffuse reflectance measurement is large, the bands in the resulting spectrum tend to broaden and the relationship between band intensity and sample concentration becomes nonlinear.

Materials that are particularly reflective, can produce bands that are completely inverted. These are called the Restrahlen bands (see “Diffuse Reflectance Sampling Technique”).

To minimize specular reflection due to particle size, grind the sample material thoroughly and mix it well with the matrix material, if one is used. The particles should be small and the mixture should be homogeneous in particle size and composition.

Factors that Affect Diffuse Reflectance Spectra

Homogeneity of the Sample

The more uniform the sample, the more linear the relationship between band intensity and sample concentration in the resulting spectrum.

You can obtain the best results when the sample material is thoroughly ground and well mixed with the matrix material, if one is used. The particles should be small and the mixture should be homogeneous in particle size and composition.

Be aware that powdered samples tend to separate by particle size when they are carried from one location to another. Make sure you mix the sample immediately before measuring it by diffuse reflectance.

Sample Concentration

The more concentrated the sample, the more likely distortions due to specular reflection will appear in the resulting spectrum. This is especially true for samples that have a high refractive index. Highly concentrated samples also tend to produce bands that are broad or totally absorbing (see “Diffuse Reflectance Sampling Technique”).

To minimize distortion due to high concentration samples, grind the sample material thoroughly. The particles should be small and the mixture should be homogeneous in particle size and composition. For best results, mix the sample with a non-absorbing matrix material such as KBr.

Factors that Affect Diffuse Reflectance Spectra

Common Problems with Diffuse Reflectance Spectra

As with any analytical technique, you will have more confidence in the data you produce if you recognize problems when they occur.

This section identifies some common problems with diffuse reflectance analysis, illustrates their effects on the spectral data and explains how to solve them.

Sample Bands are too Small

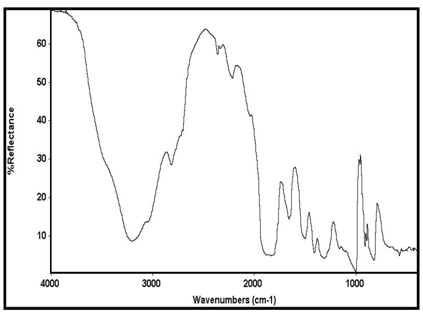
The sample bands should be large enough that you can distinguish them from the specular baseline. If this is the case with your spectrum, the following problems may have occurred:

- The sample holder was not properly positioned in the accessory.
- The sample was not ground to the correct particle size.
- The sample concentration was too low (use less matrix material).
- The sample cup was not filled to the rim.

Factors that Affect Diffuse Reflectance Spectra

Sample Bands are too Big

Bands with values below 1% R (above 49 Kubelka-Munk units) are considered totally absorbing and normally have a flattened or “squared off” end. Several totally absorbing bands are present in this spectrum.

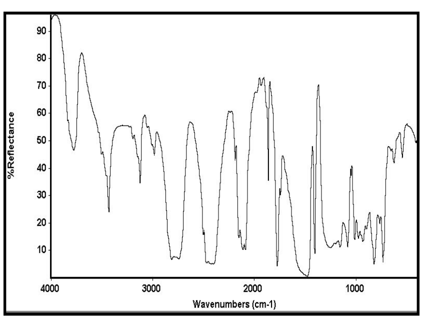


Potassium dichromate undiluted with totally absorbing bands.

When the % reflectance value of a spectral band approaches 1% R (49 Kubelka-Munk units), the intensity is so close to totally absorbing that it cannot be measured reliably.

If the absorbance is too strong when you measure an undiluted sample, mix it with a diffusely scattering matrix material such as KBr.

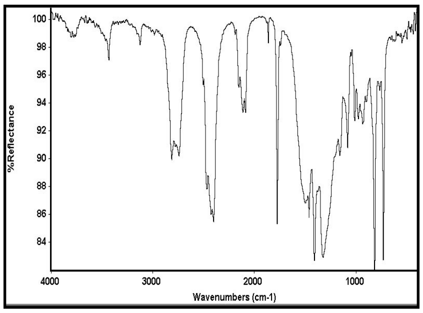
Sample Bands Are Broad or Rounded



Barium nitrate undiluted.

If you see bands that are broad or rounded, the sample particles may be too large. The larger the particle size of the sample, the more specular (mirror) reflection will occur at the sample surface.

Factors that Affect Diffuse Reflectance Spectra

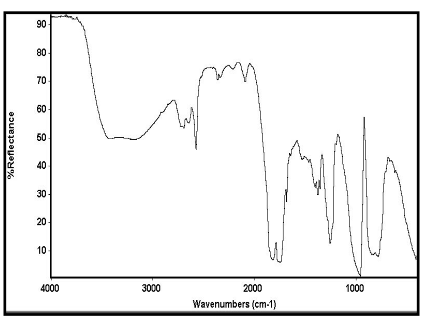


Barium nitrate ground and diluted to 1% in KBr.

To minimize specular reflection due to particle size or concentration, thoroughly grind the sample material and mix it well with the matrix material, if one is used. The particles should be small and the mixture should be homogeneous in particle size and composition.

Derivative-Shaped Bands in the Spectrum

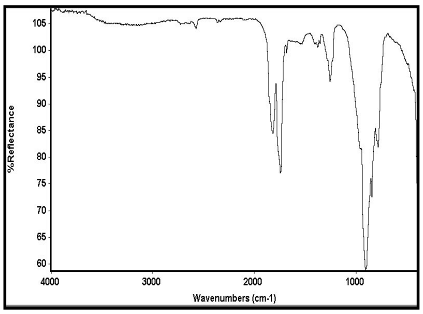
One relatively common effect in diffuse reflectance spectroscopy is bands that are inverted or derivative-like in shape. These distortions, called Restrahlen bands are caused by specular reflection of the radiation off the sample surface.



Potassium permanganate undiluted, with derivative-shaped bands at 919 cm-1.

This specular component is dependent both on particle size, and on sample packing and concentration. Restrahlen effects are especially pronounced with strongly absorbing and organic samples because the refractive index is high.

Factors that Affect Diffuse Reflectance Spectra

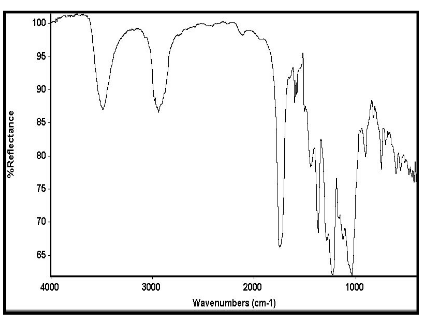


Potassium permanganate -1% in KBr.

To minimize or eliminate this effect, the sample can be further diluted with a diffusely scattering matrix such as KBr and the matrix crystals can be crushed to a smaller and more consistent size.

Spectral Baseline is Offset

When the Si-Carb Sampler is used with a reflective surface for the background measurement, the baseline of the resulting spectrum is often significantly offset. This happens when the gold disk is much more reflective than the sample. An offset baseline is one which is flat but below 0 Kubelka-Munk (or log (1/R)) units, or above 100% reflectance.



Screwdriver handle; using the Si-Carb Sampler.

If an offset occurs in spectra of powder samples, the following problems may have occurred.

- The refractive index of the sample material is higher than that of the background material (use Baseline correct).
- The sample is not ground finely enough.
- The sample material is more reflective than the background material (this can cause the baseline to be greater than 100% Reflectance). Try diluting the sample with more KBr.

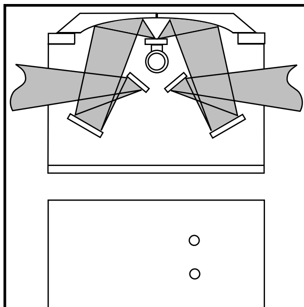
Appendix A

Mounting Methods

Spectrometer	Mating Plate	Standoffs
Analect FX6200	Yes	None
Beckman 1000, 2000	Yes	.576”
Bomem DA3	Yes	1-3/4”
Bomem Michelson	Yes	1-1/2”
Bomem MB	Yes	1”
IBM 32,85,98,38	None	1-1/2”
Digilab , FTS-14/15/20/80	Yes	3/4”
Digilab Qualimatic, FTS-50, FTS-60	Yes	None
Digilab FTS-10, 11	Yes	1”
Mattson Sirius	Yes	1”
Mattson 1000, 2000, 3000, 5000, 7000	Yes	1-1/2”
Midac	Yes	1/4”
Nicolet 5-X, MXS, 10-X, MX1,3600, 20-X	None	None
Nicolet 20-X, 60-X, 5-XB, 20-XB	None	1-1/2”
Nicolet 170-X, 7199, 6000	None	2”
Perkin Elmer 1500,	Yes	None
Perkin Elmer 1700, 2000	shuttle	1/2” or 3/4”
Perkin Elmer 1600	Slide Mount	None
Philips 9800, 9600	None	1-1/2”

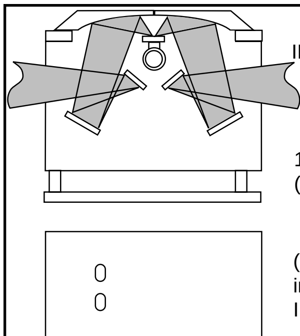
Appendix A

Mounting Methods



Nicolet 5-X, MX-S 10-X, 3600, MX-1 and old 20's with 3600 optical bench.

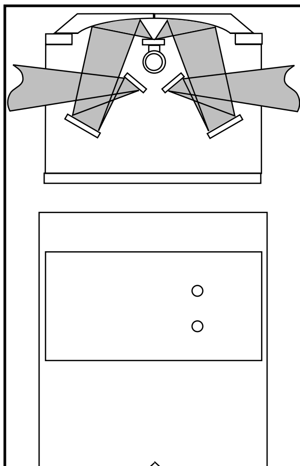
(2) 8-32 screws thru clearance holes in baseplate.



IBM spectrometers (all)

1-inch standoffs (3-inch working height)

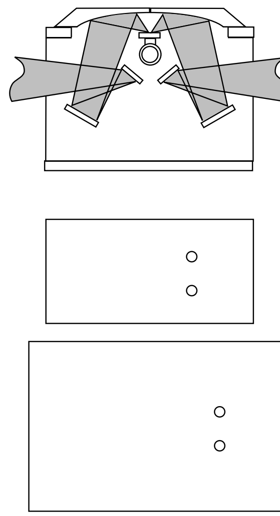
(2) M6 cap screws thru indicated holes. Clamp to IBM kinematic plate.



Perkin-Elmer 1700 Mount on supplied shuttle device as shown.

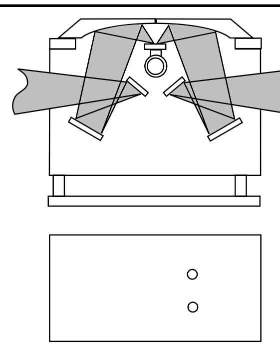
Shuttle device -- 1/2-inch standoffs for motorized shuttle.

3/4-inch standoffs for manual shuttle.



Digilab Qualimatic FTS-50, FTS-60

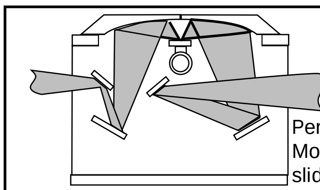
Bottom view of baseplate. Two hold-down screws to mating baseplate. Mounts on two pins into sample compartment.



Nicolet 20-X (newer) 20-XB, 60SX

(2) 1 1/2-inch standoffs to lower plate then,

(2) 8-32 screws thru clearance holes in baseplate.



Perkin-Elmer 1800 Mounts on instrument's slide mount.

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