Installation and User's Guide

VeeMax

VARIABLE ANGLE SPECULAR REFLECTANCE ACCESSORY

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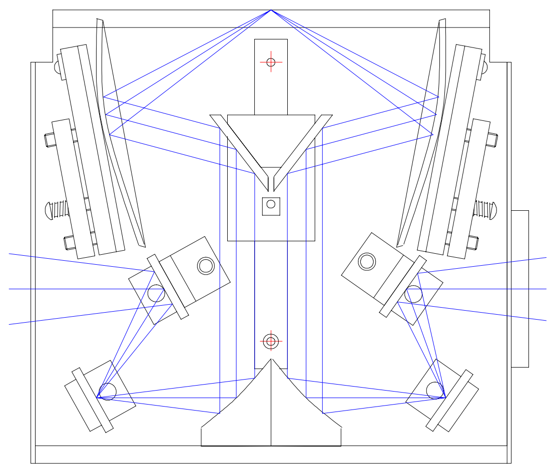
Introduction

The PIKE Technologies VeeMax™ is a high throughput variable angle specular reflectance accessory designed for use in FTIR spectrometers. The design employs a unique optical design (patent applied for) which enables samples to be analyzed over a range of incident angles from thirty to eighty degrees.

The infrared beam from the spectrometer is passed through two beam steering mirrors to a collimating parabola. The upwards traveling beam hits one side of the adjustable sliding mirror and is directed to the large parabola mirror. Since this beam is collimated, the parabola produces a focused spot at the sample position. As the sliding mirror assembly is moved, the angle of incidence of the beam is varied at the sample. The reflected beam is passed through an identical set of optics to the instrument detector.



VeeMax Optical Diagram

The sample is placed face downwards on the sample platform for analysis. Since the sample position is external to the accessory, large and bulky samples may be analyzed easily.
A polarizer mount is provided to obtain enhanced results when performing the analysis of thin films at grazing angles.

Alignment

The accessory has been aligned and tested to ensure that it performs to specification. There are two mirrors on the accessory that may be aligned to maximize performance in your spectrometer. The alignment procedure is as follows.

1. Compare your accessory with the drawings below and locate the tilt and rotation adjustments. The tilt adjustment needs a 3/32nd Allen wrench while the rotation adjustment requires a 1/8th wrench.

2. Peak up the energy of your spectrometer by adjusting the interferometer. This should be performed by following the manufacturers instructions.

3. Mount the accessory into the sample compartment.

4. Place the alignment mirror on the sample surface of the accessory

5. In alignment mode, check the signal throughput of the spectrometer with the accessory in place.

6. Set the reflectance angle to thirty degrees and place the medium mask on the sample surface.

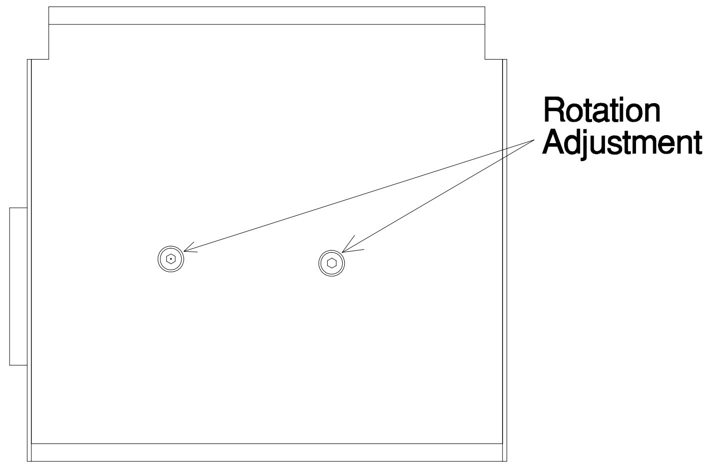
7. Using the wrenches provided adjust the rotation of the input mirror to maximize the signal.

8. Adjust the rotation of the output mirror to maximize the signal.

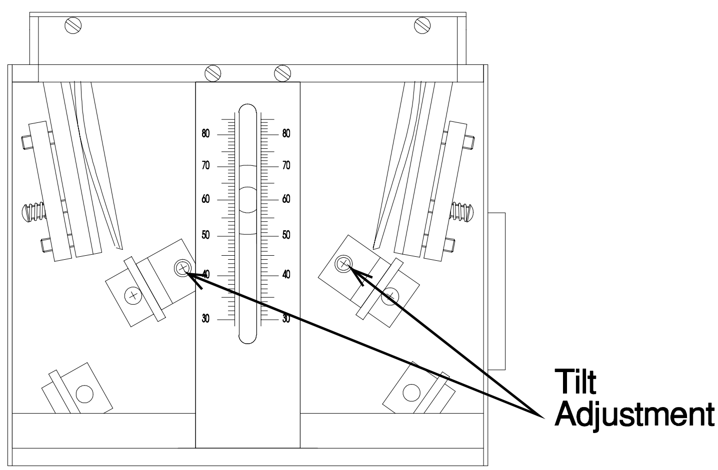
9. Adjust the tilt of the input mirror to maximize the signal.

10. Adjust the tilt of the output mirror to maximize the signal.

11. Repeat the above four steps until the signal no longer increases.



VeeMax Rear View



Veemax Front View

Sampling procedures

The sample is placed face downwards on the top surface of the accessory. a set of three masks is available to limit the size of the beam striking the sample. An alignment mirror is also provided to align the accessory and enable background scans to be taken.

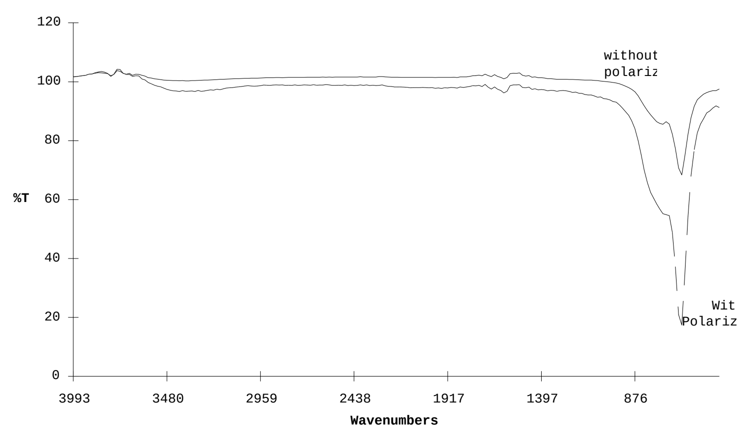
The masks have different sized holes, 1/8, 1/4 and 3/8 inch diameter. The underside of the mask contains a non reflecting surface machined into it. The masks are also coated with non-reflecting black. However, at grazing angle, the masks still reflect a little of the incident radiation. If poorly reflecting samples are being measured at grazing incidence, then the effect of mask reflections must be considered in the resulting spectrum. A candle is provided for recoating of the masks if required. If your instrument has an adjustable aperture, reducing the size of the aperture to match the mask will reduce stray reflections.

Reflectance Spectra and Applications

Reflectance spectroscopy is useful for the measurement of monolayer films on metallic substrates, the thickness of free standing transmitting films and for the characterization of samples which do not transmit IR radiation and so cannot be measured by other techniques.

Thin Films

Advantage can be taken of the enhanced absorbance of infrared energy at a large angle of incidence - a grazing angle. The theory of the grazing angle effect was explained by R.G.Greenler in his paper titled "Infrared study of adsorbed molecules on metal surfaces by reflection techniques" (J. Chem. Phys., 44, 10 (1966)). In this paper Greenler discusses the interaction of infrared energy at a metal surface. If this energy is polarized so that the electric vector is perpendicular to the surface of the sample (p polarization), and strikes the sample at a grazing incidence, the interaction of this energy with the metal surface, (and any thin surface film deposited on this surface) is greatly enhanced. The enhancement is a function of the angle of incidence of the impinging IR energy, being greatest at incidence angles close to ninety degrees. This effect is illustrated below which shows two spectra of an identical thin film measured at an eighty degree angle of incidence, with and without a polarizer.



Effect of using a polarizer when measuring thin film on a metallic substrate

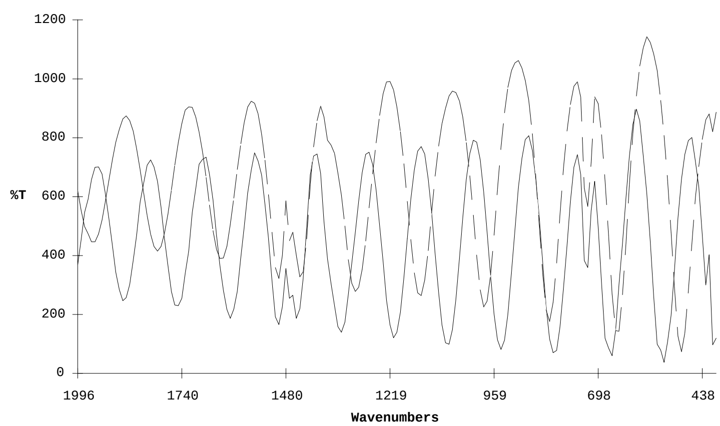
Free Standing Films

Free standing films exhibit a fringe pattern in the collected spectrum due to constructive and destructive interference by the reflection from both sides of the thin film. Measurement of the period of the fringes will give the thickness of the film as long as the refractive index is known. For an angle of incidence ϕ, and a refractive index n, the equation governing this measurement is

$$t = \frac{m}{2(n^2 - \sin^2\phi)^{1/2}\,\Delta\nu}$$

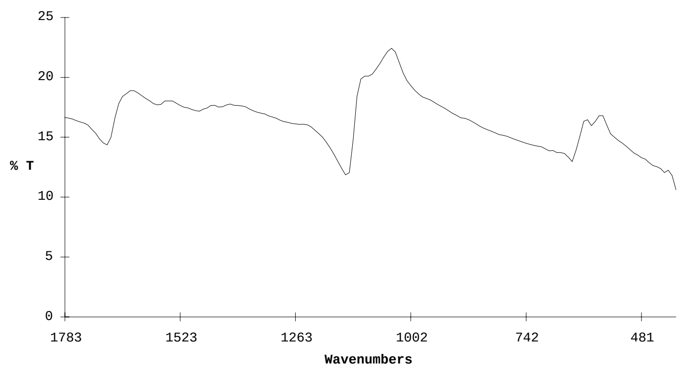
where m is the number of fringes counted and $\Delta\nu$ is the wavenumber span for this number of fringes.

If the refractive index is not known then a measurement made at two angles of incidence will furnish both the film thickness and the refractive index of the material.

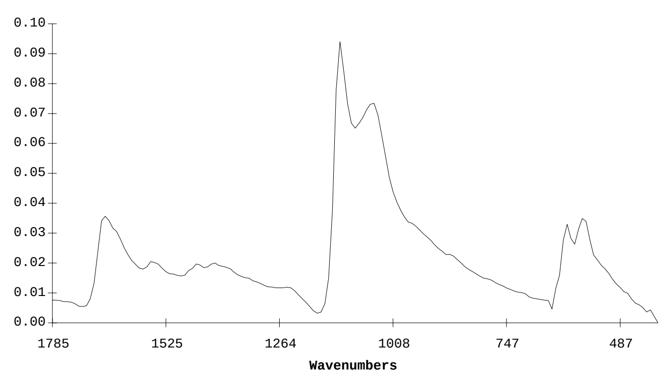


Reflectance of polythene film at thirty and seventy degrees

Effect of Dispersion
The reflectance spectra of many samples can be distorted by dispersion in the sample. In the vicinity of an absorption band the reflective index varies considerably. In order to produce a spectrum that is corrected for dispersion, a Kramers Kronig transformation must be performed on the spectrum. The two spectra below demonstrate this effect.



Raw Data



Spectrum with Kramers Kronig Transformation

Reflectance Spectra and Applications

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Free Standing Films

Free standing films exhibit a fringe pattern in the collected spectrum due to constructive and destructive interference by the reflection from both sides of the thin film. Measurement of the period of the fringes will give the thickness of the film as long as the refractive index is known. For an angle of incidence ϕ, and a refractive index n, the equation governing this measurement is

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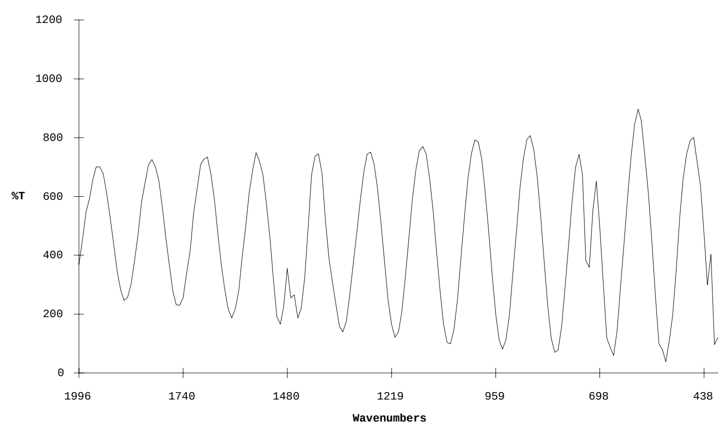
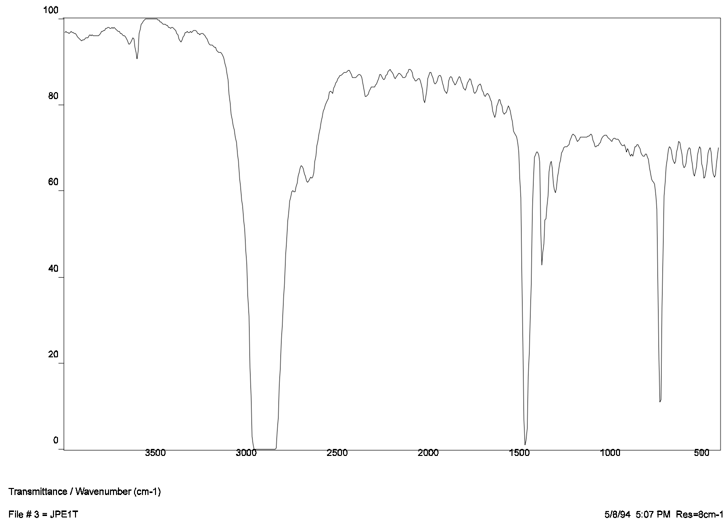
where m is the number of fringes counted and $\Delta\nu$ is the wavenumber span for this number of fringes.



Reflectance of polyethylene film at thirty degrees

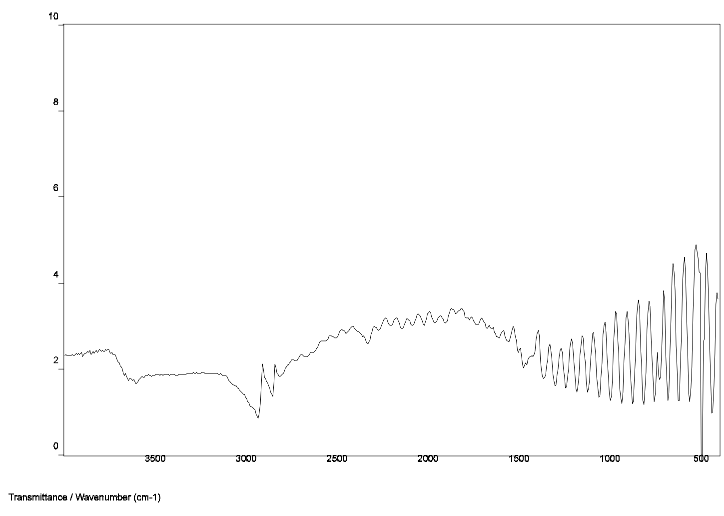
This interference fringe effect can create problems when measuring these types of samples. The interference fringes can hide spectral features and cause quantitation problems, since the fringe position and spacing is sensitive to the thickness of the sample.

Below is a transmission spectrum of a plastic film. The spectrum contains interference fringes and the fine detail in the spectrum is difficult to discern.



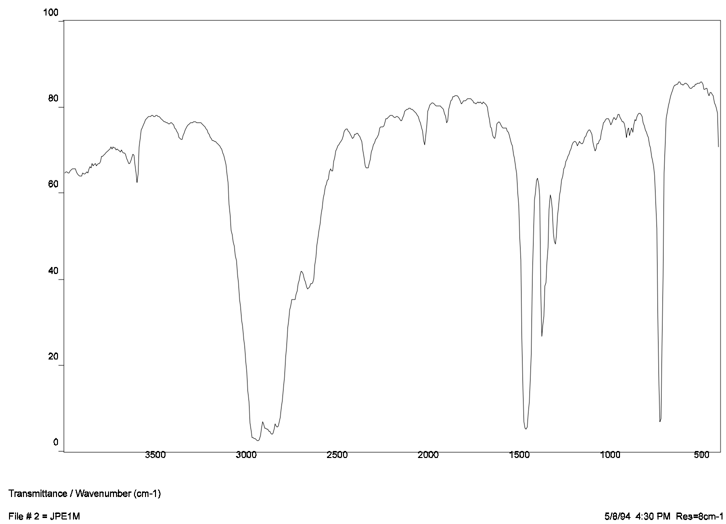
Transmission Spectrum of Plastic Film.

When the same sample is measured in reflection using the 30SPEC, the situation is significantly worse, unless the aim of the experiment is to measure the film thickness. Note the Distorted band shapes, typical of anomalous dispersion and the low level of the spectrum, since only a small percentage of the infrared energy is reflected from the sample



Reflection Spectrum of Plastic Film

The spectrum below is a reflection-absorption spectrum of the plastic film. The sample is placed on the 30SPEC and a mirror is placed on top of the sample. The spectrum shows no sign of interference fringes, the band shapes are correct and have twice the absorption of the transmission case, consistent with the double passage of the infrared beam trough the sample.



Reflection-Absorption spectrum of plastic film

Precautions

Mirrors

In order to provide the maximum transmission in the infrared, with the minimum spectral interferences, the mirrors used in this device are uncoated (bare) aluminum on a glass substrate. Since the coatings are soft, care must be taken to avoid damage. Normally, these mirrors will not need cleaning, since they are contained within the housing of the accessory. If they do need cleaning, they may be gently wiped with a lint free, abrasive free cloth, such as lens tissue, or with a camel hair brush. Under no circumstances must the mirrors be rubbed with paper products such as "Kleenex" since this will produce scratching of the mirror coating.

Packing List

The VeeMax Accessory is provided with the following:

Veemax accessory	1
Allen Wrench Set	1
Mask Set	1
Candle	1
Users Guide	1
Alignment Mirror	1