

Specular Reflection



Specular reflection is an important sampling technique for measuring thin films and reflective substrates, analyzing bulk materials and measuring mono-molecular layers on a substrate material. This technique can often be used with no special preparation, keeping the sample intact for subsequent measurements.

VeeMAX III

AT A GLANCE

- ▶ Selectable angle of incidence from 30 to 80 degrees in 1-degree increments
- ▶ Automated option for high-precision experiments
- ▶ Measurement of thin films and monolayers to relatively thick films
- ▶ Optimize spectral results with selectable angle of incidence
- ▶ Integrated position for IR polarizer – essential for monolayer analysis and study of polymer orientation
- ▶ Optional single reflection ATR crystals



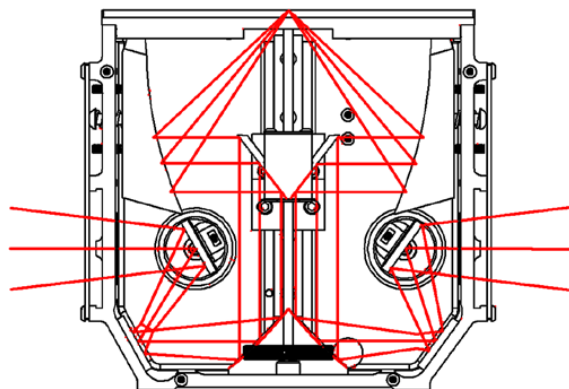
FULLY AUTOMATED
WITH AUTOPRO SOFTWARE



The VeeMAX III is a variable angle, high-performance, research-grade specular reflection accessory. A unique variable angle optical design (U.S. Patent No. 5,106,196) makes it a key accessory to analyze a wide range of samples.

Typical VeeMAX III applications include depth profiling, analysis of monolayers and ultra-thin films, determination of polymer orientation and spectroelectrochemistry. From monolayers to relatively thick films, all experiments may be optimized by varying the angle of incidence from 30 to 80 degrees. Changing the angle is as easy as turning the angle selector on the front panel of the accessory.

To make measurements, the sample is placed face down on the sampling surface. Large samples may be readily analyzed thanks to unrestricted access to the sampling area. Masks with 2", 5/8" and 3/8" apertures are provided to suit different sample geometries. Thanks to the optical design of this accessory and the quality of the diamond-turned optics, excellent throughput is realized even at high angles of incidence.



VeeMAX III optical layout.

Another important design feature of the VeeMAX III is its enclosed optics for purging, which eliminates the need to purge the entire sample compartment. This significantly decreases sampling time. It is furnished with two internal polarizer mounts allowing the PIKE polarizer to be inserted into the accessory on either the source or detector side of the spectrometer. The polarizer setting dial is easily accessed without breaking the purge when changing polarizer orientation.

VERSATILE ATR OPTIONS

The VeeMAX III accessory may be fitted with an ATR flat plate, allowing for variable angle ATR experiments. Please see the ATR section of our catalog for complete configuration options.

AUTOMATION

An optional automated version of the VeeMAX III accessory is available. It features a precision motor with USB interface and PIKE Technologies AutoPRO software. Operation of the VeeMAX III can be integrated with the spectrometer software of most FTIR instruments, which allows the operator to precisely and reliably control a wide range of angles of incidence and data collection simultaneously. Automated sampling decreases operator and measurement error, and increases workflow productivity.



Automated VeeMAX III with polarizer option. Angle of incidence and polarization angle can be set independently.

SPECTROELECTROCHEMICAL CELLS

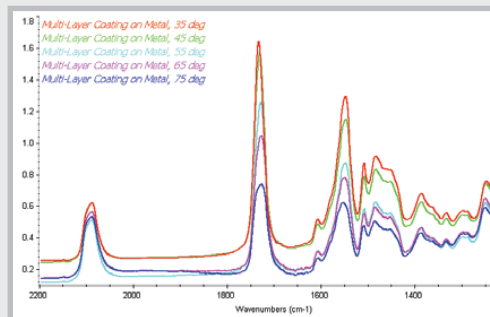
A spectroelectrochemical cell options for the VeeMAX III is also available. The PIKE cell, for applications requiring customization, allows for external reflection experiments using a flat IR window or CaF_2 prism or for ATR-SEIRAS experiments. The PIKE electrochemistry cell is equipped with a precision micrometer for electrode positioning. Alternatively, PIKE offers an out-of-the-box cell for the popular ATR-SEIRAS measurements. ATR-SEIRAS has emerged as an alternative to external reflection based IR spectroelectrochemistry, and has been shown to be a powerful tool. To learn more, See the Jackfish cell in the ATR section.



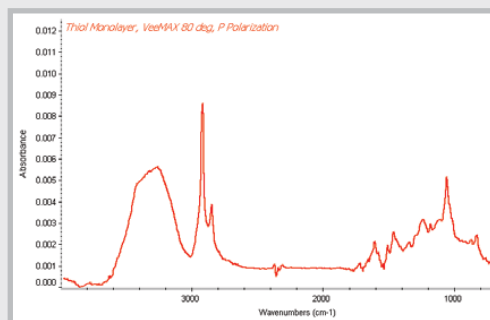
The Jackfish J2 Electrochemical Cell

APPLICATIONS

Through the unique variable angle design, the VeeMAX III angle of incidence feature provides a versatile accessory to optimize the measurement. Manual and automated options available.



Multi-layered coating on metal substrate spectra, measured using the VeeMAX III.



Thiol monolayer spectrum, measured using the VeeMAX III set at 80 degrees angle of incidence, ZnSe polarizer and MCT detector.

SPECIFICATIONS

Optics	All reflective
Angle of Incidence Range	30 to 80 degrees
Sample Masks	2", 5/8" and 3/8"
Purge Sealing	Purge tubes and purge barb included
Dimensions (W x D x H)	177 x 92 x 162 mm (excludes baseplate)
FTIR Compatibility	Most, specify model and type

PART NUMBER	DESCRIPTION
013-11XX	VeeMAX III Includes sample masks (2", 5/8" and 3/8"), gold substrate alignment mirror, FTIR base mount, and purge tubes
013-12XX	Automated VeeMAX III Includes controller, cabling, sample masks (2", 5/8" and 3/8"), gold substrate alignment mirror, FTIR base mount, and purge tubes
Note: Replace XX with your spectrometer's Instrument Code listed in the back of the catalog.	
VeeMAX III Sampling Options	
090-1000	Manual Polarizer, ZnSe
090-1200	Manual Polarizer, KRS-5
090-3000	Precision Manual Polarizer, ZnSe
090-3200	Precision Manual Polarizer, KRS-5
090-5000	Precision Automated USB Polarizer, ZnSe
090-5100	Precision Automated USB Polarizer, KRS-5
Note: Automated version includes PIKE Technologies AutoPRO software and controller. More polarizer options are found in the polarizer section of this catalog.	

PART NUMBER	DESCRIPTION
VeeMAX III Replacement Parts	
013-4010	Specular Mask Set
300-0002	Gold Substrate Alignment Mirror, 1.25 x 3.0"
Note: Please contact PIKE Technologies for items not described in this list.	
Spectroelectrochemical Configurations	
013-3300	Electrochemical Cell – PTFE
013-3370	Electrochemical Cell – PEEK
160-5527	Prism, CaF ₂ , 60 degree
013-3360	Crystal Holder, 60 degree
160-1144	Flat Window, CaF ₂ , 20 x 2 mm
160-1304	Flat Window, ZnSe, 20 x 2 mm
013-3320	Flat Window Holder
Notes: The electrochemical configuration requires Electrochemical Cell and VeeMAX III Specular Reflection accessory. Must select one or more windows. Choose appropriate window holder. More window types for Specular Reflection measurements may be found in our listing of transmission windows, 20 x 2 mm. Electrodes supplied by the end-user. See VeeMAX III with ATR and Jackfish cell product sheets for full ATR crystal and configuration options.	



Automated VeeMAX III programmed with the PIKE Technologies AutoPRO software.



Automated VeeMAX III
with polarizer option.

10Spec

AT A GLANCE

- ▶ Measure sample reflectance
- ▶ Fixed 10-degree angle of incidence
- ▶ Sample illumination using collimated beam
- ▶ Sampling mask sizes of 2", 5/8" and 3/8"
- ▶ Purge cover and purge tubes for removal of atmospheric interferences
- ▶ Extended Height 10Spec to accommodate large samples

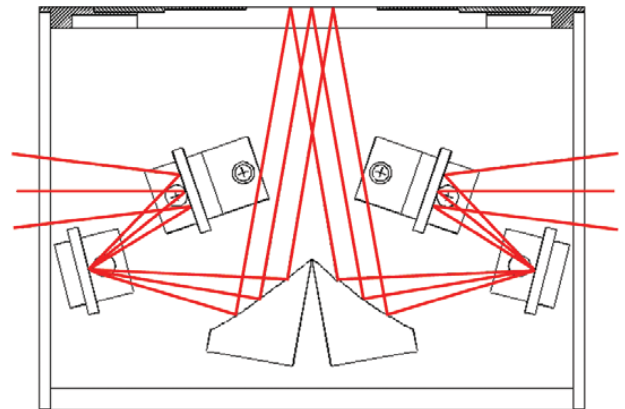
The 10Spec is an optimized specular reflection accessory designed to make high-performance measurements of sample reflectivity.

The 10Spec produces a collimated beam to illuminate the sample area such that the reflectivity measurement is made at a uniform 10-degree angle of incidence and not an average of angles produced by a focused beam accessory design. At a near-normal angle, the polarization effects on reflectivity are minimized. The optics are enclosed to allow for purging.

The 10Spec is an excellent tool for measuring the reflectivity of glass. It may also be used to measure near-normal reflectivity of a wide variety of surfaces including military devices, reflecting optics, anti-reflective (AR) coated surfaces, and other reflecting and non-reflecting materials.



Extended Height 10Spec.



Beam path within the 10Spec specular reflectance accessory.

THE 10SPEC MODELS

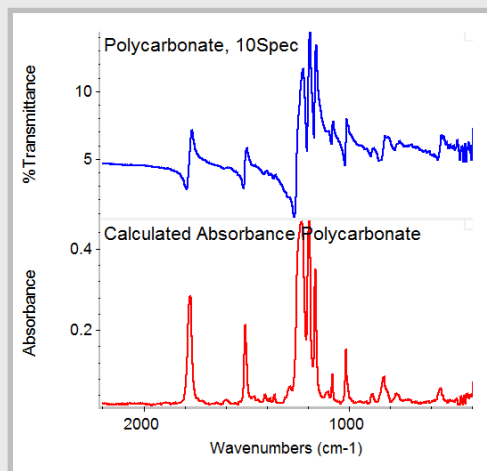
The 10Spec is available in two versions. The standard version is 118-mm tall whereas the Extended Height 10Spec is 205-mm tall, which positions the sample above the top of the FTIR instrument. The Extended Height 10Spec is designed to accommodate samples that are too large to fit within the confinements of the sample compartment.

SPECIFICATIONS

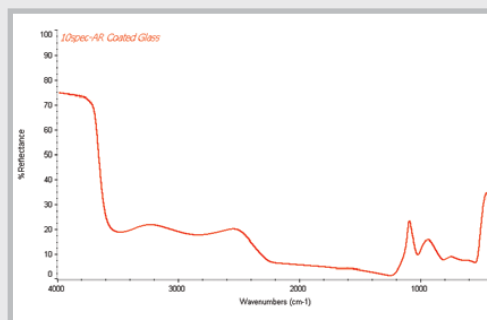
Optics	All reflective
Angle of Incidence	10 degrees
Sample Masks	2", 5/8" and 3/8"
Purge Sealing	Purge tubes and purge barb included
Dimensions (W x D x H)	
Standard	159 x 90 x 118 mm (excludes baseplate)
Extended Height	159 x 90 x 205 mm (excludes baseplate)
FTIR Compatibility	Most, specify model and type

APPLICATIONS

Near-normal specular reflection sampling is useful for the measurement of thin films on reflective substrates and the analysis of bulk materials. Often this technique offers a non-destructive testing method.

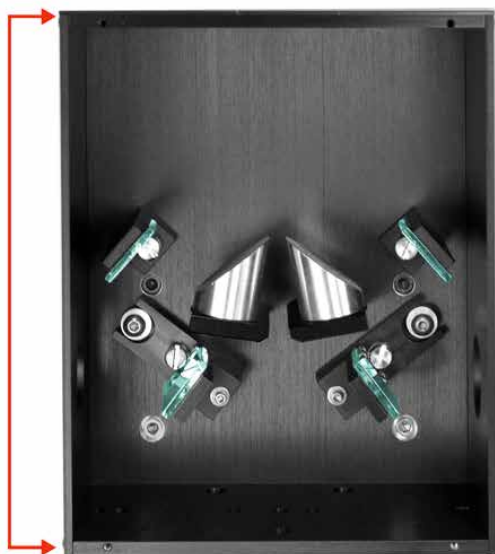


Polycarbonate spectra, collected with the 10Spec before (blue) and after Kramers-Kronig transformation.



Glass spectrum, measuring the sample's reflectivity.

205 mm



Extended Height 10Spec.

118 mm



Standard 10Spec.

30Spec and 45Spec

AT A GLANCE

- ▶ Measurement of thick films and film thickness
- ▶ Fixed angle of incidence, 30° or 45°
- ▶ Sample masks to define sampling area
- ▶ Slide-mount design for easy installation of accessory

The 30Spec and 45Spec are ideal for the measurement of relatively thick films by specular reflection.

Samples are simply laid across the top of the accessory and the spectrum of the film is measured quickly. The 30Spec and the 45Spec include sample masks of $\frac{3}{8}$ ", $\frac{1}{4}$ " and $\frac{3}{16}$ " to define specific sampling areas.

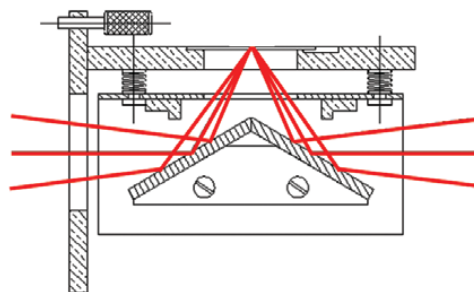
The 30Spec and 45Spec provide high-quality FTIR spectra for identification of coatings and can also be used to measure coating thickness. IR throughput is high using these accessories due to their relatively simple optical design.

These accessories are slide-mounted and are compatible with most FTIR instruments.

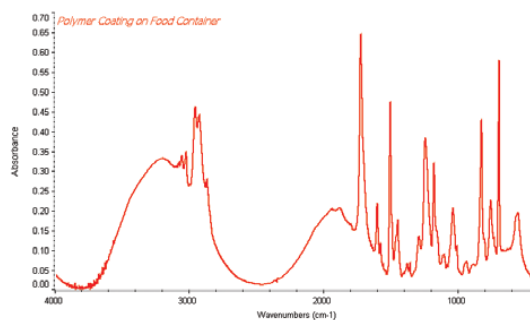
PART NUMBER	DESCRIPTION
011-1000	30Spec – 30° Specular Reflection Accessory
011-4500	45Spec – 45° Specular Reflection Accessory
Included Parts	
Sample Masks ($\frac{3}{8}$ ", $\frac{1}{4}$ ", and $\frac{3}{16}$ "), alignment mirror and slide-mount	
Replacement Parts	
011-2010	Sample Masks ($\frac{3}{8}$ ", $\frac{1}{4}$ ", and $\frac{3}{16}$ ")
300-0039	Aluminum Substrate Alignment Mirror

SPECIFICATIONS

Optics	All reflective
Angle of Incidence	30 degrees or 45 degrees
Sample Masks	$\frac{3}{8}$ ", $\frac{1}{4}$ " and $\frac{3}{16}$ "
Dimensions (W x D x H)	51 x 96 x 77 mm
Mount	2 x 3" slide mount



Optical geometry for the 30Spec and 45Spec.



FTIR spectrum of polymer coating on a food container collected using the PIKE 30Spec.

80Spec

AT A GLANCE

- ▶ Measurement of thin films and mono-molecular layers
- ▶ Fixed 80-degree angle of incidence
- ▶ Gold-coated reflective optics for highest throughput grazing angle analysis
- ▶ Integrated polarizer mounts
- ▶ Baseplate mount design for stable operation and collection of high-quality spectra

The 80Spec is ideal for the measurement of relatively thin films and mono-molecular layers by specular reflection.

Samples are simply placed face down across the top of the accessory and the spectrum is collected.

The 80Spec is available in two versions. The most popular version includes three sample masks to define smaller areas on a sample, and is recommended for smaller samples or for measurement of variations in thin film coatings. The basic configuration features a flat sampling surface with fixed sampling port (2 3/8" x 3/4"). This version is ideal for analysis of larger, uniform samples.

Generally, the measurement of ultra-thin film samples—especially monolayers—is significantly enhanced by using p-polarized light, with the electric field vector perpendicular to the sample surface. The 80Spec includes polarizer mounts on both incoming and outgoing beams for positioning optional manual or automated IR polarizers.



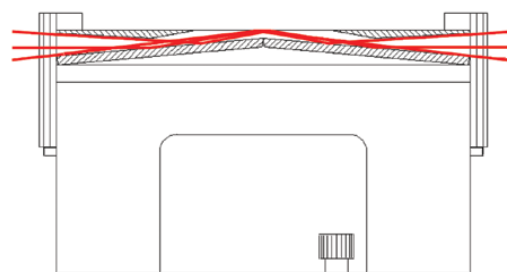
PART NUMBER	DESCRIPTION
012-11XX	80Spec – 80° Specular Reflection Accessory with Sample Masks (2", 5/8" and 3/8") Includes a gold substrate alignment mirror, dual polarizer mount and FTIR base mount
012-10XX	80Spec – 80° Specular Reflection Accessory Includes a gold substrate alignment mirror, dual polarizer mount and FTIR base mount

Notes: Replace XX with your spectrometer's Instrument Code listed in the back of the catalog. For compact-size spectrometers, only one polarizer mount may be included due to sample compartment width restriction.

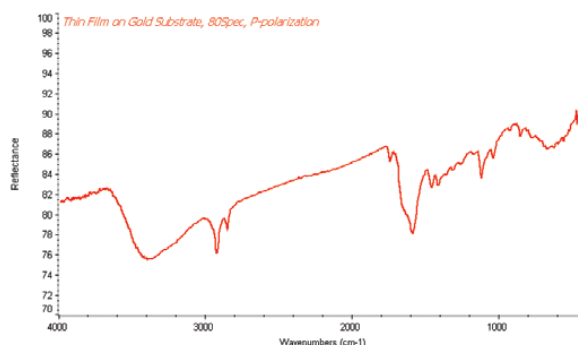
Replacement Parts and Options

010-3010	Specular Mask Set
300-0002	Gold Substrate Alignment Mirror, 1.25 x 3.0"
090-1000	Manual Polarizer, ZnSe
090-1200	Manual Polarizer, KRS-5
090-3000	Precision Manual Polarizer, ZnSe
090-3200	Precision Manual Polarizer, KRS-5
090-5000	Precision Automated USB Polarizer, ZnSe
090-5100	Precision Automated USB Polarizer, KRS-5

Note: For more polarizer options see the polarizer section of this catalog.



Beam path within the 80Spec specular reflection accessory.



FTIR spectrum of an ultra-thin film on a reflective substrate using p-polarized IR beam.

AGA Accessory

AT A GLANCE

- ▶ Quantitative measurement of small areas of thin films and mono-molecular layers
- ▶ Fixed 80-degree angle of incidence
- ▶ Measurement of lubricants on hard disks
- ▶ Sampling dimensions selectable from 1/2, 3/8, 1/4, 3/16 and 1/8 inch diameter
- ▶ Mount for optional polarizer
- ▶ Spectral range 10,000–500 cm⁻¹

The Advanced Grazing Angle (AGA) specular reflection accessory is a tool designed for quantitative measurement of spatially defined areas of thin films on reflective substrates.

Traditional grazing angle accessories produce an elliptical and non-uniform spot size on the sample area. This causes problems when quantitative analyses are performed on small sample areas. To overcome this design deficiency, the AGA accessory focuses the beam from the spectrometer onto the pin mirror. The portion of the beam that is reflected from this mirror is imaged at unit magnification onto the sample, striking it at 80 degrees. Thus, the beam at the sample position is uniform and circular in dimension.

The selectable slide-mounted pin mirrors range from 1/2" to 1/8" diameter. The AGA allows excellent quantitative results for the defined sample area.



PART NUMBER DESCRIPTION

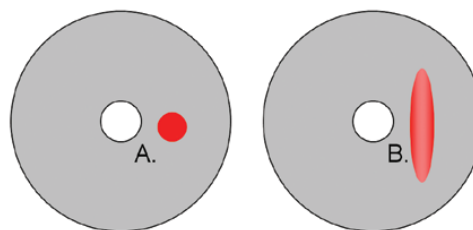
015-10XX AGA – Grazing Angle Specular Reflection Accessory
Includes 5 selectable spot sizes of 1/2", 3/8", 1/4", 3/16" and 1/8", gold substrate alignment mirror, polarizer mount and FTIR base mount

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

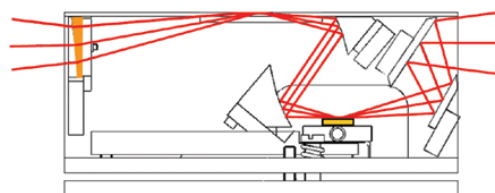
Replacement Parts and Sampling Options

300-0002	Gold Substrate Alignment Mirror, 1.25 x 3.0"
090-1000	Manual Polarizer, ZnSe
090-1200	Manual Polarizer, KRS-5

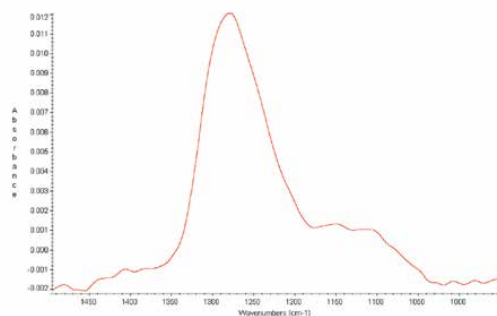
Note: For more polarizer options see the polarizer section of this catalog.



Sampling image on a hard disk surface produced by (A) the spatially resolved AGA and (B) a traditional grazing angle accessory.



Beam path within the AGA – Grazing Angle Specular Reflection Accessory.



FTIR spectrum of an 18-angstrom thick lubricant on a hard disk measured in 15 seconds using an MCT detector.

Absolute Reflection Accessory

AT A GLANCE

- ▶ Measurement of absolute reflectance of optical surfaces, windows and metallic surfaces
- ▶ Fixed 12-degree angle of incidence
- ▶ Performance evaluation of optical elements
- ▶ Evaluation of test plates in medical, industrial and military applications

The Absolute Reflection Accessory (ARA) is designed for making high precision specular reflection measurements.

Unlike traditional relative measurements where reflectance is a function of the background reference, the ARA does not require a background reflection standard due to its unique V/W optical arrangement.

When using relative reflection accessories, the sample reflectance is measured and calculated typically against a gold mirror that has a 94–99% reflectance in the infrared region. With the ARA, however, the reference mirror is integrated into both the background and sample measurements. In the V position, the beam reflects from the reference mirror in the background spectrum. In the W position, it reflects from the sample twice and the same reference mirror once at 12 degrees in the sample position. The absolute reflectance of a sample is calculated as the square root of the measured value at a given wavenumber or wavelength.



PART NUMBER DESCRIPTION

014-10XX Absolute Reflectance Accessory
Includes V / W sample holder, gold substrate mirror and FTIR base mount

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

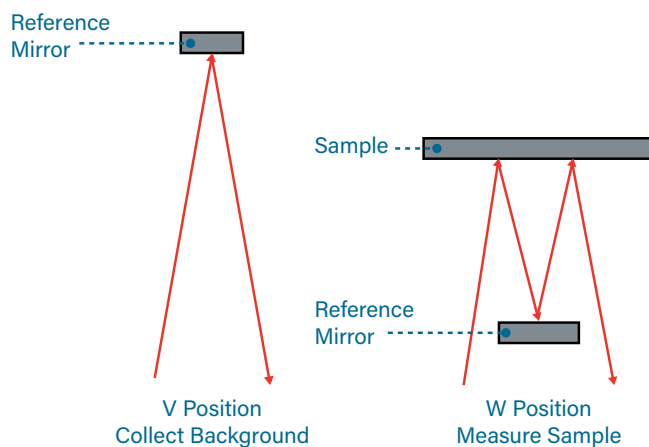
Replacement Parts and Sampling Options

300-0061 Gold Substrate Alignment Mirror, 2 x 3"

SPECIFICATIONS

Optics	All reflective
Angle of Incidence	12 degrees
Optical Configuration	V / W
Purge Sealing	Purge tubes and purge barb included
Dimensions (W x D x H)	165 x 241 x 146 mm
Sample Holder Opening	Oval, 40 mm (W) x 22 mm (H)
FTIR Compatibility	Most, specify model and type

The background and sample positions are easily selected by rotating the sample holder 180 degrees. With the ARA spring-loaded mount, sample loading and unloading is easy.



Beam path for V and W positions for the ARA.

Specular Reflection

Theory and Applications

Specular reflection sampling in the infrared is a useful technique for measuring thin films on reflective substrates, analyzing of bulk materials and measuring of mono-molecular layers on a reflective substrate. Often this technique provides a means of sample analysis with no sample preparation—keeping the sample intact for subsequent measurements.

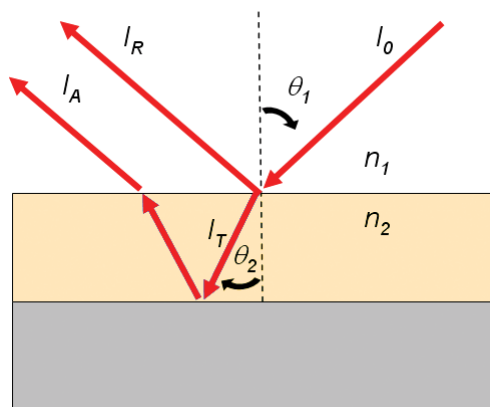
The basics of the sampling technique involve measurement of the reflected energy from a sample surface at a given angle of incidence. The electromagnetic and physical phenomena that occur at and near the surface are dependent upon the angle of incidence of the illuminating beam, refractive index and thickness of the sample and other sample and experimental conditions. A discussion of all of the physical parameters and considerations surrounding the specular reflection sampling technique is beyond the scope of this overview. We will present this technique from an applications-oriented perspective.

TYPES OF SPECULAR REFLECTION EXPERIMENTS

- ▶ Reflection-absorption of relatively thin films on reflective substrates measured at near-normal angle of incidence.
- ▶ Specular reflection measurements of relatively thick samples measured at near-normal angle of incidence.
- ▶ Grazing angle reflection-absorption of ultra-thin films or monolayers deposited on surfaces measured at a high angle of incidence.

REFLECTION-ABSORPTION MEASUREMENTS

In the case of a relatively thin film on a reflective substrate, the specular reflection experiment may be thought of as similar to a “double-pass transmission” measurement and can be represented as:



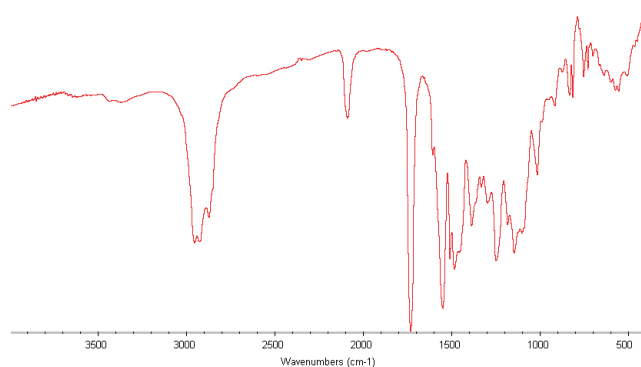
Beam path for reflection-absorption of a relatively thin film measured by specular reflection.

The incident FTIR beam represented by I_0 illuminates the thin film of a given refractive index (n_2) and at an angle of incidence (θ_1). Some of the incident beam is reflected from the sample surface, represented by I_R at the incident angle θ_1 and is also known as the specular component. Some of the incident beam is transmitted into the sample represented by I_T at an angle of θ_2 – calculated from Snell's Law.

$$n_1 \sin \theta_1 = n_2 \sin \theta_2$$

At the reflective substrate, the beam reflects back to the surface of the thin film. When the beam exits the thin film it has geometrically passed through the film twice and is now represented as I_A . Infrared energy is absorbed at characteristic wavelengths as this beam passes through the thin film and its spectrum is recorded.

The specular reflection spectra produced from relatively thin films on reflective substrates measured at near-normal angle of incidence are typically of high quality and very similar to spectra obtained from a transmission measurement. This result is expected as the intensity of I_A is high relative to the specular component (I_R).

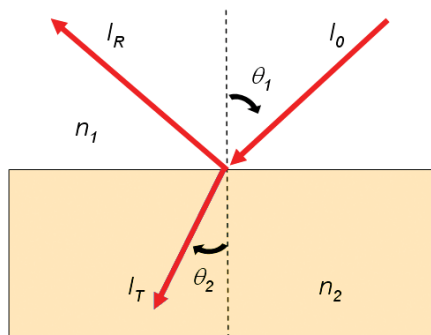


Spectrum of thin film coating on a metal surface measured at 30 degrees angle of incidence using the VeeMAX III specular reflection accessory.

REFLECTION MEASUREMENTS—THICK SAMPLES

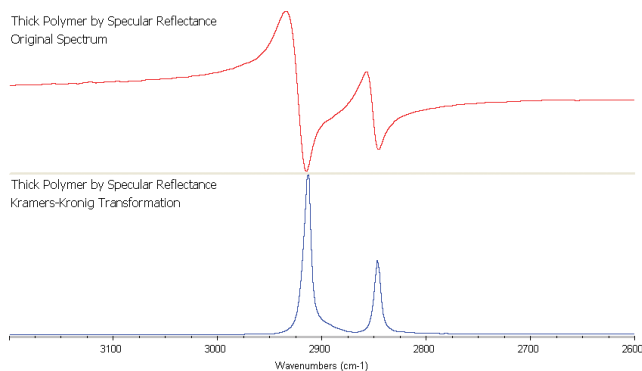
For relatively thick samples, the specular reflection experiment produces results which require additional considerations as the specular component of the total reflected radiation is relatively high.

Again, the incident FTIR beam represented by I_0 illuminates the sample of a given refractive index (n_2) and at an angle of incidence θ_1 . Some of the incident beam is reflected from the sample surface, represented by I_R at the incident angle θ_1 . Some of the incident beam is transmitted into the sample represented by I_T at an angle of θ_2 . As predicted by Fresnel equations, the percent of reflected versus transmitted light increases with higher angles of incidence of the illuminating beam. Furthermore, the refractive index of the sample, surface roughness, and sample absorption coefficient at a given wavelength all contribute to the intensity of the reflected beam.



Beam path for relatively thick sample, measured by specular reflection.

At wavelengths where the sample exhibits a strong IR absorption, the reflectivity of the sample increases. The super-position of the extinction coefficient spectrum with the refractive index dispersion results in a spectrum with derivative-shaped bands. This specular reflection spectrum can be transformed using the Kramers-Kronig conversion to a transmission-like spectrum as shown in the example below.

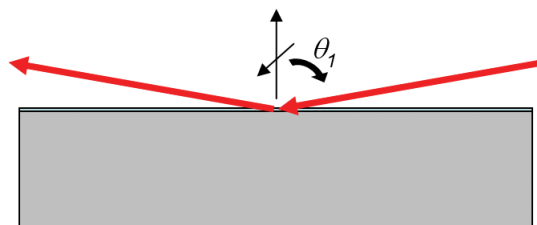


Spectrum (upper – original) of a relatively thick polymer sample measured at 30 degrees angle of incidence using the VeeMAX III; the lower spectrum has been transformed using the Kramers-Kronig software algorithm and is very similar to a transmission spectrum of the polymer polyethylene.

GRAZING ANGLE MEASUREMENTS—ULTRA-THIN FILMS AND MONOLAYERS

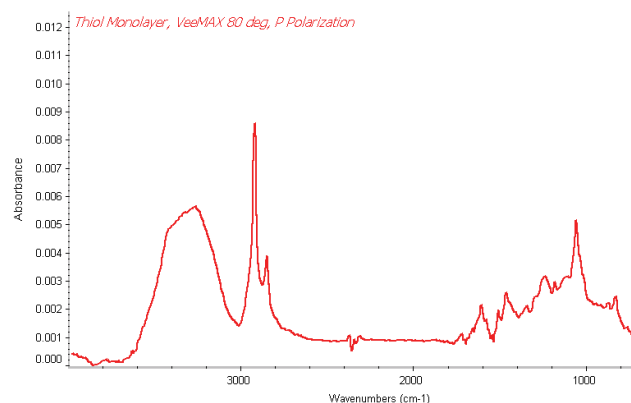
Our third application of specular reflection is the measurement of relatively thin films and mono-molecular layers at grazing angle of incidence. At high angles of incidence, between 60 and 85 degrees, the electromagnetic field in the plane of the incident and reflected radiation is greatly increased relative

to a near-normal angle of incidence. The perpendicular component of the electromagnetic field of the reflecting radiation is not enhanced.



Beam path for a relatively thick sample measured by specular reflection.

Because of the orientation of the electromagnetic field at the surface for grazing angle measurements, the use of an IR polarizer greatly improves the sampling result. By collecting the spectrum at grazing angle of incidence with p-polarization, we only examine the enhanced portion of the electromagnetic field at the sample surface, thereby producing a stronger absorbance spectrum.



Grazing angle specular reflection analysis of a thiol mono-molecular layer deposited on a gold-surfaced mirror using the PIKE VeeMAX III at 80 degrees and p-polarization; the FTIR was equipped with an MCT detector.

SUMMARY

Specular reflection is a valuable FTIR sampling technique for the analysis of thin films on reflective substrates, for relatively thick films on reflective materials and for bulk materials where no sample preparation is preferred. PIKE Technologies offers a complete line of specular reflection accessories to perform these analyses.