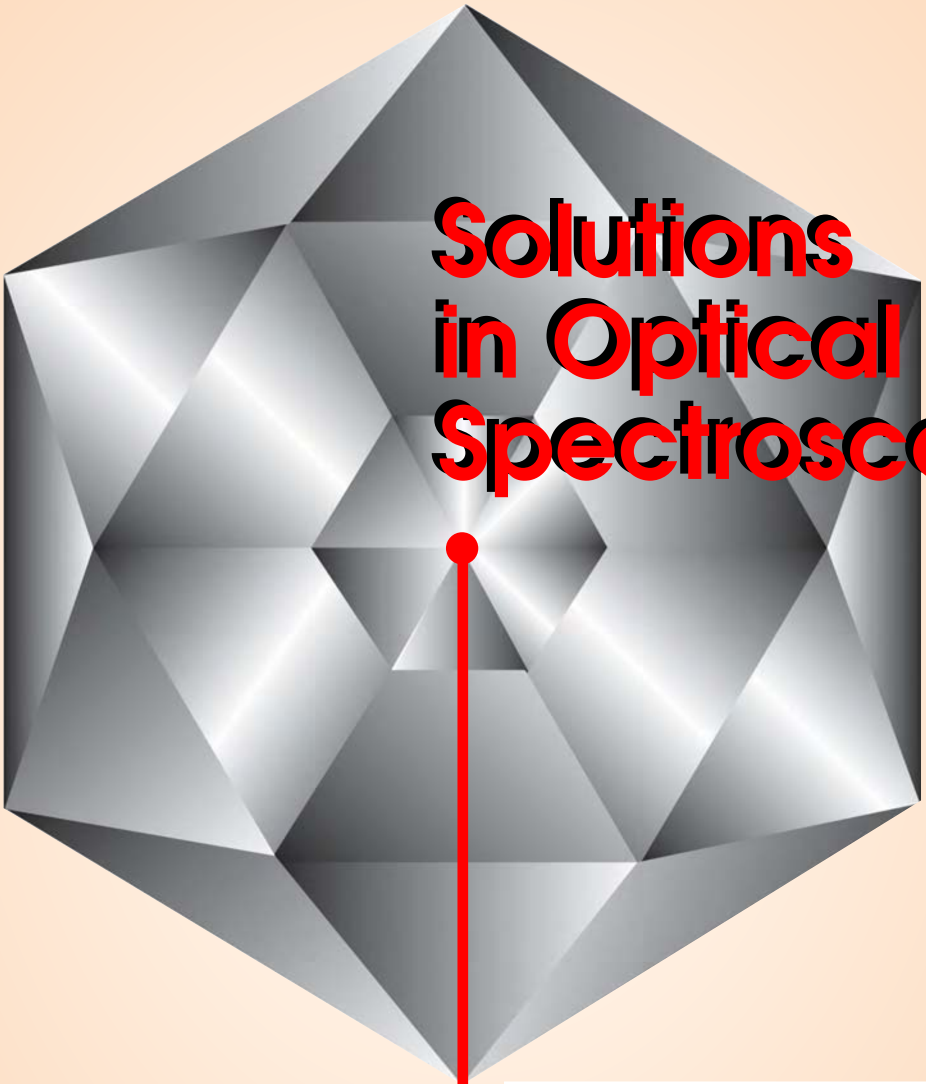
HARRICK

FTIR-VIS-UV ACCESSORIES



Solutions in Optical Spectroscopy

www.anadis.nl

ANADIS

INSTRUMENTS BENELUX B.V.

HARRICK SCIENTIFIC PRODUCTS, INC.

141 Tompkins Ave. • Box 277 • Pleasantville, NY 10562
914 / 747-7202 • FAX: 914 / 747-7209
Email: info@harrick.com • www.harricksci.com

Harrick Scientific Products, Inc. is located in Pleasantville, New York, thirty miles north of New York City, Harrick Scientific Products, Inc. specializes in designing and manufacturing instruments for optical spectroscopy.

Since its beginnings in 1969, Harrick Scientific has advanced the frontiers of optical spectroscopy through its innovations to transmission, internal reflection, external reflection, diffuse reflection, and emission spectroscopy.

Our founder, Dr. N. J. Harrick, pioneered internal reflection spectroscopy and was the principal developer of this technique.

Harrick Scientific offers a large selection of standard and custom-built attachments for IR and UV-VIS spectrometers. Many of these attachments were originally forerunners in their field and their contemporary versions are considered industry standards. Harrick Scientific continues to introduce innovative new products. In addition to these state-of-the-art attachments, Harrick Scientific supplies a complete line of optical elements, including windows, internal reflection elements, prisms, hemicylinders and hemispheres.

Harrick Scientific Products, Inc.

"Providing Solutions in Optical Spectroscopy"

Dr. N.J. Harrick began Harrick Scientific Corporation more than 30 years ago with the development of the first ATR accessory, sparking a revolution in the field of optical spectroscopy. Through the years, he and his staff worked together to solve the challenging application problems that scientists in the field were facing. Today Laurie Miller and the technical staff of Dr. Susan Berets, Lazaro Padron and Joe Lucania, the company that began with Dr. Harrick's discovery of ATR, flourishes with new products, fresh ideas and a commitment to solutions.

This new catalog introduces many new exciting products and also includes several of the standard accessories for which we are known. For more detailed product specifications, applications notes, and information on our newest developments, log on to our website. www.harricksci.com or call us at 800-248-3847.

GENERAL INFORMATION

FACILITIES

Harrick Scientific's facilities enable us to manufacture high quality spectroscopic equipment and optical components, as well as to develop future products that will lead our customers into new application areas and methodologies. Our staff is highly qualified. Their experience in a wide range of scientific and engineering fields makes them uniquely capable of both grasping problems and generating solutions.

- **RESEARCH AND DEVELOPMENT:** Our research and development staff becomes actively involved in meeting the challenges faced by our customers, because we believe that understanding our customers' needs is the only way to produce instrumentation.
- **TECHNICAL SUPPORT:** Our experts are available for consultation to determine the instrumentation that can best accomplish the required task.
- **ENGINEERING:** Working hand-in-hand with our research and development staff, engineering turns concepts into workable products. They determine how best to design the instrumentation for optimum performance.
- **MACHINING:** Our production facilities are equipped with state-of-the-art equipment for the precision production of our standard products, prototypes of new attachments, and custom instrumentation.
- **OPTICS:** Our excellent optical grinding and polishing facilities fabricate high quality optical elements. Harrick Scientific is able to offer a wide variety of windows, internal reflection elements, lenses, and mirrors.
- **ASSEMBLY/TESTING:** Harrick Scientific's attachments are assembled to specification. They are thoroughly inspected and tested prior to shipment.
- **CUSTOMER SERVICE:** Harrick Scientific's friendly and efficient customer service representatives are available to assist you.
- **WEBSITE:** www.harricksci.com displays our catalog, information on FTIR and much more.

CUSTOM INSTRUMENTATION

If you do not find the instrumentation for your specific application in our catalog, we can modify existing products or custom design the instrumentation required. Harrick Scientific has the reputation for being the problem solver in optical spectroscopy. We invite you to challenge us for the solution to your specific needs.

GUARANTEE

Harrick Scientific Corporation guarantees the performance of all items in this catalog. Should there be a defect, Harrick Scientific will, at our option, repair or replace the defective item or credit the purchaser's account in full, provided the purchaser brings the defect to our attention within 60 days of delivery.

TOXICITY

Almost any material is toxic in sufficient quantity. In compound form, many toxic elements are harmless, e.g. NaCl is an ingredient of everyone's diet and yet sodium and chlorine must be handled with extreme caution.

Many of the new optical materials with extremely desirable properties contain highly toxic elements such as Tl, Se, As, and Cd. Many of these materials in compound form are safe to handle although precautions should always be taken. Care must be exercised in dealing with the dust (e.g. Tl) and vapors (e.g. H_2Se) of these compounds. The grinding, polishing, and refinishing of these materials should be assigned to personnel familiar with appropriate handling techniques.

RECONDITIONING SERVICE

Our optical facilities also offer reconditioning services for damaged internal reflection elements and windows. Reconditioning optical elements is less costly than replacement and frequently provides the same high quality.

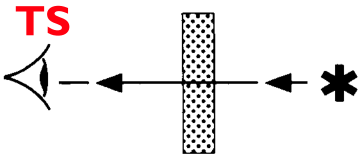
ORDERING INFORMATION

Telephone Orders:	914-747-7202 / 800-248-3847
FAX Orders:	914-747-7209
Written Orders:	Harrick Scientific Products, Inc. Box 277 / 141 Tompkins Ave. Pleasantville, NY 10562
Website:	www.harricksci.com
Terms:	Net 30 days on approved credit; FOB Pleasantville, NY; U.S. Dollars
Minimum Order:	$50; Credit Card orders accepted

- Please order by part number, whenever possible.
- We request a written confirmation for orders over $300 and for all non-standard specifications.
- If a non-standard order is canceled, the purchaser will be charged a cancellation fee for the work in progress.
- Many items are in stock and can be shipped when the order is received. Shipment dates will be quoted on non-stock items.
- Items are generally shipped via UPS with freight prepaid and added to the invoice. If an alternate carrier or faster delivery is required, notify us and the bill will be adjusted accordingly.

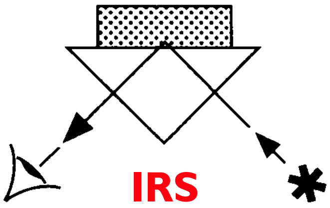
INTRODUCTION

Optical spectroscopy is one of the most powerful and widely used methods for material identification and analysis. The technique is employed over a broad spectral range from the ultraviolet to the far infrared. A wide variety of spectrometers, available from a number of suppliers, are in use daily. These instruments have many basic differences: some are dispersive (slit) and others are non-dispersive (Fourier Transform) instruments. Perhaps the single most common feature is that they are made for simple transmission of energy through samples.



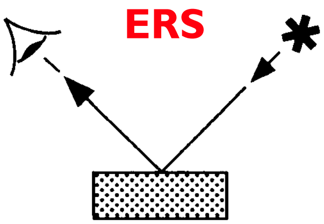
Even for simple transmission spectroscopy (TS), many accessories are needed to handle the wide variety of solid, liquid, and gas phase samples. These accessories, such as sample holders and beam attenuators, are often interchangeable for many instruments. However, this is limited by the different mounting methods employed and the windows required for different spectral ranges.

When making more sophisticated measurements, such as in internal reflection spectroscopy (IRS), external reflection spectroscopy (ERS), diffuse reflection spectroscopy (DRS), emission spectroscopy (ES), microsampling, long-path liquid or gas studies, and high or low temperature measurements, more elaborate instrumentation is required. For these purposes, conventional spectrometers often have limitations such as minimal space, magnified source image in the sample compartment and general inflexibility.



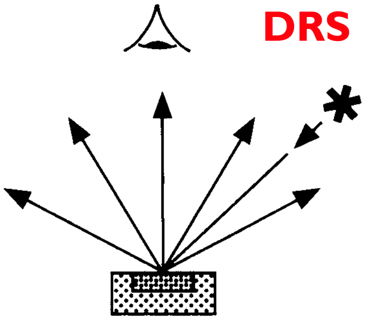
Since thousands of spectrometers are already in the field, there is a need for attachments which permit the use of existing spectrometers for many types of measurements. With good design, satisfactory results can be obtained in most cases, even though additional optical components are often required to shape and fold the light beam.

The purpose of an attachment or transfer optics, as differentiated from an accessory for that attachment, is to shape the light beam, direct it to the point of interest, and then reflect it into the spectrometer exactly as it would have entered in the absence of the attachment. The beam strikes the detector with the same angular beam spread and the beam focus is at precisely the same location with or without the attachment.



Most attachments are not interchangeable for spectrometers of different makes for both mechanical and optical reasons. Differences that must be considered are the following:

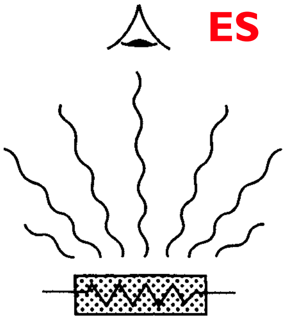
- Size of the sample compartment.
- Means of mounting.
- Location of the beam focus.
- Shape of the focus.
- Magnification of the source image.



Because of these variables, there is no simple classification of spectrometers which can be of value to the customer. Therefore, when ordering an attachment, the instrument with which it is to be used must be specified. Harrick Scientific is familiar with the various spectrometers and can assist in the selection of an attachment, or design and fabricate the attachments accordingly.

Harrick Scientific attachments have a number of significant advantages not found in many other makes. These include: simplicity, factory prealignment, minimal number of components, precisely mounted mirrors requiring only minor adjustments, maintenance of a sharper focus, and minimal beam distortion.

As will be seen from the following pages, a wide selection of attachments is offered. A guide for selecting an attachment is given on page 4. If after reviewing the features of each attachment and consulting the guide, you still have questions or you require a special solution for your needs, please call us.



CONTENTS

SAMPLING TECHNIQUES

SOLIDS

HARD				SOFT
Optically Thin[1]		*Optically Thick*[1]		
Smooth	***Rough***	***Smooth***	***Rough***	
Polymer Films, Pellets Optical Materials, Semiconductors	Laminates, Semiconductors	Plastics, Glasses	Ceramics, Ores	Flexible Polymers, Contact Lenses, Pastes, Papers
TS – Sample Holders, Beam Condenser ERS, RA[2] – Seagull, Versatile Reflection Att. ES – Emission Att.	DRS – Praying Mantis TS – Sample Holders, Beam Condenser BC – Beam Condenser ES – Emission Att.	ERS – Seagull, External Specular Reflection Att., Versatile Reflection Att., ATR – FastIR, MVP, Seagull, SplitPea, Horizon, Twin Parallel Mirror Att.	ATR – SplitPea DRS – Praying Mantis	ATR – FastIR, Horizon, Twin Parallel Mirror Att., Seagull, MVP, SplitPea, ConcentratIR

FILMS			
Free Standing		*Coatings*	
Thin	***Thick***	***Reflective Substrates***	***Non-Reflective Substrates***
Plastic Films	Polymers, Plastics	Lubricated Disks, Contaminated Wafers, Epitaxial Studies, Curing Epoxies	Coated Papers, Coatings on Agricultural Products, Epitaxial Studies, Epoxy Curing Studies
TS – Sample Holders ATR – FastIR, MVP, Seagull, Horizon, ConcentratIR, SplitPea, Twin Parallel Mirror Att. RA[2] – Versatile Reflection Att., Seagull	ATR – FastIR, MVP Seagull, ConcentratIR Horizon, Twin Parallel Mirror Att., SplitPea	ATR – GATR ERS – Focus, Refractor2, Seagull, Versatile Reflection Att., SplitPea	ATR – FastIR, MVP, Seagull, Horizon, Twin Parallel Mirror Att., SplitPea DRS – Praying Mantis, Cricket

FIBERS			POWDERS	
Hard	*Soft*	*Coated*	*Hard*	*Soft*
Plastics, Carbon Fibers, Glass Fibers	Fabrics, Wood	Plastics, Fabrics	Minerals, Catalysts	Clays, Plastics
ATR – SplitPea DRS – Praying Mantis, Cricket ES – Emission Att.	ATR – SplitPea, MVP, Horizon, Seagull, ConcentratIR, FastIR, Twin Parallel Mirror Att. DRS – Praying Mantis, Cricket	ATR – SplitPea, Seagull, Horizon, MVP, FastIR, ConcentratIR, Twin Parallel Mirror Att. ES – Emission Att.	ATR – SplitPea DRS – Praying Mantis, Cricket TS – Sample Holders, Pellet Presses	ATR – FastIR, MVP, SplitPea, Horizon, ConcentratIR, Seagull, PowderPro Twin Parallel Mirror Att. TS – Sample Holders, Pellet Presses

LIQUIDS

VISCOUS	NON-VISCOUS	
	Aqueous[1]	*Non-Aqueous*[1]
Oils, Pastes, Gels, Curing Epoxies	Beverages and Biological Samples	Oils, pastes, gels, curing epoxies
ATR – FastIR, Horizon, ConcentratIR, MVP, SplitPea RA[2] – Seagull TRS[3] – Praying Mantis, Cricket	ATR – FastIR, MVP, SplitPea, Seagull, Horizon, Twin Parallel Mirror Att.	TS – Demountable Liquid Cells ATR – Horizon, ConcentratIR, FastIR, MVP, SplitPea, Twin Parallel Mirror Att.

GASES

Gaseous Mixtures, Trace Analysis	TS – Demountable Gas Cells, Cyclops, Multiple Pass Variable Pathlength Cells

[1] The optical thickness of a material depends on the thickness and the absorption coefficient. An optically thin sample is essentially transparent, while an optically thick sample is opaque.

[2] Reflection-Absorption is a double TS technique. The radiation passes through the sample, reflects off the reflective substrate on which the sample rests, passes through the sample again, and is then detected. This technique uses external reflection attachments.

[3] Transflectance is similar to RA[2], except the sample is placed on a roughened substrate instead of a smooth one. This technique uses DRS attachments.

Harrick's sample holders, reaction chambers, and pellet makers are useful for solid sampling. The sample holders are easy to use and allow convenient mounting of pellets, films, windows, and crystals into the sample compartment of most spectrometers. The dewar and high temperature reaction chambers are well-suited to environmentally-controlled studies of pellets, films, and windows. Pellet makers are frequently used to prepare powders and grindable solids for analysis by infrared spectroscopy. The samples are finely ground to a uniform size, diluted with similarly ground KBr, and then pressed into a pellet for analysis.

BREWSTER'S ANGLE SAMPLE HOLDER

Applications

- Eliminates interference fringes which often obscure weak absorption bands when used with a polarizer.
- Handy for determining film thickness and refractive index.

Features

- Mounts samples at Brewster's angle or any other angle desired.
- A dial for accurately setting and reading the incident angle.
- Compatible with Harrick Scientific's high efficiency Single Diamond Polarizer, Wire Grid Polarizer and Glan Taylor Polarizer. (See page 36 for more information.)
- Mounts on standard sample slide holders.
- Includes two sample supports.

UNIVERSAL TRANSMISSION SAMPLE HOLDER

Applications

- Convenient for holding thin polymer films, windows, pellets and KBr pellet holders firmly in place.

Features

- Highly versatile.
- MagnaGrip˙ plate secures thin and flexible samples against a sample slide plate.
- MagnaGrip˙ plate slides on rods to accommodate samples up to 1.5" long.
- 20mm round aperture.
- Inserts with 13mm, 6mm, and 3mm apertures for securing smaller samples.
- Compatible with standard infrared sample slide holders.

Ordering Information	**Catalog No.**
Transmission Sample Holder	HUT-S1G
Brewster's Angle Sample Holder	BXH-S1G
Single Diamond Polarizer	PSD-J1R
Glan Taylor Polarizer	PGT-S1V
Wire Grid Polarizer	PWG-U1R

TRANSMISSION SAMPLING KIT

Our Transmission Sampling Kit includes all the tools and materials needed in sampling for infrared transmission spectroscopy. It contains all the accessories and supplies necessary for the analysis of liquids, both qualitative and quantitative, and solids, as mulls and pellets. This kit is packaged in a durable wooden carrying and storage case.

Applications

- General kit for transmission spectroscopy
- Process control and laboratory applications.
- Intended for analysis of liquids, solids and pastes

Features

- PTFE DLC2™ Demountable Liquid Cell with Luer Fittings. Uses standard, undrilled windows and precision PTFE spacers. Slide plate mounted to fit in all FT-IR spectrometers. Designed to examine liquids injected by syringe, solids smeared as mulls on KBr windows, viscous liquids and pastes smeared directly on the windows.
- MK Pellet Press used to prepare 13 mm diameter pellets. Pellets may be removed or left in the holder of the MK Press for analysis.
- Mortar and Pestle for grinding solids for pellet making and producing mulls. Made of highly durable alumina to prevent sample contamination during grinding.
- KBr Powder for diluting solid samples for analysis as pellets.
- Nujol and Fluorolube intended for mixing with finely ground solid samples to prepare them for analysis as mulls.
- KBr windows for use in the Liquid Cell and as supporting substrates for mulls.
- Universal Transmission Holder for supporting polymer films, pellets, the pellet press holder and mulls or pastes smeared between KBr windows. Readily slips into the sample slide holder in all FT-IR instruments.
- Sample Cards for supporting and storing samples such as pellets and polymer films.
- Spatula/Scoopula Combination and disposable Syringe for transferring samples during preparation.

Includes

- **Sample Holders**
  - PTFE Demountable Liquid Cell.
  - MK Pellet Press.
  - Universal Transmission Holder.
  - Sample Cards (10 pcs).

- **Sampling Accessories**
  - Spatula/Scoopula Combination.
  - Mortar and Pestle.

- **Supplies**
  - Fluorolube, 1 oz.
  - Nujol, 1 oz.
  - KBr powder, 100g.
  - Finger Cots (12 pcs).
  - KBr windows, 13x2 mm (8 pcs).
  - Syringe, 2ml.

Ordering Information	Catalog No.
Transmission Sampling Kit	KIT-TS1

Options and Replacement Parts

DLC2 Liquid Cell	DLC-L13	Fluorolube	KIT-FLB	Mortar and Pestle	KIT-MAP
Liquid Cell Viton O-Rings	ORV-012	MK Pellet Press with Die	MKP-S13	Nujol	KIT-NJL
Liquid Cell Kalrez O-Rings	ORK-012	KBr Powder	KBR-100	Sample Cards (100 pcs)	KIT-CRD
Liquid Cell Spacers	PSA-M13	KBr Windows (13x2mm)	WPD-U22	Spatula/Scoopula	KIT-SCP
Finger Cots (12 pcs)	KIT-COT	Universal Sample Holder	HUT-S1G	Syringes, 2 ml (10 pcs)	KIT-SYR

MK PELLET PRESS AND DIES

Applications

- ▶ Superior, inexpensive hand press.
- ▶ Non-rotating, highly polished, hardened stainless steel dies produce scratch-free pellets.

Features

- ▶ Large aperture eliminates vignetting when the pellet is left in the barrel for analysis.
- ▶ Pellets can be extracted easily without breakage.
- ▶ Evacuable for removing water prior to pressing.
- ▶ Five standard interchangeable dies.
- ▶ Includes pellet press, die, and collar of the specified size.

Ordering Information

Ordering Information	Catalog No.
Pellet Presses and Dies:	
13mm	MKP-S13

Optional and Replacement Parts for the MK Pellet Press

Dies:	Catalog No.
13 mm	MKD-S13
Spectroscopic Grade KBr Powder (100 g)	KBR-100

HIGH TEMPERATURE CELL

Applications

- Permits studies of solid samples at temperatures ranging from ambient to in excess of 500°C.

Features

- Thermally-insulated to inhibit heat loss.
- Easy to disassemble for sample exchange and cleaning.
- Fabricated from chemically-resistant 316 stainless steel.
- Accommodates samples 25mm in diameter and up to 10mm in thickness.
- Water-cooling eliminates the need for high temperature window seals.
- K-type thermocouple.
- Two 1⁄4″ 316 stainless steel VCO fittings.
- Includes two 32x3mm KBr windows and mating hardware for the specified spectrometer.

DEWARS

Applications

- Useful for examining solid samples by transmission and/or specular reflection at temperatures ranging from -196°C to 500°C.

Windows and O-rings for the High Temperature Cell and Dewars

	25x2mm	25x4mm	32x3mm
Windows:			
CaF_2	WFD-U25	WFD-U45	WFD-U36
BaF_2	WHD-U25	WHD-U45	WHD-U36
NaCl	WLD-U25	WLD-U45	WLD-U36
ZnSe	WMD-U25	WMD-U45	WMD-U36
KBr	WPD-U25	WPD-U45	WPD-U36
O-rings:			
Viton	ORV-020	ORV-020	ORV-024
Kalrez	ORK-020	ORK-020	ORK-024

Features

- Cold finger for coolant, e.g. liquid nitrogen or a dry ice and acetone mixture.
- Accommodates samples 25mm in diameter and up to 35mm in length.
- Windows mount by Viton O-ring seals.
- K-type thermocouple.
- Two 1⁄4″ 316 stainless steel VCO fittings.

Ordering Information

Ordering Information	Catalog No.
High Temperature Cell, 24Volt	HTC-3-XXX
Transmission/Specular Reflection Dewar (uses one 25x2mm or 25x4mm window, one 32x3mm window)	DER-XXX-3

Windows for the Dewar must be ordered separately.

Optional and Replacement Parts

Optional and Replacement Parts	Catalog No.
Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220V, 240 input, 24V output	ATC-024-2
Heater Assembly, 24V	HTC-HTR
Heater, 24V, 100W	HTRS-16
Heater with K-Type Thermocouple, 24W, 75V	HTRS-21

DLC2™ DEMOUNTABLE LIQUID CELLS

These affordable Demountable Liquid Cells are excellent for examining mulls, injected liquids and flowing liquids. These cells combine our time-tested leak-free design with PTFE cell body for superior chemical resistance. The Luer Lok fitting model is perfect for examining mulls and injected liquids under static conditions. With the flangeless fittings models, it is simple and straightforward to flow liquids through the cell for applications such as oil analysis. Both cells feature easily variable pathlengths.

Applications

- Ideal for infrared measurements of liquids, mulls, and smears.
- Excellent for process control and laboratory applications.

Features

- Variable pathlength from 25µm to 500µm.
- Leak-free design.
- Utilizes standard, non-drilled windows.
- Disassembles easily for cleaning and window replacement.
- Two ports for static or flow applications.
- Sample slide plate mounted for user convenience.
- Chemically resistant PTFE cell body and fittings.
- Choice of Luer Lok fittings or flangeless fittings with tubing.
- Open aperture: 8mm.
- Optional spacers available to extend the pathlength range from 6µm to 1mm.

Includes

- PTFE cell with Luer Lok or flangeless fittings.
- Viton o-rings.
- Slide plate mounting.
- Six PTFE pathlength spacers: 25, 56, 100, 150, 250 and 500 µm.
- Windows need to be ordered separately (see table).

Windows

Material	13x2mm
Al_2O_3	WBD-U22
CaF_2	WFD-U22
ZnS	WID-U22
NaCl	WLD-U22
ZnSe	WMD-U22
KBr	WPD-U22

Spacer Sets (12 per package)

Assorted		*PSA-M13*	
6µm	MSP-006-M13	250µm	MSP-250-M13
12µm	MSP-012-M13	390µm	MSP-390-M13
25µm	MSP-025-M13	500µm	MSP-500-M13
56µm	MSP-056-M13	630µm	MSP-630-M13
100µm	MSP-100-M13	750µm	MSP-750-M13
150µm	MSP-150-M13	950µm	MSP-950-M13

For additional spacer options see page 14- Spacer Sets (10 per package)

Contact Harrick Scientific for additional spacer twelve packs.

Ordering Information

	Catalog No.
DLC2™ Demountable Liquid Cell:	
Luer Lok Fittings (static)	DLC-L13
Flangeless Fittings for 1/16" Tubing (flow)	DLC-F13-062
Flangeless Fittings for 1/8" Tubing (flow)	DLC-F13-125

Options and Replacement Parts

Viton O-Ring	ORV-012
Kalrez O-Ring	ORK-012
Kalez O-Ring	ORK-012

DEMOUNTABLE LIQUID CELLS

These leak-free cells are the only Demountable Liquid Cells that you will ever need. Made from chemically resistant 316 stainless stelel with easily changed pathlengths, these cells are perfect for analyzing a wide variety of liquids and mulls under static or flow conditions.

Applications

- Infrared measurements of liquids, mulls, and smears.
- Process control and laboratory applications.
- Static or flow environments.

Features

- Variable pathlength, from 6µm to 1mm.
- Leak-free design utilizes standard, non-drilled windows.
- Easily disassembled for cleaning and window replacement.
- Two ports for static or flow applications.
- Sample slide plate mounted for user convenience.
- Chemically resistant 316 stainless steel cell body with Luer Lok or 1/8" Swagelok™ fittings.
- Available with an 8mm or 20mm diameter clear aperture.
- Maximum operating pressure with ZnSe windows 660 psi for the DLC-S13 (8 mm open aperture).
- 100 psi for the DLC-S25 (20 mm open aperture).
- Optional chemically-resistant Kalrez o-rings available.

Includes

- Stainless steel cell body with Luer Lok or Swagelok™ fittings.
- Viton o-rings.
- Slide plate mounting.
- One dozen Teflon spacers. One each for the following pathlengths: 6, 12, 25, 56, 100, 150, 250, 390, 500, 630, 750, and 950 µm.
- Windows need to be ordered separately (see spacer chart table).

Windows

Material	13x2mm	25x2mm
Al_2O_3	WBD-U22	—
CaF_2	WFD-U22	WFD-U25
ZnS	WID-U22	WID-U25
NaCl	WLD-U22	WLD-U25
ZnSe	WMD-U22	WMD-U25
KBr	WPD-U22	WPD-U25

For additional spacer options see page 14- Spacer Sets (10 per ackage)

Spacer Sets (12 per package)

	8mm aperture (13 x 2 mm)	*20mm aperture* (25 x 2 mm)
Assorted	***PSA-M13***	***PSA-M25***
6µm	MSP-006-M13	MSP-006-M25
12µm	MSP-012-M13	MSP-012-M13
25µm	MSP-025-M13	MSP-025-M13
56µm	MSP-056-M13	MSP-056-M13
100µm	MSP-100-M13	MSP-100-M13
150µm	MSP-150-M13	MSP-150-M13
250µm	MSP-250-M13	MSP-250-M25
390µm	MSP-390-M13	MSP-390-M13
500µm	MSP-500-M13	MSP-500-M13
630µm	MSP-630-M13	MSP-630-M13
750µm	MSP-750-M13	MSP-750-M13
950µm	MSP-950-M13	MSP-950-M13

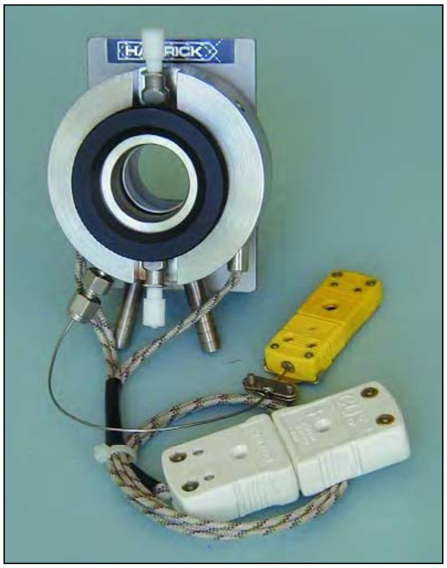
Ordering Information

Catalog No.	8mm aperture (13x2mm windows)	20mm aperture (25x2 windows)
Demountable Liquid Cell		
Luer Lok Fittings (static)	DLC-M13	DLC-M25
Swagelok™ Fittings (flow)	DLC-S13	DLC-S25

Options and Replacement Parts

Viton O-Ring)	ORV-012	ORV-020
Kalrez O-Ring	ORK-012	ORK-020

TEMPERATURE CONTROLLED DEMOUNTABLE LIQUID CELLS



Temperature Controlled Demountable Liquid Cells are intended for examining liquids and mulls at temperatures ranging from -80°C to 260°C. Their leak-free design makes them suitable for static and flow operation. In addition, these 316 stainless steel cells feature high chemical resistance and readily exchanged pathlength spacers.

Applications

- Infrared measurements of liquids, mulls and smears at elevated and reduced temperatures.
- Process control and laboratory applications.

Features

- Variable pathlength, from 6µm to 1mm.
- Heatable to 200°C with Viton o-rings or 260°C with optional Kalrez o-rings.
- Coolable to -80°C with suitable coolant, purge and o-rings.
- Flow-through cooling conduit stabilizes the cell temperature.
- Thermally isolated cell body prevents heat transfer to the spectrometer.
- Leak-free design utilizes standard, non-drilled windows.
- Easily disassembled for cleaning and window replacement.
- Two ports permit static or flow applications.
- Chemically resistant 316 stainless steel cell body with Luer Lok or 1/8" Swagelok™ fittings.
- Sample slide plate mounted for user convenience.
- Available with an 8mm or 20mm diameter open aperture.
- Maximum operating pressure with ZnSe windows
  - 660 psi for the TFC-S13 (8 mm aperture).
  - 100 psi for the TFC-S25 (20 mm aperture) (see spacer chart).

Includes

- Stainless steel cell body with Luer Lok or Swagelok™ fittings.
- Embedded K-type thermocouple, cartridge heaters and cooling conduit.
- Viton o-rings.
- Slide plate mounting.
- One dozen Teflon spacers. One each for the following pathlengths: 6, 12, 25, 56, 100, 150, 250, 390, 500, 630, 750, and 950 µm.
- Windows need to be ordered separately (see spacer chart).

Windows

Material	13x2mm	25x2mm
Al_2O_3	WBD-U22	—
CaF_2	WFD-U22	WFD-U25
ZnS	WID-U22	WID-U25
NaCl	WLD-U22	WLD-U25
ZnSe	WMD-U22	WMD-U25
KBr	WPD-U22	WPD-U25

Spacer Sets (12 per package)

	8mm aperture (13 x 2 mm)	*20mm aperture (25 x 2 mm)*
Assorted	***PSA-M13***	***PSA-M25***
6µm	MSP-006-M13	MSP-006-M25
12µm	MSP-012-M13	MSP-012-M13
25µm	MSP-025-M13	MSP-025-M13
56µm	MSP-056-M13	MSP-056-M13
100µm	MSP-100-M13	MSP-100-M13
150µm	MSP-150-M13	MSP-150-M13
250µm	MSP-250-M13	MSP-250-M25
390µm	MSP-390-M13	MSP-390-M13
500µm	MSP-500-M13	MSP-500-M13
630µm	MSP-630-M13	MSP-630-M13
750µm	MSP-750-M13	MSP-750-M13
950µm	MSP-950-M13	MSP-950-M13

For additional spacer options see page 14-Spacer Sets (10 per package)

Ordering Information

	Catalog No.	
	8mm aperture (13x2mm windows)	20mm aperture (25x2 windows)
Temperature Controlled Demountable Cells		
Liquid Cell (Luer Lok fittings)	TFC-3-M13	TFC-3-M25
Flow Cell (Swagelok™ fittings)	TFC-3-S13	TFC-3-S25

Options and Replacement Parts

Viton O-Ring)	ORV-012	ORV-020
Kalrez O-Ring	ORK-012	ORK-020
Automatic Temperature Controller, 110V input, 24V output		ATC-024-1
Automatic Temperature Controller, 220V, 240 input, 24V output		ATC-024-2
Heater Assembly, 24V		TFC-HTR
Heater, 24V, 100W		HTRS-16
Heater with K-Type Thermocouple		008-144

HIGH PRESSURE DEMOUNTABLE LIQUID CELLS

Applications

- Permits infrared transmission studies of pressurized liquids, mulls, and smears at elevated temperatures and pressures.
- Well suited for both laboratory and process control applications.

Features

- Variable pathlength (6µm to 1mm).
- Easy to disassemble, clean and replace the windows.
- 8mm clear aperture.
- Chemically resistant 316 stainless steel body.
- Includes two ports with 1/8" Swagelok™ fittings for static and flow-through applications.
- Sample slide plate mounted for easy installation in FTIR spectrometers.
- Two models available.
  - High Pressure Liquid Tranmission Cell for operation to 5000 psi
  - Temperature-Controlled High Pressure Transmission Cell. Heatable to 200°C with Viton O-rings or 260°C with Kalrez O-rings. Thermally-insulated. Features flow-through coolant conduit, K-type thermocouple, and imbedded cartridge heaters. Maximum pressure depends on operating temperature and window material.
- Optional stainless steel pathlength spacers available.
- Additional spacers available for pathlengths up to 5mm (see spacer chart table).

Includes

- Stainless steel transmission cell, Viton O-rings, and mounting hardware for the windows.
- Assorted pathlength spacers made of Teflon. One each of the following thicknesses: 6, 12, 25, 65, 100, 150, 250, 390, 500, 630, 750, and 950µm.
- Heatable model includes thermocouple, heaters, and cooling conduit.
- Slide plate mounting.
- Windows must be ordered separately.

Spacer Sets (10 per package)

	8mm aperture (13 x 2 mm)	20mm aperture (25 x 2 mm)
15µ	MSB-015-M13	MSB-015-M25
25µm	MSB-025-M13	MSB-025-M25
50µm	MSB-050-M13	MSB-050-M25
100µm	MSB-100-M13	MSB-100-M25
200µm	MSB-200-M13	MSB-200-M25
500µm	MSB-500-M13	MSB-500-M25
1000µm	MSB-000-M13	MSB-000-M25

Spacer Sets (12 per package)

6µ	MSP-006-M13
12µm	MSP-012-M13
56µm	MSP-056-M13
150µm	MSP-150-M13
250µm	MSP-250-M13
390µm	MSP-390-M13
630µm	MSP-630-M13
750µm	MSP-750-M13
950µm	MSP-950-M13

Assorted Spacers Sets

	8mm apt. (13x2 mm)	20mm apt (25 x2 mm)
Two each (25, 50, 100, 200 and 1000µm	PSB-M13	PSB-M25
One each (6, 12, 25, 56, 100, 150, 250, 390, 500 630, 750 and 950µm	PSA-M13	PSA-M25

Windows*

Zns	WID-U62
ZnSe	WIMD-U62

Ordering Information

High Pressure Liquid Cell (13x6mm windows)	HPL-C-13
Temperature Controlled High Pressure Liquid Cell, 24V (13x6mm windows)	HPL-TC-13-3

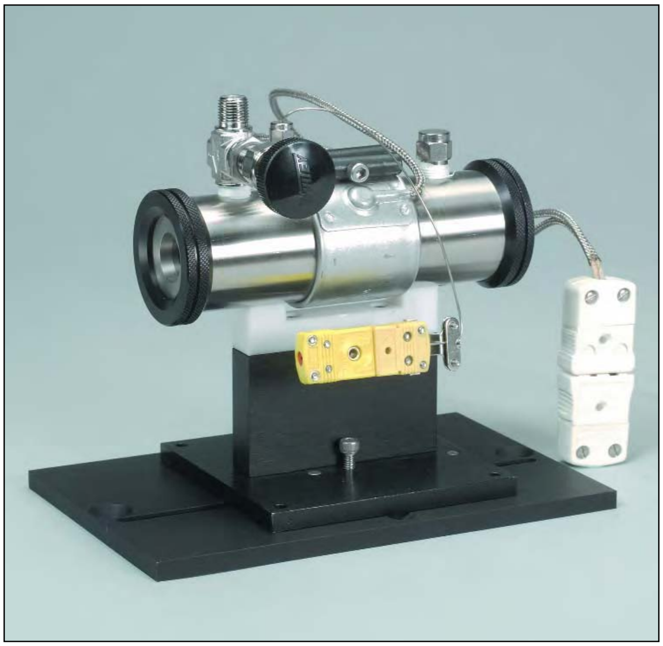
Options and Replacement Parts

Automatic Temperature Controller, 110V input, 24V output	ATC- 024-1
Automatic Temperature Controller, 220V/240V input, 24V output	ATC- 024-2
Viton O-ring	ORV-012
Kalrez O-ring	ORK-012
0.1mm thick stainless steel spacer	HPL-M01
1.0mm thick stainless steel spacer	HPL-M10
0.2mm thick stainless steel spacer	HPL-M02
2.0mm and 3.0mm thick stainless steel spacer set	HPL-M20
0.5mm thick stainless steel spacer	HPL-M05
4.0mm and 5.0mm thick stainless steel spacer set	HPL-M40
Heater Assembly for Demountable Liquid Cell, 24V	TFC-HTR
Heater, 24V, 10 0W	HTRS-16

**Windows must be ordered separately from chart*

SINGLE PASS GAS CELLS

These gas cells are ideal for examining gases and gaseous mixtures in static or flow environments. Both cells are excellent for room temperature studies and the Temperature Controlled Gas Cell is also well suited for heated measurements.



Applications

- ▶ Well-suited for spectroscopic studies of gases and vapors from room temperature to more than 200ºC.
- ▶ Ideal for interfacing to gas chromatographs for further analysis.

Features

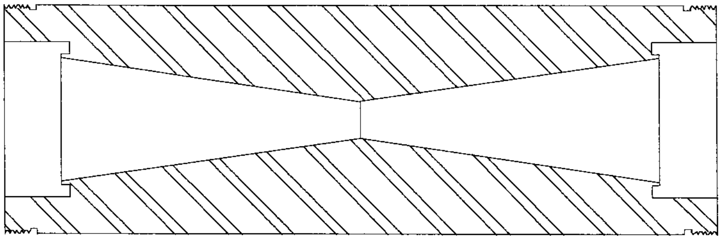
- ▶ 10cm pathlength.
- ▶ Two ports allow static and flow applications.
- ▶ Beam conforming interior maximized pathlength to volume ratio.
- ▶ Low 17ml volume (approx.)
- ▶ 20mm diameter clear entrance and exit apertures.
- ▶ Floor mounted for greater stability during analysis.
- ▶ Two models available.
  - Demountable Gas Cell featuring a Teflon body. Includes two 1⁄8″ NPT Teflon valves
  - Temperature Controlled Gas Cell, heatable to 200ºC with Viton O-rings or 260ºC with Kalrez O-rings. Thermally isolated, stainless steel cell includes embedded K-type thermocouple, band heater. Includes 1⁄8″ NPT 316 stainless steel plug and valve.

Includes

- ▶ Teflon or stainless steel cell body, Viton O-rings, and mounting hardware for the windows.
- ▶ Two ports with NPT fittings.
- ▶ Mating hardware for the specified spectrometer.
- ▶ Windows must be ordered separately.

Windows and O-rings

	25x2mm	25x4mm
Windows:		
CaF_2	WFD-U25	WFD-U45
ZnS	WID-U25	WID-U45
NaCl	WLD-U25	WLD-U45
ZnSe	WMD-U25	WMD-U45
KBr	WPD-U25	WPD-U45
O-Rings:		
Viton	ORV-020	ORV-024
Kalrez	ORK-020	ORK-024



Ordering Information

Demountable Gas Cell	DGC-PT1
Temperature Controlled Gas Cell, 24V	TGC-3-XXX

•*Windows must be ordered separately.*

Options and Replacement Parts

Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220V/240 V input, 24V output	ATC-024-2
Heater Assembly , 24V	TGC-HTR
Heaters, 24V, 200W	HTRS-22

External or specular reflectance is ideal for examining surface coatings and reflective opaque materials. Grazing incidence, as provided by the Refractor and the Disk Checker, is recommended for examining thin films. Smaller incident angles, as provided by the Versatile Reflection and External Reflection Attachments, are useful for studying thicker films and opaque materials.

EXTERNAL SPECULAR REFLECTION ATTACHMENT

Applications

- ▶ Valuable for studies of films on metallic substrates and measurements of epitaxial film thickness.

Features

- ▶ Horizontal sampling surface.
- ▶ Fixed 12.5°, 30°, 45°, or 60° incident angle. Other angles available on special order.
- ▶ Simple alignment.
- ▶ High throughput for optimum performance.
- ▶ Utilizes interchangeable masks for examining small samples or small areas of large samples.
- ▶ Includes mating hardware for the specified spectrometer and an alignment mirror.

Ordering Information

External Specular Reflection Attachment	Catalog No. Slide Plate Mounted	Floor Mounted
12.5°	ERA-12G	ERA-12-XXX
30°	ERA-30G	ERA-30-XXX
45°	ERA-45G	ERA-45-XXX
60°	ERA-60G	ERA-60-XXX

VARIABLE ANGLE REFLECTION ACCESSORY

Harrick's Variable Angle Specular Reflection Accessory is ideal for specular reflection studies of films on metallic substrates, coatings, contaminants on reflective surfaces, and measurements of film thickness. The sample stage is readily adapted for absolute reflectance measurements or convenient horizontal reflectance measurements at a 12° incident angle. Models available for use in all FT-IR spectrometers and most UV-VIS spectrometers.

Applications

- ▶ Specular reflection studies of coatings on reflective substrates in the infrared of UV-VIS.
- ▶ Films on Metallic substrates.
- ▶ Determining film thickness.
- ▶ The industry standard for variable angle specular reflectance spectroscopy.

Features

- ▶ Incident angle from 30-85°.
- ▶ Accomodates samples larger than $^{1}/_{2}$” x $^{1}/_{2}$”, up to 5” thick.
- ▶ Maintains alignment for incident angles.
- ▶ Adaptable for variable angle, fixed angle, and absolute specular reflectance studies.
- ▶ Models available for a wide range of FT-IR and UV-VIS spectrometers.

Includes

- ▶ Optical base unit with variable angle specular reflectance sample stage.
- ▶ Mating hardware for the specified UV-VIS or FT-IR spectrometer.
- ▶ Optional sample stages for absolute specular reflectance and horizontal sampling.

Ordering Information	**Catalog No.**
Variable Angle Specular Reflection Accessory	VRI-XXX
Optional Sample Stages	
12° Absolute Reflectance Sampling Stage	VR1-VWA-12
Horizontal Reflection Stage	VR1-HRS
45° Absolute Reflectance Sampling Stage	VR1-VWA-45
Options	
Wire Grid Polarizer, KRS-5 Substrate	PWG-U1R
Glan-Taylor Polarizer	PGT-S1V
Glan Thompson Polarizer	PTH-SMP

XXX denotes the spectrometer code

Absolute Reflectance Accessories
12° Absolute Reflectance Accessory

Applications

- ▶ Ideal for examining highly reflective substrates.
- ▶ Utilizes a double reflection technique to measure the square of reflectance.

Features

- ▶ Incorporates a fixed 12° incident angle.
- ▶ Alignment and pathlength are maintained for both sample and reference.
- ▶ Retains the polarization of the incident radiation.

Includes

- ▶ 12° Absolute Reflection Accessory.

45° Absolute Reflectance Accessory

Applications

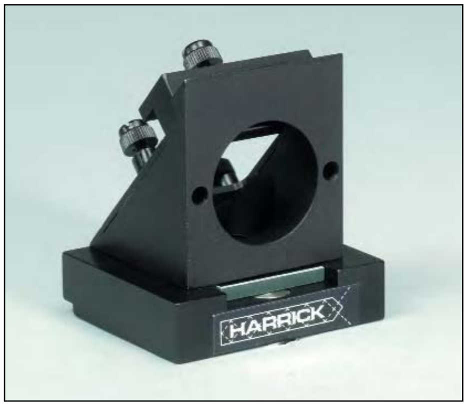
- ▶ Useful for studying highly reflective substrates.
- ▶ Measures the square of reflectance through a double reflection technique.

Features

- ▶ Incorporates a fixed 45° incident angle.
- ▶ Alignment and pathlength are maintained for both sample and reference.

Includes

- ▶ 45° Absolute Reflectance Accessory.



Horizontal Reflection Stage

Applications

- ▶ Permits reflectance measurements at a 12° incident angle.
- ▶ Horizontal sampling stage for straight-forward sample exchange and surface scanning.

Includes

- ▶ Horizontal Reflection Stage.
- ▶ Alignment mirror.

THE REFRACTOR2™

The Refractor2™ is a unique attachment for grazing incidence specular reflectance studies. The Refractor2™ incorporates two SuperCharged™ wedged windows to refract the beam to and from the sample. It also includes a pre-mounted, internal Si polarizing plate for enhanced spectral contrast. This unique design results in a compact attachment and avoids the energy losses associated with the plane mirrors typically employed.

Applications

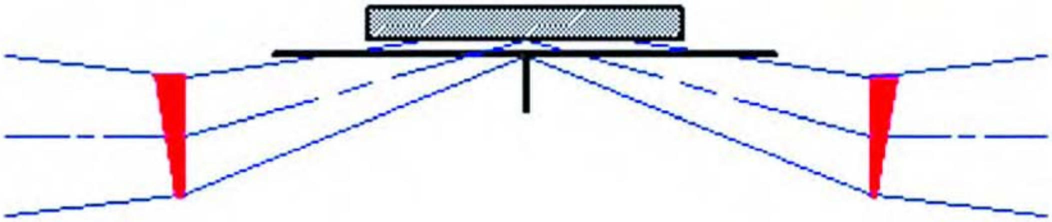
- Ideal for recording spectra of thin films on metal and semiconductor substrates.
- Well suited for laboratory and QC applications.

Features

- Low cost.
- Unobstructed, horizontal sampling surface for high sample throughput.
- Fixed 75° incident angle and built-in, removable polarizer provides high spectral sensitivity.
- Two SuperCharged™ ZnSe wedged windows refract the beam to and from the sample. KRS-5 windows available on special order.
- Compact.
- SuperCharged™ for optimal energy throughput.
- Easy to align and use.
- PermaPurge™ for rapid sample exchange without interrupting the purge.
- Removable PermaPurge™ assembly – accommodates samples up to 4.75” wide when installed and even larger when removed.

Includes

- Two SuperCharged™ ZnSe wedged windows.
- Si polarizing plate.
- Alignment mirror.
- Mating hardware for the specified spectrometer.



Ordering Information — Catalog No.

Refractor2™	RG2-XXX

Replacement Parts

Silicon Polarizing Plate	RGA-000
SuperCharged™ ZnSe Wedged Window	RGA-001

THE REFRACTOR™ REACTOR

The Refractor™ Reactor is a valuable tool for grazing angle studies of reactions on metal surfaces and on thin films on metal substrates. Samples can be heated to 400°C and pressurized up to 2 ATM. The Refractor™ Reactor incorporates many of the design features of the Refractor™ in a reaction chamber.

Applications

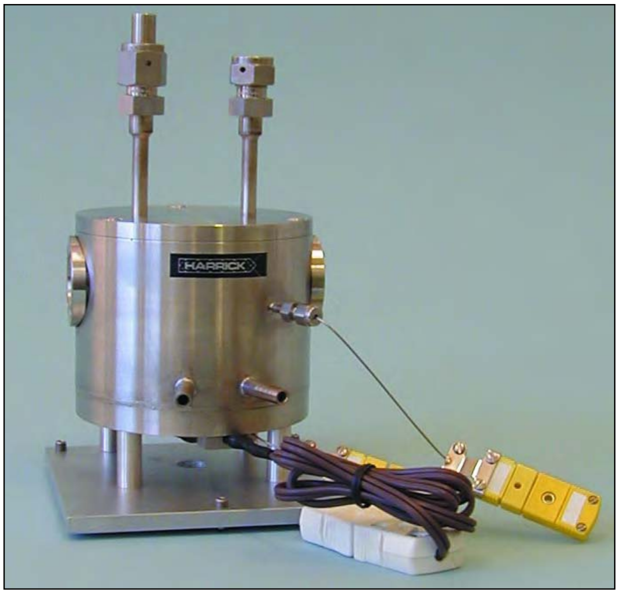
- Perfect for studying reactions on metal surfaces and on thin films on metal substrates.

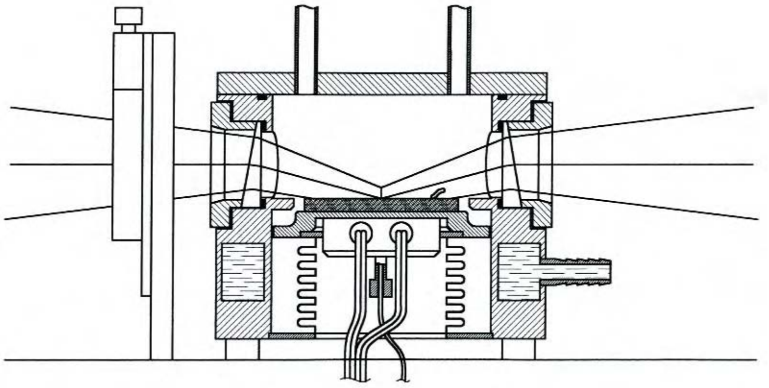
Features

- Fixed 75° incident angle for high sensitivity to films on metallic substrates.
- Incorporates two SuperCharged™ ZnSe wedged windows to refract the beam to and from the sample. KRS-5 windows available by special order.
- Maximum sample size 0.125″ thick by 1.7″ x 1″.
- Heatable to 400°C for studying high temperature reactions.
- Operable at pressures from 10^{-4} torr to 2 ATM.
- High efficiency.

Includes

- Two SuperCharged™ ZnSe wedged windows.
- Alignment mirror.
- Mating hardware for the specified spectrometer.





O-Rings

	Viton	Kalrez
Windows (#19)	ORV-019	ORK-019
Covers (#38)	ORV-038	ORK-038

Ordering Information

	Catalog No.
Refractor™ Reactor, 24V	RGR-XXX-3

Options

Wire Grid Polarizer	PWG-U1R
Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220V/240 input, 24V output	ATC-024-2

Replacement Parts

SuperCharged™ ZnSe Wedged Windows	RGA-001	Accessory Vacuum Pumps	VPE-001
Heater Assembly, 24V	RGR-HTR	Accessory Vacuum Pumps	VPE-002
Heater, 24V, 96W	HTRS-19	Oil, 1 quart	VPE-OIL

REFLECTANCE REFERENCE

The reflectance reference provides a method for calculating the reflectivity of a material from a measured spectrum of a sample. The experimental measurement is obtained in a particular wavenumber region for s-, p- or mixed polarization. The mid-infrared reflectance reference is suitable for use from 5000cm^{-1} to 400cm^{-1}.

Applications

- ▶ Absolute reflectance measurements of materials.
- ▶ Ideal for coatings on mirrors and reflective substrates.

Features

- ▶ Ge reflectance reference.
- ▶ Unique design collects only front-surface reflectance.
- ▶ Suitable for use from 5000cm^{-1} to 400cm^{-1}.
- ▶ Designed for operation with the Seagull, Variable Angle Reflection Accessory, Near-Normal Reflectance Accessory and External Specular Reflection Accessory.
- ▶ CD with an Excel spreadsheet configured to:
  - ▶ Calculate the reflectivity of the reference at various incident angles, polarizations and wavenumbers.
  - ▶ Extract the reflectivity of the sample from experimental data.

Includes

- ▶ Ge reflectance reference.
- ▶ Comes equipped to mount into the Seagull, Variable Angle Reflection Accessory, Near-Normal Reflectance Accessory and External Specular Reflection Accessory.
- ▶ Instruction manual.
- ▶ CD with reflectance data and formulas in an Excel spreadsheet.

Ordering Information	Catalog No.
Mid-Infrared Reflectance Reference (Ge)	RRF-00J

THE FOCUS™

This grazing angle specular reflectance accessory focuses on obtaining high quality spectra quickly and efficiently. It is excellent for examining a variety of films and coatings on metals or other reflective substrates. The Focus™ features a 75° incident angle and a built-in Si polarizer for grazing angle measurements with high spectral contrast. Its convenient horizontal sample stage and condensed beam make it perfect for examining a wide variety of sample sizes and shapes. The Focus™ features PermaPurge™ for rapid sample and crystal exchange without interrupting the purge of the spectrometer. For applications versatility, the Focus™ can readily be adapted for multiple reflection ATR experiments.

Applications

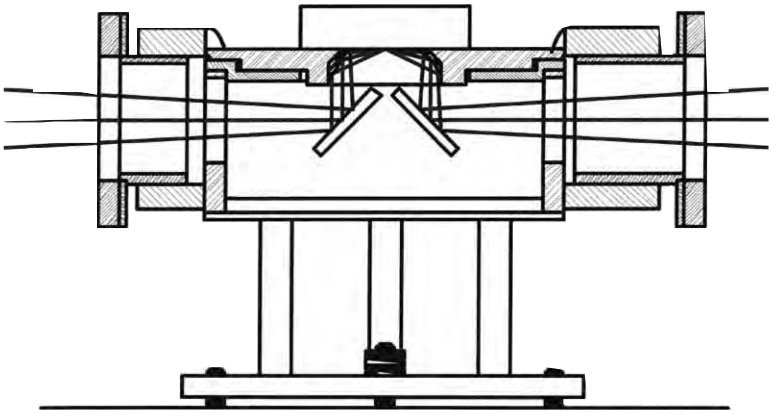
- Ideal for examining coatings on mirrors and other reflective substrates.
- Excellent for small and large samples.
- Invaluable for QC applications.

Features

- 75° grazing incident angle.
- Built-in silicon polarizing plate for optimal spectral contrast.
- Convenient horizontal stage.
- High sample throughput.
- Small sampling area.
- Optional upgrade for multiple reflection ATR.
- Harrick's exclusive PermaPurge™ allows rapid sample without interrupting the purge of the system.
- **Readily convertible to ATR with the ConcentratIR adapter (See page 19).**

Includes

- Built-in polarizer.
- Mating hardware for the specified spectrometer.



Ordering Information	Catalog No.
Focus™ Reactor	FCS-XXX
Options	
Multiple Reflection ATR Upgrade to the ConcentrateIR	CON-FOC

ACCESSATR™

The AccessATR™ makes ATR FTIR spectroscopy accessible to everyone. It is perfect for quick and accurate ATR measurements of most samples and it is affordable. Simply place the sample ñ a solid, liquid, or paste - on the ATR crystal, record the spectrum and wipe the crystal clean. The AccessATR™ is equipped with a built-in trough to contain liquids and a press for solid sampling.

Applications

- Convenient for routine analysis of liquids, pastes, and flexible solids.
- Single reflection ATR (internal reflectance) accessory.
- Convenient for routine analysis of liquids, pastes, and flexible solids.
- Designed for highly repeatable qualitative and quantitative FTIR measurements.

Features

- Convenient, horizontal sampling surface.
- Top loading.
- Fixed 45° incident angle.
- Spectral range: 20,000 cm^{-1} to 550 cm^{-1}.
- SuperCharged™ ZnSe optics for superior performance.
- Easy to use – no alignment required.
- Excellent throughput.
- Little or no sample preparation required.
- Gasket-sealed ATR crystal preserves sample integrity.
- Readily replaceable affordable ZnSe ATR crystal.
- Convenient slide plate mounting fits all FTIR spectrometers.

Includes

- Mounted ZnSe prism.
- Integral trough for analyzing liquids, powders and pastes..
- Built-in pressure applicator for solid sampling.
- Slide plate mounting.

Ordering Information

	Catalog No.
AccessATR™	ACC-ATR

Options

Sample Slide Plate Holder	HSS-XXX*
Rail Plate	HRM-XXX*

Replacement Parts

ZnSe ATR Crystal	ACC-PRM
Gasket	ACC-GSK

**XXX indicates spectrometer make and model*

FASTIR™

The FastIR™ is our newest single reflection horizontal ATR accessory. The FastIR™ incorporates a convenient horizontal sample stage for quick ATR measurements of solids, liquids, and pastes. Sample analysis is simple and straightforward: place or spread the sample on the ATR crystal, record the spectrum and wipe the crystal clean. The FastIR™ incorporates PermaPurge™ for rapid sample and crystal exchange without interrupting the purge of the system. A trough, flow-through liquid cell, and temperature controlled liquid cell are also offered.

Applications

- Convenient for routine analysis of liquids, pastes, and flexible solids.
- Excellent for both laboratory and quality control environments.

Features

- Provides highly repeatable quantitative results.
- Convenient, horizontal sampling surface.
- Single reflection ATR.
- Fixed 45° incident angle.
- Top loading.
- SuperCharged™ ZnSe optics for superior performance.
- Easy to align and use.
- High throughput (greater than 85%).
- Minimal sample preparation required.
- Readily exchangeable crystal.
- PermaPurge™ for rapid purging of the system.

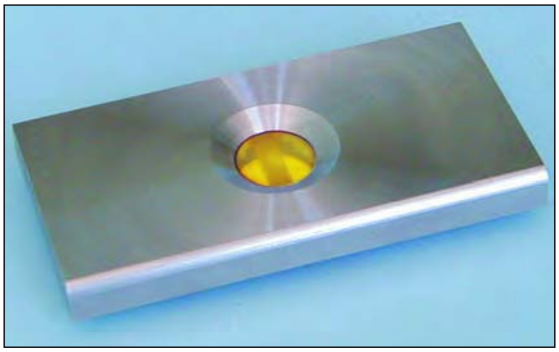
Includes

- Mounted ZnSe prism.
- Built-in pressure applicator and clamp for solid sampling.
- Glass cover slide for volatile samples.
- Mating hardware for the specified spectrometer.

Ordering Information	Catalog No.
FastIR™	FAS-XXX
Options	
Slip-Clutch	SLP-CHI
Torque Screwdriver	PTW-SXX
FasrtIR Wire Grid Polarizer (KRS-5 Substrate). Includes mount and polarizer	PWD-FAS-XXX
FastIR Wire Grid Polarizer Mount. Compatible with the PWD Wire Grid Polarizers	PWC-FAS-XXX
Flow-Through Liquid Cell	FAS-FLC
Temperature-Controlled Liquid Cell, 24V	FAS-TCC-3
Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220V/240V input, 24V output	ATC-024-2
Internal Reflection Spectroscopy	IRS-671
Internal Reflection Spectroscopy: Review and Supplement	IRS-RS1

FASTIR ACCESSORIES

Trough Liquid Cell



Applications

- ▶ Convenient for analyzing liquids and pastes.

Features

- ▶ Easily exchangeable with standard FastIR sampling stage.
- ▶ Chemically resistant construction.
- ▶ ZnSe ATR crystal (other materials available upon request).
- ▶ Contains up to 0.9 ml of liquid.
- ▶ Easily disassembled for cleaning and crystal replacement.

Flow-Through Liquid Cell

Applications

- ▶ Ideal for analyzing flowing liquids.
- ▶ Suitable for use as a sealed cell for examining volatile samples.

Features

- ▶ Fits conveniently onto the FastIR solid sampling plate.
- ▶ 0.1 ml (including the fittings) sampling volume.
- ▶ Two Luer fittings for easy injection of the sample and for adaptation for flow environments.
- ▶ Two Luer plugs for sealing the cell for analysis of volatile samples.
- ▶ Made from chemically resistant 316 stainless steel.
- ▶ No spectral interference from the O-ring sealing the cell.

ATR Crystals

Material	Catalog No.
ZnSe	FAS-ATR-M
ZnS	FAS-ATR-I
Ge	FAS-ATR-J

Temperature Controlled Liquid Cell

Applications

- ▶ Excellent for examining liquids at high and low temperatures.
- ▶ Suitable for liquid flow analyses.

Features

- ▶ Easily exchangeable with standard FastIR sampling stage.
- ▶ Chemically resistant 316 stainless steel cell and sampling stage.
- ▶ Two Swagelok fittings for easy installation in flow environments.
- ▶ Heatable to 200°C with the two imbedded cartridge heaters.
- ▶ Coolable to 0°C using the flow-through coolant channel.
- ▶ K-type thermocouple.
- ▶ No spectral interference from the Viton O-ring sealing the cell.
- ▶ Volume: 0.3 ml, including the inlet and outlet ports.

Includes

- ▶ Two heaters and a K-type thermocouple.
- ▶ Single reflection ZnSe ATR crystal.
- ▶ Mating hardware for the specified spectrometer.

Ordering Information

Ordering Information	Catalog No.
FastIR™	FAS-ATR-M
FastIR™ Reactor	FAS-R-XXX

Replacement Parts

Item	Catalog No.
ZnSe ATR Crystal	FAS-XCS
Cover Slide	FAS-XCS
Gasket	FAS-GSK
Solid Sampling Plate (includes ZnSe crystal)	FAS-SSP

THE FATIR™ ANALYSIS SYSTEM

Effective in January 2006, the FDA will require the amount of *trans* fat per serving to be indicated on the Nutrition Facts food label. To assist in meeting this requirement, Harrick Scientific has developed the FatIR™, an ATR-FTIR oil analyzer that complies with the FDA-approved AOAC method 2000.10 for routine quantitative measurements of total *trans* fat content. The FatIR™ is a straightforward analytical tool for fast, easy and accurate determinations of *trans* fat concentration. In addition, it permits real-time *in situ* monitoring of the *trans* isomer formation during the processing of edible oils at temperatures up to 175°C.

Applications

- Routine quantitative measurements of *trans* fat content, in accordance to AOAC method 2000.10.
- Real-time in situ monitoring of the *trans* fat formation during processing of edible oils at temperatures up to 175°C.
- Qualitative and quantitative analysis of liquids and pastes.

Features

- Highly repeatable, qualitative and quantitative results.
- Convenient trough for containing liquid samples.
- Fixed 45° incident angle.
- SuperCharged™ ZnSe ATR optics for superior performance.
- High throughput (greater than 85%).
- Easy to align and use.
- Imbedded heaters permit operation up to 175°C.
- K-type thermocouple directly measures the sample temperature and flips out of the way for easy cleaning.
- Low-voltage Temperature Controller allows for precise and safe regulation of the sample temperature, with options for direct, cascade and computer control.
- Secondary K-type thermocouple monitors the crystal temperature.
- Minimal sample preparation required.
- Readily exchangeable crystal.
- PermaPurge™ for rapid purging of the system.

Includes

- FatIR™ optical assembly with:
- SuperCharged™ ZnSe mounted ATR crystal.
- Two imbedded cartridge heaters.
- Two K-type thermocouples.
- Mating and PermaPurge™ hardware for the specified FT-IR spectrometer.
- Low Voltage (24V) Temperature Controller.

Additional ATR Crystals

Material	Catalog No.
ZnSe	FAS-ATR-M
Zn S	FAS-ATR-I
Ge	FAS-ATR-J

Ordering Information

Ordering Information	Catalog No.
FatIR™, 110V	FAT-1-XXX
FastIR™ 220/240V	FAT-2-XXX

Replacement Parts

Replacement Parts	
Top Plate Assembly, ZnSe	RAN-TOP-M
ZnSe Crystal	FAS-ATR-M
Viton Gasket	RAN-GSK
K-Type Thermocouple	008-148
Heater Assembly (two heaters plus connector)	RAN-HTR
Cartridge Heater, 24V	HTRS-18

THE RANGEIR™ LIQUID ANALYZER

The RangeIR™ is a powerful tool for the analysis of liquids, pastes, and gels under controlled temperatures up to 175oC. The unique design allows the safe use of the sample temperature itself in this control, an invaluable feature for rigorous studies. Reactants and reaction products can be monitored as a function of temperature. In addition, the RangeIR™ can be used for sensitive measurements on dissolved or suspended species, by using the elevated temperature to volatilize an interfering liquid.

Applications

- Routine quantitative and qualitative measurements on temperature-controlled liquids, pastes, and gels.
- time in situ monitoring of reactants and reaction products in liquids as a function of temperature.
- Analysis of dissolved or suspended species in interfering liquids.

Features

- Reproducible measurements.
- Convenient trough for containing liquid samples.
- Fixed 45° single reflection horizontal ATR configuration.
- SuperCharged™ ZnSe ATR Crystal or Gemanium ATR Crystal.
- Exchangeable ATR crystal.
- Simple to align and use.
- Safe, low-voltage heaters permit operation up to 175° C (Note: Germanium become optically opaque at temperatures exceeding 100° C.)
- K-type thermocouple directly measures the sample temperature and flips out of the way for easy cleaning of the ATR crystal.
- Secondary K-type thermocouple monitors the crystal temperature, permitting the cascade temperature regulation required as a fail-safe for high temperature operation.
- PermaPurge™ for rapid purging of the FTIR system.
- Requires the Low Voltage Temperature Controller for precise and safe regulation of the sample temperature, with options for direct, cascade, and computer control.
- Available in a specialized configuration for trans fat analysis (see Harrick's FatIRTM System data sheet).

Replacement ATR Crystals

Material	Catalog No.
ZnSe	FAS-ATR-M
Ge	FAS-ATR-J

Includes

- SuperCharged™ ZnSe mounted ATR crystal or Ge ATR mounted crystal.
- Two imbedded cartridge heaters.
- Two K-type thermocouples.
- Mating and PermaPurge™ hardware for the specified FT-IR spectrometer.

Ordering Information

Ordering Information	Catalog No.
RangeIR™ with ZnSe ATR Crystal	RAN-M-XX
RangeIR™ with Ge ATR Crystal	RAN-J-XXX
FatIR System (includes the RangeIR with ZnSe ATR and Temperature Controller), 110V	FAT-1-XXX
FatIR System (includes the RangeIR with ZnSe ATR and Temperature Controller), 220/240V	FAT-2-XXX
Automatic Temperature Controller, 110V	ATC-024-1
Automatic Temperature Controller, , 220/240V	ATC-024-2

Replacement Parts

Replacement Parts	
Top Plate Assembly, ZnSe	RAN-TOP-M
Top Plate Assembly, Ge	RAN-TOP-J
Viton Gasket	RAN-GSK
K-Type Thermocouple	008-148
Heater Assembly (two heaters plus connector)	RAN-HTR
Cartridge Heater, 24V	HTRS-18

THE CONCENTRATIR™

The ConcentratIR™ is a horizontal multiple internal reflection (ATR) accessory, designed for analyzing minute samples of liquids. The ConcentratIR™ was developed specifically for use in quality control environments. It is a durable, compact, horizontal ATR micro-sampler that utilizes a Si internal reflection element. The infrared beam interacts with the sample fourteen times within a 4mm diameter area. This concentrates the sensitivity of a regular horizontal ATR into a much smaller area and hence requires only minute quantities of sample per measurement. The ConcentratIR™ makes internal reflection simpler and more straightforward than ever before. For rapid sample exchange without interrupting the purge of the spectrometer, the ConcentratIR™ integrates Harrick's exclusive PermaPurge™*.

Applications

- ▶ Invaluable for studying minute samples of liquids and pastes.
- ▶ Uniquely suited to quality control environments.

Features

- ▶ Durable and rugged internal reflection micro-sampler.
- ▶ High sample throughput due to little or no sample preparation.
- ▶ Quick and easy clean up between samples.
- ▶ Easily accessible sampling area for rapid sample exchange.
- ▶ Fourteen reflections in a 4mm diameter.
- ▶ Highly inert and durable Si ATR crystal.
- ▶ Harrick's exclusive PermaPurge™ for rapid sample and crystal exchange without interrupting the purge of the system.
- ▶ Diamond and ZrO_2 ATR crystals available on special order.
- ▶ Pre-aligned for easy start-up.
- ▶ **Readily convertible to grazing angle specular reflectance with the Focus™ adapter. (See page 18).**

Includes

- ▶ Mating hardware for the specified spectrometer.

*Patented

Ordering Information

Ordering Information	Catalog No.
ConcentratIR™	UQC-C-XXX
Options	
Grazing Angle Focus™ Adapter	FCS-TOP
Replacement Parts	
Replacement Sampling Plate	UQC-LSP

GATR™

The GATR™ grazing angle ATR accessory is a revolutionary approach to the analysis of monolayers on semiconductor and metallic substrates. The GATR™ is optimized for high sensitivity to these types of samples. Its specially designed pressure applicator is optimized for delivering good contact between the sample and the Ge ATR crystal. This configuration provides at least an order of magnitude increase in sensitivity relative to grazing angle methods, along with the convenience of an easy to use, fully prealigned, horizontal sampling accessory.

Applications

- Analysis of monolayers and adsorbed species on semiconductors and metals.
- Rapid, repeatable measurements

Features

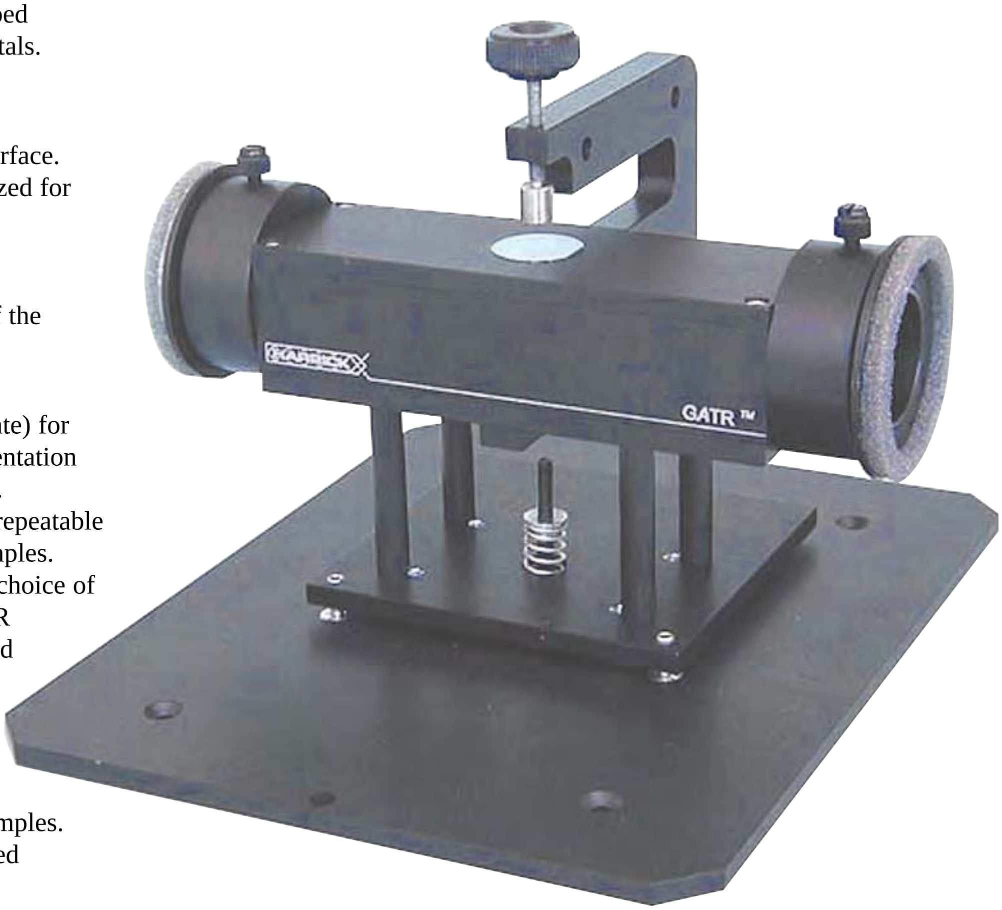
- Convenient horizontal sampling surface.
- Built-in pressure applicator optimized for contact with hard samples.
- 65º fixed incident angle.
- Ge ATR crystal.
- PermaPurge™ for rapid purging of the system.

Options available:

- Wire grid polarizer (KRS-5 substrate) for enhanced spectral contrast and orientation studies. Includes slide plate mount.
- Fixed torque Slip-Clutch to apply repeatable ATR contact pressures to solid samples.
- Torque Screwdriver to apply your choice of contact pressure for repeatable ATR spectroscopy measurements of solid samples.

Includes

- Ge hemispherical ATR crystal.
- Built-in pressure applicator, designed to accommodate large samples.
- Mounting hardware for the specified spectrometer.



Ordering Information

	Catalog No.
GATR™ Wire Grid Polarizer (KRS-5 Substrate). Includes mount and polarizer.	PWD-GATR-XXX
GATR™ Wire Grid Polarizer Mount. Compatible with the PWD Wire Grid Polarizers.	PWC-GATR-XXX
GATR™ Pads (pack of five).	GATR-PAD

Options and Replacement Parts

Slip-Clutch	SLP-CHI
Torque Screwdriver	PTW-SXX
Mounted Ge ATR Crystal	GATR-ATR-J
Internal Reflection Spectroscopy	IRS-671
Internal Reflection Spectroscopy: Review and Supplement	IRS-RS1

VARIGATR™

The VariGATR™ grazing angle ATR accessory is a revolutionary approach to the analysis of monolayers on semiconductor and metallic substrates. The VariGATR™ is variable angle, so the incident angle can be optimized for the highest sensitivity with these types of samples. Its specially designed pressure applicator is optimized for delivering good contact between the sample and the Ge ATR crystal. The VariGATR™ provides at least an order of magnitude increase in sensitivity relative to grazing angle methods, in addition to the convenience of an easy to use, fully prealigned, horizontal sampling accessory.

Applications

- Analysis of monolayers and adsorbed species on semiconductors and metals.
- Rapid, repeatable measurements.

Features

- Convenient horizontal sampling surface.
- Built-in pressure applicator with slip-clutch for reproducible pressure application.
- Continuously variable angle from 60º to 65º allowing optimization for maximum sensitivity.
- Anti-backlash mechanism allows for accurate, reproducible angle selection.
- Mounted Ge ATR crystal..
- Accommodates samples up to 8” in diameter with center-sampling of discs up to 6” in diameter..
- PermaPurge™ for rapid purging of the system.
- Options include:
  - Wire Grid Polarizer for enhanced spectral contrast and orientation studies. Includes slide plate mount.
  - Force sensor with digital read-out for precise measurement of the force applied to achieve contact between the ATR crystal and the sample.
  - Low-torque slip-clutch available.
  - Angular scale viewing aide for large samples.

Includes

- Ge hemispherical ATR crystal.
- Built-in pressure applicator, designed to accommodate large samples.
- Mounting hardware for the specified spectrometer.

Ordering Information

Ordering Information	Catalog No.
VariGATR™	GAT-V-XXX*

Options and Replacement Parts

Force Sensor with Digital Read-Out	GATR-V-FSD
Slip-Clutch, 24 in-oz	SLP-CLP
Angular Scale Viewer for Large Samples	GATR-V-VMR
Mounted Ge ATR Crystal	GATR-ATR-J1
VariGATR™ Wire Grid Polarizer (KRS-5 substrate). Includes mount and polarizer	PWD-GATR-XXX*
Wire Grid Polarizer Mount for the VariGATR™ (use with PWD Polarizer)	PWC-GATR-XXX*

HORIZON™

The Horizon™* multiple internal reflection attachment is well-suited for rapid analysis of liquids, pastes, powders, and thick films. Its unique design takes advantage of the astigmatism inherent in spherical mirrors to shape the beam to match the aperture of the internal reflection element and to keep the radiation away from the edges of the crystal. This eliminates spectral interference from the adhesive or gasket used to mount the crystal and results in higher throughput. The Horizon™ also features a horizontal sampling surface for easy sample positioning and PermaPurge™ for rapid sample and crystal exchange without interrupting the purge of the system.

Applications

- Multiple internal reflection (ATR) spectroscopy.
- Useful for examining liquids, pastes, powders and other soft surface solids.
- Perfect for quality control and other situations that require rapid sample exchange.

Features

- Top loading.
- Unobstructed, horizontal sampling surface.
- Thirteen reflections from the sample.
- PermaPurge™ provides for rapid sample and crystal exchange without interrupting the purge of the system.
- High spectral contrast.
- High throughput.
- Utilizes astigmatism to match the beam shape to that of the internal reflection element, creating insensitive edges within the crystal.
- Unique optical design eliminates spectral interference from the adhesive or gasket used to mount the crystal.
- Easily installable optional sampling adapters including flow-through liquid cells (Approx. volume: 0.35ml), cells heatable to 100°C.
- Large volume cells available on special order.
- Optional Wire Grid Polarizer and mount available.

Includes

- One mounted 50x10x2mm SPT 45° ZnSe crystal.
- Solid or liquid sampling plate. Solid sampling plate includes a pressure plate and clamp; the liquid sampling plate has a trough to contain the liquid.
- Mating hardware for the specified spectrometer.

*Patented

Mounted ATR Crystals

Material	Solid Sampling Plates	Troughs
Ge	HOR-SSP-J	HOR-LSP-J
ZnSe	HOR-SSP-M	HOR-LSP-M

Flow-Through Adapters

	Luer	Swagelok
Solid Sampling Plate	HOR-FSP-L	HOR-FSP-S
Trough	HOR-FLP-L	HOR-FLP-S

Ordering Information

	Catalog No.
Horizon™: Liquid Sampler	HOR-L-XXX
Horizon™: Solid Sampler	HOR-S-XXX

Options

Wire Grid Polarizer, Continuously Adjustable	PWG-U1R
Wire Grid Polarizer, Two Position	PWG-SEA
Torque Screwdriver	PWT-SXX
Internal Reflection Spectroscopy	IRS-671
Internal Reflection Spectroscopy: Review and Supplement	IRS-RS1
Automatic Temperature Controller, 110V inuput, 24V output	ATC-024-1
Automatic Temperature Controller, 220V, 240 input, 24V output	ATC-024-2

THE PRAYING MANTIS™

The Praying Mantis™ was the first generally available diffuse reflection attachment and remains the forerunner in the field. It incorporates two 6:1, 90° off-axis ellipsoids which form a highly efficient diffuse reflection illumination and collection system. This unique configuration deflects the specular reflectance away from the collecting ellipsoid, minimizing the associated spectral distortions. It also can be configured to study materials and reactions in controlled environments with the appropriate reaction chamber.

Applications

- For easy and reliable diffuse reflection analysis of solids and powders.
- Analysis of catalysts and other powders in a temperature and/or pressure controlled environment.

Features

- Highly efficient collection system.
- Minimizes the detection of the specular component.
- Ellipsoids pivot to provide easy access to the sampling area.
- Allows easy attachment of reaction chambers.
- Several models offered for compatibility with a wide range of IR-UV-VIS spectrometers.
- Harrick's exclusive PermaPurge™ allows rapid exchange with minimal interruption of the system purge.
- Optional references for FTIR and UV-VIS studies.
- Optional reference for FTIR and UV-VIS studies.

Includes

- Cart with two mounted alignment mirrors.
- Alignment post.
- Sample cart.
- Two sampling cups: 10mm adjustable height and 3mm adjustable microsampling.
- Funnel.
- Mating hardware for the specified spectrometer.

Ordering Information	Catalog No.
Praying Mantis Diffuse Reflectance Accessory	DRP-XXX
Praying Mantis Kit, 110V (includes HVC Chamber, Temperature Controller and sampling tools)	DRK-3-XXX
Praying Mantis Kit, 220V (includes HVC Chamber, Temperature Controller and sampling tools)	DRK-4-XXX

Options & Replacement Parts

Alignment Fixture	DRP-ALN
Sampling Accessory Kit	DRA-SAP
Micro-sampling Cup	DRP-SX3
Sampling Cup	DRP-S10
Sampling Cup	DRP-S10
Spectralon® UV-VIS Reference Disk for the Praying Mantis	DRP-SPR
Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220V/240V input, 24V output	ATC-024-2

	FT-IR	UV-VIS
Ambient Sample Chamber	DRP-ASC (KBr)	DRP-ASC-VUV(SiO2)
High Temperature, Low Pressure Reaction Chamber, 24V	HVC-DRP-3 (KBr)	HVC-VUV-3 (SiO2)
High Pressure Dome for the HVC Reaction Chambers (Max. Pressure: 500 psi)	HVC-DWI-3 (ZnS)	HVC-DWA-3 (SiO_2)
Low Temperature, Low Pressure Reaction Chamber, 24V	CHC-CHA-3(KBr)	CHC-VUV-3(SiO2)

REACTION CHAMBERS FOR THE PRAYING MANTIS™

Low Temperature Reaction Chamber

Applications

- ▶ Allows diffuse reflection measurements under controlled pressures and a wide range of temperatures.
- ▶ Used in conjunction with Praying Mantis Diffuse Reflection Accessory.

Features

- ▶ Two models available:

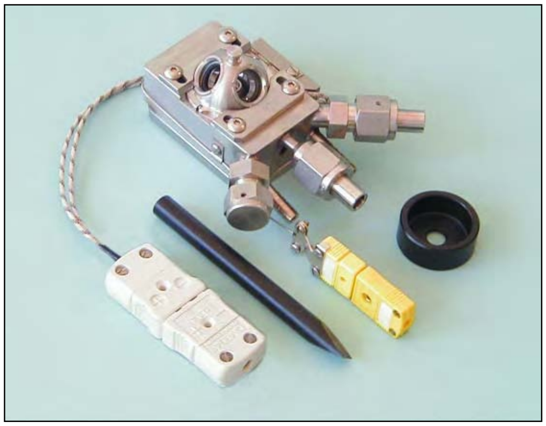
1. High-Temperature Low Pressure Reaction Chamber (HVC-DRP) for operation from high vacuum (10^{-6} torr) to two or three atmospheres and at high temperatures (up to 600°C under vacuum). Convertible for high-pressure operation, up to 500 psi with the ZnS High Pressure Dome.
2. Low Temperature Chamber (CHC-CHA) for studies from high vacuum to two or three atmospheres and at temperatures of -150°C to 600°C (under vacuum).

- ▶ Three inlet/outlet ports provided for evacuating the cell and introducing gases.
- ▶ Made of chemically-resistant 316 stainless steel.

Includes

- ▶ Reaction Chamber.
- ▶ Heating cartridge.
- ▶ K-type thermocouple.
- ▶ Dome with two KBr windows and one glass or quartz observation window.

High/Low Pressure Reaction Chamber



Windows for Vacuum Reaction Chambers

Material	Catalog No.
SiO_2	WAD-U23
Si	WED-U23
CaF_2	WFD-U23
ZnS	WID-U23
ZnSe	WMD-U23
KBr	WPD-U23

Windows for the High Pressure Dome

Material	Catalog No.
ZnS	WID-U43
ZnSe	WMD-U43
UV Quartz	WAD-U43

Ordering Information

	Catalog No.	
	FT-IR(KBr windows)	UV-VIS(SiO2 windows)
High Temperature Reaction Chamber,24V	HVC-DRP-3	HVC-VUV-3
Low Temperature Reaction Chamber,24V	CHC-CHA-3	CHC-VUV-3

Ordering Information

	Catalog No.
High Pressure Dome for the HVC-DRP with ZnS windows	HVC-DWI-3
High Pressure Dome for the HVC-DRP with ZnSe windows	HVC-DWM-3
High Pressure Dome for the HVC-DRP with SiO2 windows	HVC-DWA-3
Automatic Temperature Controller, 110V inuput, 24V output	ATC-024-1
Automatic Temperature Controller, 220V/240 input, 24V output	ATC-024-2
Screen Set, two each of three mesh sizes	116-439
Heater Assembly for the HVC and CHC, 24V	HVC-HTR
Heater, 24V	HTRS-17
Accessory Vacuum Pumps	VPE-001
Accessory Vacuum Pumps	VPE-002
Oil, 1 quart	VPE-OIL

TransFlex™

Compatible with Cricket™ sample cups, TransFlex™ disposable substrates allow quick qualitative analyses on organic and most aqueous solutions using transflectance. The convenient horizontal position allows liquids to be placed directly on the substrate without dripping. Substances in solution or suspension can be dropped onto the substrate, leaving the solute in place after evaporation.

Applications

- Quick qualitative analysis of organic liquids.
- Quick qualitative analysis of most aqueous solutions.
- Analysis of substance in solution or suspension after evaporation of the solvent.

Features

- Compatible with Cricket™ .
- Very low cost.
- Disposable.
- Ease of use.
- Ideal for routine QC laboratories where many samples are run.
- No significant spectral artifacts.
- Enhanced spectral contrast.

Ordering Information	Catalog No.
TransFlex™	CRK-TFX

THE CRICKET™

Harrick's Cricket™ is excellent for routine diffuse reflectance (DRIFTS) measurements of powders and other solid samples in quality control environments and research laboratories. Its design follows the long-standing Harrick tradition of true diffuse reflectance accessories for FT-IR and UV-VIS spectroscopy. Like its more versatile Praying Mantis cousin, the Cricket™ optimizes the collection of the diffusely reflected light while eliminating the specular component. With a flip of a switch, the Cricket™ converts from this true diffuse reflectance mode to a fixed-angle specular reflectance device. In this configuration, the Cricket™ collects 60° specular or in-line 'diffuse' reflectance. The Cricket™ is available in a downward sampling configuration for powder analysis and an upward model for cohesive solids. Both models of the Cricket™ feature PermaPurge™ for rapid sample and crystal exchange without interrupting the purge of the spectrometer.

Applications

- Ideal for routine diffuse reflectance measurements of powders, solids, and liquids.
- Excellent for quality control.

Features

- DRIFTS and specular reflection capabilities provide application versatility.
- Eliminates the collection of the specular component in the diffuse reflectance mode.
- Provides a fixed 60° incident angle in the specular reflectance mode.
- Easy to use.
- High sample throughput.
- Low cost.
- Prealigned for quick start-up.
- Harrick's exclusive PermaPurge™ allows rapid sample and crystal exchange without interrupting the purge of the system.
- Two models available:
  - Downward looking Cricket™ for diffuse reflectance measurements of powders, small cohesive solids, and liquids on TransFlex substrates.
  - Upward looking Cricket™ for cohesive solid samples of virtually unlimited sample size.
- Compatible with TransFlex substrates for quick qualitative analysis of liquids by transflectance spectroscopy.
- Compatible with most FT-IR and UV-VIS spectrometers.

Includes

- Specular Reflectance Reference.
- Diffuse Reflectance Reference.
- Three position sample holder (downward looking models only).
- Mating hardware for the specified FT-IR or UV-VIS spectrometer.

Ordering Information

	Catalog No.
Cricket™	CRK-D-XXX
Upward looking Cricket™	CRK-U-XXX

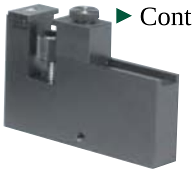
Options and Replacement Parts

Trans-Flex	CRK-TFX
Replacement Sample Holder	CRK-SAM
Replacement Specular Reflectance Reference	CRK-SPR
Replacement Difuse Reflectance Reference	CRK-DIF

THE SEAGULL™

The Seagull™ is a powerful attachment for examining numerous types of samples using a variety of reflection techniques. This unique accessory can be easily adapted for external, internal (ATR) or diffuse reflection spectroscopy. In addition, it can be operated over a broad range of incident angles without misaligning the system, defocusing the incident radiation, or changing the polarization of the incident beam. These features make the Seagull™ an extremely versatile and flexible attachment. The Seagull™ also incorporates Harrick's PermaPurge™ feature which permits changes in angle without interrupting the purge of the spectrometer.

*Patented



Applications

- Variable angle external reflectance and ATR.
- Ideal for examining a wide variety of samples, including optical coatings, opaque substrates, films on opaque substrates, and slightly curved solids.
- Excellent tool for studying liquids and films on liquids by external reflectance, for depth profiling, and for determining optical constants.

Features

- Multi-purpose, variable angle reflection attachment.
- Internal, external, and diffuse reflectance capabilities.
- Continuously variable angle of incidence, from 5° to 85°.
    - No realignment of the accessory or repositioning of the sample required when varying the incident angle.
    - Focuses the incident beam on the sample for all incident angles.
    - Always centers the incident beam on the same area of the sample.
    - PermaPurge™ permits changes in angle without interrupting the purge of the system.
- Ming-Sung™ ATR Rotator for studying oriented polymers available separately.
- Maintains polarization as the angle changes.
- High optical throughput.
- Heated liquid and solid sampling accessories available on special order.

Hemispheres

Material	Catalog No.
Ge	EJ6xBB
ZnSe	EM6xBB
Si	EE6xBB

Includes

- Sample holder for external reflectance with a sample cup for diffuse reflectance.
- Sample pressure plate.
- Optional compatible polarizer and ATR kit available.
- Mating hardware for the specified spectrometer.
- Alignment mirror.

Ordering Information	Catalog No.
Seagull™	SEA-XXX
Seagull™ Research Package (includes Seagull™, Internal Reflection (ATR) Kit, wire grid polarizer, and SOS™ software)	SRP-XXX

Options

ATR Kit (includes ZnSe hemisphere and holder, pressure plate and clamp)	SEA-IRK
Ming-Sung™ ATR Rotator (includes holder with calibrated dial for setting the angle, pressure plate and clamp)	SEA-MNP
Heated Sample Stage, 24V	SEA-HOT-3
Heated Flow-Through Liquid ATR Cell, 24V	SEA-HLC-3
Two position Wire Grid Polarizer, KRS-5 Substrate	PWG-SEA
Torque Screwdriver (See page 33)	PTW-SXX
SOS™ Software Package, 3.5" Disk	SOS-102
Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220V/240V input, 24V output	ATC-024-2
Replacement Alignment Mirror	MOP-115
Internal Reflection Spectroscopy	IRS-671
Internal Reflection Spectroscopy: Review and Supplement	IRS-RS1

DA VINCI ARM™

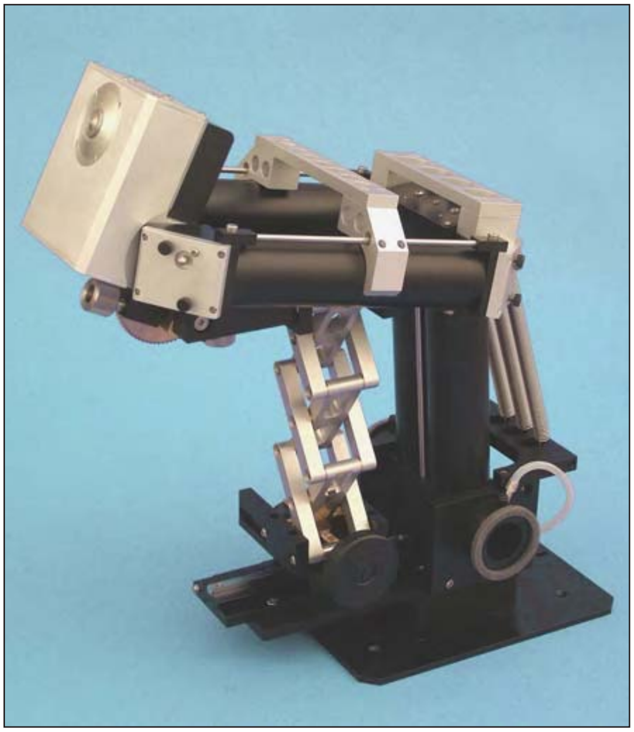
The da Vinci Arm is a unique articulated opto-mechanical FTIR accessory designed for analyzing samples that are too large to fit into the sample compartment of a spectrometer. The da Vinci Arm enables the analyses of samples by specular, diffuse and ATR reflection techniques. Since the da Vinci Arm is articulated, it simplifies analysis of samples in front of, below, or above the spectrometer sample compartment. The sampling surface can be facing in any direction – up, down, or sideways. Samples such as paintings, vases, sculptures, etc. can now be easily analyzed. The integral camera provides for magnified viewing and image capture of the sampled spot. The entire optical path of the IR beam is enclosed and integrated into the purge of the host spectrometer.

Applications

- Qualitative and quantitative analysis of samples that are too large to fit within the FTIR spectrometer sample compartment.
- Analysis of neat samples or small spots on large samples via diffuse reflectance, external reflectance or ATR spectroscopy.

Features

- Fixed 45º incident angle.
- Sampling head configurable for diffuse and specular reflectance.
- Optional diamond ATR sampling head available.
- Allows both contact and non-contact analysis, depending on the sample.
- Enables analysis outside the sample compartment.
- Always centers the incident beam on the same area of the sample.
- Articulated opto-mechanical system has:
  - 3.5” of horizontal motion
  - 5.8” of vertical motion
  - 180º of tilt
  - Fine focus adjustment
- Small sampling spot size:
  - 500µm diameter for ATR.
  - 1000 µm diameter for specular and diffuse reflectance.
- Small spot size allows analysis with high spatial resolution.
- Spectral range: 45,000 to 100 cm^{-1}.
- Video imaging system:
  - Magnifies the image for easy viewing.
  - Directly views the sample surface.
  - Integrates with your computer for real-time viewing and long-term storage of sampled images.
- Suitable for use in most FT-IR spectrometers.



Includes

- Mating hardware for the specified FTIR spectrometer.
- Alignment mirror.
- Optional compatible polarizer and ATR kit available.
- Mating hardware for the specified spectrometer.
- USB adapter and software for video image capturing, compatible with Windows 98/350MHz or higher computers.

Ordering Information

Ordering Information	Catalog No.
da Vinci Arm™	DAV-XXX
	XXX denotes the spectrometer code

Options

Diamond ATR	DAV-ATR-W

SEAGULL™ ACCESSORIES
ATR KIT

Applications

- ▶ Extremely versatile for ATR sampling.
- ▶ Excellent for depth profiling studies.

Features

- ▶ ZnSe hemispherical ATR crystal.
- ▶ Crystal holder.
- ▶ Compatible with our Torque Screwdriver for reproducible pressure application (Page 33).

HEATED STAGE

Applications

- ▶ Excellent for external or internal reflection measurements at elevated temperatures.

Features

- ▶ Useful for both external and internal reflectance.
- ▶ Heatable from room temperature to 100°C.
- ▶ Installs easily in the external or internal reflectance (ATR) sample holder.
- ▶ Requires Automatic Temperature Controller (page 33).

FLOW-THROUGH LIQUID ATR CELL

Applications

- ▶ Excellent for examining liquids in static or flow environments.

Features

- ▶ Made from chemically resistant 316 stainless steel.
- ▶ Two luer fitting and plugs for easy injection of the sample and adaptation to flow environments.
- ▶ Volume: 1 ml.
- ▶ Optional heatable model available.

TROUGH

Features

- ▶ Ideal for examining films on liquids using external reflectance.
- ▶ Volume: 3.75 ml.
- ▶ Readily placed on the external reflectance holder.

MING-SUNG ATR ROTATOR

Applications

- ▶ Ideal for examining oriented polymers using ATR.

Features

- ▶ Allows 90° rotation of the sample.
- ▶ Maintains constant pressure on the sample during sample rotation.
- ▶ Built-in adjustable sample support.
- ▶ Compatible with all Seagull ATR hemispheres.
- ▶ Use with our Torque Screwdriver (page 33) for repeatable pressurization of the sample.

THE SPLITPEA™

Harrick's SplitPea™,[1] is a horizontal internal reflection (ATR) accessory with the smallest sampling area of any ATR accessory - less than 250 µm in diameter for its Si ATR crystal. The SplitPea™ is configured to apply localized, measured pressure to produce superior contact between the sample and the ATR crystal. This makes the SplitPea™ ideal for quick and easy examination of a wide range of samples. These include: hard samples, like paint chips and combinatorial chemistry substrates; small samples, such as individual fibers and nanoliters of liquids; large samples, such as transparency film and defects thereon. For positioning samples on the sampling area, a 50X viewing microscope, the View-ThruPress™,[2] is available. In addition to making ATR nanosampling simple and straightforward, the SplitPea™ features PermaPurge™, [3] for rapid sample and crystal exchange without interrupting the purge of the spectrometer. The Harrick SplitPea™ is an innovative alternative to infrared microscopes, beam condensers, and diamond cells.

Applications

- Uniquely suitable for studying optically thick, hard samples; slightly curved samples; fibers; paint chips; nanoliters of liquids and pastes; defects on large panels; and droplets of corrosive liquids.
- Invaluable for forensic samples and combinatorial chemistry samples such as SynPhase™,[4] Lanterns and Crowns.

*Patented

Features

- Internal and external reflection capabilities provide application versatility.
- High sample throughput due to little or no sample preparation.
- Usually retains sample integrity.
- Small sampling area - less than 250µm in diameter for ATR with a silicon crystal.
- Minimizes stray light due to the small sampling area.
- Inert internal reflection elements available for use from the Near IR to the Far IR.
- Calibrated pressure applicator for reproducible ATR measurements.
- Designed for optimal contact between the ATR crystal and hard surface solids.
- Flip-up, streamlined pressure applicator for easy access to sampling area
- High energy throughput with DTGS detectors.
- Harrick's exclusive PermaPurge™ allows rapid sample and crystal exchange without interrupting the purge of the system.
- Spill-resistant cover.
- Upgrade to a 50X View-ThruPress™ for easier viewing of the sampling area.
- Options include a CombiKit™ for examining combinatorial chemistry substrates and a Flow-Through Liquid Cell

Includes

- Two ATR holders with mounted Si hemispheres.
- Sample holder adapter for studying powders by internal reflectance.
- External reflection sample holder and alignment mirror.
- Mating hardware for the specified spectrometer.

Ordering Information

Ordering Information	Catalog No.
SplitPea™ with ViewThruPress™	UNP-XXX
SplitPea™	UNS-XXX
Meridian™ -Diamond SplitPea™ with ViewThruPress™	MER-P-XXX
Meridian™ -Diamond SplitPea™	MER-XXX

Options and Replacement Parts

CombiKit™	UNS-CBK
ViewThruPress™ Upgrade	UNS-MIC
Punch Sampler	PUN-SMP
O-ring Sealed Liquid Cell with Luer Fittings	UNS-LCF
Liquid Cel O-ring	ORV-0015
Powder Adapter O-ring	ORV-012

	Catalog No.		
		Ge	UNS-ATR-0J
Mounted Crystals: Si	UNS-ATR-0E	ZnSe	UNS-ATR-0M
Diamond	UNS-ATR-0W	ZnS	UNS-ATR-01

[1] U. S. Patent 5,210,418, [2] U. S. Patent 5,308,983, [3] U. S. Patent 5,177,561, [4] Registered trademark of Mimotopes Pty. Ltd., Australia.

Meridian™: The Diamond SplitPea™,[1]

The Meridian™ is the ultimate accessory for examining small or extremely hard samples by internal reflection (ATR) spectroscopy. Harrick Scientific's Meridian™ is a horizontal ATR accessory with a sampling area less than 500 µm in diameter, the smallest area for any diamond ATR accessory. It is configured to apply localized, measured pressure to produce superior contact between the sample and its diamond ATR crystal. The Meridian™ is perfect for quick and easy examination of extremely hard samples, abrasive powders, and highly corrosive materials, in addition to routine analysis of small liquid, solid, and paste samples. For positioning samples on the small spot, a 50X viewing microscope, the ViewThruPress™,[2] is available. The Meridian™makes ATR microsampling even more simple and straightforward by integrating Harrick's PermaPurge™,[3] for rapid sample and crystal exchange with minimal purge interruption. The Meridian™ is an innovative alternative to infrared microscopes, beam condensers, and diamond cells.

Applications

- Uniquely suitable for studying extremely hard samples; highly corrosive liquids; minerals; slightly curved samples; fibers; nanoliters of liquids and pastes; and defects on large panels.
- Invaluable for forensic and combinatorial chemistry samples.

Features

- The most chemically inert, durable, and cleanable ATR crystal available: diamond.
- Small sampling area - less than 500 µm in diameter in the standard ATR configuration.
- Minimizes stray light due to the small sampling area.
- Wide spectral range: 45,000 cm-1 - 2300 cm-1, 1850 cm-1 - FIR.
- High sample throughput due to little or no sample preparation.
- Calibrated pressure applicator for reproducible ATR measurements.
- Achieves optimal contact between the ATR crystal and hard samples.
- Flip-up, streamlined calibrated pressure applicator for easy access to sampling area.
- ATR and external reflection capabilities provide application versatility.
- High energy throughput with DTGS detectors.
- Generally retains sample integrity.
- Harrick's exclusive PermaPurge™ allows rapid sample and crystal exchange without interrupting the purge of the system.
- Spill-resistant cover.
- Readily cleaned ATR crystal, due to the low adhesion properties of diamond.
- Inert ATR crystals available for use from the Near IR to the Far IR.
- Upgrade to a calibrated 50X ViewThruPress™ for easier viewing of the sampling area.

*Patented

Includes

- One ATR holder with mounted diamond hemisphere.
- Sample holder adapter for studying powders by ATR.
- External reflection sample holder and alignment mirror.
- Mating hardware for the specified spectrometer.

Ordering Information

Ordering Information	Catalog No.
CombiKit™	UNS-CBK
Punch Sampler	PUN-SMP

Options and Replacement Parts

O-ring Sealed Liquid Cell with Luer Fittings	UNS-LCF
ViewThruPress™ Upgrade	UNS-MIC
Liquid Cell O-Ring	ORV-0015
Powder Adapterl O-Ring	ORV-012

	Catalog No.		
		Ge	UNS-ATR-0J
Mounted Crystals: Sil	UNS-ATR-0E	ZnSe	UNS-ATR-0M
Diamond	UNS-ATR-0W	ZnS	UNS-ATR-01

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[1] U. S. Patent 5,210,418, [2] U. S. Patent 5,308,983, [3] U. S. Patent 5,177,561, [4] Registered trademark of Mimotopes Pty. Ltd., Australia.

VIDEO MERIDIAN™

The Video Meridian™ combines the advantages of our Meridian™ diamond micro-ATR with video imaging and force sensing capabilities, making it the ultimate accessory for sampling small, hard, corrosive or irregularly shaped samples. The Video Meridian™ features a convenient horizontal sampling surface with an active sampling area less than 500 mm in diameter on its monolithic diamond crystal. The video system images through the crystal, providing a magnified real-time picture of the sample that can be easily captured for a permanent record. The samples themselves are compressed against the sample using a force-sensing pressure applicator with an adjustable alarm that conveniently delivery reproducible contact pressure. The Video Meridian™ is perfect for routine analysis of samples as small as a single fiber or bead, as well as extremely hard samples, abrasive powders, corrosive materials, liquids, solids and pastes.

Applications

- Uniquely suitable for studying extremely hard samples; highly corrosive liquids; minerals; slightly curved samples; fibers; nanoliters of liquids and pastes; and defects on large panels.
- Invaluable for forensic and combinatorial chemistry samples.
- Ideal for samples requiring archival storage of the images as well as the spectra.

Features

- Monolithic *Diamond* ATR Prism provides:
  - The most chemically inert, durable and cleanable ATR crystal available.
  - Small sampling area - less than 500 mm in diameter.
  - Ideal for operation from 45,000 cm^{-1} to the FIR; limited S/N in the 2300 cm^{-1} to 1800 cm^{-1} region.
- Pressure applicator for optimal contact between the ATR crystal and hard samples includes:
  - Force sensor with digital display of applied force.
  - LED indicator that activates when the user set alarm point is reached.
- Video imaging and illumination system:
  - Magnifies the image for easy viewing.
  - Directly views the sampling surface though the ATR crystal.
  - Integrates with your computer for real-time viewing and long-term storage of sampled images.
  - Optional LCD display.
- Minimal stray light due to the small sampling area.
- High sample throughput, since little or no sample preparation is required.
- Generally retains sample integrity.
- High energy throughput with DTGS detectors.
- Harrick's patented PermaPurge™ allows rapid sample and crystal exchange without interrupting the purge of the system.

Includes

- Sampling accessories: one diamond ATR holder, one external reflection sample holder and an alignment mirror.
- USB adapter and software for video image capturing, compatible with Windows 98/350MHz or higher computers.
- Mating hardware for the specified spectrometer.

Ordering Information	Catalog No.
Video Meridian™	MEV-XXX
Optional 4" LCD Display	MEV-LCD
O-Ring Sealed Liquid Cell with Luer Fittings	UNS-LCF
Liquid Cell O-Ring	LORV-0015
Mounted Diamond	UNS-ATR-1W
Powder Retainer	UNS-PSC
Powder Retainer O-Ring	ORV-012

ViewThruPress™

Applications

- Extremely helpful for positioning fibers, defects in transparent media, and powder granules.
- Excellent for viewing the sample during pressure application.
- Aids in determining the minimum pressurizing force required for internal reflectance.

Features

- 50X magnifier with focusing capabilities.
- Calibrated pressure applicator for reproducible ATR spectra.
- Optimizes contact between the crystal and the sample.

Angular Mask Set

Applications

- Removes spectral anomalies associated with changing the ATR crystal material.

Features

- Easily installs onto the ATR crystal holder.
- Three sizes: 30°, 35°, and 40°.

Flow-Through Liquid Cell

Applications

- Ideal for examining volatile liquids.
- Suitable for measuring spectra from small quantities of flowing liquids.

Features

- Small volume: 20µl, including the fittings.
- Two Luer fittings and plugs for easy injection of the sample and adaptation for flow environments.
- Made from chemically resistant 316 stainless steel.
- No spectral interference from the Viton O-ring sealing the cell.

ATR Holders

Material	Catalog No.
Si	UNS-ATR-E
Diamond	UNS-ATR-W
Ge	UNS-ATR-J
ZnSe	UNS-ATR-M
ZnS	UNS-ATR-I

ATR Holders

Applications

- Excellent for changing the penetration depth into the sample.
- Allows data acquisition over a variety of spectral ranges and sampling conditions.

Features

- Inert Si and Diamond crystals available.
- Ge and Si crystals feature a small sampling volume with a low penetration depth and a 250µm diameter sampling area.
- ZnS, ZnSe, and Diamond crystals feature a larger sampling volume with a deeper penetration depth and a 500µm diameter sampling area.
- Chemically resistant holder.

Heated ATR Crystal Holders

Applications

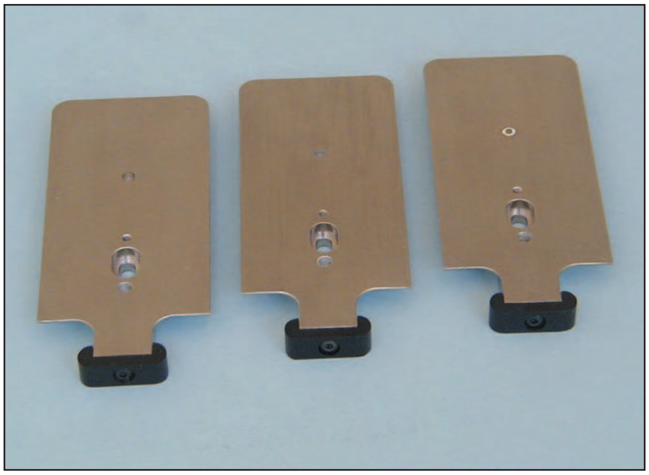
- ▶ Excellent for monitoring changes in materials as a function of temperature.
- ▶ Allows measurements of reaction kinetics
- ▶ Ideal for examining micro-chemical reactions.

Features

- ▶ Operable up to 200ºC for Si and diamond or 100ºC for Ge.
- ▶ Si, Diamond or Ge ATR hemispherical crystals available.
- ▶ Chemically resistant holder.
- ▶ Leak-free design.
- ▶ Includes K-type thermocouple and 24V heater.
- ▶ Compatible with the SplitPea™ <http://www.harricksci.com/accessories/H_splitpea.cfm> , Meridian™ <http://www.harricksci.com/accessories/H_meridian.cfm> , MVP™ <http://www.harricksci.com/accessories/H_mvp.cfm> and MVP2 Star™ <http://www.harricksci.com/accessories/H_mvpstar.cfm>
- ▶ Requires Harrick Automatic Temperature Controller.

ATR Holders

Material	Catalog No.
Si	UNS-HOT-OE
Diamond	UNS-HOT-OW
Ge	UNS-HOT-OJ



Ordering Information

Catalog No.

Heated ATR Holders <http://www.harricksci.com/accessories/H_splitpeaaccessories.cfm?R=0&A=N#atr> :

Si	UNS-HOT-0E
Diamond	UNS-HOT-0W
Ge	UNS-HOT-0J

Options

Automatic Temperature Controller, 24 V input, 110V output <http://www.harricksci.com/accessories/H_autotempcontroller.cfm?R=0&A=N>.	ATC-024-1
Automatic Temperature Controller, 24 V input, 220/240V output (CE marked) <http://www.harricksci.com/accessories/H_autotempcontroller.cfm?R=0&A=N>.	ATC-024-2
Flow-Through Liquid Cell <http://www.harricksci.com/accessories/H_flowthroughliquidcell.cfm>	UNS-LCF
Powder Retainer <http://www.harricksci.com/accessories/H_flowthroughliquidcell.cfm#powder>	UNS-PSC

THE MVP-PRO STAR™

The MVP-Pro Star™ is single reflection diamond ATR accessory. It features a convenient horizontal sampling surface with a 1.5mm diameter sampling area. The MVP-Pro Star is the choice for infrared spectroscopy measurements of extremely hard samples, abrasive powders and highly corrosive materials. Since the MVP-Pro Star™ is affordable, durable, and straightforward to use, it is an excellent choice for routine analysis of liquids, solids and powders. Small drops of liquid and minute smears of pastes can be placed directly on the monolithic diamond ATR crystal. Coatings on wire, paint chips, fabrics, powders, and miniature hard samples can be pressed against the crystal using the built-in pressure applicator. The MVP-Pro Star™ also features PermaPurge™ for rapid sample exchange without interrupting the spectrometer purge. The MVP-Pro Star™ diamond ATR makes sampling simple and straightforward.

Applications

- Excellent for studying optically thick, hard samples; slightly curved samples; fibers; paint chips; micro-liters of liquids and pastes; and powders.
- Invaluable for QC applications.

Features

- Monolithic *Diamond* ATR prism designed for operation from 45,000 cm^{-1} to the FIR.
- Small sampling area: 1.5mm in diameter.
- Convenient horizontal sampling.
- High sample throughput.
- Little or no sample preparation required.
- Excellent retention of sample integrity.
- Readily exchangeable ATR sampling plates.
- Built-in pressure applicator with slip-clutch for reproducible calibrated pressure application.
- Designed to achieve optimal contact between the ATR element and solid samples.
- Additional Ge and Si hemispherical ATR crystals available.
- Harrick's exclusive PermaPurge™ allows rapid sample exchange without interrupting the purge.
- Mechanically compatible with the SplitPea™ and Meridian™ crystal holders.
- Options include:
    - Ge, ZnSe and Si hemispherical ATR crystals.
    - Heatable sampling plates for operation up to 200°C with diamond or ZnSe ATR crystals, 100°C with Ge.
    - Powder Adapter for retaining powdered samples.
    - Flow-Through Liquid Cell for static and flow applications.
    - Force sensor with digital read-out for precise force measurements.
    - Low-torque slip-clutch available.

Crystal Material	Additional ATR Sampling Plates	Heated Sampling Plates
Diamond	MVP2-ATR-0W	UNS-HOT-0W
Ge	MVP2-ATR-J	UNS-HOT-0J
Si	MVP2-ATR-E	UNS-HOT-0E
ZnSe	MVP2-ATR-M	

Includes

- Epoxy-free Diamond ATR sampling plate.
- Built-in pressure applicator.
- Mating hardware for the specified spectrometer.

Ordering Information

	Catalog No.
MVP-Pro Star™	MVR-XXX

Options

LowTorque Slip-Clutch, 24 in-oz designed for use with ZnSe and Ge ATR crystals	SLP-CLP
Force Sensor with Digital Read-Out	MVR-FSD
O-Ring Sealed Liquid Cell with Luer Fittings	UNS-LCF
Liquid Cell Viton O-Ring	ORV-0015
Powder Adapter	UNS-PSC
Powder Adapter Viton O-Ring	ORV-012
Automatic Temperature Controller, 110V	ATC-024-1
Automatic Temperature Controller, 220/240V (CE marked)	ATC-024-2

THE MVP-PRO ™

This single reflection ATR accessory combines a convenient horizontal sampling surface with a 0.5mm diameter sampling area, making the MVP-Pro™ ideal for quick and easy examination of a wide range of samples. Small drops of liquid and minute smears of pastes can be placed directly on the ATR crystal. Coatings on wire, paint chips, fabrics, powders, and miniature hard samples can be pressed against the crystal using the built-in pressure applicator. The MVP-Pro™ also features PermaPurge™ for rapid sample and crystal exchange without interrupting the purge of the spectrometer. The MVP-Pro™ ATR makes FTIR spectroscopy of small samples simple and straightforward.

Crystal Material	Additional ATR Sampling Plates	Heated Sampling Plates
Diamond	MVP2-ATR-0W	UNS-HOT-0W
Ge	MVP2-ATR-J	UNS-HOT-0J
Si	MVP2-ATR-E	UNS-HOT-0E
ZnSe	MVP2-ATR-M	

Applications

- Excellent for studying optically thick, hard samples; slightly curved samples; fibers; paint chips; micro-liters of liquids and pastes; and powders.
- Invaluable for QC applications.

Features

- Convenient horizontal sampling.
- High sample throughput.
- Little or no sample preparation required.
- Excellent retention of sample integrity.
- Small sampling area – 0.5mm in diameter.
- Si ATR hemisphere for operation from 10,000 cm^{-1} to 100 cm^{-1}.
- Readily exchangeable ATR sampling plates.
- Built-in pressure applicator, compatible with a Torque Screwdriver for reproducible calibrated pressure application.
- Designed to achieve optimal contact between the ATR element and solid samples.
- Additional Ge and Si hemispherical ATR crystal materials are available including a diamond prism and ZnSe and Ge hemispheres.
- Harrick's exclusive PermaPurge™ allows rapid sample exchange without interrupting the purge of the system.
- Mechanically compatible with the SplitPea™ and Meridian™ crystal holders.
- Options include

Includes

- Si ATR sampling plate.
- Built-in pressure applicator.
- Mating hardware for the specified spectrometer.

Ordering Information

	Catalog No.
MVP-Pro ™	MVR-XXX

Options

LowTorque Slip-Clutch, 24 in-oz designed for use with ZnSe and Ge ATR crystals	SLP-CLP
Force Sensor with Digital Read-Out	MVR-FSD
O-Ring Sealed Liquid Cell with Luer Fittings	UNS-LCF
Liquid Cell Viton O-Ring	ORV-0015
Powder Adapter	UNS-PSC
Powder Adapter Viton O-Ring	ORV-012
Automatic Temperature Controller, 110V	ATC-024-1
Automatic Temperature Controller, 220/240V (CE marked)	ATC-024-2

VIDEO MVP™

The VideoMVP™ offers all the advantages of a high performance diamond ATR accessory plus video imaging and optional force sensing capabilities. The VideoMVP™ features a convenient horizontal sampling with an active sampling area of less than 500 µm in diameter on its monolithic diamond. This makes it ideal for infrared spectroscopy measurements of extremely hard samples, abrasive powders, fibers, beads, and even corrosive materials. The video imaging system provides a real-time magnified view of the sample through the ATR crystal. This image can be seen on the built-in display or digitally captured for a permanent record. Solid samples are compressed against the ATR crystal using the built-in pressure applicator. The pressure applicator features a slip clutch to prevent over-pressurization. An optional force sensor with digital read-out is available for precise measurement of the force applied to the sample and to reproducibly apply lower force than delivered by the built-in slip clutch.

Applications

- Extremely hard samples; highly corrosive liquids; minerals; slightly curved samples; fibers; beads; nanoliters of liquids and pastes; and defects on large panels.
- Forensics, textiles and combinatorial chemistry samples.
- Samples requiring archival storage of images as well as spectra.

Features

- Monolithic hemispherical diamond mechanically retained in a chemically resistant holder.
- All reflective optics other than the ATR crystal.
- Convenient horizontal sampling.
- Designed for optimal contact between the ATR element and solid samples.
- Minimal sample preparation required.
- Small sampling area: 500 µm in diameter with the diamond ATR crystal.
- Incident angle: 45°.
- Built-in pressure applicator with slip-clutch for reproducible calibrated pressure application.
- PermaPurge™ for rapid purging of the system.
- RoHS compliant.
- Video imaging system for viewing through the diamond ATR includes:
  - Built-in LCD display on a rotating mount for easy viewing.
  - 120X magnification of the sample.
  - USB-video adapter and software included for storing images.
- Options:
  - Readily exchangeable Ge, ZnSe, Si and diamond ATR sampling plates.
  - Heatable sampling plates for operation up to 200°C with diamond, Si or ZnSe ATR crystals, 100°C with Ge.
  - Powder adapter for retaining powdered samples.
  - Flow-through liquid cell for static and flow applications.
  - Force sensor with digital read-out for precise force measurements.

Mounted ATR Crystals

	Ambient	Temperature Controlled
Si	UNS-ATR-0E	UNS-HOT-0E
ZnSe	HOR-SSP-M	UNS-HOT-0M
Ge	UNS-ATR-0J	UNS-HOT-0J
Diamond	UNS-ATR-2W	UNS-HOT-0W

Includes

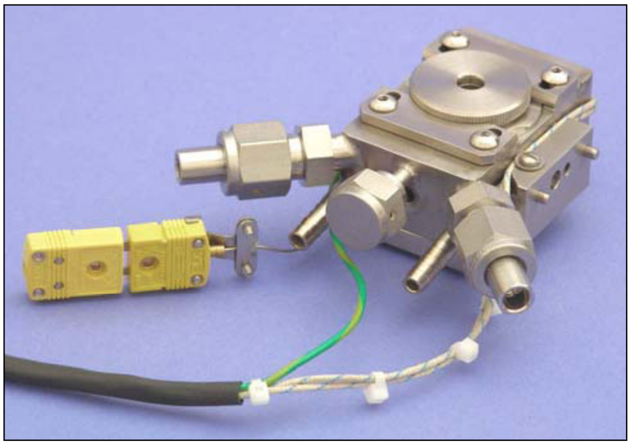
- Mounted diamond ATR.
- Built-in force limited pressure applicator for solid sampling.
- LCD display and USB-video adapter for video imaging.
- Mating hardware for the specified spectrometer.

Ordering Information

	Catalog No.
VideoMVP™, 110V	MVV-XXX-1
VideoMVP™, 220/240V	MVV-XXX-2
Force Sensor, 110V	MVV-1-FSD
Force Sensor, 220/240V	MVV-2-FSD

	Catalog No.
Liquid Cell	UNS-LCF
Liquid Cell O-Ring	ORV-0015
Powder Retainer	UNS-PSC
Powder Retainer O-Ring	ORV-012

REACTION CHAMBER FOR RAMAN SPECTROSCOPY



Applications

- Allows Raman measurements under controlled pressures and a wide range of temperatures.
- Use in conjunction with a Raman microscope or spectrometer.

Features

- Designed for operation from high vacuum (10^{-6} torr) to 25.8 ktorr.
- Operable from ambient temperatures up to 910°C (under vacuum).
- 8-mm open aperture.
- Removable 13x2mm SiO_2 window.
- Three inlet/outlet ports provided for evacuating the cell and introducing gases.
- Made from chemically resistant 316 stainless steel. minimal interruption of the system purge.
- Optional Restek Silcosteel®-CR coating available for greater corrosion resistance.
- Optional mounting available.

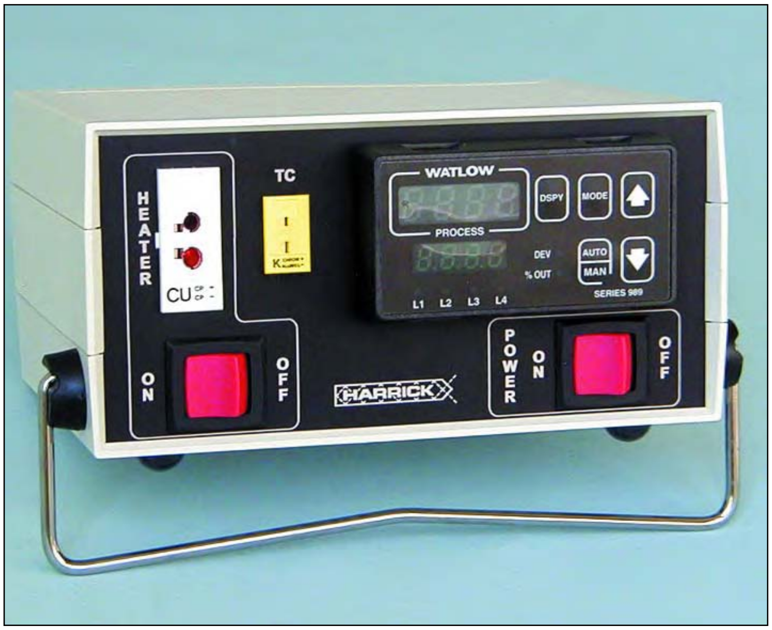
Includes

- Reaction Chamber.
- Low-voltage heating cartridge.
- K-type thermocouple.
- Window assembly with Viton o-rings and 13x2mm SiO_2 window.

Ordering Information	Catalog No.
Window Assembly for Microscopy	HVC-MRA
Reaction Chamber, 24V	HVC-000-4
Options & Replacement Parts	
Screen Set, two each of three mesh sizes	116-439
K-Type Thermocouple	008-144
13x2mm SiO_2 window	HVC-DWM-3
Heater Assembly	HVC-HT4
Heater, 24V	HTRS-26
Automatic Temperature Controller, 24V output, 110V	ATC-024-1
Automatic Temperature Controller, 24V output, 220/240V (CE marked)	ATC-024-2
Vacuum Pump, 110V	VPE-001

AUTOMATIC TEMPERATURE CONTROLLER

The Automatic Temperature Controller is a full-featured controller designed for a variety of applications. It can be manually programmed to reach a desired temperature or computer controlled for sophisticated temperature profiling. The controller automatically determines the best settings for optimal heating based on the thermal response of a particular system. It features two thermocouple inputs and one heater output, allowing regular or cascade operation. Cascade operation permits the heater output to be regulated with two thermocouple inputs. For accessories with separate heater and sample thermocouples, one set point can control the sample temperature while another provides an emergency shut off at another set point. This is particularly important for high temperature applications, where the desired sample temperature is close to the high temperature limit of the heater.



Features

- Provides accurate temperature regulation over a wide range of temperatures.
- Provides a safe, low-voltage output.
- Intended for use with 24V heaters.
- Set point programmable from -200 to 1250°C for K-type thermocouples.
- Accepts a wide variety of thermocouples and resistive temperature detectors (RTDs).
- Cascade or regular operation.
- Digital temperature readout in degrees Fahrenheit or Celsius.
- Manual or computerized programming.
- RS232 port.
- Provides a safe, low-voltage output.
- Intended for use with 24V heaters.

TORQUE SCREWDRIVER

Applications

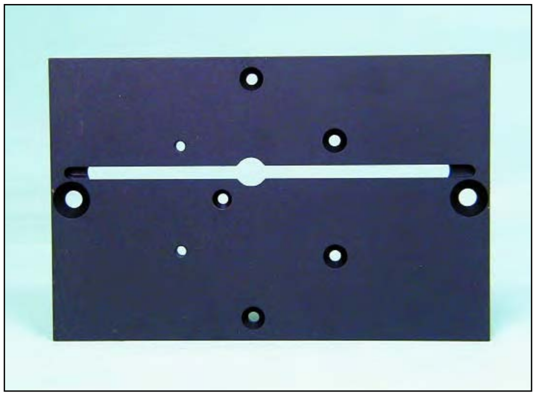
- Ideal for applying measured and reproducible pressure to crystal holders and pressure plates.

Features

- Accurate torque settings from 2 to 100 oz/in.
- Free-wheeling mode prevents over torquing and potential optical breakage.
- Includes hex keys for 4-40, 6-32, 8-32, and 10-32 screws for use with Harrick Scientific's pressure plates.

Ordering Information	Catalog No.
Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220V/240V input, 24V output (CE marked)	ATC-024-2
Torque Screwdriver	PTW-SXX

RAIL MOUNT



Features

- System for mounting attachments in sample compartments securely and precisely.
- Simplifies operations where other items, such as polarizers, must be placed co-linear with the sample beam.
- Included with some accessories.
- Permits translation of accessories along the beam path, for optimum performance.
- Compatible with Harrick Scientific's accessories.

SAMPLE SLIDE HOLDER

Features

- Convenient for holding sample slide plate mounted attachments, such as Demountable Liquid and Gas Cells, sample holders, attenuators, polarizers, and depolarizers.
- Compatible with Harrick Scientific's Rail Mount.

INFRARED BEAM FINDER

Applications

- Allows viewing of the infrared beam.
- Simplifies alignment of accessories.

Features

- Infrared sensitive paper.

Ordering Information	**Catalog No.**
Rail Mount	HRM-XXX
Sample Slide Holder	HSS-XXX
Punch Sampler	PUN-SMP
Infrared Beam Finder	IBF-000

PUNCH SAMPLER

Applications

- Excellent for sampling thin and soft substrates, including inks and defects on paper or plastic, discolorations on cloth and forensic samples.
- Efficient tool for preparing samples for analysis with the SplitPea™, Meridian™ and Video Meridian™, high pressure diamond anvil cells, and other microsampling accessories.

Features

- Simple, spring-loaded mechanism for cutting small, round samples from larger panels.
- Self-sealing cutting mat to prevent sample contamination and damage to the tip.
- Dispenses 0.5mm diameter samples.
- Individually pouched and ethylene oxide sterilized.
- Disposable punches with limited reusability; autoclavable up to 250°F.

Includes

- Five punches.
- One cutting mat.

Ordering Information	Catalog No.
Punch Sampler	PUN-SML

Replacement Parts	
Punches (25 pieces)	PUN-PAK
Cutting Mat	PUN-MAT

THE FIBERMATE2™

The FiberMate2™ links your FT-IR spectrometer to fiber optic probes. Equipped with large ellipsoidal mirrors, the FiberMate2™ efficiently transmits the FT-IR or UV-VIS beam of the spectrometer through the fiber optic probe for spectroscopy measurements. The FiberMate2™ utilizes all reflective optics, optimizing the wavelength range of the fiber optic probe employed. FiberMate2™ comes with two SMA connectors for easy installation of the fiber optic probe and it is equipped with PermaPurge™ to maintain the purge of the system.

Applications

- Mates fiber optic probes to UV-VIS or FT-IR spectrometers.
- Connects the Harrick MultiLoop-MIR™ infrared fiber optic probes to FT-IR spectrometers.
- Interface external optical systems to the spectrometer.
- Well-suited for optical testing of fiber optics for photonics applications.

Features

- Beam condensing optics efficiently transmit the UV-VIS or infrared light to and from fiber optic probes for spectroscopy measurements.
- All reflective optics maximize the wavelength range of the fiber optics employed.
- Two standard SMA connectors for easy installation of fiber optics probes.
- Designed for use with visible, near-IR and mid-IR fiber optic probes.
- Harrick's exclusive PermaPurge™ enclosure eliminates interference from atmospheric infrared absorption bands in the infrared and protects against stray light in the UV-VIS.
- Compatible with every major FT-IR spectrometer and some UV-VIS instruments.
- Prealigned and ready for the most challenging fiber optic spectroscopy application.
- Optional MultiLoop-MIR™ infrared fiber optic ATR probes available.

Ordering Information	Catalog No.
FiberMate2™	FM2-XXX*

Options

MultiLoop-MIR Mid-IR Set	FOP-MIR
MultiLoop-MIR Silver Halide Probe Set for use from 2000 cm-^{1} to 600 cm-^{1}	FOP-PIR
MultiLoop-MIR Chalcogenide Probe Set for use from 6500 cm-^{1} to 1700 cm-^{1}	FOP-CIR

**XXX denotes the spectrometer code. Varian customers receive a different model. Please contact us for details.*

MULTILOOP-MIR™ FTIR FIBER OPTIC PROBE:
SPECTROSCOPY OUTSIDE THE BOX

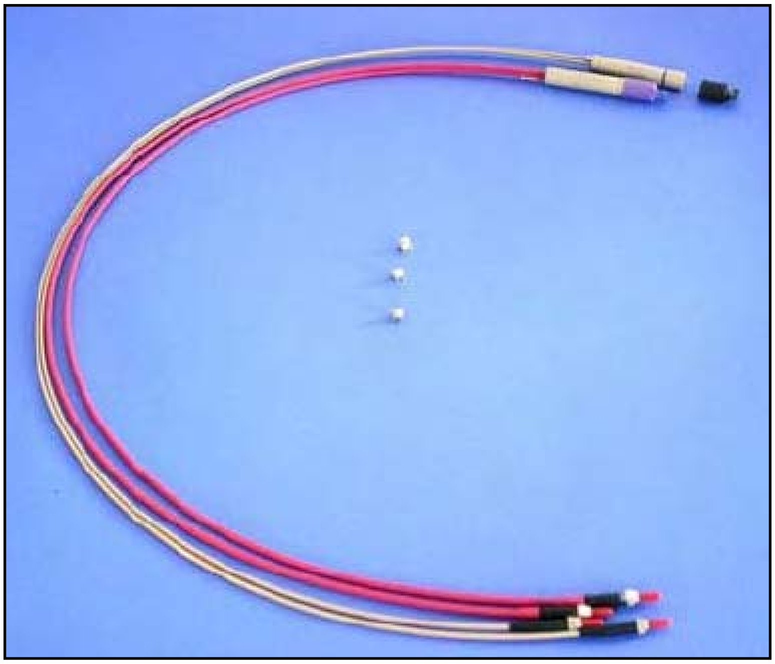
This MultiLoop-MIR™ system is perfect for analyzing liquids, pastes, and soft solids outside of the FTIR spectrometer sample compartment. It is used in conjunction with the Harrick FiberMate™ or other fiber optic coupler and includes two fiber probes with a set of ten ATR loop tips. This combination is designed for spectral data collection across the entire mid-infrared region. To analyze a sample, simply dip the ATR loop tip into the sample or press it gently against the sample. The tips are easily replaced when needed.

Applications

- Multiple reflection ATR (internal reflectance) accessory.
- In-situ analysis of a wide variety of liquids, pastes, and soft solids.
- Quantitative and qualitative analysis.

Features

- Multiple reflection ATR sampling outside the FTIR spectrometer.
- Effectively provides two reflections when compared to ATR with ZnSe at 45°.
- Wavelength range: 6500 cm^{-1} to 600 cm^{-1}:
  - Chalcogenide glass probe for use from 6500 to 1700 cm^{-1}
  - Polycrystalline Silver halide probe for use from 2000 to 600 cm^{-1}.
- Sampling tips designed for the full spectral range of 6500 to 600 cm^{-1}:
  - Dip into or press against the sample.
  - Leak-free for analysis of liquids.
  - Readily replaceable.
  - Made from silver halide fiber material.
- Easy to use.
- Suitable for aqueous solutions and organic samples.
- Standard probe length of 1 meter allows sampling outside the spectrometer sample compartment.
  - Designed for use with DTGS or MCT detectors.
  - SMA connectors for easy connection to fiber optic couplers.
  - Operable from room temperature to 100°C.
  - Use with the Harrick FiberMate™ fiber optic coupler for purged operation in most FT-IR spectrometers.



Includes

- MultiLoop-MIR™ Mid-IR Set for the entire mid-IR
  - Silver Halide Probe.
  - Chalcogenide Probe.
  - Ten Silver Halide Tips.
- MultiLoop-MIR™ Silver Halide Probe Set (2000 to 600 cm^{-1})
  - Silver Halide Probe.
  - Five Silver Halide Tips.
- MultiLoop-MIR™ Chalcogenide Probe Set (6500 to 1700 cm^{-1})
  - Chalcogenide Probe.
  - Five Silver Halide Tips

Ordering Information

Ordering Information	Catalog No.
MultiLoop-MIR™ Mid-IR Set	POP-MIR
MultiLoop-MIR Silver Halide Probe Set for use from 2000 cm^{-1} to 600 cm^{-1}	FOP-PIR
MultiLoop-MIR Chalcogenide Probe Set for use from 6500 cm^{-1} to 1700 cm^{-1}	FOP-CIR
FiberMate™ Fiber Optic Coupler	FMA-XXX*
PlasmaFlo™	PDC-FMG-1
PlasmaFlo™	PDC-FMG-2

Replacement Parts

Disposable Loop Tips (set of 5)	FOP-T05
Replacement cover kit (includes replacement fiber covers, etc.)	FOP-PRT

XXX indicates spectrometer make and model

OMNI-DIFF™ and OMNI-SPEC™

The Omni-Diff™ and Omni-Spec™ are fiber optic probes designed to interface to virtually any spectrometer via fiber optics. These miniature accessories come equipped with input and output SMA connectors that readily attach to a fiber optic coupler, like the Harrick FiberMate2™. Both are ideal for reflectance measurements up to 1.5 meters away from the spectrometer. The Omni-Diff™ measures the radiation diffusely reflected from the sample with optical elimination of the specular component. The Omni-Spec™ examines specular reflectance at a 45º incident angle. The Omni-Diff™ and Omni-Spec™ are offered with a selection of fiber optics for use in the UV-VIS, NIR and mid-IR. A digital imaging system is also available for photographic documentation of the sample.

Applications

- Reflectance measurements of solids which are too large to fit in the sample compartment.
- No sample preparation required.
- Omni-Diff™ for analysis of rough surfaced solids, contaminants and coatings.
- Omni-Spec™ for examining optical coatings, contaminants, opaque substrates and films thereon and in-line diffuse reflectance of rough-surfaced solids.
- Photographic documentation of samples undergoing reflectance measurements.

Features

- Compact and convenient to use..
- Suitable for use from the UV to the IR. For best performance, use with an MCT detector in the IR.
- Omni-Diff™ maximizes the collection of the diffusely reflected light, while minimizing the specular component.
- Omni-Spec™ features a 45º nominal incident angle.
- Two standard 950 SMA connectors for connecting suitable fiber optic cables.
- Optical fiber optics available in 1.5m long sets:
  - UV-VIS/Near-IR fiber optics for use from 350nm to 2250nm.
  - Near/Mid-IR fiber optics for use from 6500cm^{-1} to 2240cm^{-1} and 2050cm^{-1} to 1000cm^{-1}.
  - Mid-IR fiber optics for use from 2000cm^{-1} to 600 cm^{-1}.
- Optional video imaging and illumination system:
  - Magnifies the image for easy viewing.
  - Directly views the sampling surface.
  - Includes software for illumination, real-time viewing and storing the images.
  - USB2 compatible.
  - CE marked.
- Optional references for Mid-IR and UV-VIS/Near-IR studies.

Includes

- Omni-Diff™ or Omni-Spec™
- Use with a fiber optic coupler, such as the Harrick FiberMate2™, and sets of two 1.5m fibers offered separately.

Ordering Information

Ordering Information	Catalog No.
Omni-Diff™	OMN-DIF
Omni-Spec™	OMN-SPC
UV-VIS/Near-IR Fiber Set (350nm to 2250nm)	OMN-F-UVN
Near/Mid- IR Fiber Set (CIR fibers, 6500cm^{-1} to 2240cm^{-1} and 2050cm^{-1} to 1000cm^{-1})	OMN-F-CIR
Mid-IR Fiber Set (PIR fibers, 2000cm^{-1} to 600cm^{-1})	OMN-F-PIR

Options and Replacement Parts

Digital Camera for the Omni-Diff™	OMN-CAM
Reference Fixture (includes sandblasted Al diffuse reference, spectralon reference and front-surface Al mirror)	OMN-REF
FiberMate2	FM2-XXX*

**XXX indicates spectrometer make and model*

POLARIZERS

Glan-Taylor Polarizer

Harrick Scientific offers several highly efficient polarizers: Brewster's Angle Polarizer (diamond configuration) and Wire Grid Polarizers for the IR and Glan-Taylor Polarizers for the UV. High efficiency wire grid polarizers, wire grid polarizers on other substrates, e.g. ZnSe, and polarizers with larger apertures, e.g. 50mm, are available upon request.

Wire Grid Polarizer

Polarizer	Substrate	Clear Aperture Diameter (mm)	Settings	Useful Range	Efficiency	Catalog Number
Wire Grid	KRS-5	22	0-360°	5000-285cm^{-1}	95% @ 2000cm^{-1}	PWG-U1R
	KRS-5	22	0 and 90°	5000-285cm^{-1}	95% @ 2000cm^{-1}	PWG-SEA
Glan Taylor	Calcite	12	0-360°	250-2300 nm		PGT-S1V
Glan Thompson	Calcite	12	0-360°	250-2300 nm		PTH-SMP

DEPOLARIZER

Applications

- Useful for depolarizing UV-VIS radiation.

Features

- Highly efficient, regardless of the incident polarization.
- Fabricated from crystalline quartz.
- 22mm clear aperture.

DIAPHRAGM OPTICAL ATTENUATOR

Features

- Helpful for aligning FTIR spectrometers, precisely controlling the beam size, and eliminating stray radiation.
- Attenuation is achieved by varying the diameter of the iris from 25mm to 0.5mm.
- Mounts in a standard sample slide holder.

Ordering Information	**Catalog No.**
Depolarizer	DPS-R4V
Diaphragm Optical Attenuator	OAD-S1G

SLIP CLUTCH

Applications

- Ideal for applying measured and reproducible pressure to ATR crystals for FT-IR spectroscopy.

Features

- Fixed, accurate torque settings.
- Prevents over torquing and potential optical damage.
- Two slip-clutch models available:
- 24 in-oz slip-clutch for low ATR contact pressures.
- 56 in-oz slip-clutch for high ATR contact pressures.
- Built-in hex head (5/16") affixes and secures directly to ATR accessories, such as Harrick Scientific's FastIR™, MVP2™, MVP2 Star™ and GATR™.

Features

- Infrared sensitive paper.

Ordering Information	Catalog No.
Slip-Clutch, 24 oz-in	SLP-CLO
Slip-Clutch, 56 oz-in	SLP-CHI

POWDERPRO™

The PowderPro™ is specifically designed to analyze powders and is the ATR alternative to diffuse reflectance. Fine powders can be examined neat; coarser powders and hard solids can be ground prior to analysis. Simply place the sample in the cup or sample tray, slide the holder in, compress the sample against the crystal with the built-in press, and collect the spectrum. The crystal and the rest of the optical assembly rotates around the optical axis for easy access to the crystal for cleaning and replacement. In addition, the sample holders are easily disassembled for thorough cleaning. The PowderPro™ also can be adapted for examining liquids, solids, and pastes.

Applications

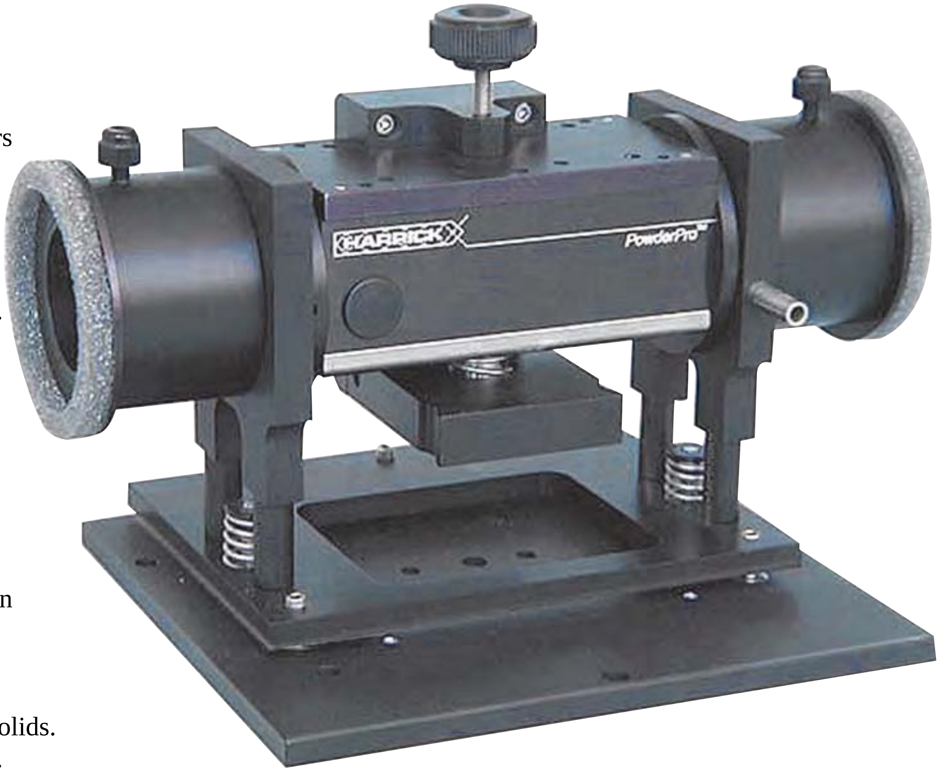
- Single, internal reflectance.
- Convenient for routine analysis of fine powders and other solid samples.
- Excellent for both laboratory and quality control environments.

Features

- Straightforward analysis of powdered samples.
- Highly repeatable, qualitative and quantitative results.
- Fixed 45° incident angle.
- SuperCharged™ ZnSe optics for superior performance.
- Easy to align and use.
- High throughput (greater than 85%).
- Minimal sample preparation required.
- Removable sample holders and rotatable design for easy clean-up.
- Powder cup retains all types of powders.
- Readily exchanged ATR crystal.
- Adaptable for analysis of liquids, pastes, and solids.
- PermaPurge™ for rapid purging of the system.

Includes

- Mounted ZnSe prism.
- Built-in pressure applicator for powder sampling.
- Powder Cup
- Mating hardware for the specified spectrometer.



Replacement ATR Crystals

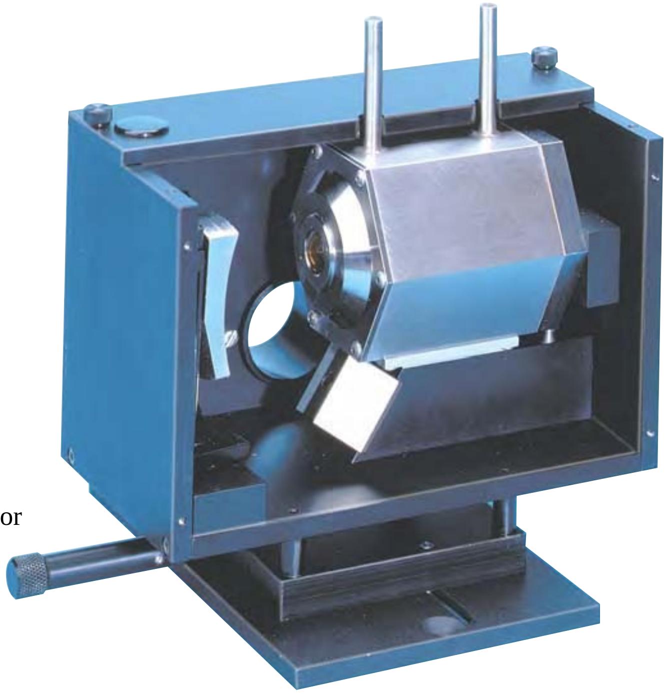
Material	Catalog No.
ZnSe	FAS-ATR-M
Zn S	FAS-ATR-I
Ge	FAS-ATR-J

CYCLOPS™

Transmission is the best method for spectral measurements of gaseous samples, including trace component analysis. Typically the cells used are single pass gas cells with pathlengths up to 10cm or multiple pass 'White' cells with much longer pathlengths. Cyclops™, with its one hemispherical window, provides an intermediate pathlength gas cell.

Applications

- Perfect for spectral measurements of gaseous samples that require intermediate pathlengths.



Features

- Low volume (0.134 l), intermediate pathlength cell (0.421m).
- Two ports provided for both flow-through and static operation.
- Chemically-resistant O-ring sealed, stainless steel body.
- Needle valve for regulating the flow through the cell and sealing the cell.
- Hemispherical window minimizes reflection losses and eliminates refraction effects.
- Conveniently fits into the sample compartment of many spectrometers.
- Heatable model available on special order.
- Two 1⁄4″ 316 stainless steel Swagelok™ fittings with teflon ferrules for inlet and outlet ports.

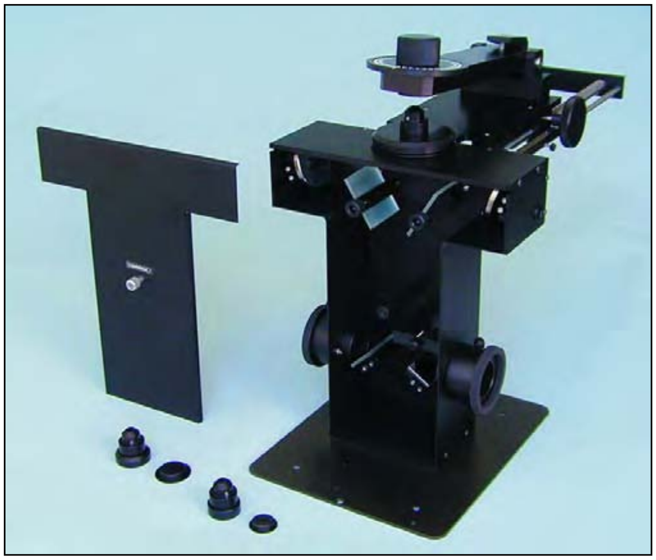
Includes

- ZnSe window and Viton O-rings.
- Transfer optics and mating hardware for the specified spectrometer.

O-Rings

	Viton	Kalrez
Lens Mount	ORV-014	ORK-014
Envelope Seals (#133)	ORV-133	ORK-133

DISK CHECKER



Applications

- Unsurpassed for examining lubricating films and surface contaminants on large magnetic disks.

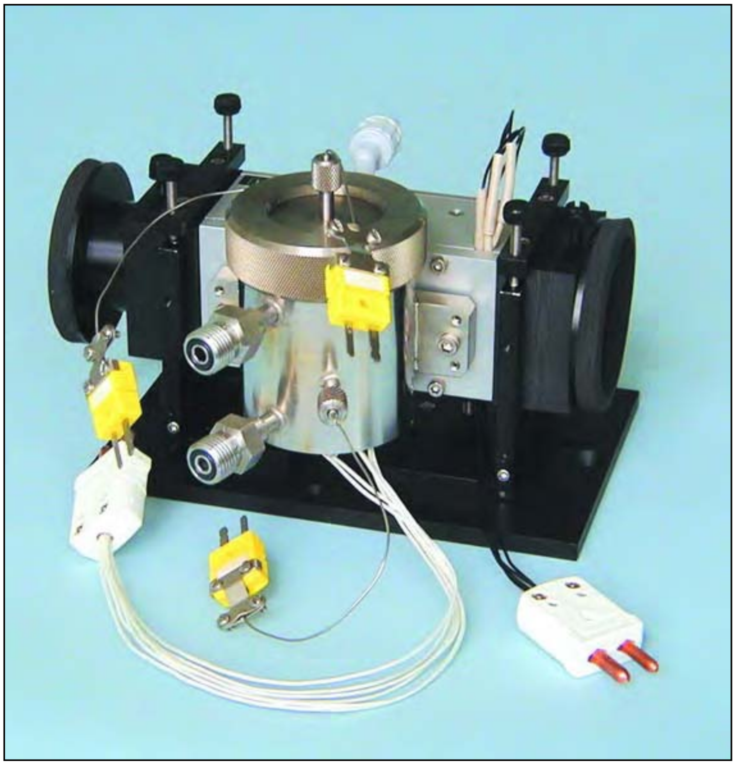
Features

- Unobstructed, horizontal sampling surface accommodates a wide range of disk sizes.
- Fixed 75° incident angle and built-in polarizer for optimum spectral sensitivity.
- Translation stage for systematic surface characterization.
- High throughput.
- Incorporates PermaPurge™ for minimal interruption of the purge when changing samples.
- Compatible with most standard disk sizes.

Includes

- Disk holders for 20mm, 25mm, and 2.5″ i.d. disks.
- Mating hardware for the specified spectrometer.
- An alignment fixture.

FASTIR™ Reactor



Applications

- Useful for real-time monitoring of liquid reactants and/or products via single reflection ATR.

Features

- Chemically resistant 316 stainless steel reaction vessel.
- Heatable to 250°C.
- Pressurizable to 800psi.
- Unique side-mounted 45° prism minimizes interference from precipitates.
- Highly efficient.
- Incorporates PermaPurge™ to maintain the system purge without closing the sample compartment cover.
- Simple to align and clean.
- Compatible with Parr MicroReactor™ reaction vessel heads.

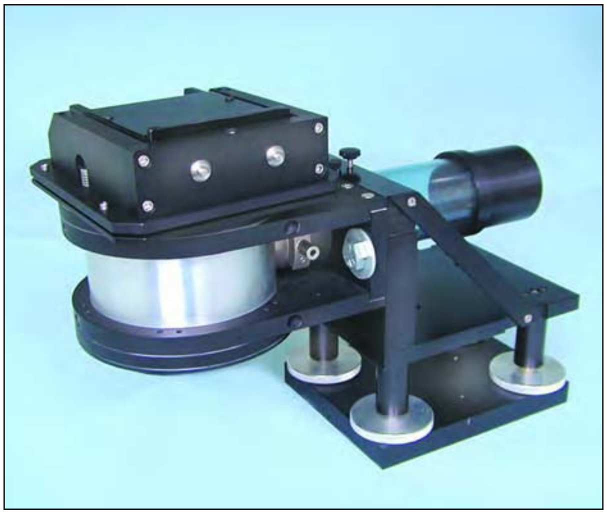
Includes

- Two heaters and a K-type thermocouple.
- Single reflection ZnSe ATR crystal.
- Mating hardware for the specified spectrometer.

CUSTOMIZED EXTERNAL REFLECTION ACCESORIES

Includes

- ▶ Various incident angles, including near-normal (8°) and Brewster's angle.
- ▶ Customized optical designs to accommodate specialized sampling needs.
- ▶ Integrated with a trough for surface analysis of liquids.



THE SPECTROPUS™ REMOTE SAMPLING SYSTEM

Spectropus™ is a versatile optical sampling system capable of performing spectroscopic analyses on remotely located and/or large samples. Specially designed Sampling Terminals, employing different spectroscopic techniques, are provided as integrated components of the system. The Sampling Terminals may be located some distance from the spectrometer and are connected to it via a purged optical Transfer System which is designed for maximum optical efficiency. Each terminal may be located in the laboratory, in a glove box, and/or at strategic places along a process line. More than one Sampling Terminal can be connected to the spectrometer.

Applications

- ▶ Custom-designed for infrared analyses of local and remotely located samples, multiple sampling, samples in glove boxes, and large samples.

Features

- ▶ Analyzes samples up to 15m away from the spectrometer.
- ▶ Highly efficient optical transfer system.
- ▶ Purgeable.
- ▶ Compatible with spectrometers with an external detector input and an external collimated beam port.
- ▶ Basic system includes Minimum Transfer System, Sampling Terminal Power Supply, and Sampling Terminal.
  - • Minimum Transfer System.
    - – Provides all equipment necessary to use the Sampling Terminals as an "external bench."
  - • Sampling Terminals feature:
    - – Dedicated detector and preamplifier.
    - – Separately purged for maximum sample throughput.
    - – Upward or downward looking mounting capabilities.
    - – Requires Harrick's external Power Supply, available separately.
- ▶ Models currently available:
  1. Barrel Ellipse Diffuse Reflectance/Emission Sampling Terminal for examining films or contaminants.
  2. External Reflection Sampling Terminal for analyses at a 75° incident angle. Suitable for analyses of thin films on reflective substrates.
  3. TurboATR™ Sampling Terminal for single reflection internal reflectance studies at a 45° incident angle and temperatures ranging from ambient to 175°C.
  4. Gas Cell Accessory for use with other Sampling Terminal detectors.

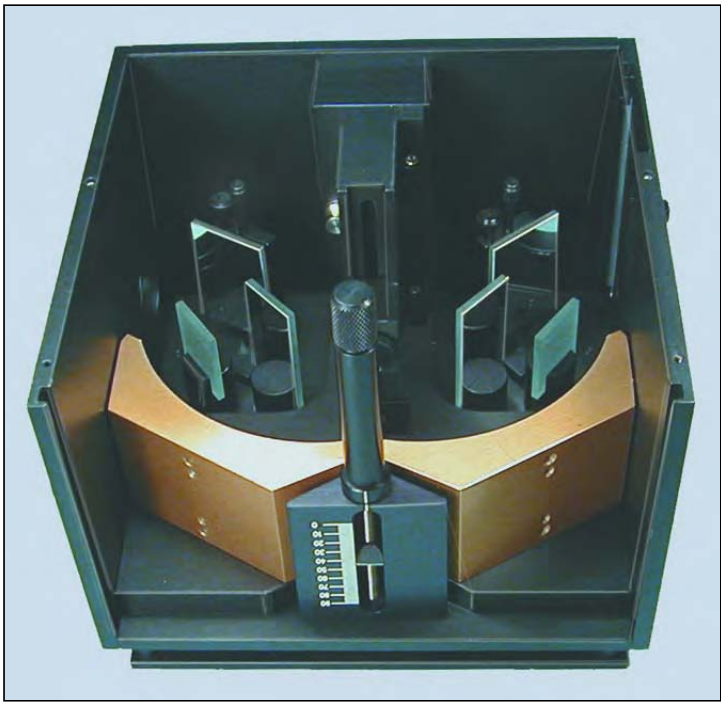
CUSTOM ACCESSORIES

Harrick Scientific's scientific, engineering, and manufacturing expertise combined with our high quality standards make us uniquely qualified to develop accessories for specialized applications. We have extensive experience in customizing for specialized applications and in developing new products to meet individualized requirements. The accessories featured below reflect a small portion of the customized products we have developed in response to the needs of our customers. Contact us for more details on these and other individualized systems.

SPECIALIZED SEAGULLS

Includes

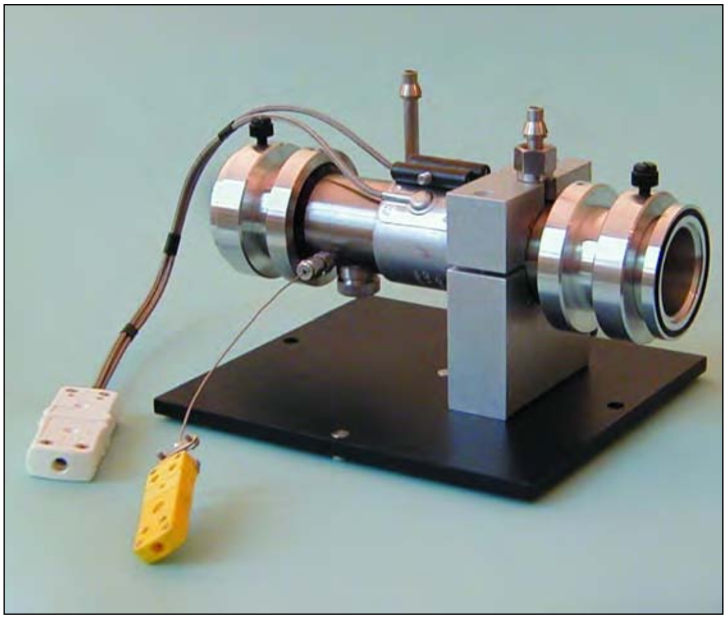
- ▶ Independently controllable angles of incidence and collection for studying bi-directional diffuse reflectance.
- ▶ Motorized for automated changes in the incident angle.
- ▶ Reoriented for vertical sampling to accommodate electrochemical cells.



CUSTOMIZED HORIZONTAL ATR

Includes

- ▶ Special crystal holders to accommodate crystals 0.25 mm to 0.5 mm thick for studies of chemically modified Si surfaces.
- ▶ Crystal holders designed for rapid crystal exchange during studies of coated or modified crystal surfaces.
- ▶ Large volume liquid cells for examining the residues of evaporated liquids.
- ▶ Integrated polarizers.



SPECIALIZED TRANSMISSION LIQUID CELLS

Includes

- ▶ Chemically inert models made from Hastelloy or Inconel.
- ▶ Longer pathlength cells, ranging from 5 mm to 25 mm.
- ▶ Specialized fittings and tubing to simplify connections for flow applications.

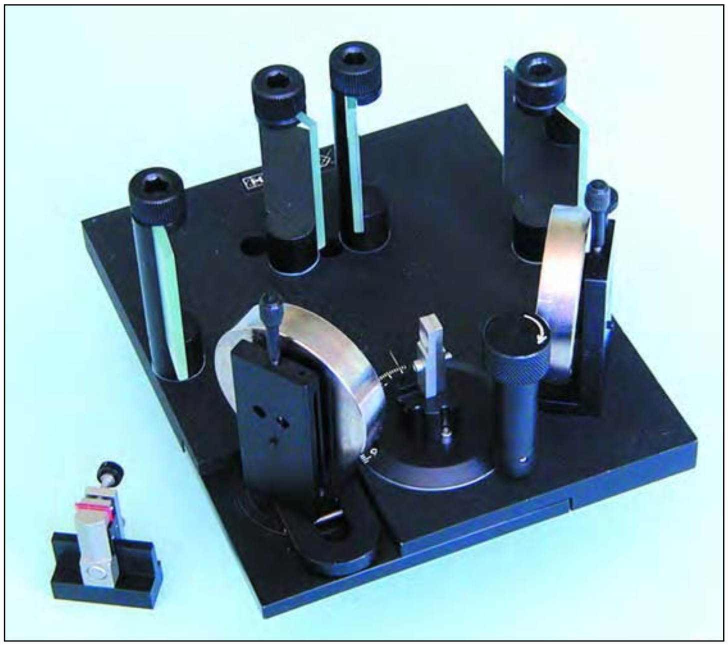
4X BEAM CONDENSER

Applications

- Transmission and internal reflection studies of small quantities of liquids, pastes, and soft or flexible solids. Continuously variable incident angle from 30° to 60°.

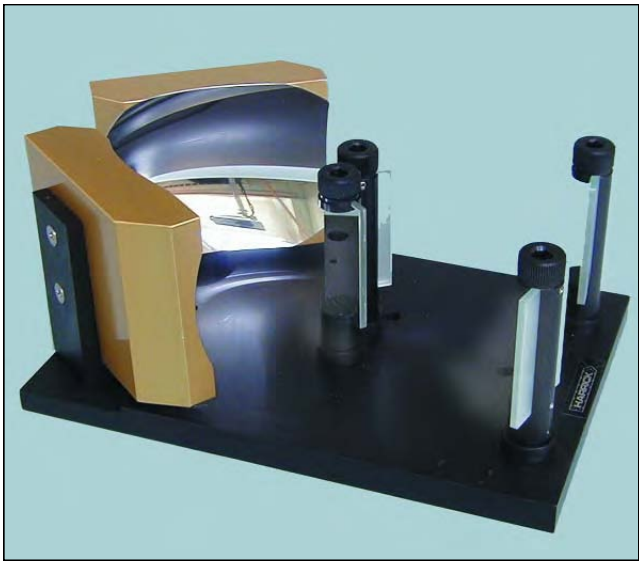
Features

- Variable Angle (30° to 60°) model. Uses 10mm and 25mm long crystals. Includes a dial for setting the angle.
- Generates a spot ¼ of the normal sample beam size for more efficient irradiation of small samples.
- Maximizes signal throughput while minimizing spectral distortions.
- Utilizes 10mm or 25mm long crystals.
- Easily converts from ATR to transmission.
- Specially designed ATR pressure plates float, eliminating unnecessary strain on the ATR crystal.
- Additional configurations of beam condensers are available by special order.



6X BEAM CONDENSER

These 6X Beam Condensers are especially suitable for examining very small quantities of samples with use of 90° off-axis ellipsoids. They also provide sufficient working space for such accessories as high-pressure diamond cells.



Applications

- Transmission and internal reflection studies of small quantities of liquids, pastes, and soft or flexible solids.
- Ideal for use with high-pressure diamond cells.

Features

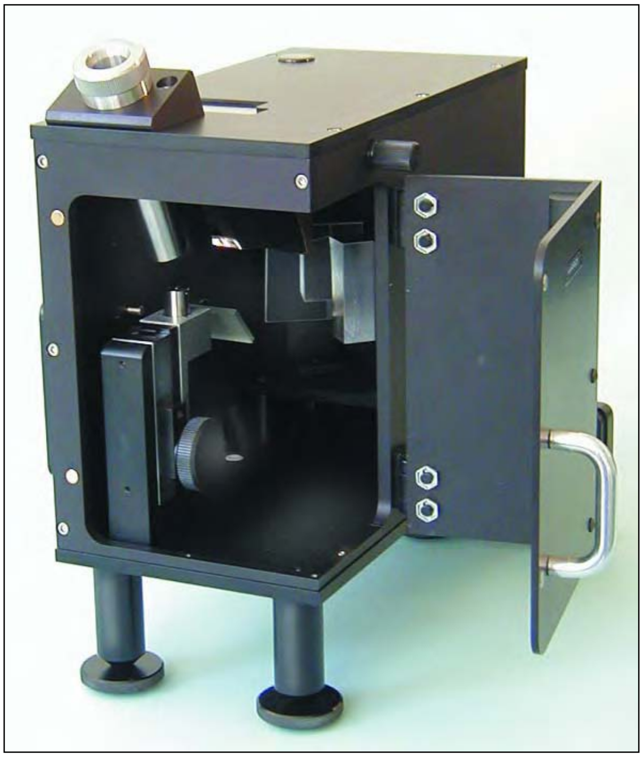
- Easily converts from internal reflectance to transmission to accommodate a variety of experiments.
- A spot 16% of the normal sample beam size, for more efficient irradiation of small samples.
- Fixed angle transmission beam condenser, providing a working space of approximately 40mm. Features large ellipsoids.
- Recommended for use with high-pressure diamond cells.

Includes

- One pellet holder
- Mating hardware for the specified spectrometer.

EMISSION ACCESSORY

Harrick's Emission Accessory is a powerful tool for examining vibrational spectra of surface species. It can be attached to any spectrometer with an emission port.



Applications

- Powerful tool for studying vibrational spectra of surface species.

Features

- Translation stage for easy sample positioning.
- Interchangeable masks define the area of the sample surface analyzed.
- Collects the emittance over a large angular distribution.
- Permits sampling of small areas.
- Use with either a room temperature or cooled detector.
- Purgeable.
- Optional viewing microscope available for identification of areas of interest.
- Compatible with any spectrometer with an emission port.

Includes

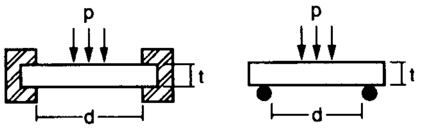
- XYZ translation stage.
- Interchangeable masks.
- Heater and K-type thermocouple.
- Mating hardware for the specified spectrometer.

UV-VIS-IR OPTICAL MATERIALS

Material	Refractive Index n	Hardness (Knoop) psi	Modulus of Rupture* psi	Useful Transmission Range		Chemical Properties
				Windows (1-2mm) cm-1 micrometers	IREs (70mm) cm-1 micrometers	
BK-7 Glass	1.50	520	2400	31,000-4300 0.32-2.3	29,000-5000 0.34-1.97	Insoluble in water.
UV Fused Silica UV SiO_2	1.44	461	7100	57,000-4600, 3500-2800 0.175-2.2, 2.9-3.6	50,000-5000 0.2-2.0	Insoluble in water; soluble in HF.
UV Sapphire Al_2O_3	1.75	1370	65,000	66,000-2000 0.15-5.0	33,000-2800 0.3-3.7	Very slightly soluble in acids and bases.
Strontium Titinate $SrTiO_3$	2.41	595	7500	25,000-1700 0.395-6	25,000-2500 0.4-4	Readily attacked by HF; resistant to most solvents.
Lithium Fluoride LiF	1.33	110	2000	90,000-1,500 0.11-7.0	50,000-2300 0.2-4.5	Slightly soluble in water; soluble in HF.
Titanium Dioxide TiO_2	2.6;2.9	800	700	24,000-1700 0.42-6	20,000-2200 0.5-4.5	Soluble in H_2SO_4 and alkalis; insoluble in water and acid.
Zirconium Dioxide ZrO_2	2.15	1250	7800	27,000-1,500 0.36-7	25,000-1800 0.4-5.5	Insoluble in water; soluble in HF and H_2SO_4.
Magnesium Oxide MgO	1.68	640	19,000	25,000-1300 0.4-8.0	20,000-1700 0.5-6.0	Soluble in acids and NH_4 salts.
Silicon Si	3.42	1150	9000	10,000-100 1.0-100	9500-1500; 350-FIR 1.06-6.7; 30-FIR	Insoluble in most acids and bases; soluble in HF and HNO_3.
Calcium Fluoride CaF_2	1.4	158	5300	66,000-1300 0.15-8.0	33,000-1500 0.3-7.0	Insoluble in water; resists most acids and bases; soluble in NH_4 salts.
Arsenic Trisulfide As_2S_3	2.4	109	2400	14,000-1200 0.7-9.0	12,500-1300 0.8-8.2	Soluble in alcohols and alkalis; slightly soluble in water.
Strontium Fluoride SrF_2	1.44	1405	500	66,000-1100 0.15-11	33,000-1100 0.3-9.5	Very slightly soluble in water; soluble in hot HCl.
Barium Fluoride BaF_2	1.45	82	3900	50,000-1000 0.2-11	33,000-1100 0.3-9.5	Low water solubility; soluble in acid and NH_4Cl.
Zinc Sulfide ZnS	2.22	355	10,000	22,000-750 0.45-14.0	14,000-1000 0.7-10	Soluble in acid; insoluble in water.
Germanium Ge	4.0	550	7000	5000-600 2.0-17	5000-900 2.0-11.4	Insoluble in water; soluble in hot H_2SO_4 and aqua regia.
Arsenic Triselenide As_2Se_3	2.8	90	500	12,500-600 0.8-17	11,000-900 0.9-11.8	Soluble in bases.
Sodium Chloride NaCl	1.5	15	350†	28,000-700 0.35-15	25,000-900 0.4-12	Hygroscopic; slightly soluble in alcohol and NH_3.
AMTIR (GeAsSe Glass)	2.5	170	2700	11,000-1000 0.9-11	11,000-1100 0.9-9.5	Insoluble in water.
Gallium Arsenide GaAs	3.14	750	11,000	10,000-600 1-17	10,000-700 1-14	Insoluble in water; slightly soluble in acids and bases.
Zinc Selenide ZnSe	2.42	150	8000	20,000-500 0.5-20	20,000-700 0.5-14.3	Soluble in strong acids; dissolves in HNO_3.
Potassium Chloride KCl	1.47	7	330†	33,000-500 0.3-20	20,000-700 0.5-15	Hygroscopic; water soluble; slightly soluble in alcohol.
Silver Chloride AgCl	2.00	10	3800†	23,000-400 0.42-27	22,000-700 0.45-16	Insoluble in water; soluble in NH_4OH.
Potassium Bromide KBr	1.52	7	160†	33,000-400 0.3-25	20,000-500 0.5-20	Soluble in water, alcohol, and glycerine; hygroscopic.
Silver Bromide AgBr	2.2	10	500†	20,000-300 0.5-35	20,000-500 0.5-22	Insoluble in water and alcohol; slightly soluble in NH_4OH.
Cadmium Telluride CdTe	2.65	45	850	10,000-350 1.0-28	10,000-500 1.0-22	Insoluble in acid and water.
Cesium Bromide CsBr	1.65	20	1220†	33,000-250 0.3-40	25,000-400 0.4-27	Soluble in water and acids; hygroscopic.
Cesium Iodide CsI	1.72	20	810	33,000-150 0.3-70	20,000-400 0.5-30	Soluble in water and alcohol; hygroscopic.
Thallium Bromoiodide KRS-5	2.35	40	3800†	16,000-200 0.6-60	14,000-400 0.7-30	Soluble in warm water; soluble in bases; insoluble in acids.
Crystal Quartz SiO_2	1.5	741	1500	50,000-4000 0.2-2.4	50,000-6,000 0.2-2.0	Soluble in HF; insoluble in water.
Polyethylene [PE] (high density)	1.55	5	400†	600-10 16-1000	600-FIR 16-FIR	Resistant to most solvents.
Diamond C	2.4	7000	54,400	45,000-2500; 1600-FIR 0.22-4; 6-FIR	45,000-2500; 1600-FIR 6-FIR**	Insoluble in water, acids, and bases.

*****The modulus of rupture, m, determines the size of the window needed to withstand a pressure, **p**. For a four times safety factor, the thickness of a mounted window, **t**, with an unsupported diameter, **d**, is:

$$t_{clamped} = 0.866\ d\ (p/m)^{1/2} \qquad t_{unclamped} = 1.05\ d\ (p/m)^{1/2}$$



** Pathlength of a few mm

† The apparent elastic limit is reported for those materials which permanently bend prior to rupturing.

COMMONLY USED INTERNAL REFLECTION ELEMENTS (ATR)

Internal reflection element (IRE) is the generic name for various optical elements, the heart of Internal Reflection Spectroscopy. The widest IRE is called an internal reflection plate (IRP). The IRP is available as a parallelepiped or trapezoid with a 30°, 45°, or 60° bevel. Standard dimensions are: lengths of 50, 25, and 10 mm; widths of 20, 10 and 5 mm; and thicknesses of 3, 2, 1, and 0.5 mm. Other IREs available include prisms, hemispheres, and hemicylinders. The most common materials for IREs are KRS-5, ZnSe, Ge, Si, and ZnS.

Sizes listed in black are available for immediate delivery. Sizes listed in green are available upon request. Other IRE sizes, shapes, bevels, and materials are available on special order. Orientation of 100 and 111 for Ge and Si are available upon request.

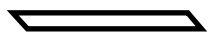
SPP (Single-Pass Parallelepiped) Plates

(lxwxt)mm	Si 45°	Si 60°	ZnS 45°	ZnS 60°	Ge 45°	Ge 60°	ZnSe 45°	ZnSe 60°	KRS-5 45°	KRS-5 60°
50x20x3	EE3131	EE3231	EI3131	EI3231	EJ3131	EJ3231	EM3131	EM3231	EU3131	EU3231
50x20x2	EE3121	EE3221	EI3121	EI3221	EJ3121	EJ3221	EM3121	EM3221	EU3121	EU3221
50x20x1	EE3111	EE3211	EI3111	EI3211	EJ3111	EJ3211	EM3111	EM3211	EU3111	EU3211
50x10x3	EE3132	EE3232	EI3132	EI3232	EJ3132	EJ3232	EM3132	EM3232	EU3132	EU3232
50x10x2	EE3122	EE3222	EI3122	EI3222	EJ3122	EJ3222	EM3122	EM3222	EU3122	EU3222
50x10x1	EE3112	EE3212	EI3112	EI3212	EJ3112	EJ3212	EM3112	EM3212	EU3112	EU3212
50x5x3	EE3133	EE3233	EI3133	EI3233	EJ3133	EJ3233	EM3133	EM3233	EU3133	EU3233
50x5x2	EE3123	EE3223	EI3123	EI3223	EJ3123	EJ3223	EM3123	EM3223	EU3123	EU3223
50x5x1	EE3113	EE3213	EI3113	EI3213	EJ3113	EJ3213	EM3113	EM3213	EU3113	EU3213
25x10x3	EE3135	EE3235	EI3135	EI3235	EJ3135	EJ3235	EM3135	EM3235	EU3135	EU3235
25x10x2	EE3125	EE3225	EI3125	EI3225	EJ3125	EJ3225	EM3125	EM3225	EU3125	EU3225
25x10x1	EE3115	EE3215	EI3115	EI3215	EJ3115	EJ3215	EM3115	EM3215	EU3115	EU3215
25x5x2	EE3126	EE3226	EI3126	EI3226	EJ3126	EJ3226	EM3126	EM3226	EU3126	EU3226
25x5x1	EE3116	EE3216	EI3116	EI3216	EJ3116	EJ3216	EM3116	EM3216	EU3116	EU3216

SPT (Single-Pass Trapezoid) Plates

(lxwxt)mm	Si 45°	Si 60°	ZnS 45°	ZnS 60°	Ge 45°	Ge 60°	ZnSe 45°	ZnSe 60°	KRS-5 45°	KRS-5 60°
50x20x2	EE2121	EE2221	EI2121	EI2221	EJ2121	EJ2221	EM2121	EM2221	EU2121	EU2221
50x20x1	EE2111	EE2211	EI2111	EI2211	EJ2111	EJ2211	EM2111	EM2211	EU2111	EU2211
50x10x3	EE2132	EE2232	EI2132	EI2132	EJ2132	EJ2232	EM2132	EM2232	EM2232	EU2132
50x10x2	EE2122	EE2222	EI2122	EI2222	EJ2122	EJ2222	EM2122	EM2222	EU2122	EU2222

SPP Microsampling Plates



(lxwxt)mm	Si 45°	Si 60°	ZnS 45°	ZnS 60°	Ge 45°	Ge 60°	ZnSe 45°	ZnSe 60°	KRS-5 45°	KRS-5 60°
10x5x1	EE 3117	EE3217	EI3117	EI3217	EJ3117	EJ3217	EM3117	EM3217	EU3117	EU3217
10x5x.5	EE 3197	EE3297	EI3197	EI3297	EJ3197	EJ3297	EM3197	EM3297	EU3197	EU3297

Other Materials, Geometries or Sizes Available on Request

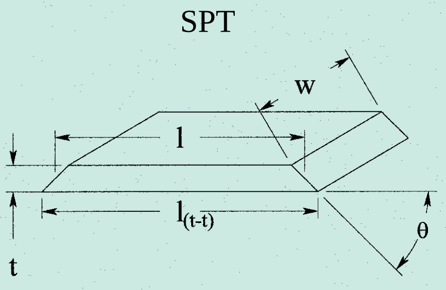
Note on IRE Dimensions

The dimension of an internal reflection element (IRE) is given by its length (l), width (w), and thickness (t). The length of the IRE, measured from the center of the entrance aperture to the center of the exit aperture, determines the number of reflections.

$$N = l/t \tan \theta$$

If l is kept constant, plates of a different thickness can be exchanged without the necessity of realignment.

Note that for l = 50mm and θ = 45°, the tip to tip length, l_{t-t} = l+t and is 52mm and 51mm, respectively, for t = 2mm and t = 1mm.



COMMONLY USED UV-VIS-IR WINDOWS

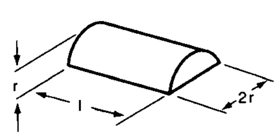
AT ONCE delivery optics are listed in black. Sizes listed in green are available upon request.

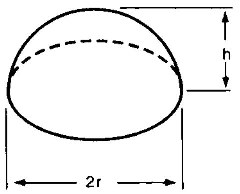
Stepped windows for ultra thin (e.g. one micron) liquid cells and wedged windows are also available. Coated windows, (e.g., KRS-5 with ZnS or ZnSe overcoat) which still have a full transmission range and have increased resistance to attack by solvents, are also available. Special sizes and materials are available upon request.

	SiO_2	Al_2O_3	Si	CaF_2	BaF_2	ZnS	Ge	NaCl	ZnSe	KBr	KRS-5
Discs	**undrilled**										
6x1mm	WAD-U11	WBD-U11	WED-U11	WFD-U11	WHD-U11	WID-U11	WJD-U11	WLD-U11	WMD-U11	WPD-U11	WUD-U11
6x6mm	—	—	WED-U61	WFD-U61	WHD-U61	WID-U61	WJD-U61	WLD-U61	WMD-U61	WPD-U61	WUD-U61
13x2mm	WAD-U12*	WBD-U12*	WED-U22	WFD-U22	WHD-U22	WID-U22	WJD-U22	WLD-U22	WMD-U22	WPD-U22	WUD-U22
13x4mm	WAD-U42	WBD-U42	WED-U42	WFD-U42	WHD-U42	WID-U42	WJD-U42		WMD-U42		
15x2mm	WAD-U13*	WBD-U13*	WED-U23	WFD-U23	WHD-U23	WID-U23	WJD-U23	WLD-U23	WMD-U23	WPD-U23	WUD-U23
19 x 2mm	WAD-U14*	WBD-U13	WED-U24	WFD-U24	WHD-U24	WID-U24	WJD-U24	WLD-U24	WMD-U24	WPD-U24	WUD-U24
25x2mm	WAD-U15*	WBD-U15*	WED-U25	WFD-U25	WHD-U25	WID-U25	WJD-U25	WLD-U25	WMD-U25	WPD-U25	WUD-U25
25x4mm	—	—	WED-U45	WFD-U45	WHD-U45	WID-U45	WJD-U45	WLD-U45	WMD-U45	WPD-U45	WUD-U45
32x3mm	WAD-U36	WBD-U36	WED-U36	WFD-U36	WHD-U36	WID-U36	WJD-U36	WLD-U36	WMD-U36	WPD-U36	WUD-U36
50x6mm	WAD-U37*	WBD-U37*	WED-U67	WFD-U67	WHD-U67	WID-U37*	WJD-U67	WLD-U67	WMD-U37*	WPD-U67	WUD-U67
Rectangles	**undrilled**										
38x19x4mm	—	—	WER-U41	WFR-U41	WHR-U41	WIR-U21*	WJR-U41	WLR-U41	WMR-U21*	WPR-U41	WUR-U41

These items can be half the stated thickness.

HEMICYLINDERS (HC) AND HEMISPHERES (HS)



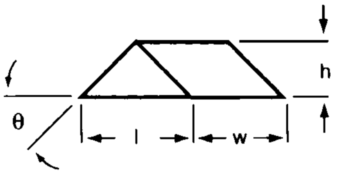


	rxl (mm) 7.5x20	rxl (mm) 12.5x25	r = h (mm) 12.5
SiO_2	EA7x89	EA7xBD	EA6xBB
Al_2O_3	EB7x89	EB7xBD	EB6xBB
Si	EE7x89	EE7xBD	EE6xBB
ZnS	EI7x89	EI7xBD	EI6xBB
Ge	EJ7x89	EJ7xBD	EJ6xBB
ZnSe	EM7x89	EM7xBD	EM6xBB
KRS-5	EU7x89	EU7xBD	EU6xBB

*Optics listed in black available for immediate delivery. Sizes listed in **green** are available upon request.*

Truncated hemicylinders and hemispheres are offered only for quartz (SiO_2) and sapphire (Al_2O_3) but not for the other materials because suitable contacting fluids of high refractive indices are unavailable. In the full hemicylinder, the height (h) and radius (r) are the same. In the truncated hemicylinder, h is less than r, and the hemicylinder becomes a full one when an OTE of thickness r-h is placed in contact with the base of the truncated hemicylinder.

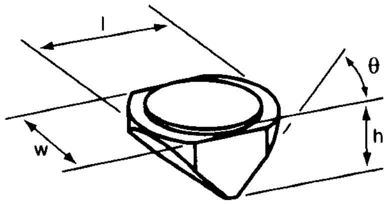
PRISMS



Single Reflection, θ = 45°

lxw	Si	ZnS	Ge	ZnSe
25x18	EE11B4	EI11B4	EJ11B4	EM11B4
25x20	EE11BN	EI11BN	EJ11BN	EM11BN

*Optics listed in black available for immediate delivery. Sizes listed in **green** are available upon request.*



OPTICAL POLISHING KIT - PART NO. OPK-1XX

Applications

- Useful for grinding, buffing and polishing many common optical materials that do not require pitch polishing. Polishing is an art; it takes expertise to obtain high quality polish.

Features

- Contains all the items needed to improve the surface finish.

Includes

- Two ground glass plates.
- Two self-adhering polishing pads.
- #400 coarse grinding compound.
- #600 fine grinding compound
- #17 very fine abrasive
- #500 polishing compound
- Instructions
- #411 KRS-5 polishing compound.
- Two camel hair brushes.

REPLACEMENT OPTICS

SPLITPEA™ ATR Holders

Material	Catalog No.
Si	UNS-ATR-E
Diamond	UNS-ATR-W
Ge	UNS-ATR-J
ZnSe	UNS-ATR-M
ZnS	UNS-ATR-I

FASTIR™ ATR Crystals

Material	Catalog No.
ZnSe	FAS-ATR-M
ZnS	FAS-ATR-I
Ge	FAS-ATR-J
Si	FAS-ATR-E

MVP™ ATR Crystals

Material	Catalog No.
ZnSe	MVP-ATR-M
ZnS	MVP-ATR-I
Ge	MVP-ATR-J

SEAGULL™ Hemispheres

Material	Catalog No.
Ge	EJ6xBB
KRS-5	EU6xBB
ZnSe	EM6xBB
ZnS	EI6xBB
Si	EE6xBB

ORIENTED SUBSTRATES

High quality substrates are available as special order items from Harrick Scientific. These are ideal for optical spectroscopy and for coating with semiconducting films. Listed below are some of the commonly used substrates with [100] orientation for cubic materials and [0001] for Al_20_3. Standard materials available are: Al_20_3, MgO, Si, CaF_2, $SrTiO_3$, ZrO_2, $LaAlO_3$, $NdGaO_3$.

Popular sizes are: Lengths: 10, 12, 15, 19, 25 mm Widths: 10, 12, 15, 19, 25 mm Thicknesses: 0.5 and 1 mm

Other materials (e.g. LiF, MgF_2, SrF_2, BaF_2, NaCl, TiO_2, crystal quartz, etc.), other sizes and orientations are also available. Wedged substrates for suppression of interface fringes are available upon request.

BICRYSTALS

Bicrystals are available as special order. The surface orientation [100] for cubic materials and [1012] for Al_2O_3.

Two sides polished upon request. Materials available are: $SrTiO_3$, MgO, Al_2O_3.

Popular sizes are: 10 x 10 x .05 mm 24 degrees 10 x 10 x .5 mm 36.8 degrees

MIRRORS

Harrick Scientific's aluminum and aluminum-coated mirrors are ideal for use in the IR-VIS-UV. These high quality mirrors have a minimum reflectivity of 85% at 200nm and 97% in the mid-IR. Other mirrors and coatings are available on request.

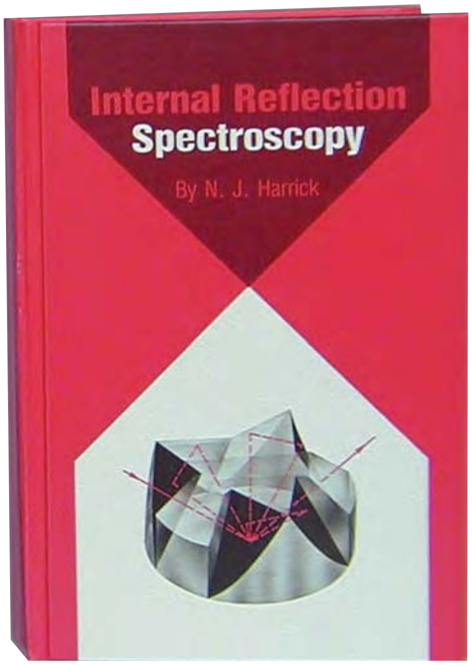
Plane Mirrors

Size (mm)	Part Number
14.3x14.3x3	MOP-111
25x32x3.2	MOP-112
25x38x3.2	MOP-113
32x50x3.2	MOP-114
50x25x3.2	MOP-115
50x38x3.2	MOP-116
50x50x3.2	MOP-117

Concave Spherical Mirrors

Radius f_1f_2(mm)	Diameter (mm)	Center Thickness (mm)	Part Number
67	50.8	6.25	MOS-514-1
132	50.8	6.25	MOS-323-1
305	63.5	6.25	MOS-234-1

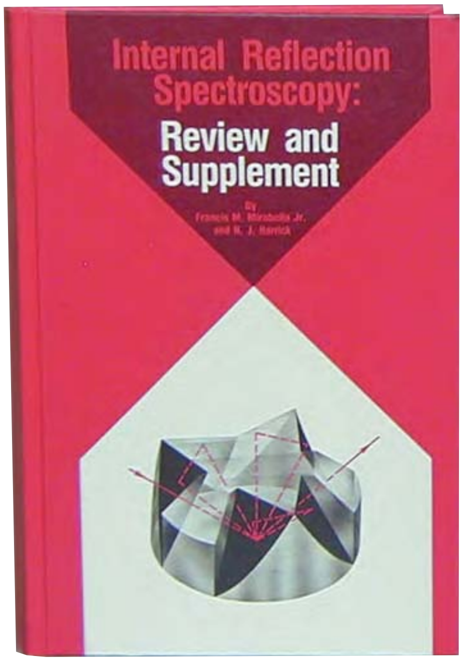
INTERNAL REFLECTION SPECTROSCOPY
by N.J. Harrick



Highlights

- The first book available devoted entirely to Internal Reflection Spectroscopy!
- Discusses the theory, instrumentation and applications of internal reflection spectroscopy.
- Provides the necessary background for the practical spectroscopist to apply the method to particular applications.
- Ò..I find Dr. HarrickÕs book a delight to read...This is an important book for the infrared spectroscopist and indeed any serious worker in practical optics will be the better for having read it...Ó
  Ñ *H. A. Willis, Trans. Faraday Soc.*

INTERNAL REFLECTION SPECTROSCOPY: REVIEW AND SUPPLEMENT
by Francis M. Mirabella, Jr. and N.J. Harrick



Highlights

- A review of internal reflection spectroscopy.
- Supplements and updates ÒInternal Reflection SpectroscopyÓ by N.J. Harrick.
- Discusses a wide range of applications of internal reflection spectroscopy.
- Provides a complete list of references to work in the field.

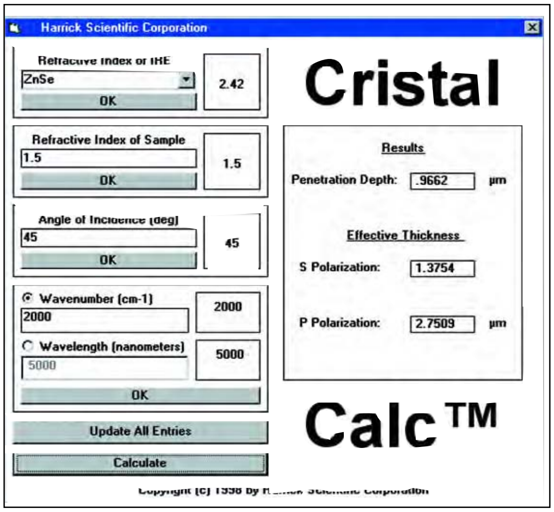
CRISTALCALC™

Features

- Ideal software for determining fundamental spectroscopic parameters for non-absorbing media and providing order of magnitude comparisons between transflectance, transmittance, and internal reflectance.
- Ascertains if internal or external reflection occurs for a given experimental configuration.
- Calculates penetration depth per wavelength, effective thickness per wavelength, polarizing angle, and critical angle for internal reflectance.
- Determines the reflectivities and transmissivities for incident radiation of parallel, perpendicular, and mixed polarizations, in addition to the transmittance through windows of the specified materials.
- Windows® '95/'98 compatible.

Includes

- N.J. Harrick's "Internal Reflection Spectroscopy" available with CCS-004



Refractive Index of IRE: ZnSe 2.42

Refractive Index of Sample: 1.5 1.5

Angle of Incidence (deg): 45 45

◉ Wavenumber (cm-1): 2000 2000

○ Wavelength (nanometers): 5000 5000

Results

Penetration Depth: .9662 µm

Effective Thickness

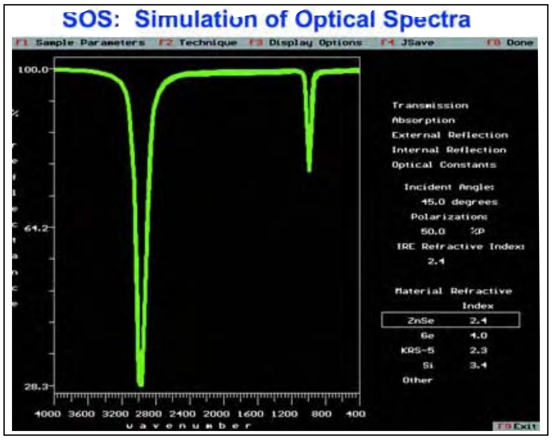
S Polarization: 1.3754

P Polarization: 2.7509 µm

SOS™: SIMULATION OF OPTICAL SPECTRA

Features

- Numerical simulation of transmission, internal reflection, and external reflection spectra of homogeneous samples and coated materials.
- Spectra can be reproduced for any incident angle, any polarization, and any sample thickness.
- Useful for determining optical constants and film thicknesses from experimental data.
- Excellent tool for selecting the best spectroscopic technique for a particular sample.
- Essential for cross-referencing spectroscopic techniques.
- IBM PC/DOS compatible.



Incident Angle:
45.0 degrees
Polarization:
50.0 %P
IRE Refractive Index:
2.4

Material	Refractive Index
ZnSe	2.4
Ge	4.0
KRS-5	2.3
Si	3.4
Other	

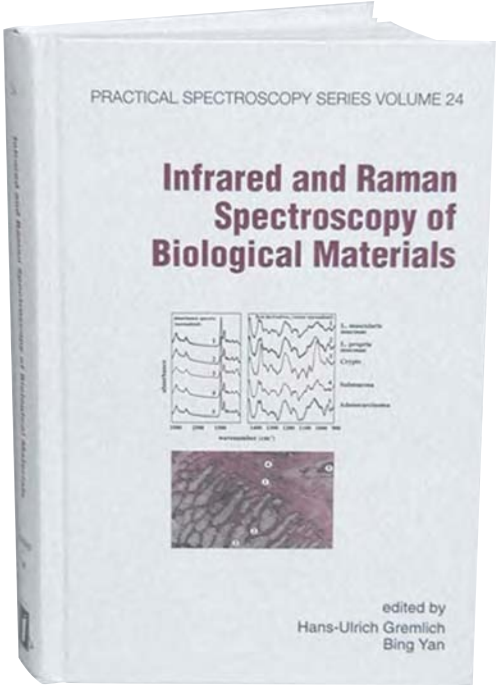
Ordering Information	Catalog No.
Internal Reflection Spectroscopy	IRS-671
Internal Reflection Spectroscopy: Review and Supplement	IRS-RS1
Optical Spectroscopy: Sampling Techniques Manual	OSP-STM
CristalCalc™:	
3.5″ Diskette (Windows® '95/'98 compatible)	CCS-003
3.5″ Diskette (Windows® '95/'98 compatible) includes N.J. Harrick "Internal Reflection Spectroscopy"	CCS-004
SOS™:	
(3.5″ Diskette)	SOS-102

Optical Spectroscopy:
Sampling Techniques Manual
Prepared by Harrick Scientific Personnel

Highlights

- ▶ Excellent introduction to and review of the six most widely used optical spectroscopy methods.
- ▶ Discusses transmission, external reflection, internal reflection, diffuse reflection, emission and photoacoustic spectroscopy.
- ▶ Presents basic principles of each method.
- ▶ Provides examples of the instrumentation required.
- ▶ Discusses advantages and disadvantages of the various techniques.
- ▶ Points to important applications.
- ▶ Includes references for further information.
- ▶ "...this is an excellent manual which could be readily used in every FT-IR laboratory..."
  — *Professor William Fately, former editor of Appl. Spectros.*

Infrared and Raman Spectroscopy of Biological Materials
Edited by Hans-Ulrich Gremlich and Bin Yang
Publisher- Marcel Dekker, Inc.



Highlights

- ▶ Supplies greatly needed chemical and structural information for identifying and characterizing biological molecules.
- ▶ Facilitates a comprehensive and thorough understanding of the latest developments in vibrational spectroscopy.
- ▶ Up-to-date accounts of the new breakthroughs in the methodologies, biological applications, and techniques for infrared, near infrared and Raman spectroscopy.
- ▶ Introduces biophysical infrared modulation for the first time.
- ▶ Discusses a variety of uses for vibrational optical activity and vibrational spectroscopic mapping and imaging; examines clinical applications of near-infrared spectroscopy as well as biological and biomedical applications of Raman spectroscopy.
- ▶ Utilizes Raman optical activity to determine multicomponent qualitative and quantitative analyses, reviews the advantages obtained by time-resolved step–scan, surface-enhanced, and imaging approaches.
- ▶ Reports on the latest software developments affecting biomedical diagnoses, such as artificial neural networks…and more.

INDEX

Visit our website,
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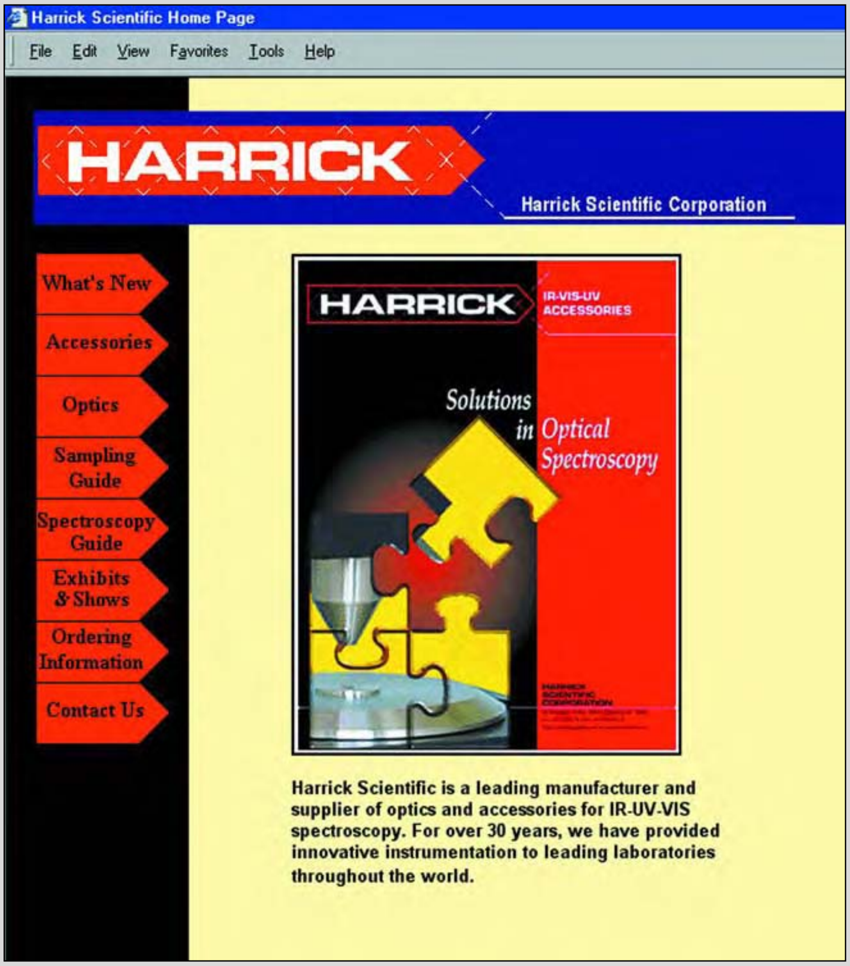

Harrick Scientific is a leading manufacturer and supplier of optics and accessories for IR-UV-VIS spectroscopy. For over 30 years, we have provided innovative instrumentation to leading laboratories throughout the world.

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Optical Material Chart
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Harrick Scientific's accessories are compatible with most spectrometers including:
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PerkinElmer Instruments Spectrum One™ with I button recognition.
Excalibur™ through Bio-Rad Instruments.

Spectrometer Code Chart for Ordering

HARRICK

FTIR SPECTROMETERS

ABB BOMEM

DA 8	BO2
MB Series	BO3
DA 3	BO4
Michelson 100 Arid Zone, FTLA 2000	BO5

ANALECT/ORBITAL

Diamond	AN6

BRUKER

113V	BR2
22, 28, 45, 48, 55, 66, 88	BR3
Quick Lock, Vector 33, Vertex, Tensor	BR4
66V	BR5

DIGILAB/BIORAD

FTS 7, 40, 50, 60, Qualimatic	DI4
375, 575, 675	DI5
155, 165, 175, 185	DI6
Excalibur w/ ART	DI7
6000, Excalibur w/o A.R.T., Scimilar, 3100, 4000, 7000, 600	DI8

HORIBA

7000 Series	HO1

JASCO

400, 600V	JA1
3000E	JA2
600, 4000, 6000	JA3

JEOL

Winspec 100	JE1

MIDAC

All Models	MI1

PERKIN ELMER

1700 Series	PE4
1600 Series, Paragon 1000, RX, BX	PE8
2000, Spectrum 2000, GX	PE9
Spectrum 1, Spectrum 100	P11

SHIMADZU

8100-8700	SH2

THERMO-MATTSON

Polaris	MA1
Galaxy, RS Infinity	MA2
Genesis, Satellite	MA3

THERMO-NICOLET

5, 20, 500. 700, 800 Impact, Magna	NI6
205, 210	NI7
Avatar, Nexus	N18

UV-VIS SPECTROMETERS

BECKMAN

DU6, DU7, DU-70	BE5
DU20, 30, 40, 50, 60	BE6
DU600, 700	BE7

HEWLETT PACKARD

8452A	HP1
8453	HP2

PERKIN ELMER

320, 330	PE6
Lambda Series	PE7

SHIMADZU

UV2101, 3101	SH1

THERMO

Evolution, 300, 600	N19

VARIAN

DMS Series, Cary 1, 3	VA1
Cary 4, 5, 400, 500, 2200, 2300, 2400,	VA2

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E- info@madisonir.com
www.madisonir.com

REFLEX ANALYTICAL
639 ALBERT PLACE
RIDGEWOOD, NJ 07450
TONY JACOBINI
T-(201)444-8958
F-(201)670-6737
E-reflexusa@att.net
www.reflexusa.com

S.A.F.I.R.
ESSENDRIES II
B-2870 PUURS
BELGIUM
DIRK VAN DEN BOSSCHE
T-32-3-8894771
F-32-3-8993965
E- safir@glo.be

SANYO TRADING CO. LTD.
11, 2-CHOME KANADANISHIKI-CHO
CHIYODA-KU, TOKYO, 101 JAPAN
T-81-3-32335844
F-81-3-32335945
E-y-matsu@sanyo-trading.co.jp

SMARTEAM SCIENTIFIC
9F 274 SEC 1, HSIANG HSANG S. RD
TAICHUNG, TAIWAN
T-886-2-43847828
F-886-2-43847829
E-smarteam@tcts.seed.net.tw
E-smartech@ms2.hinet.net

STARNA GMBH WIEN
P.O. BOX 9
A-3403 KLOSTERNEUBURG, AUSTRIA
DR. PETER SCHUSTER
T-43-3335-4850
F-43-3335-4851

WEIKE SPECTRAL DEVICES & TECHNOLOGIES,INC Republic of China
SHUDU MANSION, SUITE 1808
CHENGDU, SICHUAN 610016
CHINA
MR. CHONG WANG
86-28-6753888-51808
86-28-6749782
harrickchina@yahoo.com.cn

HARRICK

HARRICK SCIENTIFIC PRODUCTS, INC.

141 Tompkins Ave. • Box 277 • Pleasantville, NY 10562 • 914 / 747-7202 • FAX: 914 / 747-7209 • Email: info@harrrick.com • www.harricksci.com

AccessATR™

The AccessATR™ makes ATR FTIR spectroscopy accessible to everyone. It is perfect for quick and accurate ATR measurements of most samples and it is affordable. Simply place the sample – a solid, liquid, or paste - on the ATR crystal, record the spectrum and wipe the crystal clean. The AccessATR™ is equipped with a built-in trough to contain liquids and a press for solid sampling.

APPLICATIONS

- Convenient for routine analysis of liquids, pastes, and flexible solids.
- Single reflection ATR (internal reflectance) accessory.
- Excellent for university laboratories, teaching facilities and the cost-conscious.
- Designed for highly repeatable qualitative and quantitative FTIR measurements.

FEATURES

- Convenient, horizontal sampling surface.
- Top loading.
- Fixed 45° incident angle.
- Spectral range: 20,000 cm^{-1} to 550 cm^{-1}.
- Easy to use – no alignment required.
- Excellent throughput.
- Little or no sample preparation required.
- Gasket-sealed ATR crystal preserves sample integrity.
- Readily replaceable affordable ZnSe ATR crystal.
- Convenient slide plate mounting fits all FTIR spectrometers.

INCLUDES

- Mounted ZnSe prism.
- Integral trough for analyzing liquids, powders and pastes.
- Built-in pressure applicator for solid sampling.
- Slide plate mounting.

ORDERING INFORMATION

	CATALOG NO.
AccessATR™	ACC-ATR
OPTIONS	
Sample Slide Plate Holder	HSS-XXX*
Rail Plate	HRM-XXX*
REPLACEMENT PARTS	
ZnSe ATR Crystal	ACC-PRM
Gasket	ACC-GSK

**XXX indicates spectrometer make and model*

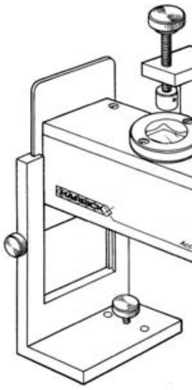


Figure 1. Access ATR™ inserted in a Slide Plate Holder.

AccessATR™ is an affordable single reflection, horizontal internal reflection (ATR) accessory. Its simple optical design and straightforward use make it a perfect introductory ATR accessory. The AccessATR™ is excellent for repeatable qualitative and quantitative studies of a wide range of samples. These include aqueous and organic liquids, pastes, and organic and inorganic powders.

ATR spectroscopy is the method of choice for many samples. ATR requires little or no sample preparation and is a much quicker method for analyzing all types of samples. For affordable analysis of solids and powders, the sample is simply placed in the trough and pressed against the ATR crystal with the built-in press. No KBr pellets or nujol mulls are required. For liquids or pastes, just smear or drip a small amount of sample on the top of the ATR crystal and record the spectrum. Avoid the typical problems with transmission liquid cells - interference fringes, peak saturation, and squeezing viscous samples into short pathlengths. The AccessATR™ produces excellent spectral quality every time.

AccessATR™ uses two planar mirrors to direct the beam to and from the ATR crystal at a 45° incident angle. The ATR crystal is made from ZnSe which has a wide useful spectral range (20,000 cm^{-1} to 550 cm^{-1}) and durable mechanical properties. The crystal is held in place with a gasket, making it straightforward to replace the crystal when needed. This design is reasonably insensitive to precise alignment. The accessory just slides into the sample slide plate holder supplied with the FTIR spectrometer and no further alignment is necessary.

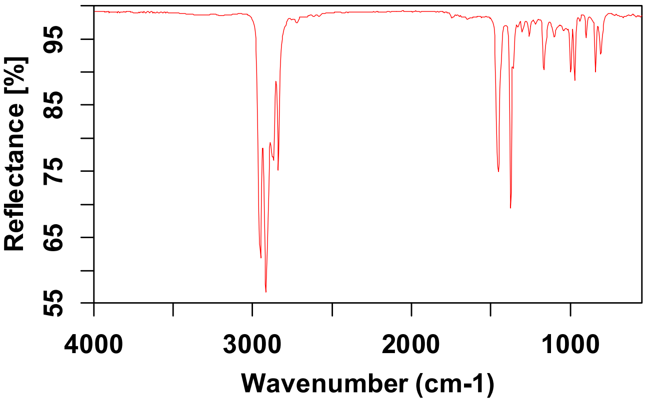


Figure 2. ATR Spectrum of a Twix Wrapper.

Figures 2 through 4 show spectra from different portions of a Twix™ (Mars, Inc.) candy bar, illustrating its use with solids and pastes. Figure 5 shows the spectrum of two liquids, rubbing alcohol and water, clearly showing that water is one of the components of rubbing alcohol. Subtraction, using commercial spectrometer software, results in a spectrum that is clearly identifiable as isopropanol. Note that, despite the strong absorbance of these samples, the ATR spectra are free from saturation effects.

AccessATR™ offers simplicity, economy, and versatility for all FTIR laboratories, enabling quick and accurate measurements for a wide range of samples.

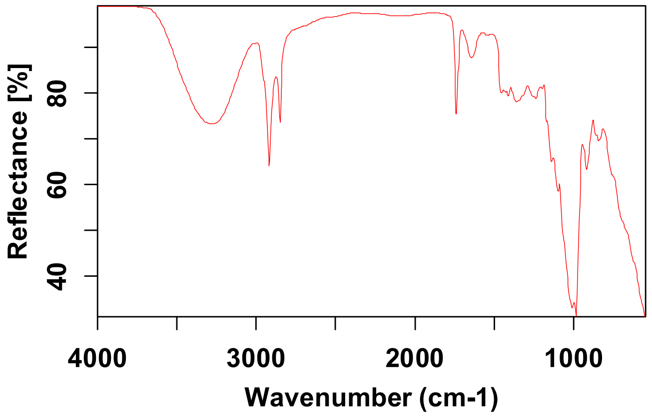


Figure 3. ATR Spectrum of the Twix Caramel.

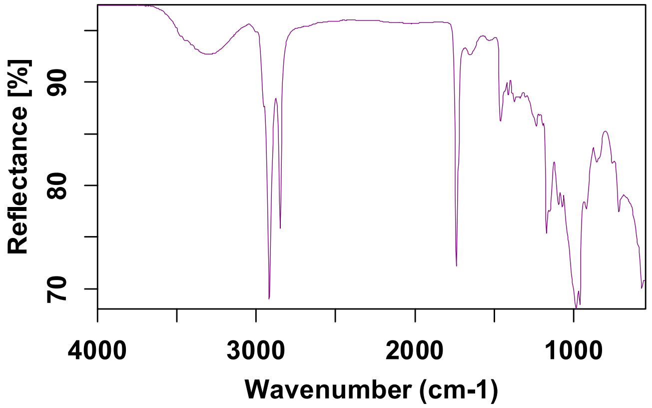


Figure 4. ATR Spectrum from a Twix Wafer.

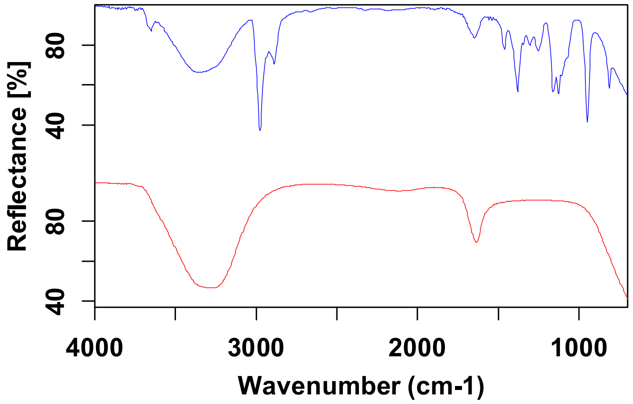


Figure 5. ATR Spectrum of Rubbing Alcohol (upper) and Water (lower).

AUTOMATIC TEMPERATURE CONTROLLER

The Automatic Temperature Controller is a full-featured tabletop controller designed for a variety of applications. It can be manually programmed to reach a desired temperature or computer controlled for sophisticated temperature profiling. The temperature controller automatically determines the best settings for optimal heating based on the thermal response of a particular system. It features two temperature sensor inputs and one low-voltage output, allowing regular or cascade operation. Cascade operation permits the output to be regulated based on two inputs from different sensors. For spectroscopy accessories with separate heater and sample thermocouples, one set point controls the sample temperature while another provides an emergency shut off at another set point. This is particularly important for high temperature applications, where the desired sample temperature is close to the upper temperature limit of the heater.

FEATURES

- Provides accurate temperature regulation over a wide range of temperatures.
- Set point programmable from -200 to 1250°C for K-type thermocouples.
- Accepts a wide variety of thermocouples and resistive temperature detectors (RTDs).
- Cascade or regular operation.
- Digital temperature readout in degrees Fahrenheit or Celsius.
- Provides a safe, low-voltage output.
- Intended for use with 24V heaters.
- Manually programmable using the integrated keypad.
- Computer programmable through an RS232 interface.

ORDERING INFORMATION

	CATALOG NO.
Automatic Temperature Controller	
110Vinput, 24V output	ATC-024-1
220/240Vinput, 24V output	ATC-024-2

THE CONCENTRATIR™

The ConcentratIR™ is a horizontal multiple internal reflection (ATR) accessory, designed for micro-liquid samples. The ConcentratIR™ was developed specifically for use in quality control environments. It is a durable, compact, horizontal ATR micro-sampler that utilizes a Si internal reflection element. The infrared beam interacts with sample seven times within a 4-mm diameter area. This concentrates the sensitivity of a regular horizontal ATR into a much smaller area and hence requires only minute quantities of sample per measurement. The ConcentratIR™ makes internal reflection micro-sampling simpler and more straightforward than ever before. For rapid sample exchange without interrupting the purge of the spectrometer, the ConcentratIR™ integrates Harrick's exclusive PermaPurge™*.

APPLICATIONS

- Invaluable for studying minute samples of liquids and pastes.
- Uniquely suited to quality control environments.

FEATURES

- Durable and rugged internal reflection micro-sampler.
- High sample throughput due to little or no sample preparation.
- Quick and easy clean up between samples.
- Easily accessible sampling area for rapid sample exchange.
- Seven reflections in a 4-mm diameter.
- Highly inert and durable Si ATR crystal.
- Harrick's exclusive PermaPurge™ for rapid sample and crystal exchange without interrupting the purge of the system.
- Pre-aligned for easy start-up.
- Optional upgrade available for grazing angle specular reflectance.

INCLUDES

- Mating hardware for the specified spectrometer.

[1] U. S. Patent No. 5,177,561

ORDERING INFORMATION

	CATALOG NO.
ConcentratIR™	UQC-XXX
REPLACEMENT PARTS	
Replacement Sampling Plate	UQC-LSP
Grazing Angle Specular Reflectance Upgrade to the Focus	FSC-TOP

THE UPWARD-LOOKING CRICKET™

Harrick Scientific's Upward-Looking Cricket™ is excellent for routine measurements of cohesive solid samples in quality control environments and research laboratories. Its design follows the long-standing Harrick tradition of *true* diffuse reflectance accessories. Like its more versatile Praying Mantis cousin, the Cricket™ optimizes the collection of the diffusely reflected light while eliminating the specular component. With a flip of a switch, the Cricket™ converts from this true diffuse reflectance mode to a fixed-angle specular reflectance device. In this configuration, the Cricket™ collects 60° specular or in-line 'diffuse' reflectance. The Cricket™ is available in a downward sampling configuration for powder analysis. Both models of the Cricket™ feature PermaPurge™ for rapid sample and crystal exchange without interrupting the purge of the spectrometer.

APPLICATIONS

- Ideal for routine diffuse reflectance measurements of powders and solid samples.
- Excellent for quality control.

FEATURES

- Diffuse and specular reflection capabilities provide application versatility.
- Eliminates the collection of the specular component in the diffuse reflectance mode.
- Provides a fixed 60° incident angle in the specular reflectance mode.
- Easy to use.
- High sample throughput.
- Low cost.
- Prealigned for quick start-up.
- Harrick’s exclusive PermaPurge™ allows rapid sample and crystal exchange without interrupting the purge of the system.
- Two models available:
  - Downward looking Cricket for examining powders and small cohesive solids.
  - Upward looking Inverted Cricket for cohesive solid samples of virtually unlimited sample size.
- Compatible with most FT-IR and UV-VIS spectrometers.

INCLUDES

- Three position sample holder.
- Specular Reflectance Reference.
- Diffuse Reflectance Reference.
- Mating hardware for the specified spectrometer.

ORDERING INFORMATION.

	CATALOG NO.
Cricket	CRK-D-XXX
Inverted Cricket	CRK-U-XXX
Replacement Sample Holder	CRK-SAM
Replacement Specular Reflectance Reference	CRK-SPR
Replacement Diffuse Reflectance Reference	CRK-DIF

The Cricket™ is an innovative reflectance accessory, featuring both true diffuse reflectance capability and 60° fixed-angle specular or in-line diffuse reflectance data collection. This is a powerful combination for collecting spectra from a wide variety of solid samples.

In its diffuse reflectance configuration, the Cricket™ optically eliminates the specularly reflected radiation and the reststrahlen bands associated therewith. With a flip of a switch, the Cricket™ converts to its specular configuation, where it illuminates the sample at a 60° incident angle and collects the radiation reflected on the same axis.

Figure 1 shows a diffuse reflectance spectrum of a powder diluted in KBr, as is commonly done to achieve diffuse reflectance spectra without saturated bands. This spectrum was recorded relative to a KBr background.

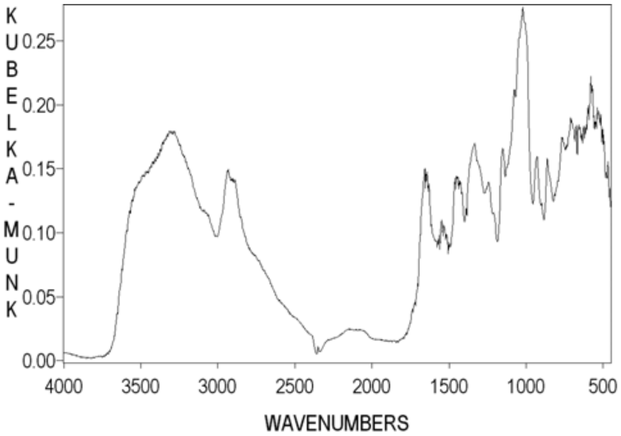


Figure 1. Diffuse Reflectance from Rye Flour Diluted in KBr.

Unfortunately, when recording true diffuse reflectance measurements, the signal levels are extremely low. This means an increased noise level. The Cricket™ allows a given sample to be examined by both true diffuse reflectance and 60° specular reflectance (in line diffuse reflectance) to determine which method is more appropriate.

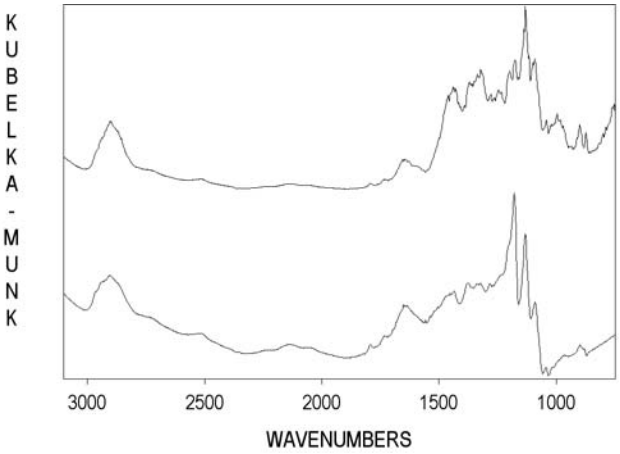


Figure 2. Diffuse (top) and Specular (bottom) Reflectance Spectra from a Yellow 3M Post-It.

Figure 2 shows the reflectance of paper, recorded in both the specular and diffuse mode of the Cricket™. Note that the sharp peak at 1132 cm^{-1} in the diffuse reflectance spectrum (upper curve) appears as a doublet in the specular reflectance spectrum (lower curve). This doublet is due to restrahlen effects. In addition, the structure of the bands around 1000 cm^{-1} is lost. Hence this sample would be better examined by diffuse reflectance, despite the smaller signal-to-noise ratio.

The Cricket™ is a powerful tool both for examining rough-surfaced solid samples and powder and for determining the best method of analysis.

THE CRICKET™

Harrick Scientific's Cricket™ is excellent for routine measurements of solid samples in quality control environments and research laboratories. Its design follows the long-standing Harrick tradition of *true* diffuse reflectance accessories. Like its more versatile Praying Mantis cousin, the Cricket™ optimizes the collection of the diffusely reflected light while eliminating the specular component. With a flip of a switch, the Cricket™ converts from this true diffuse reflectance mode to a fixed-angle specular reflectance device. In this configuration, the Cricket™ collects 60° specular or in-line 'diffuse' reflectance. The Cricket™ is available in a downward sampling configuration for powder analysis. Both models of the Cricket™ feature PermaPurge™ for rapid sample and crystal exchange without interrupting the purge of the spectrometer.

APPLICATIONS

- Ideal for routine diffuse reflectance measurements of powders and solid samples.
- Excellent for quality control.

FEATURES

- Diffuse and specular reflection capabilities provide application versatility.
- Eliminates the collection of the specular component in the diffuse reflectance mode.
- Provides a fixed 60° incident angle in the specular reflectance mode.
- Easy to use.
- High sample throughput.
- Low cost.
- Prealigned for quick start-up.
- Harrick's exclusive PermaPurge™ allows rapid sample and crystal exchange without interrupting the purge of the system.
- Two models available:
    - Downward looking Cricket for examining powders and small cohesive solids.
    - Upward looking Inverted Cricket for cohesive solid samples of virtually unlimited sample size.
- Compatible with most FT-IR and UV-VIS spectrometers.

INCLUDES

- Three position sample holder.
- Specular Reflectance Reference.
- Diffuse Reflectance Reference.
- Mating hardware for the specified spectrometer.

ORDERING INFORMATION.

	CATALOG NO.
Cricket	CRK-D-XXX
Inverted Cricket	CRK-U-XXX
Replacement Sample Holder	CRK-SAM
Replacement Specular Reflectance Reference	CRK-SPR
Replacement Diffuse Reflectance Reference	CRK-DIF

DA VINCI ARM

The da Vinci Arm is a unique articulated opto-mechanical FTIR accessory designed for analyzing samples that are too large to fit into the sample compartment of a spectrometer. The da Vinci Arm enables the analyses of samples by specular, diffuse and ATR reflection techniques. Since the da Vinci Arm is articulated, it simplifies analysis of samples in front of, below, or above the spectrometer sample compartment. The sampling surface can be facing in any direction – up, down, or sideways. Samples such as paintings, vases, sculptures, etc. can now be easily analyzed. The integral camera provides for magnified viewing and image capture of the sampled spot. The entire optical path of the IR beam is enclosed and integrated into the purge of the host spectrometer.

APPLICATIONS

- Qualitative and quantitative analysis of samples that are too large to fit within the FTIR spectrometer sample compartment.
- Analysis of neat samples or small spots on large samples via diffuse reflectance, external reflectance or ATR spectroscopy.

FEATURES

- Fixed 45º incident angle.
- Sampling head configurable for diffuse and specular reflectance.
- Optional diamond ATR sampling head available.
- Allows both contact and non-contact analysis, depending on the sample.
- Enables analysis outside the sample compartment.
- Articulated opto-mechanical system has:
  - 3.5” of horizontal motion
  - 5.8” of vertical motion
  - 180º of tilt
  - Fine focus adjustment
- Small sampling spot size:
  - 500µm diameter for ATR.
  - 1000 µm diameter for specular and diffuse reflectance.
- Small spot size allows analysis with high spatial resolution.
- Spectral range: 45,000 to 100 cm^{-1}.
- Video imaging system:
  - Magnifies the image for easy viewing.
  - Directly views the sample surface.
  - Integrates with your computer for real-time viewing and long-term storage of sampled images.
- Suitable for use in most FT-IR spectrometers.

INCLUDES

- Mating hardware for the specified FTIR spectrometer.
- Alignment mirror.
- USB adapter and software for video image capturing, compatible with Windows 98/350MHz or higher computers.

ORDERING INFORMATION

	CATALOG NO.
da Vinci Arm	DAV-XXX
	XXX denotes the spectrometer code

OPTIONS

Diamond ATR	DAV-ATR-W

The daVinci Arm is a high performance accessory designed for spectroscopic analysis of samples outside of the box. Many samples are too large to fit within the confines of the sample compartment. The daVinci Arm is articulated so that it reaches out of the sample compartment to measure samples facing virtually any direction.

The daVinci has two modes of operation: ATR and in-line diffuse reflectance. Both modes utilize all front-surface mirrors to direct the beam to and from the sample at a 45º incident angle.

Its ATR mode utilizes a monolithic diamond ATR crystal and the moveable sampling head is used to apply the pressure required to obtain good contact between the ATR crystal and the sample. The video-imaging system views through the ATR crystal for easy visual confirmation of the wetting of the crystal that occurs when good contact is obtained and also for photographic documentation of the sampling area.

The diffuse reflectance mode allows non-contact measurements of a variety of samples. This mode collects both the diffusely and specularly reflected components reflected from the sample and the video-imaging system can be used as a aid in sample alignment and for photographic documentation.

Although primarily intended for analysis of large solid samples, the daVinci Arm can also be used to examine powders, pastes, gels and liquids. Figures 1 through 3 show sample spectra recorded with the daVinci Arm. Figure 1 are spectra recorded from two locations on the vents on an in-tact automobile dashboard. The vent itself is shown in to the right. There are small spectra differences between the two locations on the vent. Figures 2 and 3 show spectra of paper and ink thereon, as measured by diffuse reflectance and ATR spectroscopy respectively. Both sets of spectra show differences between the ink-coated paper and the paper itself.

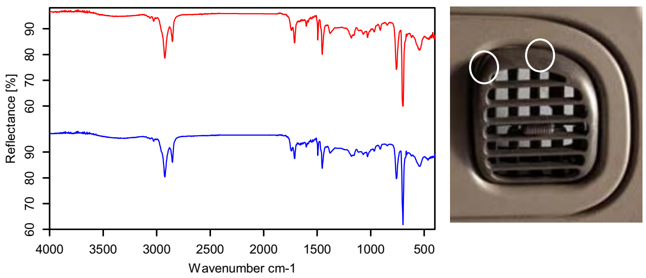


Figure 1. ATR spectra (right) of an automobile dashboard vent (left), measured from two areas.

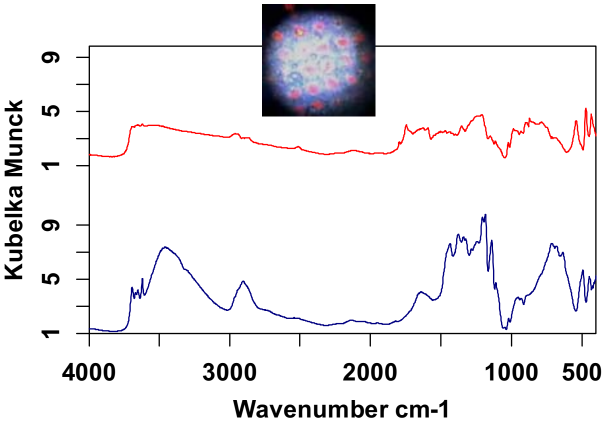


Figure 2. Diffuse reflectance of colored ink on 1940s paper (top) and the paper itself (bottom).

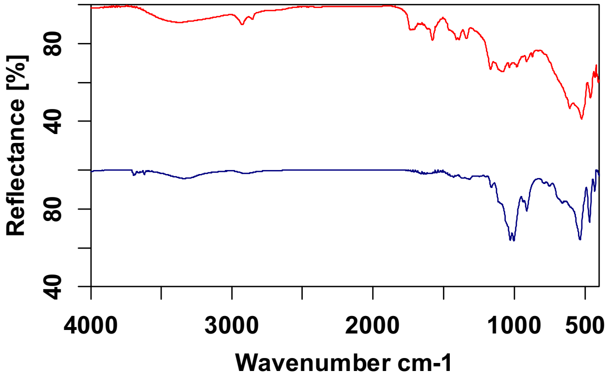


Figure 3. ATR spectra of 1940s ink on paper (top) and the paper itself (bottom).

DLC 2™

These affordable Demountable Liquid Cells are excellent for examining mulls, injected liquids and flowing liquids. These cells combine our time-tested leak-free design with an all-PTFE cell body for superior chemical resistance. The Luer Lok fitting model is perfect for examining mulls and injected liquids under static conditions. With the flangeless fittings models, it is simple and straightforward to flow liquids through the cell for applications such as oil analysis. Both cells feature easily variable pathlengths.

APPLICATIONS

- Ideal for infrared measurements of liquids, mulls, and smears.
- Excellent for process control and laboratory applications.

FEATURES

- Variable pathlength from 25µm to 500mm.
- Leak-free design.
- Utilizes standard, non-drilled windows.
- Disassembles easily for cleaning and window replacement.
- Two ports for static or flow applications.
- Sample slide plate mounted for user convenience.
- Chemically resistant PTFE cell body and fittings.
- Choice of:
  - Static cell with Luer Lok fittings.
  - Flow cell with flangeless fittings for 1/8" or 1/16" tubing. Operable to 200 psi with the appropriate windows.
- Open aperture: 8mm.
- Optional spacers available to extend the pathlength range from 6µm to 1mm.

INCLUDES

- PTFE cell with Luer Lok or flangeless fittings.
- Viton o-rings.
- Slide plate mounting.
- Six PTFE pathlength spacers: 25, 56, 100, 150, 250 and 500 µm.
- Windows need to be ordered separately (see table).

Windows	13x2 mm
Al_2O_3	WBD-U22
CaF_2	WFD-U22
ZnS	WID-U22
NaCl	WLD-U22
ZnSe	WMD-U22
KBr	WPD-U22

Spacer Sets (10 per package)			
Assorted (two each of 25, 50, 100, 200 and 1000 µm)	PSB-M13	50µm	MSB-050-M13
		100µm*	MSB-100-M13
		200µm	MSB-200-M13
15µm	MSB-015-M13	500µm*	MSB-500-M13
25µm	MSB-025-M13	1000µm	MSB-000-M13
Spacer Sets (12 per package)			
Assorted (one each of 6, 12, 25, 56, 100, 150, 250, 390, 500, 630, 750 and 950 µm)	PSA-M13	150µm	MSP-150-M13
		250µm	MSP-250-M13
		390µm	MSP-390-M13
		630µm	MSP-630-M13
6µm	MSP-006-M13	750µm	MSP-750-M13
12µm	MSP-012-M13	950µm	MSP-950-M13
56µm	MSP-056-M13	*Also available in 12 pks	

ORDERING INFORMATION	CATALOG NO.
DLC 2™	DLC-L13
DLC 2™ Flow Cell (1/16" tubing)	DLC-F13-062
DLC 2™ Flow Cell (1/8" tubing)	DLC-F13-125

OPTIONS AND REPLACEMENT PARTS

Viton O-Ring	ORV-012
Kalrez O-Ring	ORK-012

Demountable Liquid Cells

These leak-free cells are the only Demountable Liquid Cells that you will ever need. Made from chemically resistant 316 stainless steel with easily changed pathlengths, these cells are perfect for analyzing a wide variety of liquids and mulls under static or flow conditions.

APPLICATIONS

- Infrared measurements of liquids, mulls, and smears.
- Process control and laboratory applications.
- Static or flow environments.

FEATURES

- Variable pathlength, from 6µm to 1mm.
- Leak-free design utilizes standard, non-drilled windows.
- Easily disassembled for cleaning and window replacement.
- Two ports for static or flow applications.
- Sample slide plate mounted for user convenience.
- Chemically resistant 316 stainless steel cell body with Luer Lok or 1/8" Swagelok™ fittings.
- Available with an 8mm or 20mm diameter clear aperture.
- Maximum operating pressure with ZnSe windows
  - 660 psi for the DLC-S13 (8 mm open aperture).
  - 100 psi for the DLC-S25 (20 mm open aperture).
- Optional chemically-resistant Kalrez o-rings available.

INCLUDES

- Stainless steel cell body with Luer Lok or Swagelok™ fittings.
- One dozen Teflon spacers. One each for the following pathlengths: 6, 12, 25, 56, 100, 150, 250, 390, 500, 630, 750, and 950 µm.
- Viton o-rings.
- Slide plate mounting.
- Windows need to be ordered separately (see table).

Windows	13x2 mm	25x2 mm
Al_2O_3	WBD-U22	--
CaF_2	WFD-U22	WFD-U25
ZnS	WID-U22	WID-U25
NaCl	WLD-U22	WLD-U25
ZnSe	WMD-U22	WMD-U25
KBr	WPD-U22	WPD-U25

Assorted Spacers Sets		
	8 mm apt. (13x 2 mm)	**20 mm apt. (25 x 2 mm)**
Two each (25, 50, 100, 200 and 1000 µm)	PSB-M13	PSB-M25
One each (6, 12, 25, 56, 100, 150, 250, 390, 500, 630, 750 and 950 µm)	PSA-M13	PSA-M25

	8 mm apt. (13x 2 mm)	20 mm apt. (25 x 2 mm)
Spacers Sets (10 per package)		
15µm	MSB-015-M13	MSB-015-M25
25µm	MSB-025-M13	MSB-025-M25
50µm	MSB-050-M13	MSB-050-M25
100µm	MSB-100-M13	MSB-100-M25
200µm	MSB-200-M13	MSB-200-M25
500µm	MSB-500-M13	MSB-500-M25
1000µm	MSB-000-M13	MSB-000-M25
Spacers Sets (12 per package)		
6µ	MSP-006-M13	MSP-006-M25
12µm	MSP-012-M13	MSP-012-M25
56µm	MSP-056-M13	MSP-056-M25
150µm	MSP-150-M13	MSP-150-M25
250µm	MSP-250-M13	MSP-250-M25
390µm	MSP-390-M13	MSP-390-M25
630µm	MSP-630-M13	MSP-630-M25
750µm	MSP-750-M13	MSP-750-M25
950µm	MSP-950-M13	MSP-950-M25

ORDERING INFORMATION

CATALOG NO.

Demountable Liquid Cell	8 mm aperture (13x2 mm windows)	20 mm aperture (25x2 mm windows)
Luer Lok Fittings (static)	DLC-M13	DLC -M25
Swagelok™ Fittings (flow)	DLC -S13	DLC -S25

OPTIONS AND REPLACEMENT PARTS

Viton O-Ring	ORV-012	ORV-020
Kalrez O-Ring	ORK-012	ORK-020

HARRICK

DIFFUSE REFLECTANCE KIT

This Diffuse Reflectance Kit is a special offer, packaging our premier Praying Mantis with its High Temperature Reaction Chamber, Temperature Controller and all the sampling tools required. The Praying Mantis was the first generally available diffuse reflection attachment and remains the forerunner in the field. Its unique and highly efficient optical system deflects specular reflectance away from the collecting ellipsoid, minimizing spectral distortions. With its Reaction Chamber and Temperature Controller, the Praying Mantis is ideal for examining samples in low to ambient pressures and at high temperatures. Upgradeable for high pressure operation.

APPLICATIONS

- Straightforward and reliable diffuse reflection analysis of solids and powders.
- Analysis of catalysts and other powders in a temperature and/or pressure controlled environments.

FEATURES

- Praying Mantis Diffuse Reflectance Accessory
  - Highly efficient collection system.
  - Minimal collection of the specular component.
  - Easy access to the sampling area.
  - Straightforward attachment of reaction chambers.
  - Includes PermaPurge™ for rapid exchange with minimal interruption of the purge.
- High Temperature, Low Pressure Reaction Chamber
  - Designed for operation from high vacuum (10^{-6} torr) to two or three atmospheres and at high temperatures (up to 600°C under vacuum).
  - Readily adapted for high-pressure operation up to 500 psi with a High Pressure Dome.
  - Three inlet/outlet ports provided for evacuating the cell and introducing gases.
  - Made from chemically resistant 316 stainless steel.
- Automatic Temperature Controller
  - Accurate temperature regulation from -200 to 1250°C for K-type thermocouples.
  - Cascade or regular operation.
  - Programmable through the keypad or the supplied RS232 port.
  - Digital temperature readout in degrees Fahrenheit or Celsius.
- Sampling Tools for a quick start.

INCLUDES

- Diffuse Reflectance accessory with alignment fixture, alignment post, 10mm and 3mm sampling cups, funnel and mating hardware for the specified spectrometer.
- Reaction Chamber with K-type thermocouple, two KBr or UV quartz windows, a glass observation window and a screen set.
- Automatic Temperature Controller.
- Sampling Tools: mortar and pestle, spatula, KBr powder and finger cots.

ORDERING INFORMATION

ORDERING INFORMATION	CATALOG NO.
Praying Mantis Kit, 110V	DRK-3-XXX*
Praying Mantis Kit, 220/240V	DRK-4-XXX*

*XXX indicates spectrometer make and model; compatible with a wide range of IR-UV-VIS spectrometers.

OPTIONS

OPTIONS		
KBr powder, 100g (FTIR reference and dilution material)		KBR-100
Spectralon® UV-VIS Reference Disk for the Praying Mantis		DRP-SPR
Vacuum Pump, 110V		VPE-001
Vacuum Pump, 220/240V		VPE-002
	FT-IR	*UV-VIS*
Ambient Sample Chamber	DRP-ASC (KBr)	DRP-ASC-VUV(SiO_2)
High Pressure Dome for the HVC Reaction Chambers (Max. Pressure: 500 psi)	HVC-DWI-3 (ZnS)	HVC-DWA-3 (SiO_2)
Low Temperature, Low Pressure Reaction Chamber, 24 V	CHC-CHA-3 (KBr)	CHC-VUV-3 (SiO_2)

Spectralon® is a registered trademark of Labsphere.

Harrick Scientific Products, Inc.
141 Tompkins Ave, 2nd Floor, Pleasantville NY 10570
Ph: 800-248-3847 or 914-747-7202, FAX: 914-747-7209, web site: www.harricksci.com, e-mail: info@harricksci.com

THE PRAYING MANTIS

The Praying Mantis was the first generally available diffuse reflection attachment and remains the forerunner in the field. It incorporates two 6:1, 90° off-axis ellipsoids that form a highly efficient diffuse reflection illumination and collection system. This unique configuration deflects the specular reflectance away from the collecting ellipsoid, minimizing the associated spectral distortions. It also can be configured to study materials and reactions in controlled environments with the appropriate reaction chamber.

APPLICATIONS

- For easy and reliable diffuse reflection analysis of solids and powders.
- Analysis of catalysts and other powders in a temperature and/or pressure controlled environment.

FEATURES

- Highly efficient collection system.
- Minimizes the detection of the specular component.
- Ellipsoids pivot to provide easy access to the sampling area.
- Allows easy attachment of reaction chambers.
- Several models offered for compatibility with a wide range of IR-UV-VIS spectrometers.
- Harrick's exclusive PermaPurge™ allows rapid exchange with minimal interruption of the system purge.
- Optional references for FTIR and UV-VIS studies.

INCLUDES

- Cart with two mounted alignment mirrors.
- Alignment post.
- Sample cart.
- Two sampling cups: 10mm adjustable height and 3mm adjustable microsampling.
- Funnel.
- Mating hardware for the specified spectrometer.

ORDERING INFORMATION

	CATALOG NO.
Praying Mantis Diffuse Reflectance Accessory	DRP-XXX
Praying Mantis Kit, 110V (includes HVC Chamber, Temperature Controller and sampling tools)	DRK-3-XXX
Praying Mantis Kit, 220V (includes HVC Chamber, Temperature Controller and sampling tools)	DRK-4-XXX

OPTIONS & REPLACEMENT PARTS

Alignment Fixture	DRP-ALN
Sampling Accessory Kit	DRP-SAP
Micro-sampling Cup	DRP-SX3
Sampling Cup	DRP-S10
KBr powder, 100g (FTIR reference and dilution material)	KBR-100
Spectralon® UV-VIS Reference Disk for the Praying Mantis	DRP-SPR
Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220/240V input, 24V output (CE marked)	ATC-024-2

	FT-IR	*UV-VIS*
Ambient Sample Chamber	DRP-ASC	DRP-ASC-VUV
High Temperature, Low Pressure Reaction Chamber, 24V	HVC-DRP-3 (KBr)	HVC-VUV-3 (SiO_2)
High Pressure Dome for the HVC Reaction Chambers (Max. Pressure: 500 psi)	HVC-DWI-3 (ZnS)	HVC-DWA-3 (SiO_2)
Low Temperature, Low Pressure Reaction Chamber, 24V	CHC-CHA-3 (KBr)	CHC-VUV-3 (SiO_2)

Spectralon® is a registered trademark of Labsphere.

The Praying Mantis™ was the first generally available diffuse reflection accessory (DRA) and remains the forerunner in the field today. It is ideal for reliable diffuse reflectance studies of powders and other rough surface solid samples.

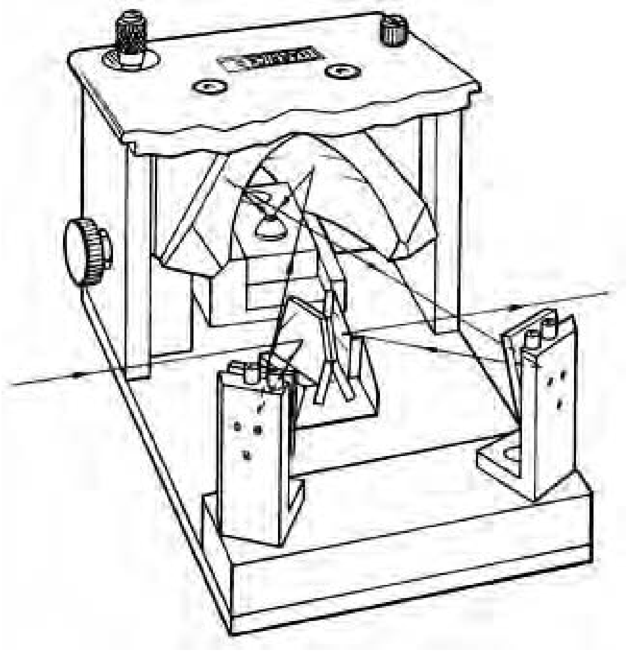


Figure 1. Interior View of the DRP.

The typical optical configuration of the Praying Mantis™ Diffuse Reflectance Attachments is shown in Fig. 1. The DRA incorporates two 6:1 90° off-axis ellipsoidal mirrors. One ellipsoid focuses the incident beam on the sample while the second collects the diffusely reflected radiation from the sample. Both ellipsoidal mirrors are tilted forward so the diffusely reflected radiation is collected at an azimuthal angle of 120°. This deflects the specularly reflected component behind the collection ellipsoid, minimizing the intensity of restrahlen bands caused by the specularly reflected light. Most other commercially available attachments collect the diffusely reflected light at 180°, where the restrahlen bands have maximum intensity**.** This optical geometry permits collection of up to 20% of all the diffusely reflected radiation, making the DRA quite practical for routine measurements.

The Praying Mantis™ can be used to examine powders and small solid samples. The sample is placed in one of the supplied sampling cups on the Praying Mantis™ sampling stage. The height of the stage can then be adjusted for optimal performance. The micrometer-style height adjust allows for accurate and reproducible positioning of the sample. For easy access to the sampling area, the Praying Mantis™ features the ability to flip its illumination and collection ellipsoids away from optical plane of the attachment.

The Praying Mantis™ also features PermaPurge™ and hence is enclosed in a purgeable box for rapid sample exchange with minimal interruption of the purge. This eliminates interference from water and carbon-dioxide bands in the infrared.

The Praying Mantis™ is ideal for studying samples in a controlled environment and several optional chambers are offered. Our Ambient Sample Chamber is designed for analysis of air-sensitive samples. The samples can be loaded in a glove box or similar enclosed environment. The chamber can then be sealed, removed from the glove box, and inserted in the Praying Mantis™ for analysis. This chamber features a removable stainless steel dome with two KBr or UV quartz windows and a glass observation window.

Two reaction chambers (see separate data sheet) are also available. These reaction chambers are designed for operation in static or flow conditions. Our Low Temperature Chamber is designed for operation up to 1-2 ATM and for temperatures ranging from -150ºC to 600ºC. Our High Temperature, Low Pressure Chamber operates at temperature up to 600ºC (under vacuum) and from pressures of 10^{-6} torr to 2 ATM. With its optional High Pressure Dome, this chamber can withstand pressures up to 500 psi.. This chamber can be purchased as part of a kit including the Praying Mantis™, Automatic Temperature Controller and comprehensive sampling tools (mortar and pestle, KBr powder, finger cots and spatula).

Representative spectra recorded with the Praying Mantis are shown here in Figures 2 and 3. Note that Figure 3 was recorded using the DRA with its HVC reaction chamber.

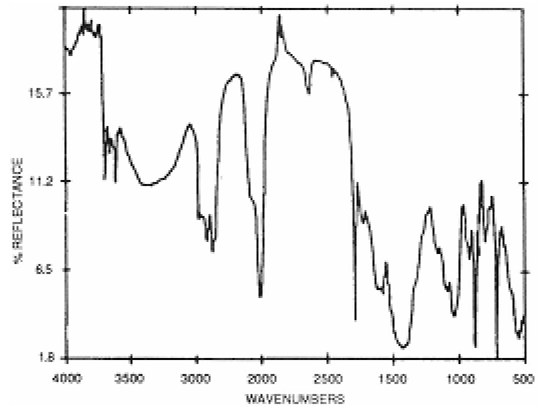


Figure 2. Diffuse Reflection of Chalk.

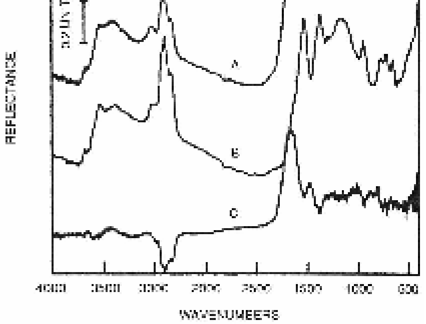


Figure 3. Diffuse Reflection of Wyodak Coal (A) after 24 hrs of oxidation at 2.4KPa at 393°C, (B) dried unoxidized samples, and (C) the difference spectrum (A-B).

FastIR™

The FastIR* is a horizontal, single ATR accessory. It was originally designed for quick internal reflection (ATR) measurements of liquids and pastes. It is also extremely convenient for examining solids. Simply place the sample (liquid, paste or soft solid) on the prism, spread it over the horizontal sampling surface, record the spectrum, and wipe the prism clean.

APPLICATIONS

- Single reflection ATR spectroscopy.
- Convenient for routine analysis of liquids, pastes, and flexible solids.
- Excellent for both laboratory and quality control environments.

FEATURES

- Provides highly repeatable, qualitative and quantitative results.
- Convenient, horizontal sampling surface.
- Fixed 45° incident angle.
- Top loading.
- SuperCharged™ ZnSe optics for superior performance.
- Easy to align and use.
- High throughput (greater than 85%).
- Minimal sample preparation required.
- Readily exchangeable crystal.
- Optional polarizer for enhanced spectral contrast and orientation studies. Includes slide plate mount.
- PermaPurge™ for rapid purging of the system.

INCLUDES

- Mounted ZnSe prism.
- Built-in pressure plate and clamp for solid sampling.
- Glass cover slide for volatile samples.
- Mating hardware for the specified spectrometer.

Material	Catalog No.
ZnSe	FAS-ATR-M
ZnS	FAS-ATR-I
Ge	FAS-ATR-J

*Patented.

ORDERING INFORMATION

	CATALOG NO.
FastIR™	FAS-XXX*
OPTIONS	
Slip-Clutch, 56 in-oz	SLP-CHI
Slip-Clutch, 24 in-oz	SLP-CLO
FastIR™ Wire Grid Polarizer (KRS-5 substrate). Includes mount and polarizer	PWD-FAS-XXX*
Wire Grid Polarizer Mount for the GATR™ (use with PWD Polarizer)	PWC-FAS-XXX*
Torque Screwdriver	PTW-SXX
Flow-Through Liquid Cell	FAS-FLC
Temperature-Controlled Liquid Cell, 24V	FAS-TCC-3
Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220/240V input, 24V output	ATC-024-2
REPLACEMENT PARTS	
Cover Slide	FAS-XCS

**XXX indicates spectrometer make and model*

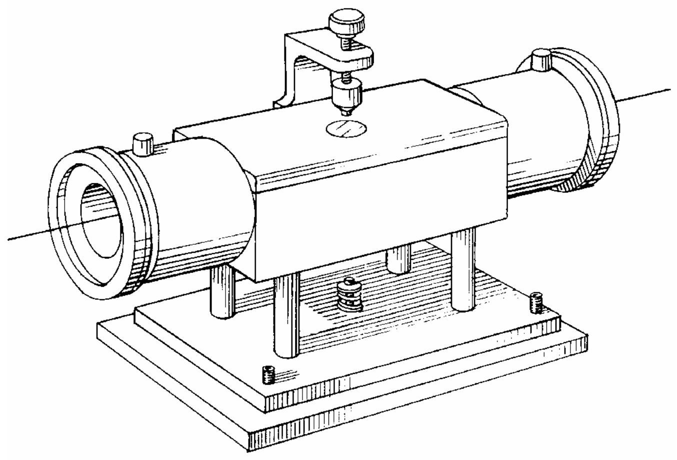


Figure 1. The FastIR. Shown with PermaPurge.

This single reflection, horizontal internal reflection (ATR) attachment is a powerful analytical tool for repeatable qualitative and quantitative studies of a wide range of samples. These include aqueous and organic liquids, pastes, and organic and inorganic powders

The simple efficient optical design of this attachment combines high optical throughput with the convenience of horizontal sampling. Installation and alignment are simple. Sample preparation is virtually eliminated. The high energy throughput allow excellent spectra to be obtained even with weakly absorbing samples. Conventional transmission cells can cause a degradation of spectral information obtained on highly absorbing samples due to severe energy losses, making accurate analytical work impossible. For such samples, the attenuation of this single reflection horizontal internal reflection attachment is essential.

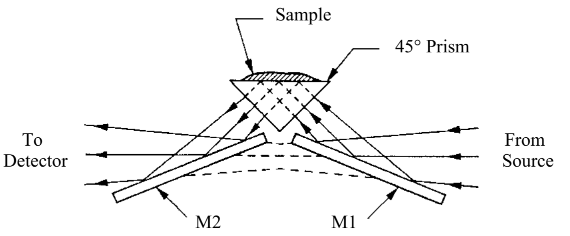


Figure 2. Optical Drawing of the FastIR™.

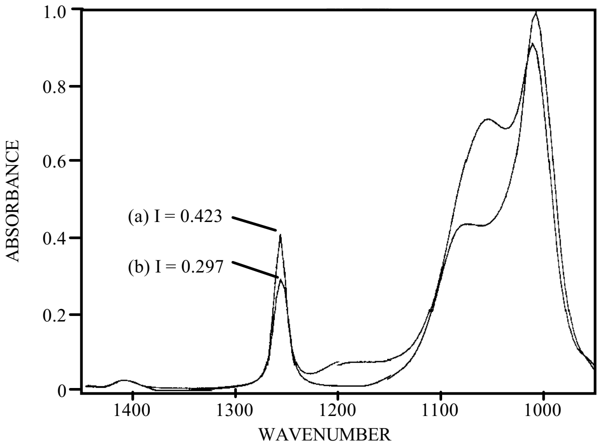
An optical drawing of the FastIR™ is shown in Figure 2. Light from the spectrometer source strikes mirror M1. The reflected light then strikes the 45° single reflection crystal at normal incidence and passes through the element until it is internally reflected at the sampling surface. The light interacts with the sample and exits the other 45° face at normal incidence. The radiation then reflects from mirror M2 to the detector of the spectrometer. This design provides a high degree of insensitivity to precise alignment. It also allows the mirror configuration of the attachment to be used not only for internal reflection (ATR) but also for external or specular reflection at a fixed 45° angle of incidence.

The standard internal reflection element is made from ZnSe. This has a wide useful spectral range (20,000 cm^{-1} to 500 cm^{-1}) and durable mechanical properties. Alternative materials are also available. The crystal is held into its mounting block using a Kalrez gasket. This gasket does not interact with the sampling radiation. Thus spectra are free from any spurious peaks or energy degradation due to the interaction of the light with the adhesive.

Liquids and pastes may be analyzed by simply placing the sample on top of the crystal. For volatile samples, a glass cover slide is provided. The o-ring of the sealed liquid cell does not come in contact with the sampling light, so spectra are free from any interference or degradation associated with the o-ring. Additional accessories for the FastIR™ include flow-through liquid reservoirs and trough-style liquid cells. Heatable solid and liquid cells are available on special order.

Solid materials, such as fine powders, flexible polymers, and soft films, may also be analyzed using this attachment. In order to assure uniform and repeatable compression of the sample, a pressure plate and clamp are provided.

As one example of the versatility of this attachment, quantitative analyses of silicone oil were performed. The ability to analyze these samples directly eliminated the need for a tedious chromatographic separation. The samples exhibit high absorbance in the infrared and high viscosity. Use of short pathlength transmission liquid cells would prove difficult due to the viscosity of the samples. The short effective pathlength and ease of sample introduction for this attachment, however, make analyses of samples of this nature extremely straightforward. Figures 3a-c show the almost identical absorbance spectra of the silicone oil samples: pure component 1, pure component 2, and a mixture of the two. Using the absorbance at 1257 cm^{-1}, the concentration of components 1 and 2 in the mixture were quickly and accurately determined.



Figures 3a and 3b. Silicone Oil.
Components (a) 1 and (b) 2.

A second example is given in Figure 4. Here, the spectrum of talcum powder is shown. This spectrum was taken using the attachment with the pressure plate and clamp.

These two examples demonstrate the utility and flexibility of this single reflection FastIR™ accessory.

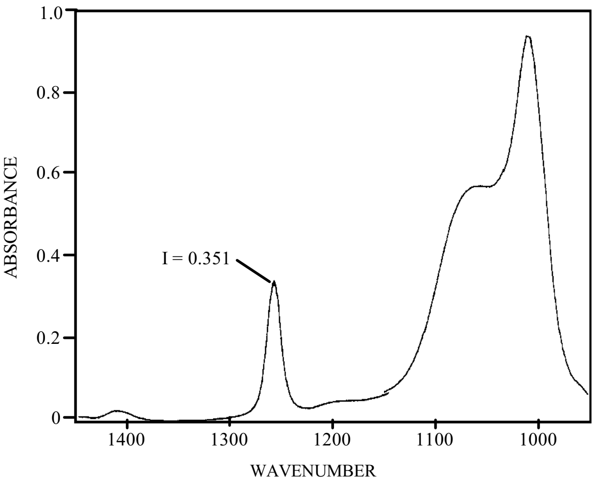


Figure 3c. Silicone Oil Mixture.

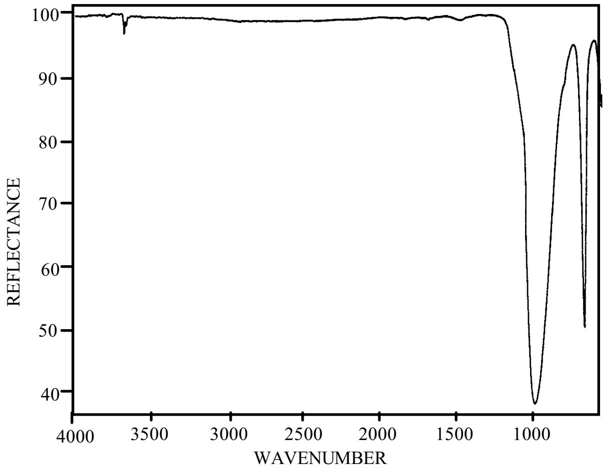


Figure 4. Internal Reflectance of Talcum Powder.

The FatIR™ Analysis System

The FDA now requires the amount of *trans* fat per serving to be indicated on the Nutrition Facts food label. To assist in meeting this requirement, Harrick Scientific has developed the FatIR™ System. This ATR FTIR oil analyzer complies with the FDA-approved AOAC method 2000.10 for routine quantitative ATR measurements of total *trans* fat content. The FatIR™ System is a straightforward analytical tool for fast, easy, and accurate determinations of *trans* fat concentration. In addition, it permits real-time *in situ* monitoring of the *trans* isomer formation during the processing of edible oils at temperatures up to 175°C.

APPLICATIONS

- Routine quantitative measurements of *trans* fat content, in accordance to AOAC method 2000.10.
- Real-time *in situ* monitoring of the *trans* fat formation during processing of edible oils at temperatures up to 175°C.
- Qualitative and quantitative analysis of liquids, pastes, and gels.

FEATURES

- Reproducible measurements.
- Convenient trough for containing liquid samples.
- Fixed 45° single reflection horizontal ATR configuration.
- SuperCharged™ ZnSe ATR optics for high throughput (greater than 85%)
- Simple to align and use.
- Safe, low-voltage heaters permit operation up to 175°C.
- K-type thermocouple directly measures the sample temperature and flips out of the way for easy cleaning of the ATR crystal.
- Low-voltage Temperature Controller allows for precise and safe regulation of the sample temperature, with options for direct, cascade and computer control.
- Secondary K-type thermocouple monitors the crystal temperature, permitting the cascade temperature regulation required for high temperature operation.
- Minimal sample preparation.
- Exchangeable ATR crystal**.**
- PermaPurge™ for rapid purging of the FTIR system.

INCLUDES

- RangeIR™ Liquid Analyzer with:
  - SuperCharged™ ZnSe mounted ATR crystal.
  - Two imbedded cartridge heaters.
  - Two K-type thermocouples.
  - Mating and PermaPurge™ hardware for the specified FT-IR spectrometer.
- AutomaticTemperature Controller

ORDERING INFORMATION

	CATALOG NO.
FatIR™, 110V	FAT-1-XXX
FatIR™, 220/240V	FAT-2-XXX

REPLACEMENT PARTS

Top Plate Assembly, ZnSe	RAN-TOP-M
ZnSe Crystal	FAS-ATR-M
Viton Gasket	RAN-GSK
K-Type Thermocouple	008-148
Heater Assembly (two heaters plus connector)	RAN-HTR
Cartridge Heater, 24V	HTRS-18

Under new FDA regulations, the amount of *trans* fat in a serving must be included in the Nutrition Facts panel. The Association of Official Analytical Chemists (AOAC) has developed an ATR-FTIR based method (AOAC Official Method 2000.10) that is in compliance with the new FDA regulations. This ATR-FTIR method is not only the simplest analytical method for the routine determination of the total trans fat content of fats, but it also allows for real-time *in situ* monitoring of *trans* isomer formation during the processing of vegetable oils and fats.

In light of this, Harrick Scientific has developed the FatIR^{TM} Oil Analysis System, which complies with the AOAC requirements. The FatIR^{TM} System is optimized for fast, easy, and accurate infrared measurements of the total *trans* fat content of oils and fats. Since much of the oil processing occurs at high temperatures, the FatIR^{TM} Oil Analysis System was designed for real-time *in situ* ATR studies of oils and fats at temperatures up to 175°C.

The FatIR^{TM} Oil Analysis System consists of a temperature-controlled trough style sampling accessory (the RangeIR^{TM}) and a low-voltage temperature controller. The RangeIR^{TM} is a single reflection ATR accessory, based on our time-proven FastIR^{TM}. This design has the highest optical throughput (>85%) of any ATR accessory. Its simple optical configuration, consisting of only two fixed flat mirrors and a triangular prism ZnSe ATR crystal, provides optimum performance without the need for sensitive optical alignment adjustments. The design is inherently stable, an essential feature for operation of a system used at the extreme temperatures simulating oil processing conditions.

The FatIR^{TM} System utilizes a trough configuration to simplify sample introduction and crystal cleaning. This configuration is required to contain oils and fats, especially at the higher temperatures where oil viscosity is lowered.

The two 24V cartridge heaters, in thermal contact with the crystal mounting plate, are operated by our automatic low-voltage temperature controller. Two thermocouples are provided, one in the sample and one in contact with the ATR crystal, to enable cascade operation of the temperature controller. Cascade control is necessary at higher temperatures because the lag between the heater and sample temperatures can cause the controller to otherwise overshoot the set point, potentially resulting in damage.

When studying heat-induced changes in oils, it is important to precisely and accurately know the sample temperature. The FatIR^{TM} System measures and controls the sample temperature directly, instead of recording the crystal or heater temperature, as is commonly done by other ATR accessories. This is important because the sample is exposed to a different thermal environment and hence is at a slightly lower temperature. With the FatIR^{TM}, the sample thermocouple is placed directly into the sample, providing the true value (and control) desired.

The FatIR^{TM} System incorporates easily adjustable and highly stable kinematic mounting, for optimum alignment in the particular spectrometer being used. In addition, the FatIR^{TM} System is equipped with Harrick's patented PermaPurge^{TM}. This allows samples to be exchanged without interrupting the purge of the FTIR spectrometer, which, in turn, greatly enhances sample throughput.

Figures 1 and 2 show the ATR spectra of various readily available oils. These spectra were recorded at 65°C, the temperature used by AOAC method 2000.10. From Figure 1, it is clear that pizza oil has the highest concentration of species with *trans* double bonds, while olive oil has the lowest. Figure 2 shows the *cis* bond region and indicates that the pizza oil has the highest concentration of species with *cis* double bonds.

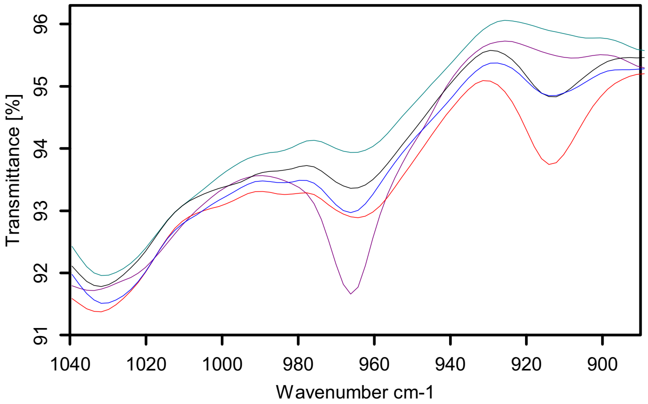


Figure 1. ATR spectra of the *trans* double bond region of several oils: extra virgin olive oil (green), peanut oil (black), canola oil (blue), corn oil (red) and pizza oil (purple).

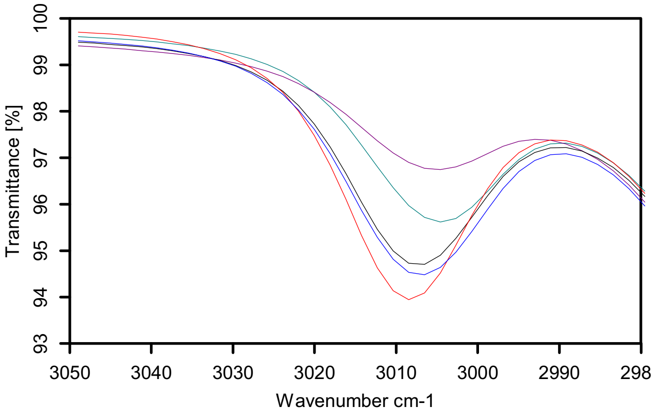


Figure 2. ATR spectra of the *cis* bond region of several oils: extra virgin olive oil (green), peanut oil (black), canola oil (blue), corn oil (red) and pizza oil (purple).

FiberMate2™

The FiberMate2™ links your FT-IR spectrometer to fiber optic probes. Equipped with large ellipsoidal mirrors, the FiberMate2™ efficiently transmits the infrared beam of the spectrometer through the fiber optic probe. The FiberMate2™ utilizes all reflective optics, optimizing the wavelength range of the fiber optic probe employed. FiberMate2™ comes with two SMA connectors for easy installation of the fiber optic probe and it is equipped with PermaPurge™ to maintain the purge of the system.

APPLICATIONS

- Mates fiber optic probes to FT-IR and UV-VIS spectrometers.
- Connects the Harrick MultiLoop-MIR™ infrared fiber optic probes to FT-IR spectrometers.
- Interfaces external optical systems to the spectrometer.
- Well-suited for optical testing of fiber optics for photonics applications.

FEATURES

- Beam condensing optics efficiently transmit light to and from fiber optic probes.
- All reflective optics maximize the wavelength range of the fiber optics employed.
- Two standard SMA connectors for easy installation of fiber optics probes.
- Designed for use with visible, near-IR and mid-IR fiber optic probes.
- and protects against stray light in the UV-VIS.
- Compatible with every major FT-IR spectrometer and some UV-VIS instruments.
- Prealigned and ready for the most challenging fiber optic application.
- Optional MultiLoop-MIR™ infrared fiber optic ATR probes available.

ORDERING INFORMATION

FiberMate2™ ---------- FM2-XXX*

OPTIONS

MultiLoop-MIR Mid-IR Set ---------- FOP-MIR
MultiLoop-MIR Silver Halide Probe Set for use from 2000 cm^{-1} to 600 cm^{-1} ---------- FOP-PIR
MultiLoop-MIR Chalcogenide Probe Set for use from 6500 cm^{-1} to 1700 cm^{-1} ---------- FOP-CIR

**XXX indicates spectrometer make and model. Varian customers receive a different model. Please contact us for details.*

THE FOCUS™

This grazing angle specular reflectance accessory focuses on obtaining high quality spectra quickly and efficiently. It is excellent for examining a variety of films and coatings on metals or other reflective substrates. The Focus™ features a 75º incident angle and a built-in Si polarizer for grazing angle measurements with high spectral contrast. Its convenient horizontal sample stage and condensed beam make it perfect for examining a wide variety of sample sizes and shapes. The Focus™ features PermaPurge™ for rapid sample exchange without interrupting the purge of the spectrometer. *For applications versatility, the Focus™ can readily be adapted for multiple reflection ATR experiments.*

APPLICATIONS

- Ideal for examining coatings on mirrors and other reflective substrates.
- Excellent for small and large samples.
- Invaluable for QC applications.

FEATURES

- 75º grazing incident angle.
- Built-in silicon polarizing plate for optimal spectral contrast.
- Convenient horizontal stage.
- High sample throughput.
- Small sampling area.
- Optional upgrade for multiple reflection ATR.
- Harrick's exclusive PermaPurge™ allows rapid sample without interrupting the purge of the system.

INCLUDES

- Built-in polarizer.
- Mating hardware for the specified spectrometer.

ORDERING INFORMATION

	CATALOG NO.
Focus™	FCS-XXX
OPTIONS	
Multiple Reflection ATR Upgrade to the ConcentratIR	CON-FOC

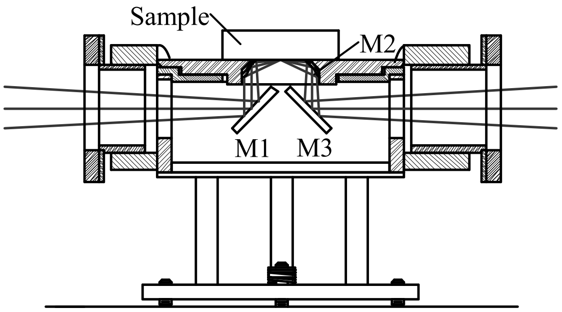


Figure 1. Schematic of the Focus.

The Focus™ is an excellent tool for analyzing coated materials of a variety of sizes and shapes. Such samples include coated mirrors, wafers, and disks. Coatings on flat machined metals can also be effectively examined.

This accessory, shown in Figure 1, utilizes a planar mirror to direct the beam to a parabolic reflector (M2) that focuses through a Si polarizing plate onto the sample. The reflected radiation is directed from the parabolic reflector and a second plane mirror to the detector of the spectrometer.

The Focus™ features a convenient horizontal sampling stage to accommodate a wide range of sample sizes. The Focus also includes a built-in Si polarizing plate for optimal spectral contrast. The accessory is enclosed in a purgable box for rapid sample exchange without interrupting the purge of the system.

Representative spectra recorded with the Focus™ are shown in Figures 2 through 4. Figure 2 is the reflectance from a golden film on a Si wafer. Note that the high level of the baseline indicates that the sample is a better reflector than the reference mirror used.

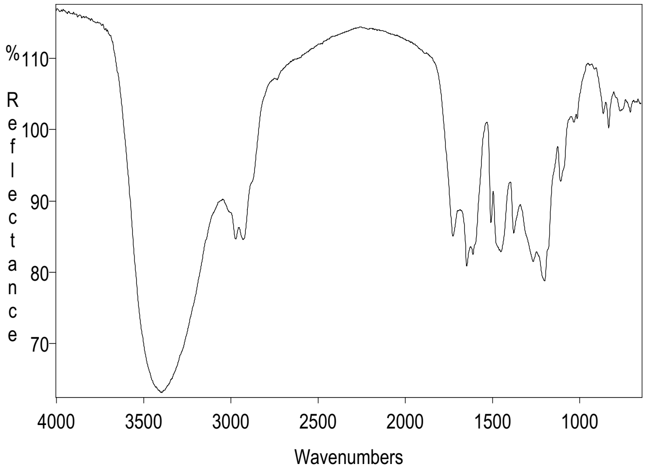


Figure 2. Grazing Angle Specular Reflectance of a Golden Colored Coated Si Wafer.

Figure 3 shows the spectra of two SiO_2 coated aluminum mirrors. The band intensities in the two spectra differ, indicating that the two coatings are not the same thickness.

Figure 4 shows the spectrum recorded from a machined surface coated with polyethylene. This demonstrates the effectiveness of the Focus™ for examining samples for coatings on flat, non-polished materials.

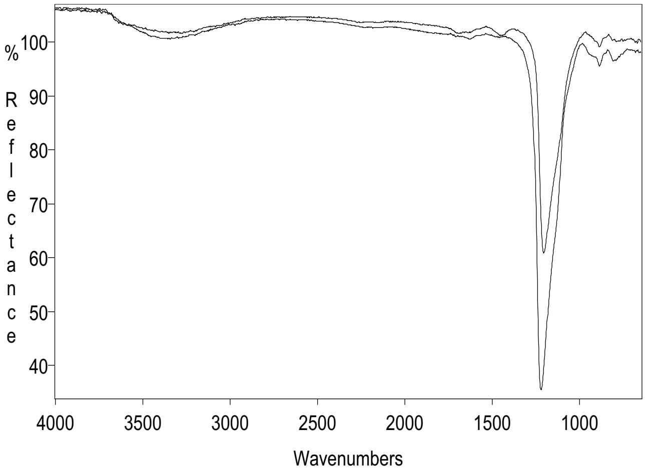


Figure 3. Grazing Angle Reflectance from Two SiO_2 Coated Aluminum Mirrors.

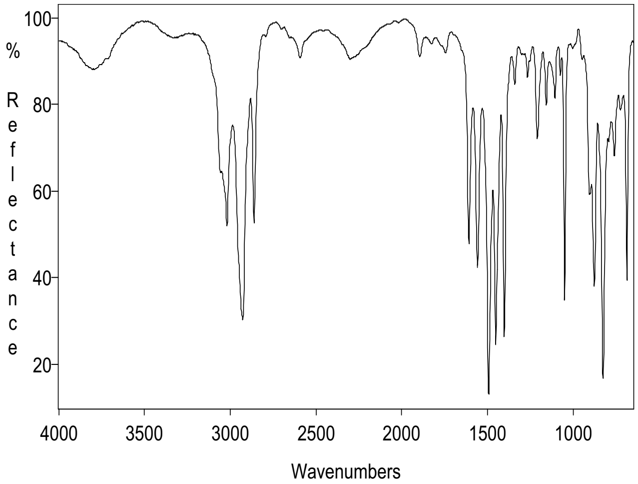


Figure 4. Grazing Angle Specular Reflectance of Polyethlyene on Machined Metal.

This accessory records grazing angle spectra quickly and accurately, allowing the user to *focus* on the results. Furthermore, the Focus™ is ATR-ready. Simply exchange its sampling plate with the optional ConcentratIR (see separate data sheet) top plate for multiple reflection ATR studies!

GATR™

The GATR™ grazing angle ATR accessory is a revolutionary approach to the analysis of monolayers on semiconductor and metallic substrates. The GATR™ is optimized for high sensitivity to these types of samples. Its specially designed pressure applicator is optimized for delivering good contact between the sample and the Ge ATR crystal. The GATR™ provides at least an order of magnitude increase in sensitivity relative to grazing angle methods, in addition to the convenience of an easy to use, fully prealigned, horizontal sampling accessory.

APPLICATIONS

- Analysis of monolayers and adsorbed species on semiconductors and metals.
- Rapid, repeatable measurements.

FEATURES

- Convenient horizontal sampling surface.
- Built-in pressure applicator optimized for contact with hard samples.
- 65° fixed incident angle.
- Ge ATR crystal.
- Accommodates samples up to 8” in diameter with center-sampling of discs up to 6” in diameter.
- PermaPurge™ for rapid purging of the system.
- Optional polarizer for enhanced spectral contrast and orientation studies. Includes slide plate mount.
- Optional torque screwdriver and slip-clutch for repeatable pressure application.

INCLUDES

- Ge hemispherical ATR crystal.
- Built-in pressure applicator, designed to accommodate large samples.
- Mounting hardware for the specified spectrometer.

ORDERING INFORMATION

	CATALOG NO.
GATR™	GATR-XXX*

OPTIONS AND REPLACEMENT PARTS

Slip-Clutch, 56 in-oz	SLP-CHI
Torque Screwdriver	PTW-SXX
Mounted Ge ATR Crystal	GATR-ATR-J
GATR™ Wire Grid Polarizer (KRS-5 substrate). Includes mount and polarizer	PWD-GATR-XXX*
Wire Grid Polarizer Mount for the GATR™ (use with PWD Polarizer)	PWC-GATR-XXX*

*XXX indicates spectrometer make and model

The G*A*TR™ is a single reflection ATR accessory designed for analyzing monolayers and adsorbed species on semiconductor and metallic substrates.

The G*A*TR™ integrates the theoretical conditions that provide the highest sensitivity to these extremely thin films[1,2] in a convenient horizontal ATR sampler. The G*A*TR features a 65° incident angle and a Ge ATR crystal for use from 5000 to 650 cm^{-1}. Its specially designed pressure applicator optimizes contact between the sample and the relatively small active portion of the crystal. For greater sensitivity, a polarizer can be added to the G*A*TR and, for repeatable measurements, a torque screwdriver or slip-clutch can be used to maintain consistent contact.

Figures 1 and 2 demonstrate the high sensitivity of the G*A*TR to monolayers. Figure 1 is a spectrum of an organic monolayer on a polished silicon surface. Figure 2 shows the spectrum of a monolayer on a gold-coated glass substrate.

For sampling versatility, the G*A*TR can also be used to analyze liquids, powders, pastes, and other solids. It is especially useful for samples with intense spectral bands. Such samples might otherwise exhibit too high absorbance or band distortions.

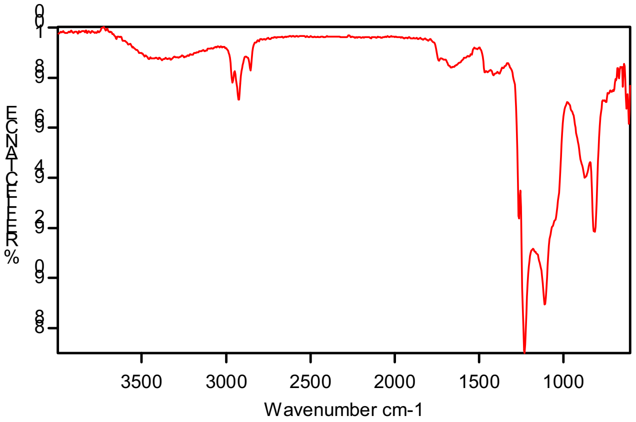


Figure 1. ATR Spectrum of an Organic Monolayer on Silicon.

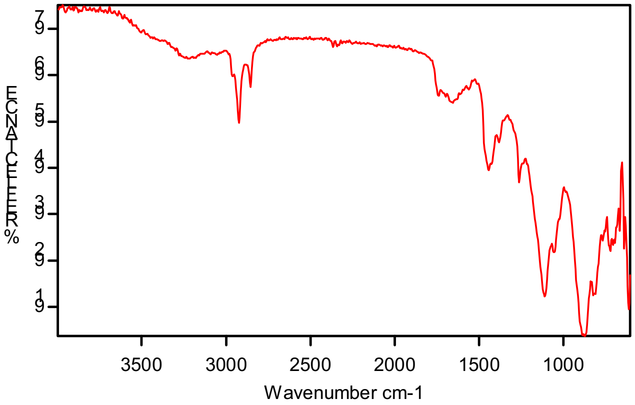


Figure 2. ATR Spectrum of an Organic Monolayer on Gold.

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[1] M. Milosevic and S. L. Berets, 'ATR of Monolayers on Si and Neat Powders by Single Reflection ATR,' PittCon 2002 invited paper.

[2] S.L. Berets and M. Milosevic, 'ATR Spectroscopy of Thin Films on Silicon,' paper in preparation.

HORIZON™

The Horizon™ multiple internal reflection attachment is well suited for rapid analysis of liquids, pastes, powders, and thick films. Its unique design takes advantage of the astigmatism inherent in spherical mirrors to shape the beam to match the aperture of the internal reflection element and to keep the radiation away from the edges of the crystal. This eliminates spectral interference from the adhesive used to mount the crystal and results in higher throughput. The Horizon™ also features a horizontal sampling surface for easy sample positioning and PermaPurge for rapid sample and crystal exchange without interrupting the purge of the system.

APPLICATIONS

- ► Multiple internal reflection (ATR) spectroscopy.
- ► Useful for examining liquids, pastes, powders and other soft surface solids.
- ► Perfect for quality control and others requiring rapid sample exchange.

FEATURES

- ► Top loading.
- ► Unobstructed, horizontal sampling surface.
- ► Thirteen reflections from the sample.
- ► PermaPurge provides for rapid sample and crystal exchange without interrupting the purge of the system.
- ► High spectral contrast.
- ► High throughput (25-40%)
- ► Utilizes astigmatism to match the beam shape to that of the internal reflection element, creating insensitive edges within the crystal.
- ► Unique optical design eliminates spectral interference from the adhesive used to mount the crystal.
- ► Easily exchangeable optional sampling plates including flow-through liquid cells with swagelok fittings. (Approx. volume: 0.35ml and large volume trough cells (approx. volume: 10ml). Heatable cells available on special order.
- ► Optional polarizer mount and compatible Wire Grid Polarizer.

INCLUDES

- ► One mounted 50x10x2mm SPT 45° ZnSe crystal.
- ► Solid or liquid sampling plate. Solid sampling plate includes a pressure plate and clamp; the liquid sampling plate has a trough to contain the liquid.

FLOW-THROUGH ADAPTERS

	Luer	Swagelok
Solid Plate	HOR-FSP-L	HOR-FSP-S
Trough	HOR-FLP-L	HOR-FLP-S

MOUNTED ATR CRYSTALS

Material	Solid Plate	Trough
Ge	HOR-SSP-J	HOR-LSP-J
KRS-5	HOR-SSP-U	HOR-LSP-U
ZnSe	HOR-SSP-M	HOR-LSP-M

ORDERING INFORMATION

	CATALOG NO
Horizon™ Liquid Sampler	HOR-L-XXX
Horizon™ Solid Sampler	HOR-S-XXX

OPTIONS

Polarizer Mount compatible with the PWG-U1R and PWG-SEA	HOR-PMT
Wire Grid Polarizer, Continuously Adjustable	PWG-UIR
Wire Grid Polarizer, Two Position	PWG-UIR
Torque Screwdriver	PWT-SXX

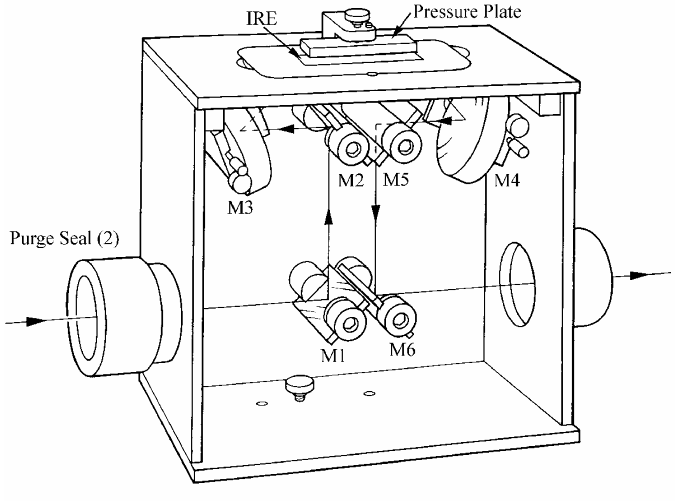


Figure 1. The Horizon™ Solid Sampler.

The Horizon™ multiple internal reflection attachment is a powerful tool for examining liquids, pastes, powders, and soft surface solids.

The unobstructed, horizontal sampling surface makes analysis easy. Samples of various sizes may be simply placed on top of the ATR element. For high quality spectra without atmospheric interference, this accessory features PermaPurge™. This permits sample and crystal exchange while maintaining the purge through the spectrometer and the accessory. This is ideal for quality control and other applications, which require rapid sample exchange.

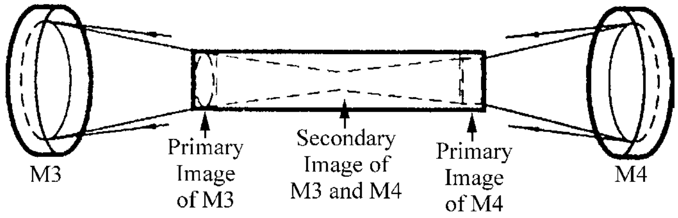


Figure 2. Schematic of the Beam Profile as the Beam Passes through the Internal Reflection Element.

The Horizon™ is shown in Figure 1. Mirrors M1 and M2 direct the incident beam to mirror M3. This spherical mirror focuses the beam and, due to its off-axis orientation, elongates the beam to match the rectangular entrance aperture of the crystal. The beam is internally reflected along the length of the crystal, where it interacts with the sample, and then exits the crystal. The beam, which has interacted with the sample, is then reflected from spherical mirror M4 to mirror M5. Mirror M5 reflects the beam to mirror M6 and onto the detector of the spectrometer. The crystal is positioned relative the focal points of the two spherical mirrors so that the beam shape is elongated to match the aperture of the crystal at both its entrance and exit (see Figure 2). Upon entering the crystal, the light continues converging in the direction parallel to the width of the crystal. When it reaches the center of the crystal, it starts diverging. Thus within the crystal, the radiation is kept away from the edges of the crystal. This eliminates spectral interference from the adhesive used to seal the crystal onto the mounting plate. This optical design results in a highly efficient multiple reflection accessory.

Representative spectra obtained using this attachment are shown in Figures 3 through 6.

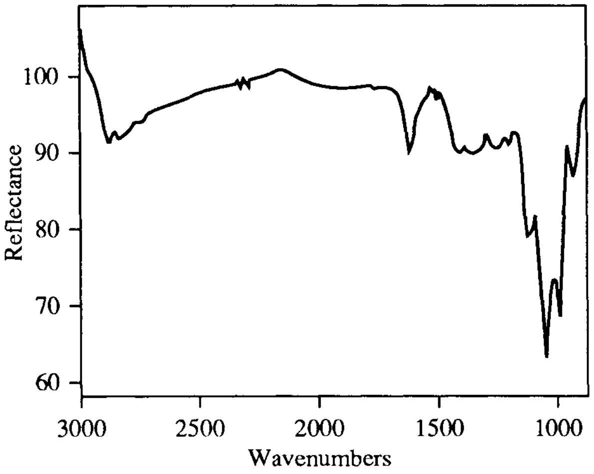


Figure 3. Internal Reflection Spectrum of Sweetened Coffee (without Milk) Relative to Water.

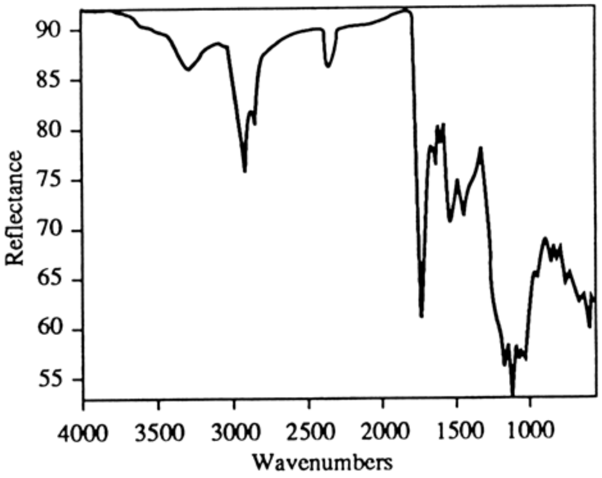


Figure 4. Internal Reflectance of an Adhesive on Paper.

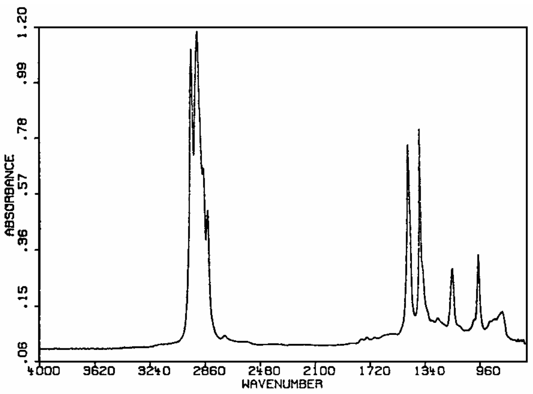


Figure 5. Internal Reflectance of Anhydrous Maleic Acid in Molten Polypropylene Recorded at 185°C.

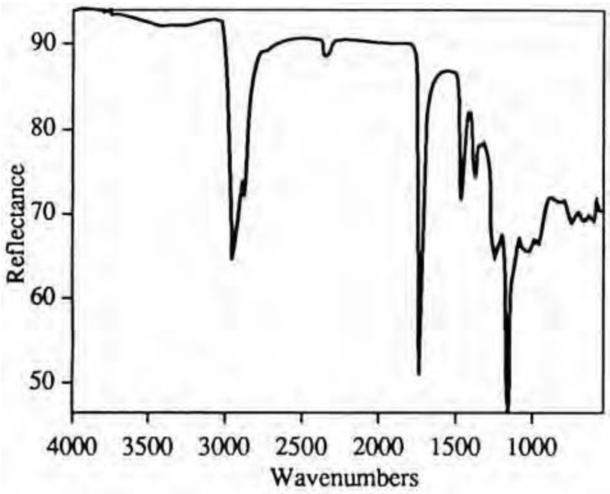


Figure 6. Internal Reflectance of Black Foam Packing Material.

REACTION CHAMBERS FOR THE PRAYING MANTIS

APPLICATIONS

- Allows diffuse reflection measurements under controlled pressures and a wide range of temperatures.
- Used in conjunction with the Praying Mantis Diffuse Reflection Accessory.

Low Temperature
Reaction Chamber

FEATURES

- Two models available:
  1. High Temperature, Low Pressure Reaction Chamber (HVC-DRP) for operation from high vacuum (10^{-6} torr) to two or three atmospheres and at high temperatures (up to 600°C under vacuum). Readily adapted for high-pressure operation with a high-pressure dome assembly. Use with the high-pressure domes for operation to 500 psi.
  2. Low Temperature Reaction Chamber (CHC-CHA) for studies from high vacuum to two or three atmospheres and at temperatures from -150°C to 600°C (under vacuum).
- Three inlet/outlet ports provided for evacuating the cell and introducing gases.
- Made of chemically resistant 316 stainless steel.

INCLUDES

- Reaction Chamber.
- Low-voltage heating cartridge.
- K-type thermocouple.
- Dome with two windows, KBr for theinfrared or SiO_2 for theUV-VIS, and one glass observation window.

High Temperature Reaction Chamber

WINDOWS FOR VACUUM CHAMBERS

MATERIAL	CATALOG NO.
SiO_2	WAD-U23
Si	WED-U23
CaF_2	WFD-U23
ZnS	WID-U23
ZnSe	WMD-U23
KBr	WPD-U23

WINDOWS FOR HVC-DRP HIGH PRESSURE DOMES

MATERIAL	CATALOG NO.
ZnS	WID-U43
ZnSe	WMD-U43
UV Quartz	WAD-U43

ORDERING INFORMATION

CATALOG NO

	FT-IR	*UV-VIS*
High Temperature Reaction Chamber, 24V	HVC-DRP-3	HVC-VUV-3
Low Temperature Reaction Chamber , 24V	CHC-CHA-3	CHC-VUV-3

OPTIONS & REPLACEMENT PARTS

Heater Assembly for the HVC and CHC, 24V	HVC-HTR
Heater, 24V	HTRS-17
Screen Set, two each of three mesh sizes	116-439
K-Type Thermocouple	008-144
High Pressure Dome for the HVC-DRP, ZnS Windows	HVC-DWI-3
High Pressure Dome for the HVC-DRP, ZnSe Windows	HVC-DWM-3

	110V	*220/240V*
Automatic Temperature Controller, 24V output	ATC-024-1	ATC-024-2
Vacuum Pump	VPE-001	VPE-002

Diffuse reflection spectroscopy is a very sensitive method for detecting changes at the surface of rough materials. It is particularly effective for powders with a high surface area. This makes diffuse reflectance a valuable tool for catalysis, oxidation, and photochemical studies. However, measurements for these applications must be obtained in a carefully controlled environment.

For this purpose, Harrick Scientific offers two reaction chambers for use in conjunction with the Praying Mantis Diffuse Reflection Attachment. Both reaction chambers are constructed of chemically resistant 316 stainless steel and can be heated up to at least 600°C under vacuum. The reaction chambers feature a sample cup that is part of a temperature-controlled sample stage. This stage incorporates a cartridge heater and K-type thermocouple. The electrical leads to the heater and thermocouple are located on the outside of the chamber for convenience. The stage is thermally isolated from the outer chamber. A water-cooling jacket is provided to control the temperature of the outer chamber and windows during high and/or low temperature operation. The reaction chamber also features three gas ports for evacuating, pressurizing or flowing gas through the sample. These ports have 1/4" VCO fittings with Viton o-rings. One of these ports leads directly under the sample cup; the other two lead into the sides of the chamber.

Optically, the reaction chambers are designed to minimize reflection losses from the windows and maximize light interaction with the sample. The radiation enters and exits the chamber perpendicular to the two optical apertures. A third aperture is provided for viewing, illuminating, or irradiating the sample. This makes the reaction chambers useful for photochemical studies. Both reaction chambers include two KBr windows and a glass observation window. These windows are mounted in a removable stainless steel dome using o-ring seals. Both reaction chambers can be operated from high vacuum to two or three atmospheres with the KBr and glass windows provided. Other window materials are available for higher operating pressures and for different wavelength regions. Low refractive index window materials should be selected to minimize reflection losses.

The High Temperature Reaction Chamber, HVC-DRP, is designed for operation from room temperature up to at least 600°C under vacuum. It can be adapted for use at high pressure by simply replacing the dome assembly. The high-pressure dome assembly utilizes two optical windows and a UV quartz observation window mounted in a stainless steel dome. ZnS, ZnSe and UV quartz are offered for operation to 500 psi. Note that for high-pressure operation, the maximum temperature limit could be lower due to heat losses that depend on properties of the gas, characteristics the sample, and the operating pressure. For optimal temperature control, Harrick Scientific recommends our Automatic Temperature Controller for gradual heating of the system. Heater and thermocouple connectors are compatible with this controller.

The Low Temperature Reaction Chamber, CHC-CHA, is designed for operation from -150°C to 600°C under vacuum. In addition to the heater incorporated in the sample stage, the CHC also features a cooling conduit connected to a dewar. The dewar can be filled with liquid nitrogen or other coolant to cool the sample stage below room temperature. For optimal temperature control, Harrick Scientific recommends our Automatic Temperature Controller for gradual heating of the system, both above and below room temperature. Heater and thermocouple connectors are compatible with this controller.

To evacuate these chambers, use our highly reliable, compact, low-noise level Vacuum Pump with a minimum pumping speed of 1.4m^3/hr. Compliant with international standards, it is ideally suited both for use with Harrick equipment and for general use in chemistry and research laboratories. Hermetically sealed by oil pressure controlled valves, the vacuum chamber is protected against inadvertent venting and oil backstreaming. This vacuum pump is supplied with a three foot length of ½" ID vacuum tubing and with the hardware to connect this tubing to the pump. Connections between this tubing and the HVC or CHC fittings are included.

Figure 1 shows several spectra recorded with the High Temperature Reaction Chamber in the Praying Mantis Diffuse Reflection Attachment. Harrick's Automatic Temperature Controller was used to regulate the temperature.

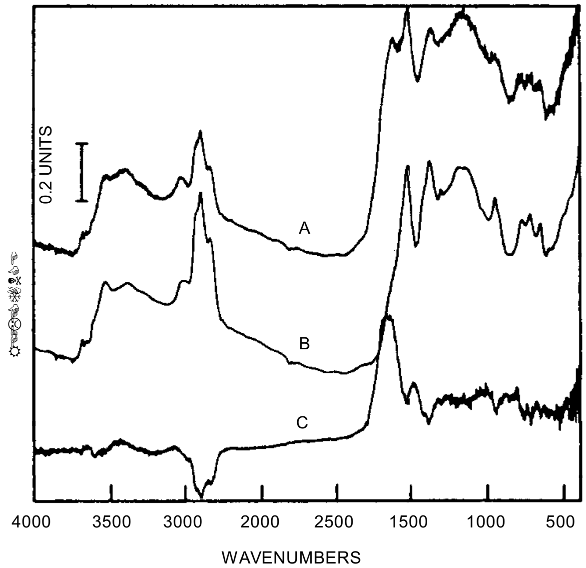
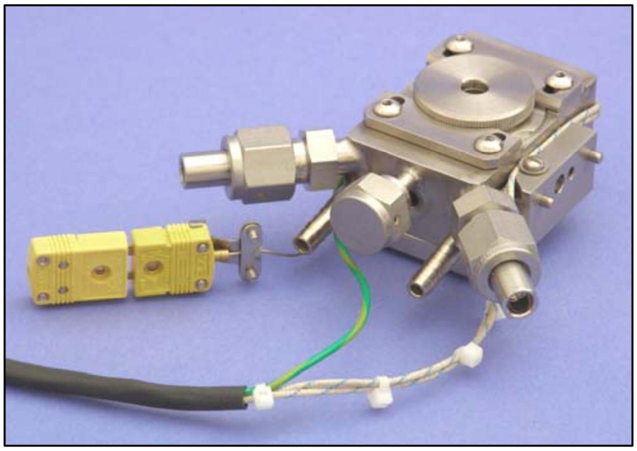


Figure 1. Diffuse Reflection of Wyodak Coal a) after 24 hrs. of oxidation at 2.4KPa at 393°C, b) dried unoxidized samples, and c) the difference spectrum (a-b).

REACTION CHAMBER FOR RAMAN SPECTROSCOPY



APPLICATIONS

- Allows Raman measurements under controlled pressures and a wide range of temperatures.
- Use in conjunction with a Raman microscope or spectrometer.

FEATURES

- Designed for operation from high vacuum (10^{-6} torr) to 25.8 ktorr
- Operable from ambient temperatures up to 910°C (under vacuum).
- 8-mm open aperture.
- Removable 13x2mm SiO_2 window.
- Three inlet/outlet ports provided for evacuating the cell and introducing gases.
- Made from chemically resistant 316 stainless steel.
- Optional Restek Silcosteel®-CR coating available for greater corrosion resistance.
- Optional mounting available.

INCLUDES

- Reaction Chamber.
- Low-voltage heating cartridge.
- K-type thermocouple.
- Window assembly with Viton o-rings and 13x2mm SiO_2 window

ORDERING INFORMATION	CATALOG NO
Window Assembly for Microscopy	HVC-MRA
Reaction Chamber, 24V	HVC-000-4
OPTIONS & REPLACEMENT PARTS	
Screen Set, two each of three mesh sizes	116-439
K-Type Thermocouple	008-144
13x2mm SiO_2 window	HVC-DWM-3
Heater Assembly	HVC-HT4
Heater, 24V	HTRS-26
Automatic Temperature Controller, 24V output, 110V	ATC-024-1
Automatic Temperature Controller, 24V output, 220/240V (CE marked)	ATC-024-2
Vacuum Pump, 110V	VPE-001

TRANSMISSION SAMPLING KIT

Our Transmission Sampling Kit includes all the tools and materials for sampling by infrared transmission spectroscopy. It contains accessories and supplies necessary for the analysis of liquids, both qualitative and quantitative, and solids, as mulls and pellets. This kit is packaged in a durable wooden carrying and storage case.

APPLICATIONS

- General kit for transmission spectroscopy.
- Intended for analysis of liquids, solids and pastes.

FEATURES

- DLC 2™, a PTFE Demountable Liquid Cell with Luer Lok Fittings. Uses standard, undrilled windows and precision PTFE spacers. Slide plate mounted to fit in all FT-IR spectrometers. Designed to examine liquids injected by syringe, solids smeared as mulls on KBr windows, viscous liquids and pastes smeared directly on the windows.
- MK Pellet Press used to prepare 13 mm diameter pellets. Pellets may be removed or left in the holder of the MK Press for analysis.
- Mortar and Pestle for grinding solids for making KBr pellets and producing mulls. Made of highly durable alumina to prevent sample contamination during grinding.
- KBr Powder for diluting solid samples for analysis as pellets.
- Nujol and Fluorolube for mixing with finely ground solid samples for analysis as mulls.
- KBr windows for use in the Liquid Cell and as supporting substrates for mulls, viscous liquids and pastes.
- Universal Transmission Holder for supporting polymer films, pellets, the pellet press holder and mulls or pastes smeared between KBr windows. Readily slips into the sample slide holder in all FT-IR instruments.
- Sample Cards for supporting and storing samples such as pellets and polymer films.
- Spatula/Scoopula Combination and disposable Syringe for transferring samples during preparation.

INCLUDES

- Sample Holders
  - PTFE Demountable Liquid Cell.
  - MK Pellet Press.
  - Universal Transmission Holder.
  - Sample Cards (10 pcs).
  - Sampling Accessories
  - Spatula/Scoopula Combination.
  - Mortar and Pestle.
- Supplies
  - Fluorolube, 1 oz.
  - Nujol, 1 oz.
  - KBr powder, 100g.
  - Finger Cots (12 pcs).
  - KBr windows, 13x2 mm (8 pcs).
  - Syringe, 2ml.

ORDERING INFORMATION

ORDERING INFORMATION	CATALOG NO.
Transmission Sampling Kit	KIT-TS1

OPTIONAL AND REPLACEMENT PARTS

DLC 2™	DLC-L13	Fluorolube	KIT-FLB	Mortar and Pestle	KIT-MAP
Liquid Cell Viton O-Rings	ORV-012	MK Pellet Press with Die	MKP-S13	Nujol	KIT-NJL
Liquid Cell Kalrez O-Rings	ORK-012	KBr Powder	KBR-100	Sample Cards (100 pcs)	KIT-CRD
Liquid Cell Spacers	PSA-M13	KBr Window (13x2mm)	WPD-U22	Spatula/Scoopula	KIT-SCP
Finger Cots (100 pcs)	KIT-COT	Universal Sample Holder	HUT-S1G	Syringes, 2 ml (10 pcs)	KIT-SYR

MERIDIAN™: THE DIAMOND SPLITPEA™,[1]

The Meridian™ is the ultimate accessory for examining small or extremely hard samples by internal reflection (ATR) spectroscopy. Harrick Scientific's Meridian™ is a horizontal ATR accessory with a sampling area less than 500 µm in diameter, the smallest area for any diamond ATR accessory. It is configured to apply localized, measured pressure to produce superior contact between the sample and its diamond ATR crystal. The Meridian™ is perfect for quick and easy examination of extremely hard samples, abrasive powders, and highly corrosive materials, in addition to routine analysis of small liquid, solid, and paste samples. For positioning samples on the small spot, a 50X viewing microscope, the ViewThruPress™,[2] is available. The Meridian™ makes ATR microsampling even more simple and straightforward by integrating Harrick's PermaPurge™,[3] for rapid sample and crystal exchange with minimal purge interruption. The Meridian™ is an innovative alternative to infrared microscopes, beam condensers, and diamond cells.

APPLICATIONS

- Uniquely suitable for studying extremely hard samples; highly corrosive liquids; minerals; slightly curved samples; fibers; nanoliters of liquids and pastes; and defects on large panels.
- Invaluable for forensic and combinatorial chemistry samples.

FEATURES

- The most chemically inert, durable, and cleanable ATR crystal available: diamond.
- Small sampling area - less than 500 µm in diameter in the standard ATR configuration.
- Minimizes stray light due to the small sampling area.
- Wide spectral range: 45,000 cm^{-1} to the FIR.
- High sample throughput due to little or no sample preparation.
- Calibrated pressure applicator for reproducible ATR measurements.
- Achieves optimal contact between the ATR crystal and hard samples.
- Flip-up, streamlined calibrated pressure applicator for easy access to sampling area
- ATR and external reflection capabilities provide application versatility.
- High energy throughput with DTGS detectors.
- Generally retains sample integrity.
- Harrick's exclusive PermaPurge™ allows rapid sample and crystal exchange without interrupting the purge of the system.
- Spill-resistant cover.
- Readily cleaned ATR crystal, due to the low adhesion properties of diamond.
- Inert ATR crystals available for use from the Near IR to the Far IR.
- Upgrade to a calibrated 50X ViewThruPress™ for easier viewing of the sampling area.

INCLUDES

- One ATR holder with mounted diamond hemisphere.
- Sample holder adapter for studying powders by ATR.
- External reflection sample holder and alignment mirror.
- Mating hardware for the specified spectrometer.

ORDERING INFORMATION

	CATALOG NO.
Meridian™ Diamond SplitPea™ with ViewThruPress™	MER-P-XXX
Meridian™ Diamond SplitPea™	MER-XXX
CombiKit™	UNS-CBK
Punch Sampler	PUN-SMP

OPTIONS AND REPLACEMENT PARTS.

	CATALOG NO.
O-ring Sealed Liquid Cell with Luer Fittings	UNS-LCF
ViewThruPress™ Upgrade	UNS-MIC
Liquid Cell O-Ring	ORV-0015
Powder Adapter O-Ring	ORV-012

Mounted Crystals:	CATALOG NO.
Si	UNS-ATR-0E
Diamond	UNS-ATR-0W
Ge	UNS-ATR-0J
ZnSe	UNS-ATR-0M
ZnS	UNS-ATR-0I

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[1] U. S. Patent 5,210,418
[2] U. S. Patent 5,308,983
[3] U. S. Patent 5,177,561

This diamond, horizontal micro-ATR accessory is ideal for analyzing a variety of samples, including hard solids, corrosive liquids and abrasive materials. Samples, which are difficult to analyze by conventional spectroscopic methods, such as fibers, rocks, paints, and microliters of liquids or pastes, are easily examined with the Meridian™.

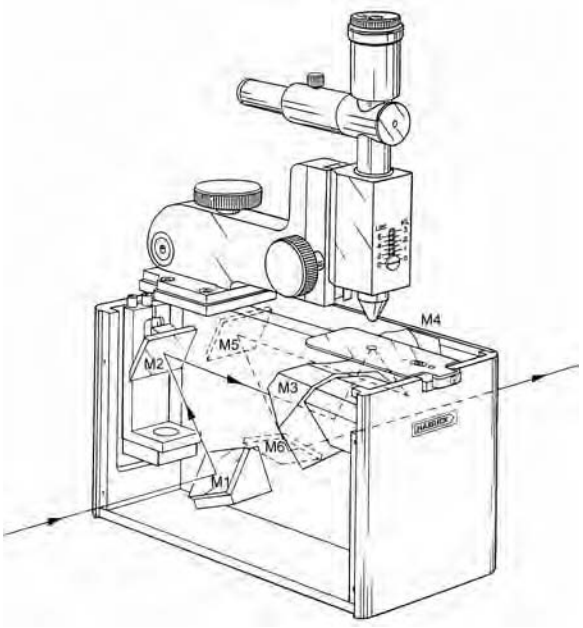

Figure 1. The Meridian™.

The Meridian™ can be configured for internal or external reflectance, simply by changing the sample holder. In its ATR mode, the Meridian™ features a removable holder for easy sample insertion and crystal cleaning. For external reflection and pre-alignment, the Meridian™ features a removable sample holder and reference mirror. The Meridian™ is enclosed in a purgable box for rapid sample exchange without interrupting the purge of the system.

An illustration of the Meridian™ is shown in Figure 1. Two mirrors, M1 and M2, direct the beam to an ellipsoidal mirror, M3, which focuses the light onto the sample. The radiation reflected from the sample is collected by a second ellipsoid, M4. Mirrors M5 and M6 direct radiation reflected from M4 to the detector of the spectrometer. This configuration provides a six times linear reduction of the source image on the sampling surface.

For ATR, the Meridian™ comes with one diamond ATR crystal. Diamond is extremely inert, allowing the analysis of highly corrosive materials. It also has a low friction coefficient, so samples do not adhere strongly to the surface, making sample clean-up simple and straightforward. Furthermore, diamond is an extremely hard material. This permits the application of high clamping pressures to ensure good contact between the IRE and the sample, even for samples as hard as rocks. Generally, lower pressures are required to achieve the good contact needed in ATR than are needed to flatten the sample for transmission studies. Thus fewer chemical and physical changes will be induced in the sample with the Meridian™ than with a high pressure diamond anvil cell.

The Meridian™ ATR crystal is a 3-mm diameter hemisphere. This further focuses the incident radiation onto the sample (see Figure 2), providing an additional reduction of the source image. For such a short pathlength, diamond is virtually transparent in the useful range of the far and mid-infrared. The diamond crystal is faceted on the edge of its flat surface to provide a sampling area that is 500 µm in diameter, slightly larger than the hot spot on the crystal. This makes it easier to position small samples and maximize the clamping pressure. Because of the small size of this island, the pressure plate applies localized pressure to the sample improving contact between the sample and the crystal, allowing high contact pressures to be achieved.

For applications versatility, ZnSe, Ge, and Si crystals are also available. This selection offers a variety of sampling depths and volumes.

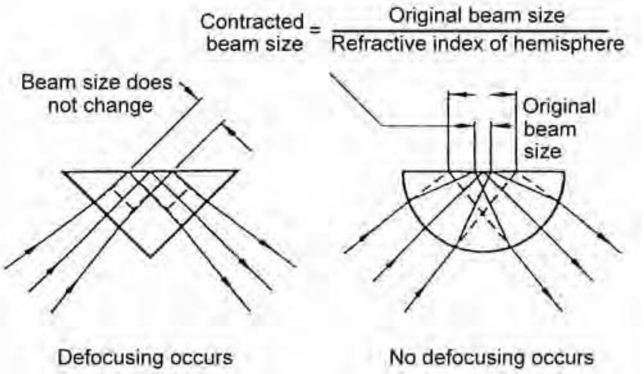

Figure 2. Focusing Effects of a Hemisphere.

The Meridian™ is available with a ViewThruPress™. This configuration is recommended for analyzing samples such as fibers, spots on transparent substrates, and powders. The ViewThruPress™ provides a 50X magnification of the sampling area and features precision adjustments for aligning the viewer over the active sampling area of the crystal. Its unique design permits the sample to be viewed as it is compressed against the crystal. The ViewThruPress™ also features an independent adjustment for focusing on the image. For liquid sampling, a flow-through liquid cell is available for use with the Meridian™. This cell is o-ring sealed and features two luer fittings. In addition, a kit is now available for dedicated sampling of combinatorial chemistry samples. This kit includes sample holders and pressure plates specially designed to accommodate the SynPhase™,[1] Lanterns and Crowns as well as the beads used as substrates for combinatorial chemistry.

Representative spectra recorded with the Meridian™ are shown in Figures 3 through 6. Figure 3 shows the spectra of

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[1] SynPhase™ is a registered trademark of Mimotopes Pty. Ltd., Australia.

Malachite ($CuCO_3{\cdot}Cu(OH)_2$), and Marble ($CaCO_3$ or $CaCO_3{\cdot}MgCO_3$). The Meridian™ readily exposes the characteristic features of these materials.

Figure 4 shows spectra from a clam shell collected from a beach on Cape Cod. The spectrum of the exterior surface has a greater contribution from the C-H and O-H bands, as would be expected from its greater exposure to the underwater plant life.

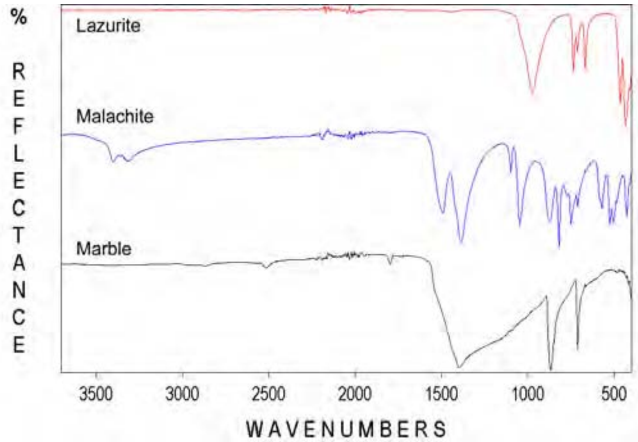

Figure 3. The ATR Spectra of Several Different Types of Rocks.

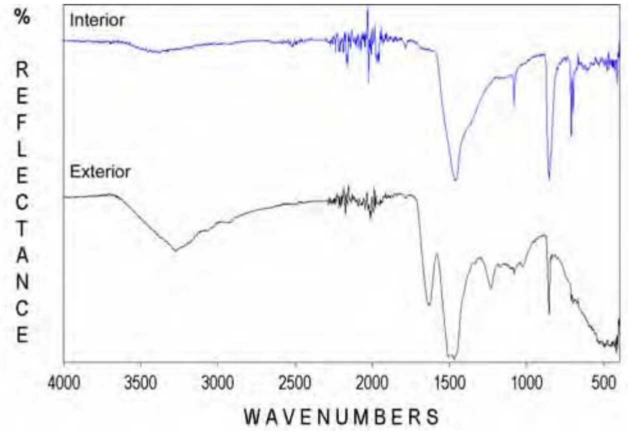

Figure 4. The ATR Spectra of the Interior and Exterior of a Weathered Clam Shell.

In addition to physically hard samples, the Meridian can be used to examine corrosive materials. Figure 5 shows the spectrum of Easy Off®[1] Oven Cleaner, whose active ingredient is sodium hydroxide. This highly corrosive material attacks most ATR crystal materials but does not effect diamond. For this measurement, a small drop of oven cleaner was placed on the sampling surface of the diamond ATR crystal. Note that this type of sample could also be examined using the Sealed Liquid Cell (UNS-LCF) for the Meridian™.

Figures 3 through 5 illustrate the use of the Meridian™ with materials that are typically difficult to examine. The Meridian™ easily analyzes more routine samples, as demonstrated in Figure 6. Here, small spectral differences in the chemical composition of two waxes are evident.

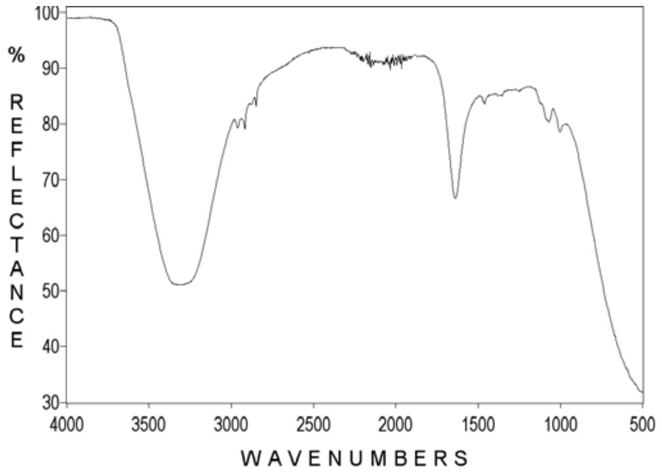

Figure 5. The ATR Spectrum of Easy Off® Oven Cleaner.

As demonstrated by the above examples, the Meridian™ diamond ATR micro-sampler is a powerful tool for examining a wide range of samples, including corrosive liquids, abrasive powders, and extremely hard solids in addition to those samples routinely analyzed by ATR.

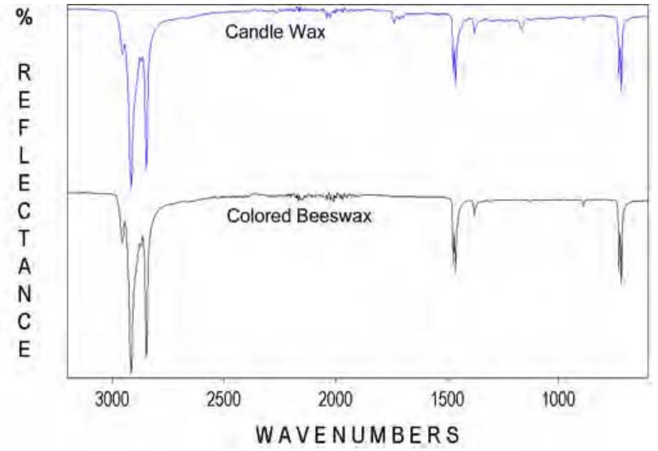

Figure 6. The ATR Spectra of Two Types of Wax.

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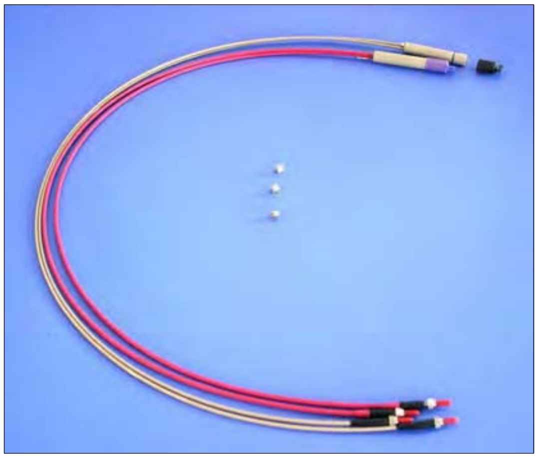
[1] Registered Trademark of Reckitt and Colman, Inc.

MultiLoop-MIR$^{\text{TM}}$ FTIR Fiber Optic Probe:
Spectroscopy Outside the Box

This MultiLoop-MIR system is perfect for analyzing liquids, pastes, and soft solids outside of the FTIR spectrometer sample compartment. It is used in conjunction with the Harrick FiberMate or other fiber optic coupler and includes two fiber probes with a set of ten ATR loop tips. This combination is designed for spectral data collection across the entire mid-infrared region. To analyze a sample, simply dip the ATR loop tip into the sample or press it gently against the sample. The tips are easily replaced when needed.

APPLICATIONS

- Multiple reflection ATR (internal reflectance) accessory.
- In-situ analysis of a wide variety of liquids, pastes, and soft solids.
- Quantitative and qualitative analysis.



FEATURES

- Multiple reflection ATR sampling outside the FTIR spectrometer.
- Effectively provides two reflections when compared to ATR with ZnSe at 45º.
- Wavelength range: 6500 cm^{-1} to 600 cm^{-1}:
  - Chalcogenide glass probe for use from 6500 to 1700 cm^{-1}.
  - Polycrystalline Silver halide probe for use from 2000 to 600 cm^{-1}.
- Sampling loop tips designed for the full spectral range of 6500 to 600 cm^{-1}:
  - Dip into or press against the sample.
  - Leak-free for analysis of liquids.
  - Readily replaceable.
  - Made from silver halide fiber material.
- Easy to use.
- Suitable for aqueous solutions and organic samples.
- Standard probe length of 1meter allows sampling outside the spectrometer sample compartment.
- Designed for use with DTGS or MCT detectors.
- SMA connectors for easy connection to fiber optic couplers.
- Operable from room temperature to 100°C.
- Use with the Harrick FiberMate™ fiber optic coupler for purged operation in most FT-IR spectrometers.

INCLUDES

- MultiLoop-MIR Mid-IR Set for the entire mid-IR
  - Silver Halide Probe.
  - Chalcogenide Probe.
  - Ten Silver Halide Tips.
- MultiLoop-MIR Silver Halide Probe Set (2000 to 600 cm^{-1})
  - Silver Halide Probe.
  - Five Silver Halide Tips.
- MultiLoop-MIR Chalcogenide Probe Set (6500 to 1700 cm^{-1})
  - Chalcogenide Probe.
  - Five Silver Halide Tips.

ORDERING INFORMATION

CATALOG NO.

MultiLoop-MIR Mid-IR Set	FOP-MIR
MultiLoop-MIR Silver Halide Probe Set for use from 2000 cm^{-1} to 600 cm^{-1}	FOP-PIR
MultiLoop-MIR Chalcogenide Probe Set for use from 6500 cm^{-1} to 1700 cm^{-1}	FOP-CIR
FiberMate Fiber Optic Coupler	FMA-XXX*

**XXX indicates spectrometer make and model*

REPLACEMENT PARTS

Disposable Loop Tips (set of 10)	FOP-T10
Disposable Loop Tips (set of 5)	FOP-T05
Replacement cover kit (includes replacement fiber covers, etc.)	FOP-PRT

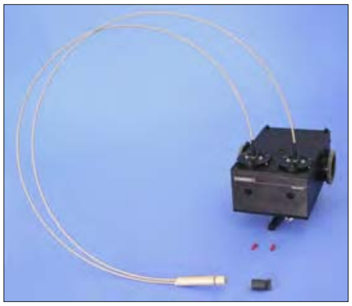


Figure 1. MultiLoop-MIR installed on the FiberMate.

New fiber manufacturing techniques combined with the high performance of modern FTIR spectrometers now make it possible to offer a fiber optic system for use with the DTGS detectors that are standard with most FTIR spectrometers. This system allows data collection over the entire mid-IR.

This system, the MultiLoop-MIR, features two fiber optic probes with readily replaceable sampling tips. One probe operates over the fingerprint region, from 2000 to 600 cm^{-1}, while the second covers the remaining mid-IR, from 6500 to 1700 cm^{-1}. The probes connect to a fiber optic coupler, such as Harrick's FiberMate, using standard SMA connectors. Probe tips are affixed onto the end of the probes to provide a leak-free probe assembly that can be dipped into or pressed gently into liquid, pastes, and soft solids. These tips are the only part of the MultiLoop-MIR that comes in contact with the sample, and they are easily replaced as needed.

The MultiLoop-MIR effectively provides two reflections from the sample compared to single reflection ATR at 45º with a ZnSe crystal. It has excellent throughput for infrared fiber optics, allowing data collection with a DTGS detector.

Several examples are shown below using the MultiLoop-MIR with the FiberMate (see Figure 2). For these measurements, the spectrometer set for 64 scans at 8cm^{-1} resolution. The entire mid-infrared spectrum was measured using the two fiber probes and tips. The chalcogenide probe was used for 4000cm^{-1} to 1520cm^{-1} and the silver halide probe for the 1530cm^{-1} to 600cm^{-1} region. The spectra were baseline corrected and joined together to show the full spectral range.

Figures 2 and 3 show spectra of three different liquids. The two children's analgesics shown in Figure 2 are clearly distinguishable from each other in the fingerprint region. The spectrum of a wine sample (Figure 3) shows characteristics from the organic component of the grapes, in additional to the strong aqueous bands.

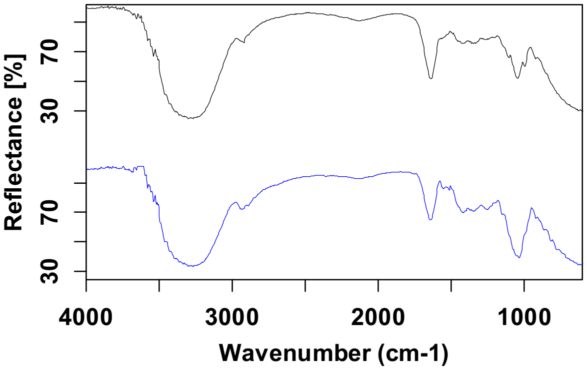


Figure 2. ATR Spectra of Children's Liquid Pain-Relievers Motrin (black) and Tylenol (blue).

Figures 4 and 5 demonstrate using the MultiLoop-MIR for solid sampling. The silicone rubber tape is soft and flexible, so good contact was readily obtained between the sampling tip and the tape, as shown by the strong band intensities. The peak intensities in Figure 5 are much weaker, indicating that the contact with this sample was not as good. Yet the spectrum is clearly identifiable as polyester. Note: pressing the sampling tip against solid samples reduces the lifetime of the tip and is only recommended for use with soft solids.

The MultiLoop-MIR provides a quick and easy method for quantitative and qualitative in-situ analysis of liquids, pastes, and soft solids within one meter of the spectrometer.

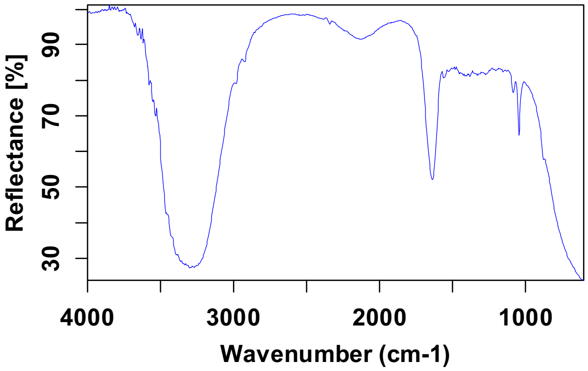


Figure 3. ATR Spectrum of Kouros White Wine.

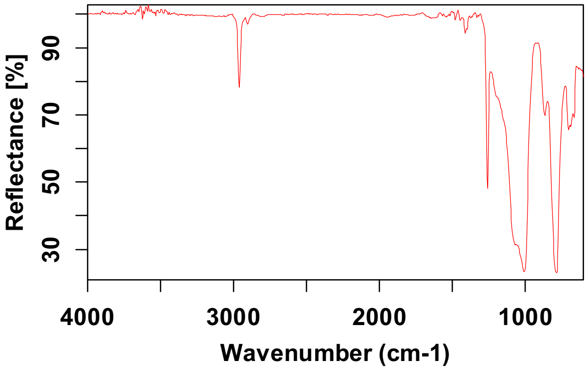


Figure 4. ATR Spectrum of Silicone Rubber Tape.

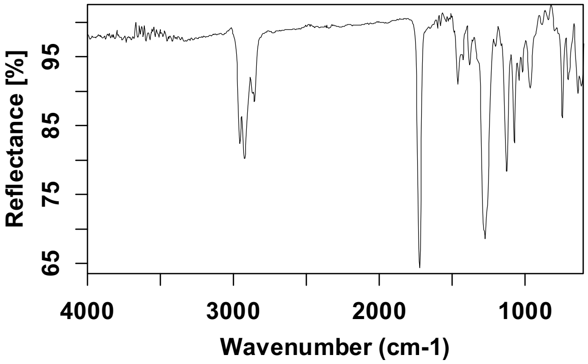


Figure 5. ATR Spectrum of a Plastic Toy Fish.

THE MVP-PRO STAR™

The MVP-Pro Star™ is single reflection *diamond* ATR accessory. It features a convenient horizontal sampling surface with a 1.5mm diameter sampling area. The MVP-Pro Star is *the choice* for infrared spectroscopy measurements of extremely hard samples, abrasive powders and highly corrosive materials. Since the MVP-Pro Star™ is affordable, durable, and straightforward to use, it is an excellent choice for routine analysis of liquids, solids and powders. Small drops of liquid and minute smears of pastes can be placed directly on the monolithic diamond ATR crystal. Coatings on wire, paint chips, fabrics, powders, and miniature hard samples can be pressed against the crystal using the built-in pressure applicator. The MVP-Pro Star™ also features PermaPurge™ for rapid sample exchange without interrupting the spectrometer purge. The MVP-Pro Star™ diamond ATR makes sampling simple and straightforward.

APPLICATIONS

- Excellent for studying optically thick, hard samples; slightly curved samples; fibers; paint chips; micro-liters of liquids and pastes; and powders.
- Invaluable for QC applications.

FEATURES

- Monolithic *Diamond* ATR prism designed for operation from 45,000 cm^{-1} to the FIR.
- Small sampling area: 1.5mm in diameter.
- Convenient horizontal sampling.
- High sample throughput.
- Little or no sample preparation required.
- Excellent retention of sample integrity.
- Readily exchangeable ATR sampling plates.
- Built-in pressure applicator with slip-clutch for reproducible calibrated pressure application.
- Designed to achieve optimal contact between the ATR element and solid samples.
- Harrick's exclusive PermaPurge™ allows rapid sample exchange without interrupting the purge.
- Mechanically compatible with the SplitPea™ and Meridian™ crystal holders.
- Options include:
  - Ge, ZnSe and Si hemispherical ATR crystals.
  - Heatable sampling plates for operation up to 200°C with diamond or ZnSe ATR crystals, 100°C with Ge.
  - Powder Adapter for retaining powdered samples.
  - Flow-Through Liquid Cell for static and flow applications.
  - Force sensor with digital read-out for precise force measurements.
  - Low-torque slip-clutch available.

Crystal Material	Additional ATR Sampling Plates	Heated Sampling Plates
Diamond	MVP2-ATR-0W	UNS-HOT-0W
Ge	MVP2-ATR-J	UNS-HOT-0J
Si	MVP2-ATR-E	UNS-HOT-0E
ZnSe	MVP2-ATR-M	

INCLUDES

New
- Epoxy-free Diamond ATR sampling plate.
- Built-in pressure applicator.
- Mating hardware for the specified spectrometer.

ORDERING INFORMATION

	CATALOG NO.
MVP-PRO Star™	MVS-XXX

XXX denotes the spectrometer code

OPTIONS

LowTorque Slip-Clutch, 24 in-oz designed for use with ZnSe and Ge ATR crystals	SLP-CLP
Force Sensor with Digital Read-Out	MVR-FSD
O-Ring Sealed Liquid Cell with Luer Fittings	UNS-LCF
Liquid Cell Viton O-Ring	ORV-0015
Powder Adapter	UNS-PSC
Powder Adapter Viton O-Ring	ORV-012
Automatic Temperature Controller, 110V	ATC-024-1
Automatic Temperature Controller, 220/240V (CE marked)	ATC-024-2

This MVP-Pro Star™ is a unique horizontal ATR mini-sampler, ideal for analyzing samples that are difficult to analyze by conventional spectroscopic methods. Such samples include solids such as coatings on slightly curved materials and paints and micro-liters of liquids or pastes.

The MVP-Pro Star™ utilizes all front-surface mirrors to focus the beam to and from the ATR crystal. The MVP-Pro Star™ features a monolithic diamond ATR prism. This provides a 1.5 mm diameter sampling area on the top of the diamond that can be used for spectroscopic measurements from 45,000 cm^{-1} to the FIR, with limited signal-to-noise in the 2300 cm^{-1} to 1850 cm^{-1} region.

The ATR crystal is mounted in a removable sampling plate for easy cleaning and crystal replacement. This sampling plate is part of the purge enclosure around the MVP-Pro Star™ that allows rapid sample exchange without interrupting the purge of the system. For applications requiring different penetration depths or chemical resistances, sampling plates with Ge, Si, and ZnSe hemispherical ATR crystals are also available.

Heatable sampling plates are also available for operation up to 200°C with diamond and ZnSe or up to 100°C with a Ge ATR crystal. The Heatable Sampling Plates feature a K-type thermocouple and cartridge heater. These are intended for operation with the Harrick Automatic Temperature Controller.

For making contact with solid and powder samples, the MVP-Pro Star™ is equipped with a built-in pressure applicator with slip-clutch for consistent and reproducible results. For more fragile ATR crystals, i.e. ZnSe and Ge, a compatible low-torque slip-clutch is available. For more precise force measurements, a pressure applicator with built-in force sensor and display is offered.

For easy powder and liquid sampling, an optional Powder Retainer and Flow-Through Liquid Cell are available. Both attach to the sampling plates and are sealed with a Viton o-ring. The Powder Retainer contains the powder in a restricted volume and requires approx. 0.1 ml for operation. The Flow-Through Liquid Cell is a micro-cell with a volume of approx. 0.15 ml and Luer Lok fittings for sample introduction.

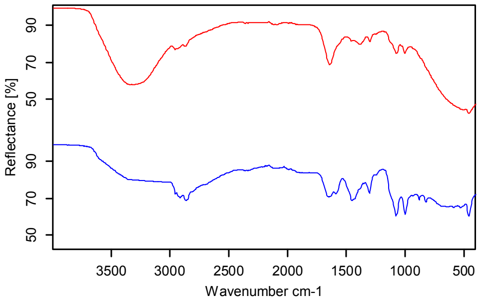


Figure 1. ATR Spectra of Easy-Off™ Oven Cleaner Freshly Applied (top) and After 15 min. of Drying (bottom).

Representative spectra recorded with the MVP-PRO Star™ are shown in Figures 1 through 4. Figure 1 shows the chemical changes in a corrosive cleansing agent as it dries. Over time, the water evaporates and the organic components and NaOH becomes concentrated.

Figure 2 shows spectra of an abrasive cleaner. The pad is impregnated with soap, as shown by the black spectrum. The spectra show that the concentration of soap in the pad is lower after the pad has been used.

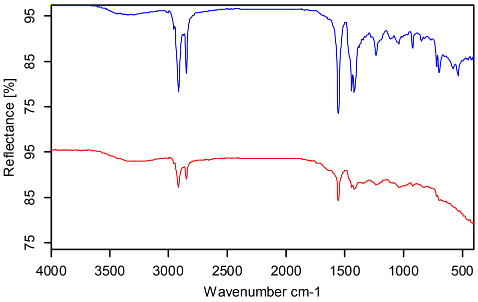


Figure 2. ATR Spectra of an SOS™ Steel Wool Cleaning Pad, New (top) and Dried After Use (bottom).

Figure 3 shows the ATR spectra of powdered dishwasher detergent. The detergent was a white powder with a low (<1%) concentration of green particles. A few grains of the green grains were separated from the bulk for analysis. The spectra show that the green material differs in composition from the bulk.

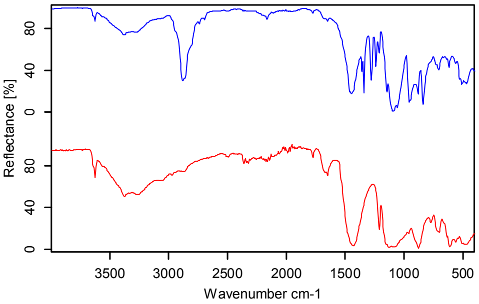


Figure 3. Normalized ATR Spectra of Green Specks in Cascade™ (top) and Cascade™ (bottom).

Figure 4 shows spectra of the inside and outside of an antihistamine table. The differences between the two are due to a coating on the outside of the tablet.

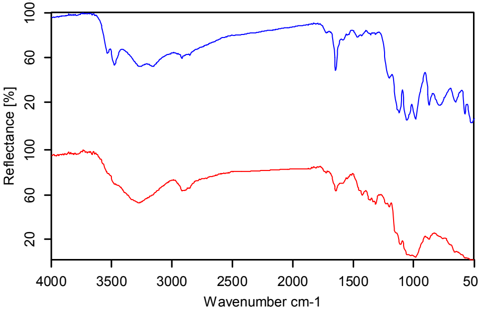


Figure 4. Normalized ATR Spectra of ChlorTrimeton™ antihistamine, inside of tablet (top) and outside of tablet (bottom).

The MVP-PRO Star™ diamond ATR is excellent for examining a wide range of samples, including liquids, pastes and solids. It is ideal for analyzing samples that would damage other crystals – highly acidic or basic liquids and extremely hard solids.

THE MVP-PRO™

This single reflection ATR accessory combines a convenient horizontal sampling surface with a 0.5mm diameter sampling area, making the MVP-Pro™ ideal for quick and easy examination of a wide range of samples. Small drops of liquid and minute smears of pastes can be placed directly on the ATR crystal. Coatings on wire, paint chips, fabrics, powders, and miniature hard samples can be pressed against the crystal using the built-in pressure applicator. The MVP-Pro™ also features PermaPurge™ for rapid sample and crystal exchange without interrupting the purge of the spectrometer. The MVP-Pro™ ATR makes FTIR spectroscopy of small samples simple and straightforward.

APPLICATIONS

- Excellent for studying optically thick, hard samples; slightly curved samples; fibers; paint chips; micro-liters of liquids and pastes; and powders.
- Invaluable for QC applications.

FEATURES

- Convenient horizontal sampling.
- High sample throughput.
- Little or no sample preparation required.
- Excellent retention of sample integrity.
- Small sampling area – 0.5mm in diameter.
- Si ATR hemisphere for operation from 10,000 cm^{-1} to 100 cm^{-1}.
- Readily exchangeable ATR sampling plates.
- Built-in pressure applicator with slip-clutch for reproducible pressure application.
- Designed to achieve optimal contact between the ATR element and solid samples.
- Additional ATR crystal materials available include a diamond prism and ZnSe or Ge hemispheres.
- Harrick's exclusive PermaPurge™ allows rapid sample exchange without interrupting the purge of the system.
- Mechanically compatible with the SplitPea™ and Meridian™ crystal holders.
- Options include:
  - Ge, ZnSe and diamond hemispherical ATR crystals.
  - Heatable sampling plates for operation up to 200°C with diamond or ZnSe ATR crystals, 100°C with Ge.
  - Powder Adapter for retaining powdered samples.
  - Flow-Through Liquid Cell for static and flow applications.
  - Force sensor with digital read-out for precise force measurements.
  - Low-torque slip-clutch available.

Crystal Material	Additional ATR Sampling Plates	Heated Sampling Plates
Diamond	MVP2-ATR-0W	UNS-HOT-0W
Ge	MVP2-ATR-J	UNS-HOT-0J
Si	MVP2-ATR-E	UNS-HOT-0E
ZnSe	MVP2-ATR-M	

INCLUDES

- Si ATR sampling plate.
- Built-in pressure applicator.
- Mating hardware for the specified spectrometer.

ORDERING INFORMATION

	CATALOG NO.
MVP-Pro™	MVR-XXX
OPTIONS	
LowTorque Slip-Clutch, 24 in-oz designed for use with ZnSe and Ge ATR crystals	SLP-CLP
Force Sensor with Digital Read-Out	MVR-FSD
O-Ring Sealed Liquid Cell with Luer Fittings	UNS-LCF
Liquid Cell Viton O-Ring	ORV-0015
Powder Adapter	UNS-PSC
Powder Adapter Viton O-Ring	ORV-012
Automatic Temperature Controller, 110V	ATC-024-1
Automatic Temperature Controller, 220/240V (CE marked)	ATC-024-2

This novel horizontal ATR mini-sampler is perfect for examining samples that are difficult to analyze by conventional spectroscopic methods. Such samples include solids, such as slightly curved and inflexible materials, powders, and micro-liters of liquids and pastes.

The MVP-Pro™ utilizes all front-surface aluminum mirrors to focus the spectrometer beam to and from the ATR crystal. The Si ATR crystal is hemispherical, additionally focusing the beam to produce a 0.5mm diameter sampling area. The accessory is enclosed in a purgable box for rapid sample exchange without interrupting the purge of the system.

For applications requiring different penetration depths or chemical resistance, ZnSe and Ge hemispherical ATR crystals are also offered, premounted in their sampling plates. In addition, a monolithic diamond prism ATR crystal is available.

For making contact with solid and powder samples, the MVP-Pro™ is equipped with a built-in pressure applicator with slip-clutch for consistent and reproducible results. For more fragile ATR crystals, i.e. ZnSe and Ge, a compatible low-torque slip-clutch is available. For more precise force measurements, a pressure applicator with built-in force sensor and display is offered.

Heatable sampling plates are also available for operation up to 200°C with diamond and ZnSe or up to 100°C with a Ge ATR crystal. The Heatable Sampling Plates feature a K-type thermocouple and cartridge heater. These are intended for operation with the Harrick Automatic Temperature Controller. For easy sampling of powders and liquids, an optional Powder Retainer and Flow-Through Liquid Cell are available. Both of these sampling accessories attach to the sampling plates and are sealed with a Viton o-ring. The Powder Retainer is designed to contain the powder in a restricted volume and requires approx. 0.1 ml for operation. The Flow-Through Liquid Cell is a micro-cell with a volume of approx. 0.15 ml and features Luer Lok fittings for introduction of the sample.

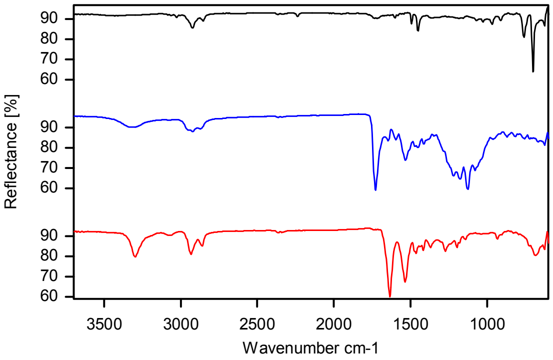


Figure 1. ATR Spectra of a Painting Pad Showing the Stiff Backer (top), the Compressible Sponge (middle) and the Soft Bristles (bottom).

Representative spectra recorded are shown in Figures 1 through 4. Figure 1 shows the ATR spectra of various parts of a Shur-Line Trim and Touch-Up Pad for painting. As seen in the spectra, all three components have different compositions.

Figure 2 shows the spectra from two parts of a traditional paint brush. Spectra from the hard plastic handle and a clump of the bristles are both readily measured with the MVP-Pro™.

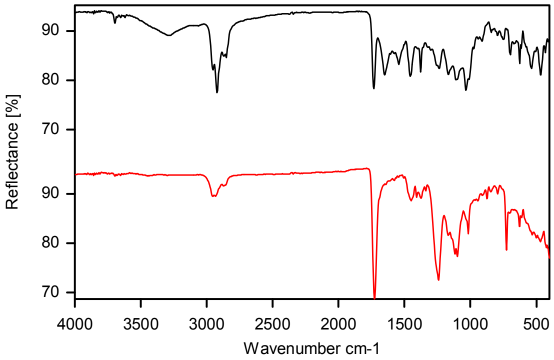


Figure 2. Anatomy of a Paint Brush: ATR Spectra of the Plastic Handle (top) and Bristles (bottom).

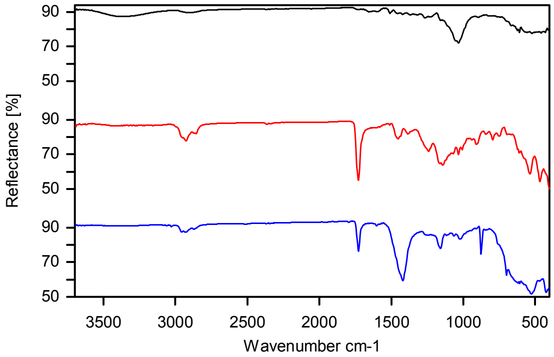


Figure 3. ATR Spectra of Wooden Paint Stirrer Unused (top), Covered with Dry Brown Paint (middle) and Covered with Dry White Paint (bottom).

Figure 3 shows the spectra of the paint stirrer and dried paint thereon. Spectral differences are clearly visible between the two types of paint and their substrate.

Figure 4 shows Zinsser Primer Sealer Stain Killer Bond Coat. The changes in the relative intensities of the bands at 3000 cm^{-1} and those at 1400 cm^{-1} are indicative of the evaporation of the organic solvent used in the primer.

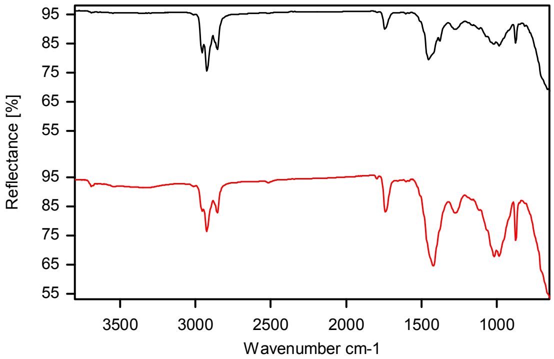


Figure 4. ATR spectra of Oil-Based Primer Wet (top) and Dried Residue (bottom).

The MVP-Pro™ is perfect for routine analysis of a variety of materials. Liquids, pastes, powders and solids are all readily examined.

OMNI-DIFF™ and OMNI-SPEC™

The Omni-Diff™ and Omni-Spec™ are fiber optic probes designed to interface to virtually any spectrometer via fiber optics. These miniature accessories come equipped with input and output SMA connectors that readily attach to a fiber optic coupler, like the Harrick FiberMate2™. Both are ideal for reflectance measurements up to 1.5 meters away from the spectrometer. The Omni-Diff™ measures the radiation diffusely reflected from the sample with optical elimination of the specular component. The Omni-Spec™ examines specular reflectance at a 45º incident angle. The Omni-Diff™ and Omni-Spec™ are offered with a selection of fiber optics for use in the UV-VIS, NIR and mid-IR. A digital imaging system is also available for photographic documentation of the sample.

APPLICATIONS

- Reflectance measurements of solids which are too large to fit in the sample compartment.
- No sample preparation required.
- Omni-Diff™ for analysis of rough surfaced solids, contaminants and coatings.
- Omni-Spec™ for examining optical coatings, contaminants, opaque substrates and films thereon and in-line diffuse reflectance of rough-surfaced solids.
- Photographic documentation of samples undergoing reflectance measurements.

FEATURES

- Compact and convenient to use.
- Suitable for use from the UV to the IR. For best performance, use with an MCT detector in the IR.
- Omni-Diff™ maximizes the collection of the diffusely reflected light, while minimizing the specular component.
- Omni-Spec™ features a 45º nominal incident angle.
- Two standard 950 SMA connectors for connecting suitable fiber optic cables.
- Optical fiber optics available in 1.5m long sets:
  - UV-VIS/Near-IR fiber optics for use from 350nm to 2250nm.
  - Near/Mid-IR fiber optics for use from 6500cm^{-1} to 2240cm^{-1} and 2050cm^{-1} to 1000cm^{-1}.
  - Mid-IR fiber optics for use from 2000cm^{-1} to 600 cm^{-1}.
- Optional video imaging and illumination system:
  - Magnifies the image for easy viewing.
  - Directly views the sampling surface.
  - Includes software for illumination, real-time viewing and storing the images.
  - USB2 compatible.
  - CE marked.
- Optional references for Mid-IR and UV-VIS/Near-IR studies.

INCLUDES

- Omni-Diff™ or Omni-Spec™
- Use with a fiber optic coupler, such as the Harrick FiberMate2™, and sets of two 1.5m fibers offered separately.

ORDERING INFORMATION

	CATALOG NO.
Omni-Diff™	OMN-DIF
Omni-Spec™	OMN-SPC
UV-VIS/Near-IR Fiber Set (350nm to 2250nm)	OMN-F-UVN
Near/Mid- IR Fiber Set (CIR fibers, 6500cm^{-1} to 2240cm^{-1} and 2050cm^{-1} to 1000cm^{-1})	OMN-F-CIR
Mid-IR Fiber Set (PIR fibers, 2000cm^{-1} to 600cm^{-1})	OMN-F-PIR

OPTIONS AND REPLACEMENT PARTS

Digital Camera for the Omni-Diff™	OMN-CAM
Reference Fixture (includes sandblasted Al diffuse reference, spectralon reference and front-surface Al mirror)	OMN-REF
FiberMate2	FM2-XXX*

**XXX indicates spectrometer make and model*

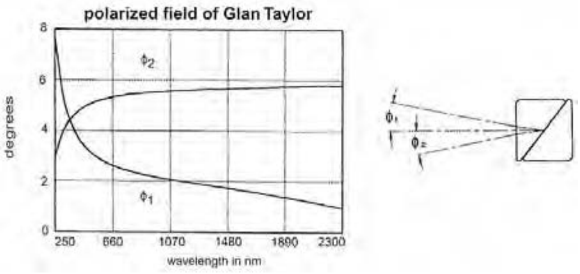
GLAN TAYLOR POLARIZER

Our Glan-Taylor Polarizer has a wavelength range from 250 nm to 2300 nm. This efficient UV-VIS-NIR polarizer is made of two calcite prisms assembled with an air spaced interface. The Glan-Taylor Polarizer is thin, making it perfect for applications where there are space constraints.

This polarizer is ideal for removing interference fringes from transmission spectra recorded at Brewster's angle and for obtaining optimum sensitivity for reflectance measurements

FEATURES

- Wavelength range: 250 nm to 2300 nm.
- Made from UV selected calcite.
- Extinction Ratio: <5x10^{-5}.
- Useful Field Angle (see graph)
- Beam Deviation: 0º5'.
- Clear Aperture: 12 mm.
- Slide plate mounted in a fully rotatable holder with an angular scale.
- Slide Plate Mounted. Length in the beam direction: 0.93 mm.



ORDERING INFORMATION

	CATALOG NO.
Glan Taylor Polarizer	PGT-S1V

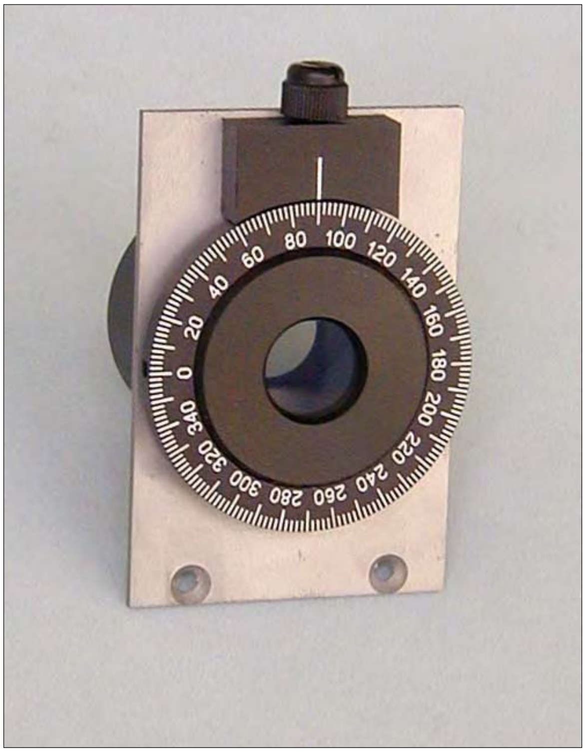
GLAN THOMPSON POLARIZER

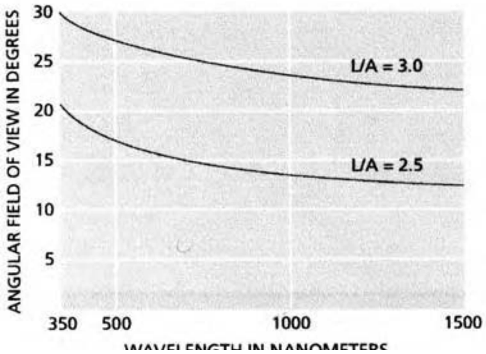
Our Glan-Thompson Polarizer has a wavelength range from 250 nm to 2300 nm. This UV-VIS-NIR polarizer is recommended for low-to-medium power applications (up to 2W cw) requiring a large field of view and the highest degree of polarization purity. The Glan-Thompson Polarizer is made from two calcite elements cemented together to form a prism. This configuration reduces its power-handling capability but results in an increased field of view. The angular field of view of the polarizer is asymmetrical about the mechanical axis and varies as a function of wavelength as shown below. The rejected (ordinary) beam is absorbed by the prism housing.

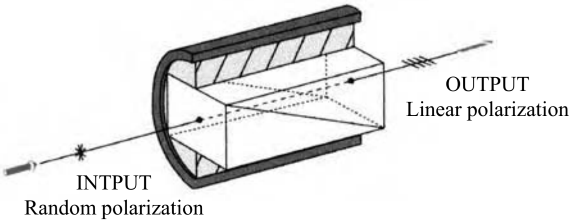
This polarizer is ideal for removing interference fringes from transmission spectra recorded at Brewster's angle and for obtaining optimum sensitivity for reflectance measurements.

FEATURES

- ► Wavelength range: 250 nm to 2300 nm.
- ► Made from calcite.
- ► Transmission (Ratio of Total Output to Total Unpolarized Input): ½ $(k_1 + k_2) = 36\%$.
- ► Extinction Ratio: $<1 \times 10^{-5}$.
- ► Useful Field Angle (see graph)
- ► Maximum Operating Temperature: 60ºC.
- ► Clear Aperture: 15x15 mm.
- ► Slide plate mounted in a fully rotatable holder with an angular scale.
- ► Length in the beam direction: 1.75"







ORDERING INFORMATION

	CATALOG NO.
Glan-Thompson Polarizer	PTH-SMP

REFLECTANCE REFERENCE

The reflectance reference provides a method for calculating the reflectivity of a material from a measured spectrum of a sample. The experimental measurement is obtained in a particular wavenumber region for s-, p- or mixed polarization. The mid-infrared reflectance reference is suitable for use from 5000cm^{-1} to 400cm^{-1}.

APPLICATIONS

- Absolute reflectance measurements of materials.
- Ideal for coatings on mirrors and reflective substrates.

FEATURES

- Ge reflectance reference.
- Unique design collects only front-surface reflectance.
- Suitable for use from 5000cm^{-1} to 400cm^{-1}.
- Designed for operation with the Seagull, Variable Angle Reflection Accessory, Near-Normal Reflectance Accessory and External Specular Reflection Accessory.
- CD with an Excel spreadsheet configured to:
  - Calculate the reflectivity of the reference at various incident angles, polarizations and wavenumbers.
  - Extract the reflectivity of the sample from experimental data.

INCLUDES

- Ge reflectance reference.
- Comes equipped to mount into the Seagull, Variable Angle Reflection Accessory, Near-Normal Reflectance Accessory and External Specular Reflection Accessory
- Instruction manual.
- CD with reflectance data and formulas in an Excel spreadsheet.

ORDERING INFORMATION

	CATALOG NO.
Mid-Infrared Reflectance Reference (Ge)	RRF-00J

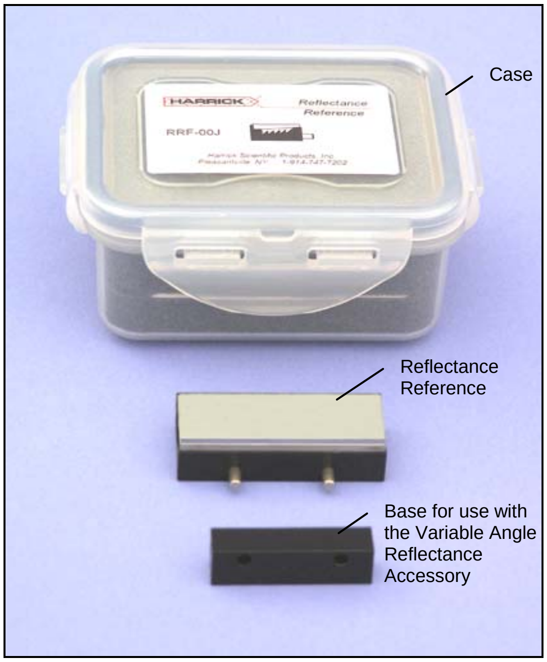


Figure 1. The Reflectance Reference.

The reflectivity of a material can be measured in one of two ways: directly, using VW absolute reflectance equipment, or indirectly, using relative reflectance equipment. The VW configuration is highly sensitive to the alignment of both the accessory and the spectrometer.

The other method is less direct. The sample spectrum is recorded relative to a reference with a well-known reflectivity under controlled experimental conditions and the reflectivity of the sample is then calculated from the experimental spectrum and the reflectivity of the reference.

Harrick's Reflectance Reference (see Figure 1) includes a specially designed optical element which provides only front-surface specular reflectance. The Reflectance Reference comes with the hardware needed to adapt it to Harrick reflection accessories and software to calculate the reflectivity of the substrate for incident angles over the useable wavelength range of the material for the s-, p- or mixed polarization. The software also can be used to extract the reflectivity of the sample from the experimental data.

Sample reflectivity data for the reference are shown in Figures 2 and 3. Figure 4 shows the reflectance spectrum of ZnSe measured relative to the reflectance reference and the resulting calculated absolute reflectance of ZnSe. The experiemental data **were** collected at an 8cm^{-1} resolution using the Seagull at a 30º incident angle with a wire grid polarizer.

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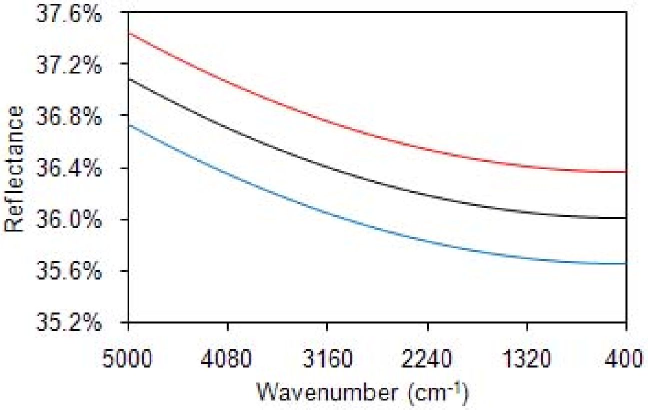


Figure 2. Theoretical Reflectivity of Ge at a near-normal (8º) incident angle for s (red), p (blue) and mixed (black) polarization.

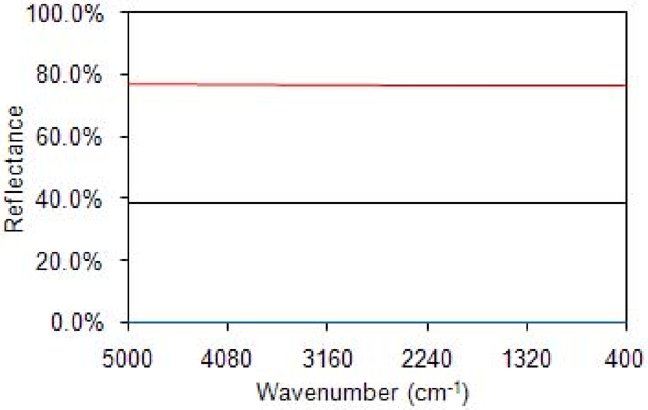


Figure 3. Theoretical Reflectivity of Ge at a grazing (75º) incident angle for s (red), p (blue) and mixed (black) polarization.

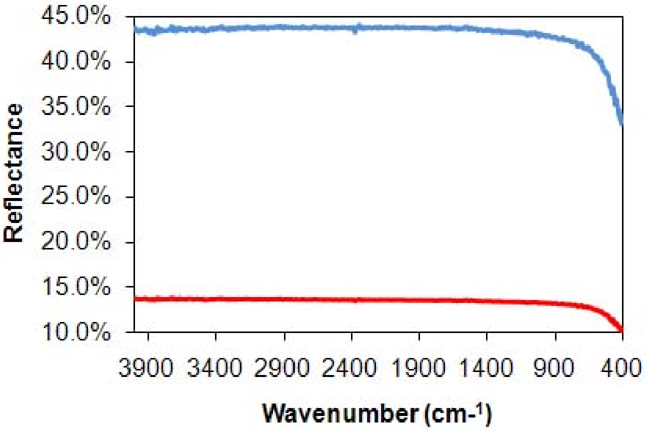


Figure 4. The reflectance of ZnSe extracted (red) from an experimental spectrum (blue) at a 30º incident angle with p-polarization.

The RangeIR$^{\mathrm{TM}}$ Liquid Analyzer

The RangeIR$^{\mathrm{TM}}$ is a powerful tool for the analysis of liquids, pastes, and gels under controlled temperatures up to 175°C. The unique design allows the safe use of the sample temperature itself in this control, an invaluable feature for rigorous studies. Reactants and reaction products can be monitored as a function of temperature. In addition, the RangeIR™ can be used for sensitive measurements on dissolved or suspended species, by using the elevated temperature to volatilize an interfering liquid.

APPLICATIONS

- Routine quantitative and qualitative measurements on temperature-controlled liquids, pastes, and gels.
- Real-time *in situ* monitoring of reactants and reaction products in liquids as a function of temperature.
- Analysis of dissolved or suspended species in interfering liquids.

FEATURES

- Reproducible measurements.
- Convenient trough for containing liquid samples.
- Fixed 45° single reflection horizontal ATR configuration.
- SuperCharged™ ZnSe ATR Crystal or Gemanium ATR Crystal.
- Exchangeable ATR crystal.
- Simple to align and use.
- Safe, low-voltage heaters permit operation up to 175°C (Note: Germanium becomes optically opaque at temperatures exceeding 100°C.)
- K-type thermocouple directly measures the sample temperature and flips out of the way for easy cleaning of the ATR crystal.
- Secondary K-type thermocouple monitors the crystal temperature, permitting the cascade temperature regulation required as a fail-safe for high temperature operation.
- PermaPurge™ for rapid purging of the FTIR system.
- Requires the Low Voltage Temperature Controller for precise and safe regulation of the sample temperature, with options for direct, cascade, and computer control.
- Available in a specialized configuration for *trans* fat analysis (see Harrick's FatIR$^{\mathrm{TM}}$ System data sheet).

Replacement ATR Crystals	
Material	Catalog No.
ZnSe	FAS-ATR-M
Ge	FAS-ATR-J

INCLUDES

- SuperCharged™ ZnSe mounted ATR crystal or Ge ATR mounted crystal.
- Two imbedded cartridge heaters.
- Two K-type thermocouples.
- Mating and PermaPurge™ hardware for the specified FT-IR spectrometer.

ORDERING INFORMATION

	CATALOG NO.
RangeIR$^{\mathrm{TM}}$ with ZnSe ATR Crystal	RAN-M-XXX
RangeIR$^{\mathrm{TM}}$ with Ge ATR Crystal	RAN-J-XXX

	110V	*220/*240V
FatIR™ System (includes the RangeIR with ZnSe ATR and Temperature Controller)	FAT-1-XXX	FAT-2-XXX
Automatic Temperature Controller, 110V	ATC-024-1	ATC-024-2

REPLACEMENT PARTS

Top Plate Assembly, ZnSe	RAN-TOP-M
Top Plate Assembly, Ge	RAN-TOP-J
Viton Gasket	RAN-GSK
K-Type Thermocouple	008-144
Heater Assembly (two heaters plus connector)	RAN-HTR
Cartridge Heater, 24V	HTRS-18

The RangeIR™ Liquid Analyzer is optimized for fast, easy, and accurate infrared measurements of liquids, pastes, and gels at controlled temperatures up to 175°C. The RangeIR™ may also be used to study reactants and reaction products in liquids at elevated temperatures. Further, this equipment is extremely convenient for studying dissolved or suspended species by using elevated temperatures to volatilize an otherwise interfering solvent.

The RangeIR™ is a trough-style single-reflection ATR FTIR accessory. It requires the use of Harrick's Low-Voltage Temperature Controller and is based on our time-proven FastIR™. With the SuperCharged™ ZnSe crystal, this design has the highest optical throughput (>85%) of any ATR accessory. Its simple optical configuration, consisting of only two fixed flat mirrors and a triangular prism ATR crystal, provides optimum performance without the need for sensitive optical alignment adjustments. The design is inherently stable, an essential feature for operation of a system used at the extreme temperatures simulating processing conditions.

The RangeIR™ utilizes a trough configuration to simplify sample introduction and crystal cleaning. This configuration is required to contain substances which have low viscosities at elevated temperatures.

The two 24V cartridge heaters, in thermal contact with the crystal mounting plate, are operated by our automatic low-voltage temperature controller. Two thermocouples are provided, one in the sample and one in contact with the ATR crystal, to enable cascade operation of the temperature controller. Cascade control is necessary at higher temperatures because the lag between the heater and sample temperatures can cause the controller to otherwise overshoot the set point, potentially resulting in damage.

When studying heat-induced changes in liquids, it is important to precisely and accurately know the sample temperature. The RangeIR™ measures and controls the sample temperature directly, instead of recording the crystal or heater temperature, as is commonly done by other ATR accessories. This is important because the sample is exposed to a different thermal environment and hence is at a slightly lower temperature. With the RangeIR™, the sample thermocouple is placed directly into the sample, providing the true value (and control) desired.

The RangeIR™ incorporates easily adjustable and highly stable kinematic mounting, for optimum alignment in the particular spectrometer being used. In addition, the RangeIR™ is equipped with Harrick's patented PermaPurge™. This allows samples to be exchanged without interrupting the purge of the FTIR spectrometer, which, in turn, greatly enhances sample throughput.

THE REFRACTOR2™

The Refractor2™ is a unique attachment for grazing incidence specular reflectance studies. The Refractor2™ incorporates two SuperCharged™ wedged windows to refract the beam to and from the sample. It also includes a pre-mounted, internal Si polarizing plate for enhanced spectral contrast. This unique design results in a compact attachment and avoids the energy losses associated with the plane mirrors typically employed.

APPLICATIONS

- Ideal for recording spectra of thin films on metal and semiconductor substrates.
- Well suited for laboratory and QC applications.

FEATURES

- Low cost.
- Unobstructed, horizontal sampling surface for high sample throughput.
- Fixed 75° incident angle and built-in, removable polarizer provides high spectral sensitivity.
- Two SuperCharged™ ZnSe wedged windows refract the beam to and from the sample. KRS-5 windows available on special order.
- Compact.
- SuperCharged™ for optimal energy throughput.
- Easy to align and use.
- PermaPurge™ for rapid sample exchange without interrupting the purge.
- Removable PermaPurge™ assembly - accommodates samples up to 4.75" wide when installed and even larger when removed.

INCLUDES

- Two SuperCharged™ ZnSe wedged windows.
- Si polarizing plate.
- Alignment mirror.
- Mating hardware for the specified spectrometer.

ORDERING INFORMATION

	CATALOG NO.
Refractor2™	RG2-XXX

REPLACEMENT PARTS

Silicon Polarizing Plate	RGA-000
SuperCharged™ ZnSe Wedged Window	RGA-001

The Refractor2™ is a simple, low cost, in-line grazing incidence attachment. It is ideal for recording spectra of thin films on metal substrates. The angle of incidence is fixed at 75°. Excellent spectral contrast results. Large panel samples can be easily analyzed, since the sampling surface is above all of the optical components.

Conventional in-line grazing incidence attachments have two major drawbacks. First, the short plane mirrors employed do not totally intercept the beam of the spectrometer, resulting in a significant loss in optical throughput. Second, an external polarizer is typically required. Such an external polarizer is expensive. Both of these drawbacks are overcome with the Refractor2™.

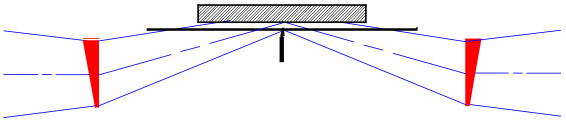


Figure 1. The Optical Drawing of the Refractor2™

In the Refractor2™, the optical beam is deflected to and from the sample via wedged ZnSe windows (see Figure 1). This design results in a compact accessory with optimal throughput. The two ZnSe windows are SuperCharged™, which nearly doubles the performance of the Refractor2™. A single Brewster's angle silicon polarizer plate is located below the sampling plane to provide the required parallel polarization.

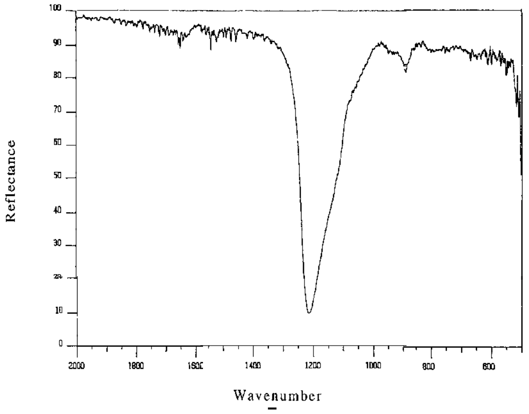


Figure 2. Spectrum of SiO_2 on an Al Substrate

Figure 2 shows the spectrum of a silicon dioxide film, approximately 500Å thick, on an aluminum substrate. In Figure 3, the spectrum of a 100Å layer of silicone lubricant on an aluminum substrate is given. Here, an external wire-grid polarizer is used in place of the internal silicon polarizer.

The Refractor2™ is supplied with all mounting hardware required to install it directly in the spectrometer and is compatible with most FTIR instruments. Alignment is fast and simple. For analyses that require spectral information at frequencies below 500 cm^{-1}, replacement KRS-5 windows are available on a special order. A reaction chamber model of the Refractor2™ is also available, on special order, for analysis of samples in a vacuum or pressurized environment.

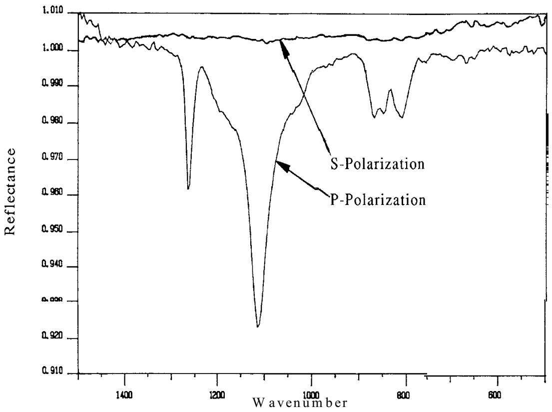


Figure 3. Spectra of Silicone Lubricant on an Al Substrate.

THE REFRACTOR REACTOR

The Refractor™ Reactor is a valuable tool for grazing angle studies of reactions on metal surfaces and on thin films on metal substrates. Samples can be heated to 400°C and pressurized up to 2 ATM. The Refractor™ Reactor incorporates two wedged windows to refract the beam to and from the sample. This unique design results in a compact reaction chamber and avoids the energy losses associated with the short plane mirrors typically employed in grazing angle accessories.

APPLICATIONS

- Perfect for studying reaction on metal surfaces and on thin films on metal substrates.

FEATURES

- 75º grazing incident angle for high sensitivity to films on metallic substrates.
- Incorporates two Supercharged™ ZnSe wedged windows to refract the beam to and from the sample.
- Ideal for samples up to 1"x2"x⅛".
- Heatable up to 400°C (under vacuum) for studying high temperature reactions.
- Operable at pressures from 10^{-4} torr to 2 ATM.
- Manufactured from chemically resistant 316 stainless steel.
- High efficiency.
- Designed for in-line use of a wire grid polarizer.

INCLUDES

- Two ZnSe wedged windows.
- Alignment mirror.
- Mating hardware for the specified spectrometer.

ORDERING INFORMATION

	CATALOG NO.
Refractor Reactor (24V)	RGR-XXX-3
OPTIONS AND REPLACMENT PARTS	
Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220/240V input, 24V output	ATC-024-2
Supercharged™ ZnSe Wedged Window	RGA-001
Heater Assembly, 24V	RGR-HTR
Heater, 24V, 96W	HTRS-19
Wire Grid Polarizer	PWG-U1R
Vacuum Pump, 110V	VPE-001
Vacuum Pump, 220/240V	VPE-002

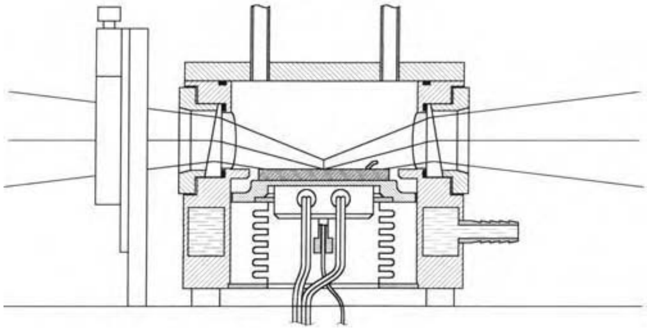


Figure 1. The Refractor Reactor.

The Reactor Refractor is a reaction chamber for in-line grazing incidence studies. It is ideal for recording spectra of thin films on metal substrates. The angle of incidence is fixed at 75 degrees and can accommodate samples up to 1"x2"x⌨" thick. It can be heated up to 400°C under vacuum, evacuated to 10^{-4} torr, and pressurized up to 2 ATM with the standard ZnSe windows. Excellent spectral contrast results.

Conventional in-line grazing incidence attachments have a significant drawback. The short plane mirrors employed do not totally intercept the beam of the spectrometer, resulting in a significant loss in optical throughput. In the Refractor, these losses do not occur, since the optical beam is deflected (15°) to and from the sample via wedged (10°) ZnSe windows.

The Refractor Reactor has two ports for flowing reactant over the sample, evacuating the sample, and pressurizing the sample with a reactant. Heating cartridges are imbedded below the sample and two thermocouples are supplied to measure the sample and heater temperatures. For precise temperature control, the heater and thermocouples should be connected to an Automatic Temperature Controller (Harrick's ATC-001 or ATC-002).

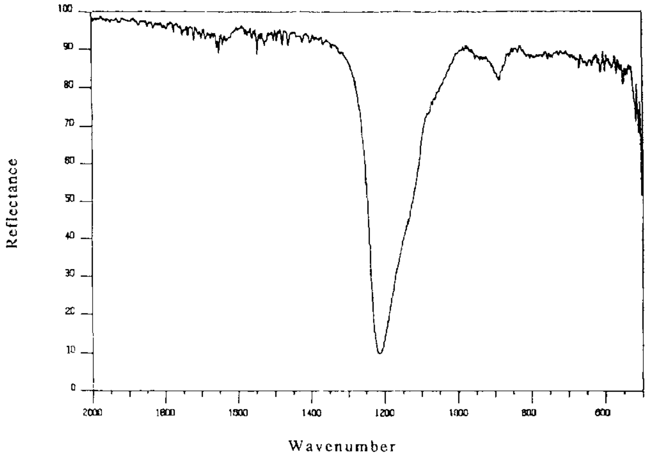


Figure 2. External Reflectance of SiO_2 on Aluminum.

Figure 2 shows the spectrum of a silicon dioxide film, approximately 500 Angstroms thick, on an aluminum substrate. In Figure 3, the spectrum of a 100 Angstrom layer of silicone lubricant on an aluminum substrate is given. Here, an external wire-grid polarizer is used to generate P-polarized incident radiation.

The Refractor is supplied with all mounting hardware required to install it directly in the spectrometer and is compatible with most center focus FTIR instruments. Alignment is fast and simple. For measurements that require spectral information at frequencies below 500cm^{-1}, replacement KRS-5 windows are available on special order.

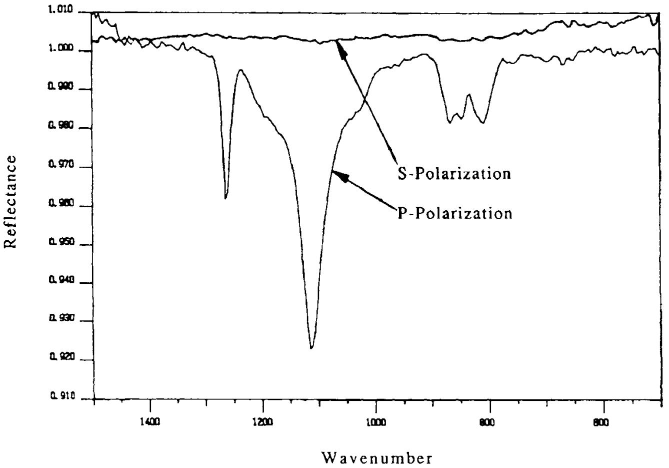


Figure 3. Grazing Angle Spectrum of Silicon Lubricant on Aluminum.

THE SEAGULL™

The Seagull™* is a powerful attachment for examining numerous types of samples using a variety of reflection techniques. This unique accessory can be easily adapted for external, internal or diffuse reflection spectroscopy. In addition, it can be operated over a broad range of incident angles without misaligning the system, without defocusing the incident radiation, and without changing the polarization of the incident beam. This makes the Seagull™ extremely versatile and flexible. The Seagull™ also incorporates Harrick's PermaPurge™ feature which permits changing the angle of incidence without interrupting the purge of the spectrometer.

APPLICATIONS

- Variable angle external and internal reflectance.
- Ideal for examining a wide variety of samples, including powders, optical coatings, opaque substrates, films on opaque substrates, and slightly curved solids.
- Excellent tool for studying liquids and films on liquids by external reflectance, for depth profiling, and for determining optical constants.

FEATURES

- Multi-purpose, variable angle reflection attachment.
- Internal, external, and diffuse reflectance capabilities.
- Continuously variable angle of incidence, from 5° to 85°.
- Focuses the incident beam on the sample for all incident angles.
- Always centers the incident beam on the same area of the sample.
- Maintains polarization for angles.
- No realignment of the accessory or repositioning of the sample required when varying the incident angle.
- High optical throughput.
- PermaPurge™ permits changes in angle without interrupting the purge of the system.
- Ming-Sung™ATR Rotator for studying oriented polymers available separately.
- Convenient liquid, powder, and heated sampling accessories available.

HEMISPHERES	
Material	Catalog No.
Ge	EJ6XBB
ZnSe	EM6XBB
Si	EE6XBB

INCLUDES

- Sample holder for external reflectance with a sample cup for diffuse reflectance.
- Sample pressure plate.
- Mating hardware for the specified spectrometer.
- Alignment mirror.
- Optional compatible polarizer and ATR kit available.

*Patented.

ORDERING INFORMATION

	CATALOG NO.
Seagull™	SEA-XXX
Seagull™ Research Package (includes Seagull™, internal reflection kit, wire grid polarizer, and SOS™ software)	SRP-XXX
OPTIONS	
Flow-Through Liquid Cell	SEA-FLC
Heatable Flow-Through Liquid ATR Cell, 24V	SEA-HLC-3
Heated Sample Stage, 24V	SEA-HOT-3
ATR Kit (includes ZnSe hemisphere and holder, pressure plate, and clamp)	IRK-SEA
Ming-Sung™ ATR Rotator (includes holder with calibrated dial for setting the angle, pressure plate and clamp)	MNP-SEA
Powder Sample Holder	SEA-PSH
SOS™ Software Package	SOS-102
Torque Screwdriver	PTW-SXX
Trough Liquid Cell	SEA-TLC
Two Position Wire Grid Polarizer, KRS-5 Substrate	PWG-SEA

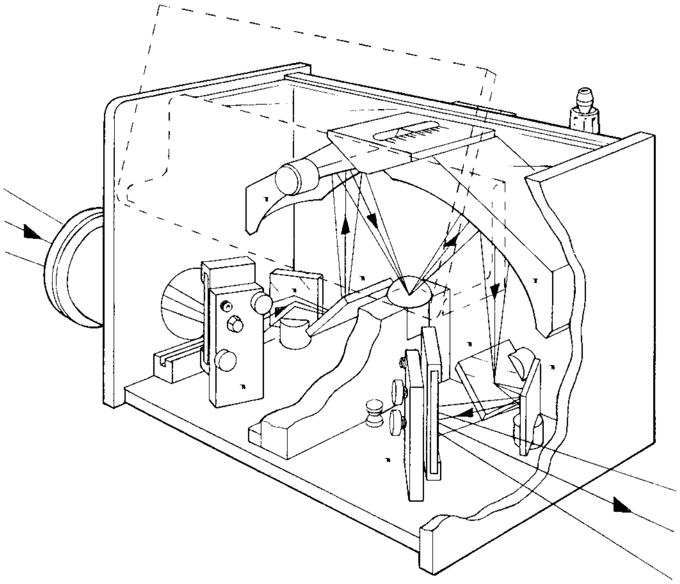


Figure 1. The Seagull™ variable angle reflection attachment.

Variable angle reflection spectroscopy is becoming increasingly important. Certain samples such as powders, opaque substances, films on opaque substrates, and films on liquids are tedious or practically impossible to analyze with conventional transmission spectroscopic equipment. The analysis of such samples with reflection spectroscopy, however, is straightforward.

The Seagull™ is shown in Figure 1. Three mirrors (M1, M2, and M3) direct the beam to an ellipsoid (E1) that focuses the beam onto the sample. The sample reflects the light onto a second ellipsoid (E2). Ellipsoid E2 reflects the beam from mirrors M4, M5, and M6 onto the detector of the spectrometer. Mirrors M3 and M4 are coupled to rotate together, in mirror image fashion. This directs the beam to and from different portions of the ellipsoids, changing the incident angle of the beam on the sample. This configuration automatically preserves the optical alignment for any selected angle of incidence.

The Seagull™ is ideal for examining surface and optical coatings, for measuring optical constants, and for liquid or electrochemical analysis with the appropriate cells. Sampling accessories are available for convenient sampling of a variety of types of samples.

For ATR studies, the Seagull can be equipped with our Internal Reflection Kit (SEA-IRK). This kit includes a ZnSe hemispherical ATR crystal mounted in a holder that slides into the Seagull in place of the specular reflectance holder. The holder has a built-in pressure applicator. The kit also includes a compatible pressure plate.

For specular reflectance of liquids, the trough liquid (SEA-TLC) cell provides an open reservoir to contain the sample. The trough readily mounts onto the standard specular reflectance holder.

For heated studies, a sample stage (SEA-HOT) is available that can be heated to 150°C. This stage can be used to support samples for either ATR or specular reflectance measurements.

For ATR studies of liquids, a flow-through liquid cell (SEA-FLC) is offered. This cell is equipped with two luer ports and seals against the ATR crystal with an o-ring. The cell can be used for both flow-through measurements and for applications requiring a sealed liquid cell. A temperature-controlled version of the cell (SEA-HLC) is also available for examining liquids from room temperature to 150°C.

For ATR measurements of powders, a convenient powder holder (SEA-PSH) is available. This holder provides a spring-loaded removable cup that minimizes spillage of the sample while optimizing contact with the ATR crystal.

For studying oriented polymers and determining ordering in systems with molecules on inorganic surfaces, our Ming-Sung™ ATR Rotator (MNP-SEA) is available. This rotator is a specially designed crystal holder that permits a 0° to 90° rotation of the sample without dismounting the sample. It is compatible with the hemispheres that can be purchased separately or with the Internal Reflection Kit.

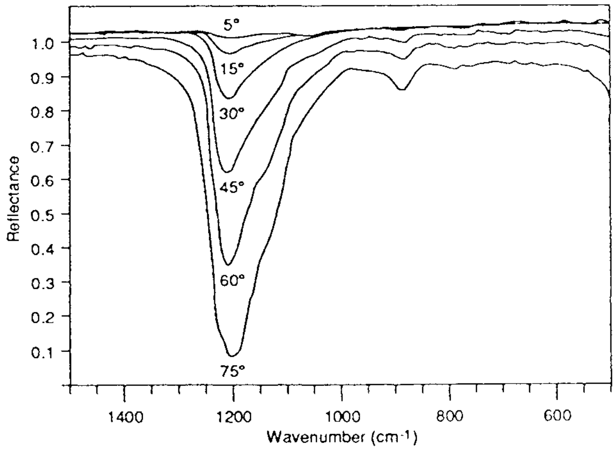


Figure 2. External reflectance of 0.05 µm SiO_2 on aluminum (p-polarization).

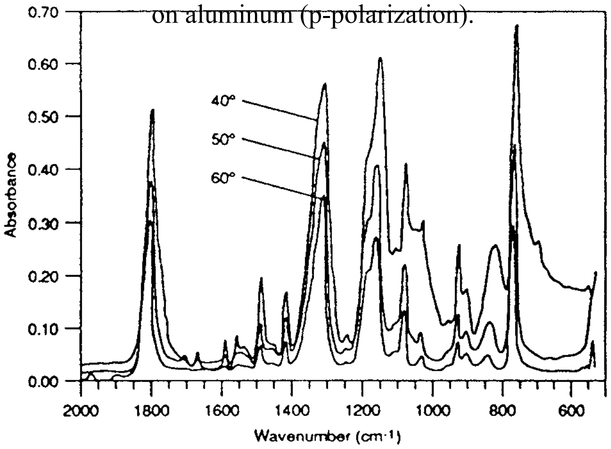


Figure 3. Internal reflectance of a 2.5 µm mylar film on a silicon substrate.

In addition, a model of the Seagull™ with independently variable angles of incidence and collection is available on special order.

The versatility of the Seagull™ makes it a powerful tool for the analysis of a number of different sample types using a variety of reflection techniques. Representative spectra are shown in Figures 2 through 5.

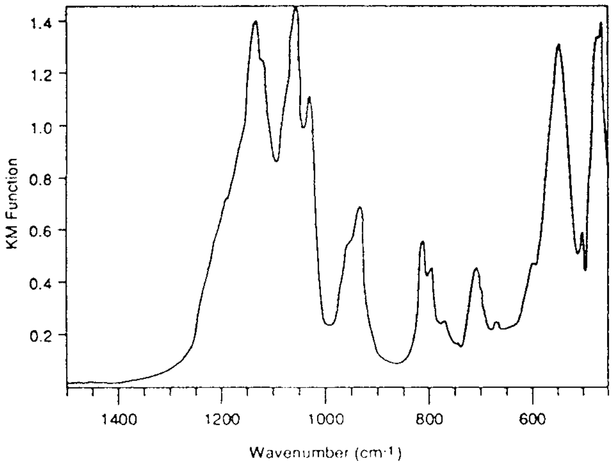


Figure 4. Diffuse reflectance of kaolinite diluted in KBr at a 15° angle of incidence.

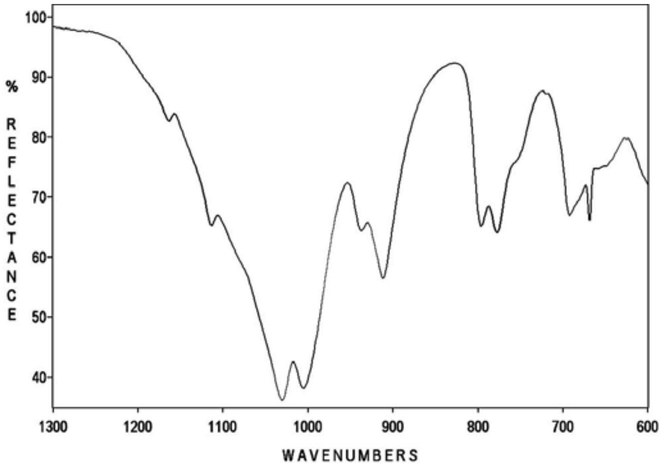


Figure 5. ATR spectrum of 10 µm silica powder, recorded at a 45° angle of incidence using the powder sample holder.

THE SPLITPEA™

Harrick's SplitPea™[1] is a horizontal internal reflection (ATR) accessory with the smallest sampling area of any ATR accessory - less than 250 µm in diameter for its Si ATR crystal. The SplitPea™ is configured to apply localized, measured pressure to produce superior contact between the sample and the ATR crystal. This makes the SplitPea™ ideal for quick and easy examination of a wide range of samples. These include: hard samples, like paint chips and combinatorial chemistry substrates; small samples, such as individual fibers and nanoliters of liquids; large samples, such as transparency film and defects thereon. For positioning samples on the sampling area, a 50X viewing microscope, the View-ThruPress™[2] is available. In addition to making ATR nanosampling simple and straightforward, the SplitPea™ features PermaPurge™[3] for rapid sample and crystal exchange without interrupting the purge of the spectrometer. The Harrick SplitPea™ is an innovative alternative to infrared microscopes, beam condensers, and diamond cells.

APPLICATIONS

- Uniquely suitable for studying optically thick, hard samples; slightly curved samples; fibers; paint chips; nanoliters of liquids and pastes; defects on large panels; and droplets of corrosive liquids.
- Invaluable for forensic samples and combinatorial chemistry samples such as SynPhase™[4] Lanterns and Crowns.

FEATURES

- Internal and external reflection capabilities provide application versatility.
- High sample throughput due to little or no sample preparation.
- Usually retains sample integrity.
- Small sampling area - less than 250 µm in diameter for ATR with a silicon crystal.
- Minimizes stray light due to the small sampling area.
- Inert internal reflection elements available for use from the Near IR to the Far IR.
- Calibrated pressure applicator for reproducible ATR measurements.
- Designed for optimal contact between the ATR crystal and hard surface solids.
- Flip-up, streamlined pressure applicator for easy access to sampling area
- High energy throughput with DTGS detectors.
- Harrick's exclusive PermaPurge™ allows rapid sample and crystal exchange without interrupting the purge of the system.
- Spill-resistant cover.
- Upgrade to a 50X View-ThruPress™ for easier viewing of the sampling area.
- Options include Flow-Through Liquid Cell and Heatable Sampling Plates for operation to 200°C.

INCLUDES

- Two ATR holders with mounted Si hemispheres or one ATR holder with a mounted diamond hemisphere.
- Sample holder adapter for studying powders by internal reflectance.
- External reflection sample holder and alignment mirror.
- Mating hardware for the specified spectrometer.

[1] U. S. Patent 5,210,418
[2] U. S. Patent 5,308,983
[3] U. S. Patent 5,177,561
[4] Registered trademark of Mimotopes Pty. Ltd., Australia.

ORDERING INFORMATION

	CATALOG NO.
SplitPea™ with ViewThruPress™	UNP-XXX
SplitPea™	UNS-XXX
Meridian™ Diamond SplitPea™ with ViewThruPress™	MER-P-XXX
Meridian™ Diamond SplitPea™	MER-XXX
ViewThruPress™ Upgrade	UNS-MIC

OPTIONS AND REPLACEMENT PARTS.

	CATALOG NO.
Flow-Through Liquid Cell with Luer Fittings	UNS-LCF
Liquid Cell O-Ring	ORV-0015
Powder Adapter O-Ring	ORV-012
Mounted Crystals: Si	UNS-ATR-0E
Diamond	UNS-ATR-0W
ZnSe	UNS-ATR-0M

	CATALOG NO.
Mounted Crystals: Ge	UNS-ATR-0J
ZnS	UNS-ATR-0I
Heatable Sampling Plates:	
Diamond	UNS-HOT-0W
Si	UNS-HOT-0E
Ge	UNS-HOT-0J

This horizontal reflection accessory is ideal for analyzing optically thick, hard materials. Samples, which are difficult to analyze by conventional spectroscopic methods, such as fibers, paints, and microliters of liquids or pastes, are easily examined with the SplitPea™.

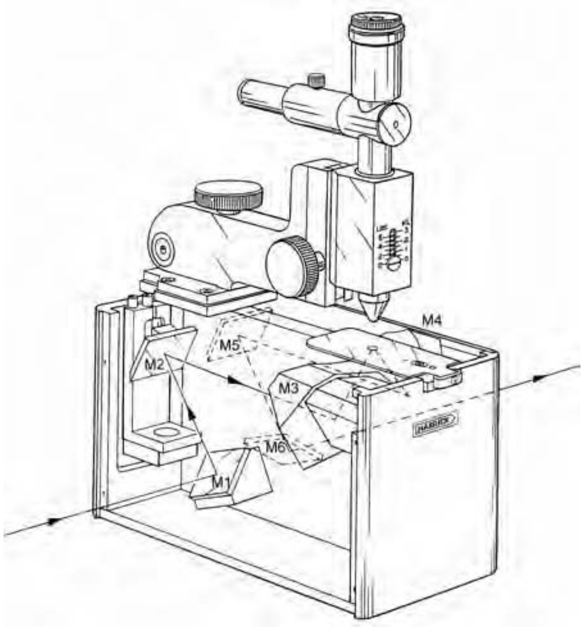


Figure 1. The SplitPea™.

The SplitPea™ can be configured for internal or external reflectance, simply by changing the sample holder. In its internal reflection mode, the SplitPea™ features a removable crystal/sample holder for easy sample insertion and crystal cleaning. For the external reflection and pre-alignment, the SplitPea™ features a removable sample holder and reference mirror. The SplitPea™ is enclosed in a purgable box for rapid sample exchange without interrupting the purge of the system.

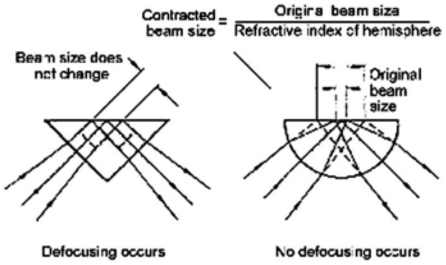


Figure 2. Focusing Effects of a Hemisphere.

An illustration of the SplitPea™ is shown in Figure 1. Two mirrors, M1 and M2, direct the beam to an ellipsoidal mirror, M3, which focuses the light onto the sample. The radiation reflected from the sample is collected by a second ellipsoid, M4. Mirrors M5 and M6 direct radiation reflected from M4 to the detector of the spectrometer. This configuration provides a six times linear reduction of the source image on the sampling surface.

For internal reflection, the SplitPea™ comes with two silicon internal reflection elements (IRE). Silicon is an excellent material for the IRE of the SplitPea™ since it is extremely inert, allowing the analysis of even highly corrosive materials. In addition, it is a very hard material. This permits the application of high clamping pressures to ensure good contact between the IRE and the sample. Generally, lower pressures are required to achieve the good contact needed in internal reflectance than are needed to flatten the sample for transmission studies. Thus fewer chemical and physical changes will be induced in the sample with the SplitPea™ than with a high pressure diamond anvil cell.

The Si IRE is a 3-mm diameter hemisphere. This further focuses the incident radiation onto the sample (see Figure 2), providing an additional 3.4 times linear reduction of the source image. For such a short pathlength, Si is virtually transparent in the far and mid-infrared. The Si IRE is beveled on the edge of its flat surface to provide a sampling area slightly larger than the 150-200 µm diameter hot spot on the crystal. This makes it easier to position small samples and maximize the clamping pressure. Because of the small size of this island, the pressure plate applies localized pressure to the sample improving contact between the sample and the IRE and allowing high contact pressures to be achieved.

For applications versatility, ZnSe, Ge, and diamond IREs are also available. This IRE selection offers a variety of sampling depths of volumes.

The SplitPea™ is available with a ViewThruPress™. This configuration is recommended for analyzing samples such as fibers, spots on transparent substrates, and powders. The ViewThruPress™ provides a 50X magnification of the sampling area and features precision adjustments for aligning the viewer over the active sampling area of the IRE. Its unique design permits the sample to be viewed as it is compressed against the IRE. The ViewThruPress™ also features an independent adjustment for focusing on the image.

For liquid sampling, a flow-through liquid cell is available for use with the SplitPea™. This cell is o-ring sealed and features two luer fittings.

In addition, heatable sampling plates are available for

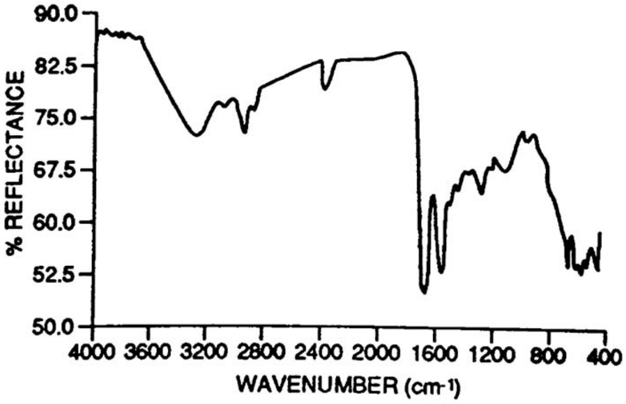


Figure 3. Internal Reflectance of a Human Hair.

sampling at temperatures up to 200°C with the diamond or Si ATR crystals and to 100°C with a Ge ATR crystal. These sampling plates feature a gasket-sealed ATR crystal, K-type thermocouple, and 24V heater. These are designed for use with the Harrick Automatic Temperature Controller.

Representative spectra recorded with the SplitPea™ are shown in Figures 3 through 6.

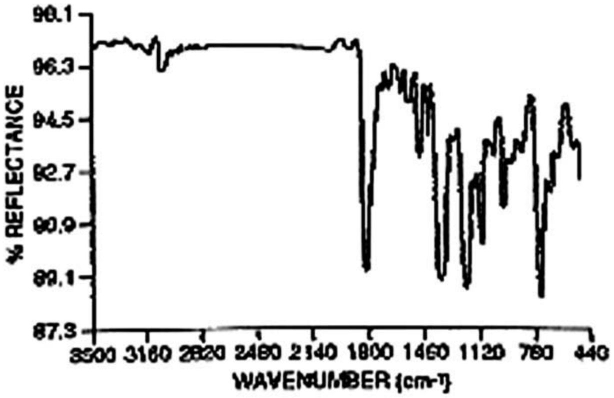


Figure 4. Internal Reflectance of a 20µm Poly(ethylene terephthalate) Fiber. ZnSe IRE.

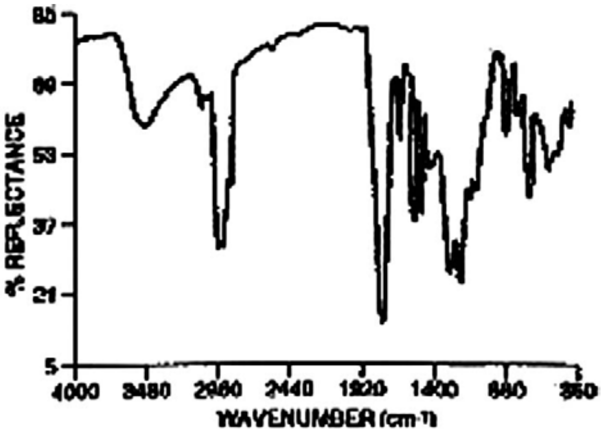


Figure 5. Internal Reflectance of a Paint Chip.

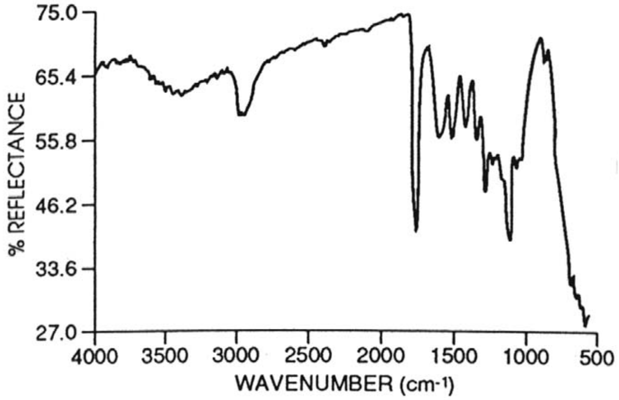


Figure 6. External Reflectance of a Lottery Ticket.

Temperature Controlled Demountable Liquid Cells

Temperature Controlled Demountable Liquid Cells are intended for examining liquids and mulls at temperatures ranging from -80°C to 260°C. Their leak-free design makes them suitable for static and flow operation. In addition, these 316 stainless steel cells feature high chemical resistance and readily exchanged pathlength spacers.

APPLICATIONS

- Infrared measurements of liquids, mulls and smears at elevated and reduced temperatures.
- Process control and laboratory applications.

FEATURES

- Variable pathlength , from 6μm to 1mm.
- Heatable to 200°C with Viton o-rings or 260°C with optional Kalrez o-rings.
- Coolable to -80°C with suitable coolant, purge and o-rings.
- Flow-through cooling conduit stabilizes the cell temperature.
- Thermally isolated cell body prevents heat transfer to the spectrometer.
- Leak-free design utilizes standard, non-drilled windows.
- Easily disassembled for cleaning and window replacement.
- Two ports permit static or flow applications.
- Chemically resistant 316 stainless steel cell body with Luer Lok or 1/8" Swagelok™ fittings.
- Sample slide plate mounted for user convenience.
- Available with an 8mm or 20mm diameter open aperture.
- Maximum operating pressure with ZnSe windows
    - 660 psi for the DLC-S13 (8 mm aperture).
    - 100 psi for the DLC-S25 (20 mm aperture).

INCLUDES

- Stainless steel cell body with Luer Lok or Swagelok™ fittings.
- Embedded K-type thermocouple, low-voltage cartridge heaters and cooling conduit.
- Viton o-rings.
- Slide plate mounting.
- One dozen Teflon spacers. One each for pathlengths of 6, 12, 25, 56, 100, 150, 250, 390, 500, 630, 750, and 950 μm.
- Order windows separately (see table).

Windows	13x2 mm	25x2 mm
Al_2O_3	WBD-U22	--
CaF_2	WFD-U22	WFD-U25
ZnS	WID-U22	WID-U25
NaCl	WLD-U22	WLD-U25
ZnSe	WMD-U22	WMD-U25
KBr	WPD-U22	WPD-U25

Assorted Spacers Sets		
	8 mm apt. (13x 2 mm)	**20 mm apt. (25 x 2 mm)**
Two each (25, 50, 100, 200 and 1000 μm)	PSB-M13	PSB-M25
One each (6, 12, 25, 56, 100, 150, 250, 390, 500, 630, 750 and 950 μm)	PSA-M13	PSA-M25

	8 mm apt. (13x 2 mm)	20 mm apt. (25 x 2 mm)
Spacers Sets (10 per package)		
15μm	MSB-015-M13	MSB-015-M25
25μm	MSB-025-M13	MSB-025-M25
50μm	MSB-050-M13	MSB-050-M25
100μm	MSB-100-M13	MSB-100-M25
200μm	MSB-200-M13	MSB-200-M25
500μm	MSB-500-M13	MSB-500-M25
1000μm	MSB-000-M13	MSB-000-M25
Spacers Sets (12 per package)		
6μ	MSP-006-M13	MSP-006-M25
12μm	MSP-012-M13	MSP-012-M25
56μm	MSP-056-M13	MSP-056-M25
150μm	MSP-150-M13	MSP-150-M25
250μm	MSP-250-M13	MSP-250-M25
390μm	MSP-390-M13	MSP-390-M25
630μm	MSP-630-M13	MSP-630-M25
750μm	MSP-750-M13	MSP-750-M25
950μm	MSP-950-M13	MSP-950-M25

ORDERING INFORMATION

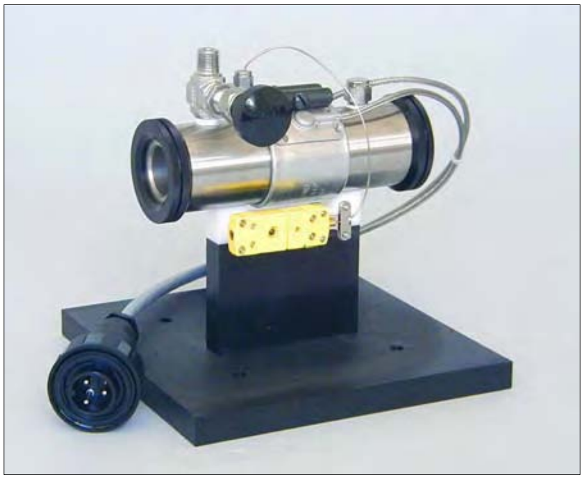
	CATALOG NO.	
	8 mm aperture (13x2 mm windows)	20 mm aperture (25x2 mm windows)
Temperature Controlled Demountable Cells, 24V		
Liquid Cell (Luer Lok fittings)	TFC-3-M13	TFC-3-M25
Flow Cell (Swagelok™ fittings	TFC-3-S13	TFC-3-S25
OPTIONS AND REPLACEMENT PARTS		
Viton O-Ring	ORV-012	ORV-020
Kalrez O-Ring	ORK-012	ORK-020
Automatic Temperature Controller, 110V input, 24V output		ATC-024-1
Automatic Temperature Controller, 220/240V input, 24V output		ATC-024-2

TEMPERATURE CONTROLLED GAS CELL

Gas cells are ideal for examining gases and gaseous mixtures for static or flow environments. The Temperature Controlled Gas Cell is excellent for both room temperature studies and heated measurements.

APPLICATIONS

- Well-suited for spectroscopic studies of gases and vapors from room temperature to more than 200°C.
- Ideal for interfacing to gas chromatographs for further analysis.

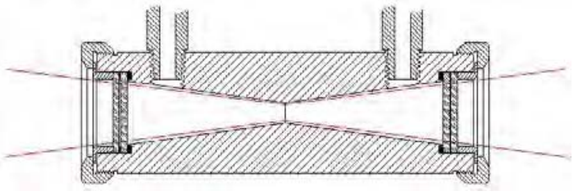


FEATURES

- 10 cm pathlength.
- Heatable to 200°C with Viton O-rings and to 260°C with Kalrez o-rings. Higher temperatures achievable with metal o-rings.
- Beam conforming interior maximizes pathlength to volume ratio.
- Cell volume: 17 ml.
- 20-mm diameter clear entrance and exit apertures.
- Two ports allow both static and flow-through applications.

INCLUDES

- Stainless steel cell body, Viton O-rings, and mounting hardware for the windows.
- Embedded thermocouple and low-voltage band heater.
- One port fitted with a valve and a second plugged port.
- Mating hardware for the specified spectrometer.



Cell Body Cross-Section Showing Beam Conforming Shape.

WINDOWS AND O-RINGS

	25 x 2 mm	25 x 4 mm
Windows:		
CaF_2	WFD-U25	WFD-U45
ZnS	WID-U25	WID-U45
NaCl	WLD-U25	WLD-U45
ZnSe	WMD-U25	WMD-U45
KBr	WPD-U25	WPD-U45
O-Rings		
Viton	ORV-020	ORV-020
Kalrez	ORK-020	ORK-020

ORDERING INFORMATION

	CATALOG NO.
Temperature Controlled Gas Cell, 24V	TGC-3-XXX

OPTIONS AND REPLACEMENT PARTS

Automatic Temperature Controller, 110V input, 24V output	ATC-024-1
Automatic Temperature Controller, 220/240V input, 24V output	ATC-024-2
Heater Assembly, 24V	TGC-HTR
Heater, 24V	HTRS-22

VACUUM PUMPS

These dual-stage oil-sealed rotary vacuum pumps are intended for operation with our environmental chambers. The pumps are capable of evacuating the vessels and vacuum systems down to the 100mtorr range. They are not intended for use with oxygen, hazardous gases or corrosive materials.

FEATURES

- Two-stage operation.
- Safety valves to prevent inadvertent venting of the vacuum system and back-streaming of the oil.
- Pumping speed: 10.3 m^3/hr.
- Ultimate total pressure: 20 mtorr.
- Motor power: 325W
- Weight (with oil): 27 lbs.
- Dimensions: 9.75”W x 15”H x 5.625”D.

INCLUDES

- Vacuum pump.
- Vacuum tubing (3’) to connect the pump.
- Vacuum pump oil.
- Two hose clamps to secure the vacuum tubing.
- An adapter to connect to ¼” VCO fittings.

ORDERING INFORMATION.

	CATALOG NO.
Vacuum Pump, 110V	VPE-001
Vacuum Pump, 220/240V	VPE-002
Oil, 1 quart	VPE-OIL

VariGATR ™

The *VariGATR*™ grazing angle ATR accessory is a revolutionary approach to the analysis of monolayers on semiconductor and metallic substrates. The *VariGATR*™ is variable angle, so the incident angle can be optimized for the highest sensitivity with these types of samples. Its specially designed pressure applicator is optimized for delivering good contact between the sample and the Ge ATR crystal. The *VariGATR*™ provides at least an order of magnitude increase in sensitivity relative to grazing angle methods, in addition to the convenience of an easy to use, fully prealigned, horizontal sampling accessory.

APPLICATIONS

- Analysis of monolayers and adsorbed species on semiconductors and metals.
- Rapid, repeatable measurements.

FEATURES

- Convenient horizontal sampling surface.
- Built-in pressure applicator with slip-clutch for reproducible pressure application.
- Continuously variable angle from 60º to 65º allowing optimization for maximum sensitivity.
- Anti-backlash mechanism allows for accurate, reproducible angle selection.
- Mounted Ge ATR crystal.
- Accommodates samples up to 8” in diameter with center-sampling of discs up to 6” in diameter.
- PermaPurge™ for rapid purging of the system.
- Options include:
    - Wire Grid Polarizer for enhanced spectral contrast and orientation studies. Includes slide plate mount.
    - Force sensor with digital read-out for precise measurement of the force applied to achieve contact between the ATR crystal and the sample.
    - Low-torque slip-clutch available.
    - Angular scale viewing aide for large samples.

INCLUDES

- Ge hemispherical ATR crystal.
- Built-in pressure applicator, designed to accommodate large samples.
- Mounting hardware for the specified spectrometer.

ORDERING INFORMATION

	CATALOG NO.
VariGATR™	GAT-V-XXX*

Options and Replacement Parts

Force Sensor with Digital Read-Out	GAT-V-FSD
Slip-Clutch, 24 in-oz	SLP-CLP
Angular Scale Viewer for Large Samples	GAT-V-VMR
Mounted Ge ATR Crystal	GATR-ATR-J1
VariGATR™ Wire Grid Polarizer (KRS-5 substrate). Includes mount and polarizer	PWD-GATR-XXX*
Wire Grid Polarizer Mount for the *VariGATR*™ (use with PWD Polarizer)	PWC-GATR-XXX*

The *VariGATR™* is a single reflection ATR accessory designed for analyzing monolayers and adsorbed species on semiconductor and metallic substrates.

The *VariGATR™* integrates the theoretical conditions that provide the highest sensitivity to these extremely thin films[1,2,3] in a convenient horizontal ATR sampler. The *VariGATR™* features a Ge ATR crystal for use from 5000 to 650 cm^{-1}. Its incident angle can be varied from 60º to 65º, making it possible to optimize the angle for any given spectrometer and sample. Its specially integrated Slip-Clutch pressure applicator optimizes contact between the sample and the relatively small active portion of the crystal. For greater sensitivity, a polarizer can be added to the *VariGATR™* and, for more precise measurements of the force applied to the sample by the pressure applicator, a force sensor with digital read-out is available.

Figures 1 and 2 demonstrate the high sensitivity of the *VariGATR™* to monolayers. Figure 1 is a spectrum of an organic monolayer on a polished silicon surface. Figure 2 shows the spectrum of a monolayer on a gold-coated glass substrate. Figure 3 shows spectra of a thin film on Si for two different angles. For this particular film, the lower angle was closer to the experimental critical angle and hence provided stronger band intensities.

For sampling versatility, the *VariGATR™* can also be used to analyze liquids, powders, pastes, and other solids. It is especially useful for samples with intense spectral bands. Such samples might otherwise exhibit too high absorbance or band distortions.

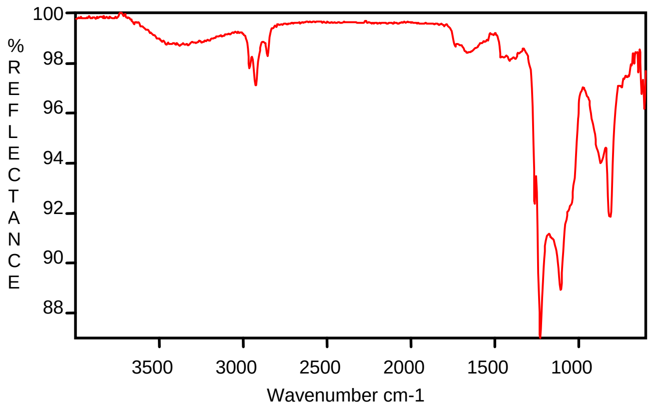


Figure 1. ATR Spectrum of an Organic Monolayer on Silicon, Measured at a 65º Incident Angle.

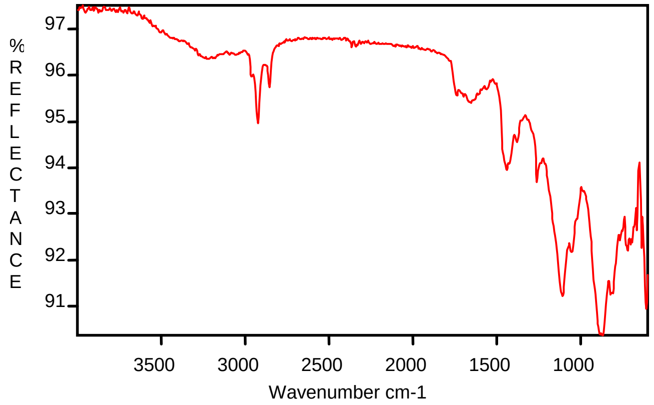


Figure 2. ATR Spectrum of an Organic Monolayer on Gold, Measured at a 65º Incident Angle.

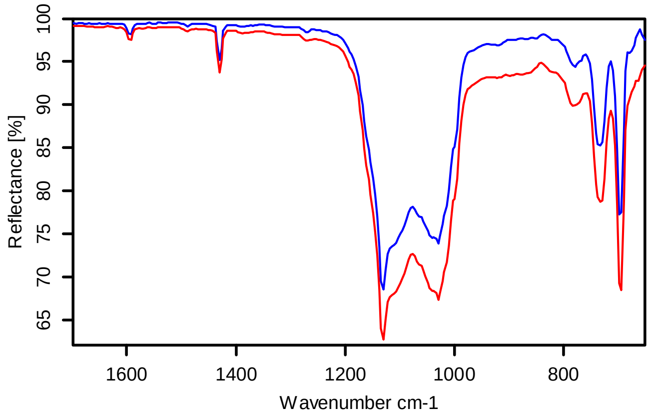


Figure 3. ATR Spectrum of a Thin Film on Si, Measured at Incident Angles of 60˚ (red) and 65˚ (blue).

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[1] M. Milosevic and S. L. Berets, '*ATR of Monolayers on Si and Neat Powders by Single Reflection ATR*,' PittCon 2002 invited paper.

[2] M. Milosevic, S. L. Berets, and Y. Fadeev, '*Single Reflection Attenuated Total Reflection of Organic Monolayers on Silicon*', *Appl. Spectros.*, 57 (6), 4724 (2003).

[3] Milan Milosevic, Violet Milosevic, and S. L. Berets, '*Grazing Angle Attenuated Total Reflection Spectroscopy: Fields at the Interface and Source of the Enhancement*', *Appl. Spectros*, 61(5), 530 (2007).

Video Meridian™

The Video Meridian™ combines the advantages of our Meridian™ diamond micro-ATR with video imaging and force sensing capabilities, making it the *ultimate* accessory for sampling small, hard, corrosive or irregularly shaped samples. The Video Meridian™ features a convenient horizontal sampling surface with an active sampling area less than 500 µm in diameter on its monolithic diamond crystal. The video system images through the crystal, providing a magnified real-time picture of the sample that can be easily captured for a permanent record. The samples themselves are compressed against the sample using a force-sensing pressure applicator with an adjustable alarm that conveniently delivery reproducible contact pressure. The Video Meridian™ is perfect for routine analysis of samples as small as a single fiber or bead, as well as extremely hard samples, abrasive powders, corrosive materials, liquids, solids and pastes.

APPLICATIONS

- ► Extremely hard samples; highly corrosive liquids; minerals; slightly curved samples; fibers; nanoliters of liquids and pastes; and defects on large panels.
- ► Forensic and combinatorial chemistry samples.
- ► Samples requiring archival storage of the images as well as the spectra.

FEATURES

- ► Monolithic Type IIA Diamond ATR Prism provides:
  - The most chemically inert, durable and cleanable ATR crystal available.
  - Small sampling area - less than 500 µm in diameter.
  - Operates from 45,000 cm^{-1} to the FIR.
  - Concentrated pressure application due to its unique crown design.
- ► Pressure applicator for optimal contact between the ATR crystal and hard samples includes:
  - Force sensor with digital display of applied force.
  - LED indicator that activates when the user set alarm point is reached.
- ► Video imaging and illumination system:
  - Magnifies the image for easy viewing.
  - Directly views the sampling surface *through* the ATR crystal.
  - Integrates with your computer for real-time viewing and long-term storage of sampled images.
  - Optional LCD display provides a 44X magnification of the image.
- ► Minimal stray light due to the small sampling area.
- ► High sample throughput, since little or no sample preparation is required.
- ► Generally retains sample integrity.
- ► High energy throughput with DTGS detectors.
- ► Harrick's patented PermaPurge™ allows rapid sample and crystal exchange without interrupting the purge of the system.

INCLUDES

- ► Sampling accessories: one diamond ATR holder, one external reflection sample holder and an alignment mirror.
- ► USB adapter and software for video image capturing, compatible with Windows 98/350MHz or higher computers.
- ► Mating hardware for the specified spectrometer.

ORDERING INFORMATION

	CATALOG NO.		CATALOG NO.		CATALOG NO.
Video Meridian™	MEV-XXX	Liquid Cell	UNS-LCF	Powder Retainer	UNS-PSC
Optional 4" LCD Display	MEV-LCD	Liquid Cell O-Ring	ORV-0015	Powder Retainer O-Ring	ORV-012
Mounted Diamond	UNS-ATR-1W				

This accessory integrates advantages of our Meridian™ diamond micro-ATR with video imaging and force sensing capabilities, making the Video Meridian™ *the* accessory for analyzing small, hard, corrosive or irregularly shaped samples.

The Video Meridian™ can be configured for either ATR or external reflectance, simply by changing the sample holder. In its ATR mode, it features a sample holder with a monolithic hemispherical diamond ATR crystal for operation from 45,000 cm^{-1} to the FIR. The sampling surface on the diamond is less than 500 µm in diameter and is horizontally oriented for convenient sampling. A video system images through the crystal, providing a magnified real-time picture of the true sampling surface. This image can be viewed on the optional LCD display or a computer screen. Software is included to capture the image for a permanent record. For analysis, the samples are compressed against the sample using a force-sensing pressure applicator. This pressure applicator is equipped with an adjustable alarm, to easily deliver repeatable and reproducible contact pressures. For external reflectance and re-alignment, the Video Meridian™ features a removable sample holder and reference mirror. The Video Meridian™ is enclosed in a purgable box for rapid sample exchange without interrupting the purge of the system.

Optional accessories for the Video Meridian™ include:

- An LCD display with a 4” monitor. The display provides a 44X magnification of the image and can be affixed to the front of the accessory or rested on its stand nearby.
- A liquid cell which seals to the crystal holder with an o-ring and features two luer fittings for flow or static applications.
- A powder retainer that o-ring seals to the crystal holder and contains powders during analysis

Representative spectra recorded with the Video Meridian™ are shown in Figures 1 through 4. All spectra were recorded with 32 scans and 8 cm^{-1} resolution with a DTGS detector.

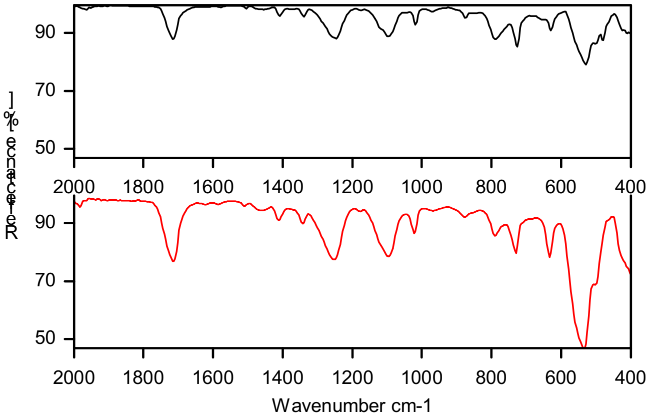

Figure 1. ATR Spectra of a 20 um PET Fiber compressed with 20 (top) and 302 (bottom) units of pressure.

Figures 1 and 2 show spectra and photographs of a 20 um diameter PET fiber compressed with different contact pressures. The photographs clearly show how much the fiber was flattened during pressure application.

Figure 2. Photographs of the 20 um PET Fiber compressed with 20 (left) and 302 (right) units of pressure.

Figure 3 shows spectra of two pulverized rocks. The spectra show that the white rock is composed mostly of quartz and the brown rock has a significant amount of chromite. The inserts show the differences between the two in texture and color.

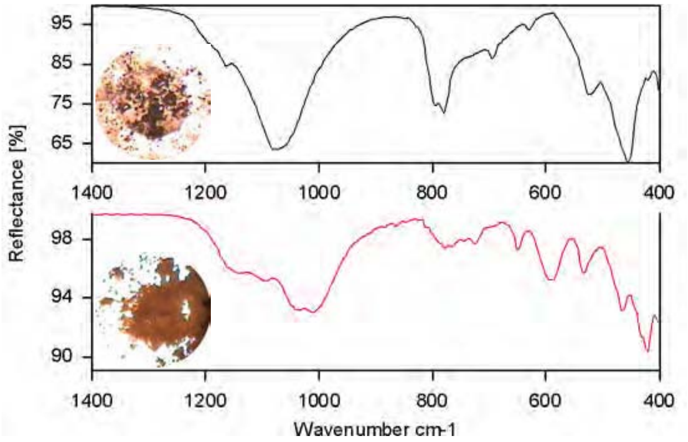

Figure 3. ATR Spectra of White (top) and Brown (bottom) Rocks.

For Figure 4, spectra were measured from the exposed side and underside of a paint chip. The two surfaces have different chemical compositions, and the spectra indicating that the top surface is an alkyd resin while the bottom is mostly cellulose.

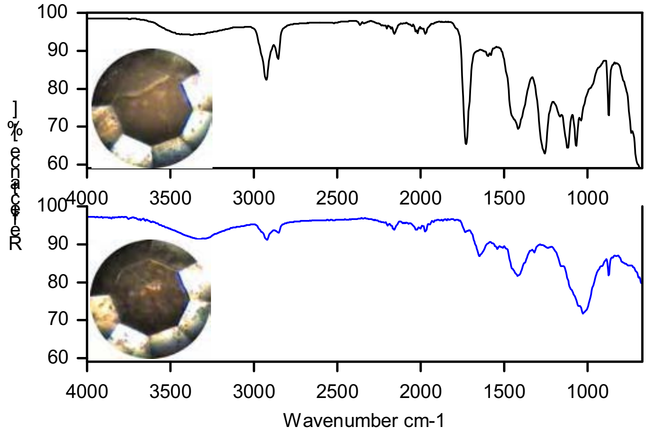

Figure 4. ATR Spectrum of the Bottom (lower) and Top (upper) Surface of a Gray Floor Paint Chip.

As demonstrated by the above examples, the Video Meridian™ is a versatile sampling tool for examining a wide range of solids, liquids and pastes.

HARRICK

VideoMVP™

The VideoMVP™ offers all the advantages of a high performance diamond ATR accessory plus video imaging and optional force sensing capabilities. The VideoMVP™ features a convenient horizontal sampling with an active sampling area of less than 500 µm in diameter on its monolithic diamond. This makes it ideal for infrared spectroscopy measurements of extremely hard samples, abrasive powders, fibers, beads, and even corrosive materials. The video imaging system provides a real-time magnified view of the sample through the ATR crystal. This image can be seen on the built-in display or digitally captured for a permanent record. Solid samples are compressed against the ATR crystal using the built-in pressure applicator. The pressure applicator features a slip clutch to prevent over-pressurization. An optional force sensor with digital read-out is available for precise measurement of the force applied to the sample and to reproducibly apply lower force than delivered by the built-in slip clutch.

APPLICATIONS

- ▶ Extremely hard samples; highly corrosive liquids; minerals; slightly curved samples; fibers; beads; nanoliters of liquids and pastes; and defects on large panels.
- ▶ Forensics, textiles and combinatorial chemistry samples.
- ▶ Samples requiring archival storage of images as well as spectra.

FEATURES

- ▶ Monolithic hemispherical diamond mechanically retained in a chemically resistant holder.
- ▶ All reflective optics other than the ATR crystal.
- ▶ Convenient horizontal sampling.
- ▶ Designed for optimal contact between the ATR element and solid samples.
- ▶ Minimal sample preparation required.
- ▶ Small sampling area: 500 µm in diameter with the diamond ATR crystal.
- ▶ Incident angle: 45°.
- ▶ Built-in pressure applicator with slip-clutch for reproducible calibrated pressure application.
- ▶ PermaPurge™ for rapid purging of the system.
- ▶ RoHS compliant.
- ▶ Video imaging system for viewing through the diamond ATR includes:
    - ▶ Built-in LCD display on a rotating mount for easy viewing.
    - ▶ 120X magnification of the sample.
    - ▶ USB-video adapter and software included for storing images.
- ▶ Options:
    - ▶ Readily exchangeable Ge, ZnSe, Si and diamond ATR sampling plates.
    - ▶ Heatable sampling plates for operation up to 200°C with diamond, Si or ZnSe ATR crystals, 100°C with Ge.
    - ▶ Powder adapter for retaining powdered samples.
    - ▶ Flow-through liquid cell for static and flow applications.
    - ▶ Force sensor with digital read-out for precise force measurements.

INCLUDES

- ▶ Mounted diamond ATR.
- ▶ Built-in force limited pressure applicator for solid sampling.
- ▶ LCD display and USB-video adapter for video imaging.
- ▶ Mating hardware for the specified spectrometer.

Mounted ATR Crystals		
	Ambient	Temperature Controlled
Si	UNS-ATR-0E	UNS-HOT-0E
ZnSe	UNS-ATR-0M	UNS-HOT-0M
Ge	UNS-ATR-0J	UNS-HOT-0J
Diamond	UNS-ATR-2W	UNS-HOT-0W

ORDERING INFORMATION

	CATALOG NO.		CATALOG NO.		CATALOG NO.
VideoMVP™, 110V	MVV-XXX-1	Force Sensor, 220/240V	MVV-2-FSD	Powder Retainer	UNS-PSC
VideoMVP™, 220/240V	MVV-XXX-2	Liquid Cell	UNS-LCF	Powder Retainer O-Ring	ORV-012
Force Sensor, 110V	MVV-1-FSD	Liquid Cell O-Ring	ORV-0015		

Harrick Scientific Products, Inc.
141 Tompkins Ave., 2nd floor, Pleasantville NY 10570
Ph: 800-248-3847, FAX: 914-747-7209, web site: www.harricksci.com, e-mail: info@harricksci.com

The VideoMVP is a high performance diamond ATR microsampler with real-time imaging of the sample. This makes it ideal for examining fibers and other small samples via infrared spectroscopy. Not only is positioning minute samples simple and straightforward, but the imaging system allows observation of the ‘wetting’ of the crystal as pressure is applied in addition to photographic documentation of the area sampled.

The VideoMVP comes with a diamond ATR crystal with a 500 µm sampling area. This ATR crystal is mechanically retained in a chemically resistant sample holder. The integrated LCD display provides a 120X magnified image of the sample and the display tilts for easy viewing. The imaging system can be connected to a computer using the supplied adapter to record and store the image.

The basic configuration comes with a built-in slip clutch which is designed to apply sufficient force for quality spectra with a over-pressurization limit. An optional force sensor is available for more accurate and repeatable pressure application.

Sample spectra recorded with the VideoMVP are shown in Figure 1 through 3. Figure 1 shows the spectrum of cotton fibers, recorded with the maximum pressure applied by the VideoMVP. From the imaging system, it is possible to see the fibers compress against the crystal as pressure is applied. Figure 2 shows the spectrum from a ridge on a plastic bag. From the spectrum, it is clear that the ridge is composed of polyethylene. Figure 3 features spectra recorded from two different locations on a credit card. The two sections are visibly different, but the spectroscopic differences are small.

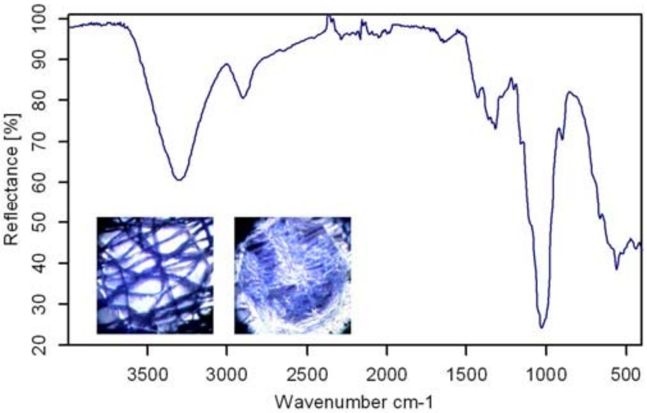

Figure 1. The ATR Spectrum of Cotton. The inset photographs show the cotton fibers before (left) and after (right) compression.

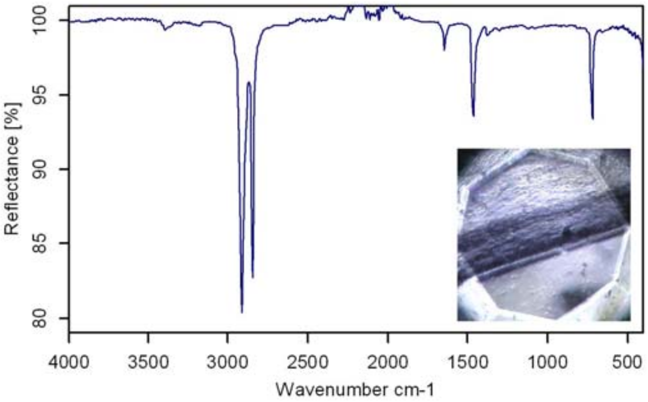

Figure 2. ATR Spectrum of a Ridge on a 2”x3” Ziplock Bag.

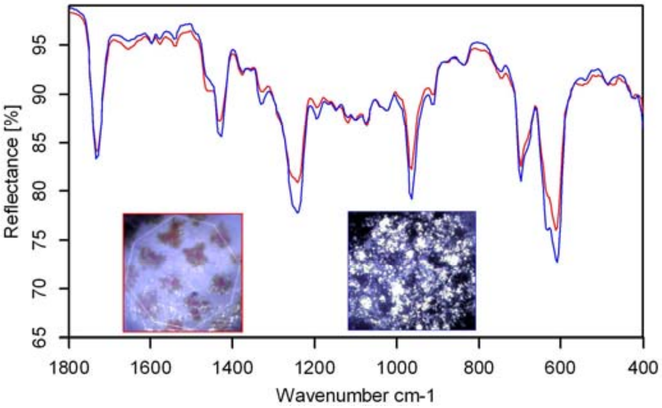

Figure 3. ATR Spectra from two locations on a credit card. The inset photographs are bordered by the color of the corresponding spectrum

As demonstrated, the VideoMVP is ideal for examining a wide variety of small samples in addition to focusing in on details on larger samples.

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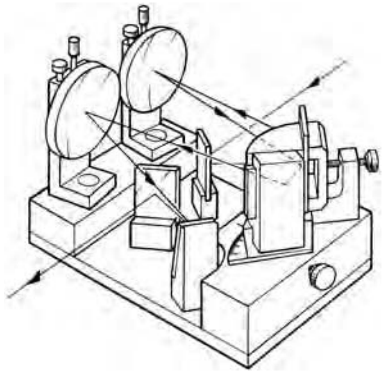
HARRICK

VARIABLE ANGLE REFLECTION ACCESSORY

Harrick's Variable Angle Accessory is ideal for specular reflection studies of films on metallic substrates, coatings, contaminants on reflective surfaces, and measurements of film thickness. The sample stage is readily adapted for absolute reflectance measurements or convenient horizontal sampling at a 12° incident angle.

APPLICATIONS

- ► Specular reflection studies of coatings on reflective substrates.
- ► Films on metallic substrates.
- ► Determining film thickness.
- ► The industry standard for variable angle specular reflectance.



FEATURES

- ► Incident angle variable from 30° to 85°.
- ► Accommodates samples larger than ½" x ½", up to 0.5" thick.
- ► Maintains alignment for all incident angles.
- ► Adaptable for variable angle, fixed angle, and absolute reflection studies.
- ► Models available for a wide range of FT-IR and UV-VIS spectrometers.

INCLUDES

- ► Optical base unit with variable angle reflectance sample stage.
- ► Mating hardware for the specified spectrometer.
- ► Optional sample stages for absolute reflectance and horizontal sampling.

OPTIONAL SAMPLE STAGES

12° ABSOLUTE REFLECTANCE SAMPLING STAGE

- Ideal for highly reflective substrates.
- Utilizes a double reflection technique.
- Alignment and pathlength are maintained for both sample and reference.
- Retains the incident polarization.

45° ABSOLUTE REFLECTANCE SAMPLING STAGE

- Useful for examining highly reflective substrates.
- Measures the square of the reflectance using a double reflection technique.
- Alignment and pathlength are maintained for both the sample and reference.

HORIZONTAL REFLECTION STAGE

- Fixed 12 ° incident angle.
- Convenient horizontal sampling.

ORDERING INFORMATION	**CATALOG NO.**
Variable Angle Reflection Accessory	VR1-XXX

XXX denotes the spectrometer code

OPTIONAL SAMPLE STAGES

12° Absolute Reflectance Sampling Stage	VR1-VWA-12	Horizontal Reflection Stage	VR1-HRS
45° Absolute Reflectance Sampling Stage	VR1-VWA-45		

OPTIONS

Wire Grid Polarizer, KRS-5 Substrate	PWG-U1R	Glan-Taylor Polarizer	PGT-S1V
Single Diamond Brewster's Angle Polarizer	PSD-J1R		

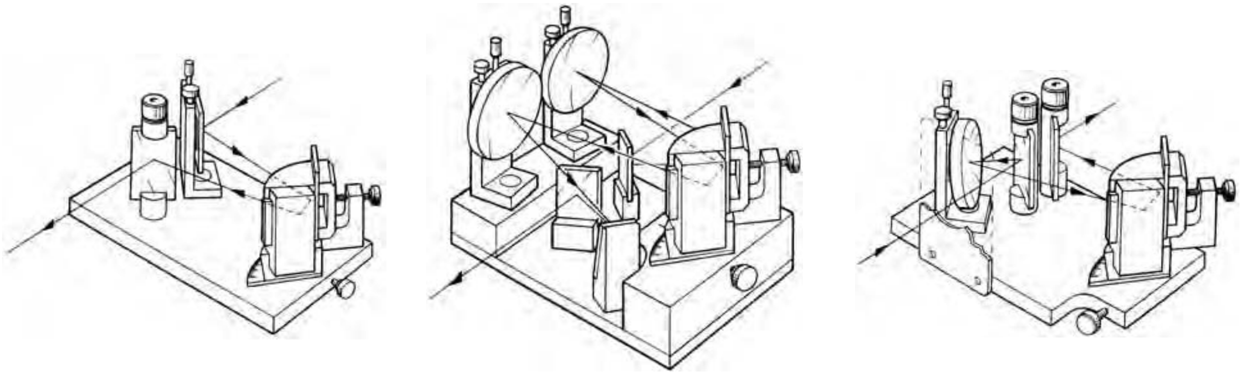


Figure 1. Three Models of the Variable Angle Reflection Accessory. Center model is most commonly used for FT-IR spectrometer, the other two for UV-VIS instruments.

The Variable Angle Reflection Accessory is ideal for specular reflection studies of films on metallic substrates, coatings, contaminants on reflective surfaces, and measurements of film thickness. This accessory is the industry standard for variable angle specular reflectance studies. Three different models, shown in Figure 1, are available to suit the various optical designs of commercial FT-IR and UV-VIS spectrometers.

The incident angle can be continuously varied from approximately 30° to 80°. Samples with a minimum size of ½" x ½" and up to 0.5" thick can be readily examined. This RMA works best with samples up to 0.5" thick.

The incoming radiation is directed by mirrors to the sampling stage, where it reflects from the sample stage mirror and the sample. This mirror and the sample are coupled to rotate together. This configuration ensures that, once the Variable Angle Reflection Accessory is aligned, it remains aligned for all incident angles. Some typical applications are shown in Figures 2 and 3.

For polarization measurements, Harrick Scientific's Brewster's Angle or Wire Grid Polarizers can be easily mounted onto the sample stage. For sampling versatility, there are four different reflectance sampling stages available: a continuously variable angle rotational stage, included with the accessory; 12º absolute reflectance stage; 45º absolute reflectance stage and a 12º horizontal stage.

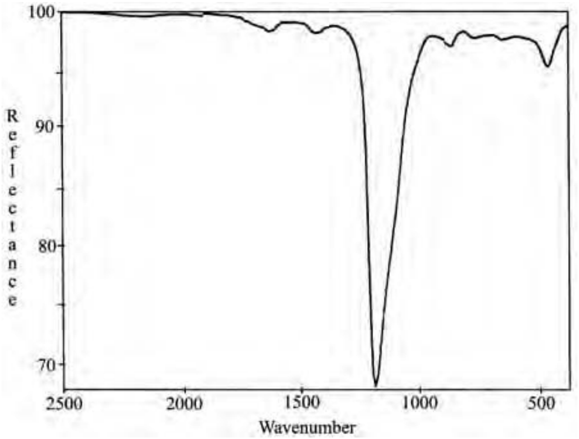


Figure 2. External Reflectance of an SiO2 Coated Mirror at an Incident Angle of 75°.

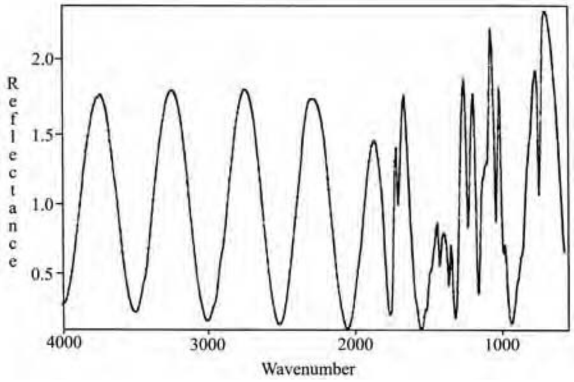
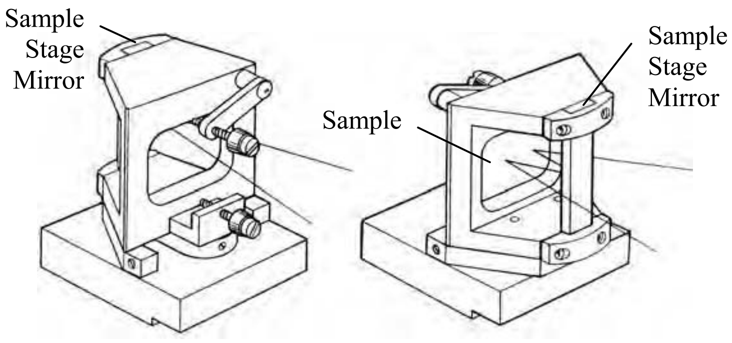


Figure 3. External Reflectance of a Mylar Film at an Incident Angle of 20°.

Absolute Reflectance Sample Stages

Two models are available. One, particularly well suited for examining highly reflective samples, features a 12º incident angle and retains the polarization of the incident beam (see Figure 4). The other, shown in Figure 6, offers a 45° incident angle.

The two fixed angle absolute reflectance sample stages available feature incident angles of 12º and 45º. Both utilize the 'V-W' double reflection technique illustrated in Figures 4 and 5. Using this technique, the reference spectrum is obtained in the 'V' mode. The beam is directed by mirrors to the sample stage mirror and back to the detector. To collect the sample spectrum, the stage is rotated (12º model) or

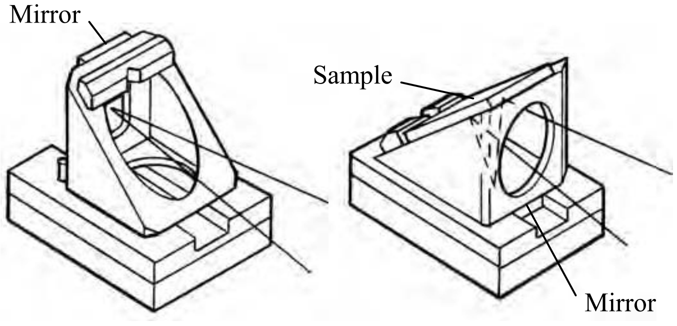


V - Reference Mode W - Sample Mode

Figure 4. The 12° Absolute Reflectance Accessory.

inverted (45º model) to the 'W' mode. In this mode, the beam is directed from the sample to the stage mirror. The sample stage mirror reflects the beam back to the sample. The beam is then directed via mirrors to the detector of the spectrometer.

This configuration maintains the alignment and optical pathlength for both the sample and the reference spectra. Note that the quantity measured is the ratio of the spectrum with the sample to the spectrum without the sample, is R^2.



V - Reference Mode W - Sample Mode

Figure 5. The 45° Absolute Reflectance Stage.

Horizontal Reflection Stage

The Horizontal Reflection Stage (see Figure 6) is used in combination with the Variable Angle Reflection Accessory for reflectance measurements at a 12° angle of incidence (near normal). The sample rests sample side down on a horizontal stage, making surface scanning and sample exchange straightforward.

This sample stage incorporates a single mirror that reflects the radiation from the sample stage mirror to the horizontal sample and then directs the light reflected from the sample back to the sample stage mirror.

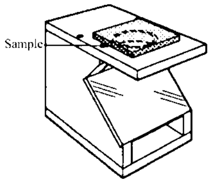


Figure 7. Horizontal Reflection Stage.

Aperture Design for Near Critical Angle FTIR ATR Spectroscopy

Joseph P. Lucania and Susan L. Berets
Harrick Scientific Products, Inc., P.O. Box 277, Pleasantville, NY 10570

Poster Paper No.2090-5P, 2007 Pittsburgh Conference, February 28, 2007.

Figure 1. Diaphragm aperture installed in the entrance beam side of the Seagull™.

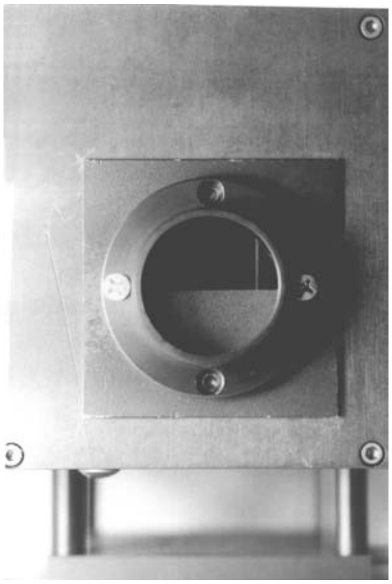


Figure 2. Half-moon aperture installed in the exit beam side of the Seagull™.

OBJECTIVE

The objective of this study is to investigate the effects of geometry changes of apertures on the sensitivity, baseline, and throughput of ATR data acquired near the critical angle.

ABSTRACT

In a previous report,[1] experimental equipment and techniques were discussed that empirically demonstrated the effects of approaching the critical angle in FTIR ATR spectroscopy. Using a variable angle ATR accessory in a modern FTIR spectrometer, it was shown that sensitivity does increase as the critical angle is approached. Contrary to a first order theoretical prediction, however, the increase is limited. Although spectral distortion with certain intense peaks plays a part in this limitation, the chief cause for the apparent discrepancy is the beam spread of the FTIR instrument. This spread is exacerbated by refraction in the ATR element of the accessory. For example, with the reflection angle of the accessory set slightly above the critical angle, almost half of the actual optical rays are below the critical angle. These rays exit the optical system, resulting in an overall increase in the absorbance baseline with no contribution to increased sensitivity. Furthermore, with the same setting, absorbance contributions from rays that are at significantly greater angles than the setting will attenuate those at or near the setting. For this study, a variable aperture in the ATR accessory is used to reduce the beam spread. The effect of decreasing the aperture diameter on sensitivity and throughput will be studied. Similarly, a new “half-moon” aperture, designed to eliminate rays at angles lower than the setting, will be investigated for effects on the baseline and throughput.

EXPERIMENTAL

All spectra were taken with a Thermo/Nicolet Nexus™ 670 FTIR spectrometer equipped with a DTGS detector and a standard mid-IR beamsplitter and using Thermo/Nicolet Omnic™ Version 6.1a software. All spectra were run at 4000cm^{-1} to 400cm^{-1}, 4 cm^{-1} resolution, and a gain equal to 1. Spectra were derived from 64 co-added scans, except where indicated. The spectrometer aperture was set to 100. A background spectrum (air) was taken prior to each sample spectrum, with the exception of the throughput scans taken for all aperture configurations. For these, only one background spectrum (the open beam sample compartment) was used for all sample scans. The purge inputs of the spectrometer and the installed accessory were connected to filtered air (water and CO_2 removed) produced by a Parker Balston Model 75-62 FT-IR Purge Gas Generator at 30 SCFH.

The Harrick Seagull^{TM} variable angle reflection accessory was installed in the sample compartment of the FTIR spectrometer, except where noted. The Seagull^{TM} accessory allows the angle of incidence to be changed continuously from 5^o to 85^o. The ATR kit was installed in the accessory along with a Luer-compatible liquid cell. All runs were taken using a hemispherical ZnSe ATR element. A Hamilton Model 1002 2.5ml Luer-tipped syringe was used to inject samples into the liquid cell.

All angles were set in the Seagull^{TM} by first moving the angle several degrees lower than the desired angle and then carefully moving the adjustment continuously higher until the desired angle was reached. This was to assure the consistent removal of any backlash.

Spectrophotometric grade Toluene (Alpha Aesar Stock No. 19376) was used as the sample in all tests, except where noted.

Transmission spectra were converted to absorbance spectra using the Omnic^{TM} "Absorb" function. Baseline corrections on the absorbance spectra, where required, were performed using the Omnic^{TM} "Aut Bsln" function. Absorbance peak maxima and associated wavelengths were found using the Omnic^{TM} "Analyze Find Peaks" function. The Harrick CristalCalc^{TM} software was used to calculate some depths of penetration used for the Discussion section.

Two custom apertures were installed in the Seagull^{TM} accessory. An iris diaphragm aperture (Part No. NT41-973; Edmund Optics Inc.; Barrington, NJ) was installed in the entrance beam side of the accessory. See Figure 1. Five precision metal shafts were used to adjust this aperture to exact diameter positions. For the three smaller sizes (4.95mm, 9.93mm, and 14.9mm), the shank ends of three metric drills were used (Part No.'s 029050, 029100, and 016150 Series 2AB Jobber Length Drills; Precision Twist Drill Co.; Crystal Lake, IL). For the two larger sizes (20.0mm and 25.0mm), 7/8" and 1" aluminum rods were machined to the desired sizes. The largest aperture size, with the iris diaphragm fully open, is actually formed by the purge input fitting at 33.0mm.

A half-moon aperture was installed in the exit beam side of the accessory. See Figure 2. The plate used in the half-moon aperture was painted with Sherwin Williams Krylon Semi-Flat Black No. 1613.

RESULTS

In the first experiment, the diaphragm aperture was fully opened (33.0mm diameter). The angle on the Seagull^{TM} was set to 38^o. This is the critical angle calculated using 2.4295 as the refractive index of zinc selenide at 2000cm^{-1} and 1.4961 as the refractive index of toluene.[2] Spectra were then taken with and without the half-moon aperture. The results are presented in Figure 3. The spectrum taken with the unapertured equipment has an overall elevation in its baseline of over 0.2 absorbance units. Perhaps more seriously, the same spectrum has a negative distortion preceding the intense peak at 725 cm^{-1} and a positive distortion following the adjacent intense peak at 692 cm^{-1}. These types of distortion are attributed to dispersion. The negative distortion makes baseline correction difficult. Both the negative distortion and the elevated baseline were removed by using the half-moon aperture.

Aperture Design for Near Critical Angle FTIR ATR

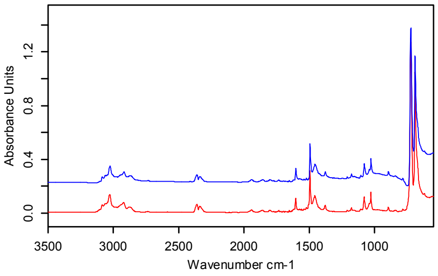


Figure 3. Comparison of toluene spectra at 38° with (red) and without (blue) half-moon aperture.

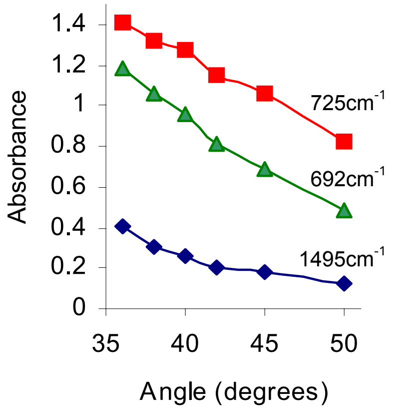


Figure 4. Determination of optimum angle for highest sensitivity.

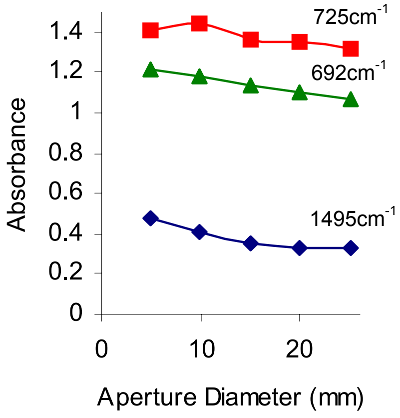


Figure 5. Determination of the optimum aperture for highest sensitivity.

In the second experiment, the half-moon aperture was left in place, the diaphragm aperture was left fully open, and the absorbances at the peaks located at 1495, 725, and 692 cm^{-1} were monitored as the angle was decreased from 50° to 36° (50, 45, 42, 40, 38, and 36 degrees). A summary of the data is presented in Figure 4. A significant increase in sensitivity is observed as the angle is decreased. The spectrum at 36° exhibited negative distortion preceding the 725 cm^{-1} peak, calling the associated baseline corrected absorbance data in question. For this reason, 38° was chosen as the angle for the next experiment.

In the third experiment, the half-moon aperture was left in place, the angle was set to 38°, and the same three peak absorbances were monitored as the diaphragm aperture was decreased from 25.0mm to 4.95mm (25.0, 20.0, 14.9, 9.93, and 4.95mm). Since the noise increased significantly with the 4.95mm aperture, the number of co-added scans was increased from 64 to 256. Figure 5 summarizes the data. A modest increase in sensitivity is observed as the aperture is decreased. The 9.93mm aperture was chosen as the smallest practical size.

DISCUSSION

In ATR spectroscopy, as the angle of incidence is decreased, an increase in sensitivity is expected. However, the beam spread in modern FTIR spectrometers causes some problems as the critical angle is approached and this beam spread is exacerbated by refraction in the high refractive index ATR element material. First, those rays lower than the selected angle that are also lower than the critical angle are lost. This causes an overall decrease in the energy that reaches the detector, resulting in an elevated absorbance baseline. Second, rays higher than the selected angle contribute to a decrease in sensitivity.

Use of the half-moon aperture to eliminate rays lower than the selected angle minimizes absorbance baseline shifts as the selected angle is decreased towards the critical angle. This type of aperture also greatly reduces dispersive negative shifts preceding intense absorbance peaks.

Use of the diaphragm aperture reduces rays higher than the selected angle which contribute to lowered sensitivity. The smaller the diameter of this diaphragm, the higher the sensitivity.

Aperture Design for Near Critical Angle FTIR ATR

Lowering the angle, as seen in Figure 4, had a significant effect in increasing the sensitivity. Reducing the aperture had a more modest effect. See Figure 5. The most sensitive, practical configuration was with the half-moon aperture in place, the angle set at 38°, and the diaphragm set at 9.93mm. A comparison of the spectrum obtained with these parameters set in the Seagull™ and one obtained using the fixed 45° angle ZnSe ATR Harrick AccessATR™ is given in Figure 6. Table 1 compares the absorbances of peaks obtained with the two setups. The apertured 38° Seagull™ results are 1.3 to 2.9 times higher than those obtained from the 45° AccessATR™ results. Different peaks have different sensitivity increases, probably due to different localized differences in the refractive indices of the sample near the peak maxima. It can be shown that a greater sensitivity increase will take place at any given wavelength for samples of higher refractive indices. In general, the absorbances of weaker peaks were increased more than those of stronger peaks. No correlation can be seen between sensitivity increases and wavelength. Additional work with alternate samples would be required before making further generalizations. Also of interest, three of the peaks detected by the FTIR software for the 38° results were not found by the same software in the 45° results.

One disadvantage of using apertures to increase sensitivity is that the throughput is simultaneously lowered. Figure 7 plots the throughput at 2600 cm^{-1} versus the aperture area for different diaphragm settings with and without the half-moon aperture. (This wavelength was chosen since it lies in an area that is relatively free of spectral artifacts. The open beam sample compartment was run for the background. The sample was the Seagull™ accessory set at 45° with the ZnSe ATR kit and air as the sample.) From aperture openings of 33 to 25mm, there is essentially no change in throughput, indicating that the FTIR beam diameter is smaller than 25mm. Between 25 and 15mm, there is a nonlinear drop in throughput, indicating that the beam is not uniform. Between 15 and 5mm, there is a

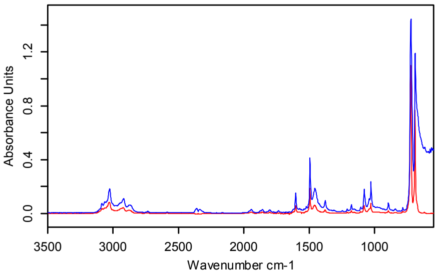


Figure 6. Comparison of toluene ATR spectra taken with a standard 45° accessory (red) and the experimentally apertured Seagull™ at 38° (blue).

Table 1. Comparison of sensitivity of fixed 45° ATR accessory vs. apertured variable angle ATR accessory at 38°.

Peak Wavelength (cm^{-1})	Absorbance		Sensitivity Increase
	45°	38°	
3028	0.0850	0.179	2.11x
2919	------	0.107	------
1604	0.0612	0.149	2.43x
1495	0.187	0.407	2.18x
1457	------	0.179	------
1378	------	0.0891	------
1080	0.0620	0.169	2.73x
1030	0.0781(5)	0.227	2.91x
725	1.103	1.444	1.31x
692	0.768	1.185	1.54x

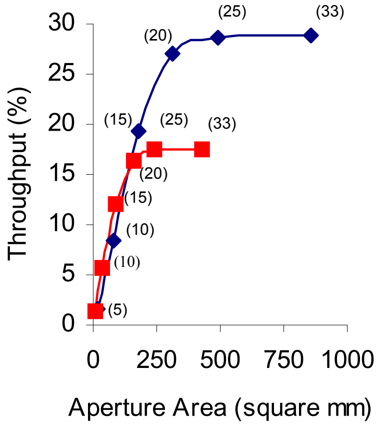


Figure 7. Throughput at 2600cm^{-1} vs. aperture area for configurations with (red) and without (blue) the half-moon aperture. Numbers in parentheses are diaphragm aperture diameters in mm.

steep linear drop in throughput, indicating that the beam is strong and uniform. It is in this latter area, where the most precipitous drop in throughput occurs, that one would expect the best increases in sensitivity to be derived over unapertured equipment. Loss of throughput, of course, will decrease Signal/Noise, so that eventually any benefits of increased sensitivity will be lost. This did in fact occur during the course of this work, at least at the 64 coadded scan level.

Although further work with alternate samples is clearly indicated, one can propose a possible direct method of obtaining the highest possible sensitivity with the equipment here described. First, the critical angle is calculated based on the bulk refractive index of the sample. Then, the Seagull$^{\text{TM}}$ is set to this angle. The half-moon and diaphragm apertures are installed with the latter set to 10mm.

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2. N.J. Harrick, *Internal Reflection Spectroscopy*, Harrick Scientific Corporation, Ossining (1987).

No. 50415

ANALYSIS OF HAIR USING THE SEAGULL™ VARIABLE ANGLE REFLECTION ACCESSORY

Figure 1. The Seagull™ Variable Angle Reflectance Accessory.

INTRODUCTION

The presence of breast cancer appears to alter the hair growth process, resulting in changes in the hair fiber structure.[1] A normal hair fiber is essentially dead tissue made up of alpha keratin, related proteins, and lipid materials.[2] The hair fiber is composed of an outer cuticle layer surrounding the central fiber core, or cortex. The cuticle is composed of flat, overlapping scales with an average cuticle layer thickness of about 2.5µm. At the molecular level, the cuticle material is a heavily cross-linked, not highly organized, protein-lipid material that provides a chemical-resistant barrier to the cortex. The cortex (average diameter of 30 to 60 µm) is composed of a number of elongated, keratinized cells and intercellular membrane binding material, primarily protein and lipid acids and esters. The cells are symmetrically arranged so that their keratin macrofibrils are aligned along the hair shaft axis. The macrofibrils are aggregated structures of coiled-coil alpha keratin molecules. A gel-like proteinaceous matrix encapsulates these keratin filaments and macrofibrils.

Subtle changes have been observed in the synchrotron X-ray diffraction patterns of hair from breast cancer patients.[1] In the cancer hair, one or more rings of comparatively low intensity were superimposed at specific locations on the normal hair alpha keratin pattern. These changes suggest that compositional and/or conformational changes have occurred in the lipid and/or protein materials within the matrix associated with the intermediate filaments of the hair fiber. In order to demonstrate whether these changes could be seen on the infrared spectra of these hairs (without the necessity of physically removing the cuticle, which could alter the cortex), the effective penetration depth of the evanescent wave of the IR beam into the composite

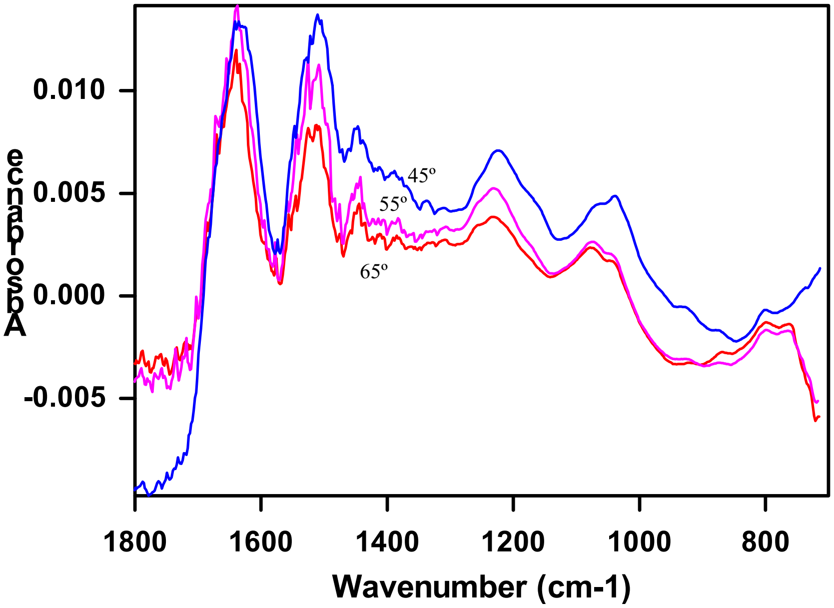

Figure 2. The ATR spectra of a sample of three human scalp hairs at incident light angles of 45º (showing cuticle and cortex material), 55º (showing primarily cuticle material) and 60º (showing only cuticle material).

structure of the hair fiber had to be determined. The Seagull^{TM} variable angle ATR cell[3] was used to study whether a spectrum of the cortex portion of the hair fiber could be obtained without having to physically remove the overlying cuticle layer.[4]

EXPERIMENTAL

Spectra were obtained on a FTIR spectrometer equipped with a liquid nitrogen cooled MCT detector using the Seagull^{TM} variable angle ATR cell (see Figure 1) with both germanium (Ge) and zinc selenide (ZnSe) IREs. Three scalp hair fibers from each individual were mounted on a sample card designed to hold the fibers in an alignment parallel to the IR beam. Spectra were collected from 4000 to 700 cm^{-1} using 128 scans at a resolution of 4 cm^{-1} (see Figure 2).

RESULTS AND DISCUSSION

Examination of the baseline corrected and smoothed (Grams 386 Savitsky-Golay program, 11 pts) spectra of adult hair fibers at 30° and 45° using a Ge IRE showed no significant differences in the spectra for the region above 1300 cm^{-1}. With the deeper penetration of the IR beam at an incident light angle of 30°, there were increased absorptions at 1180 (C-O ester stretch), 1077 (C-OH), 1043 (C-O ester stretch), and 950-850 cm^{-1} regions. This suggests that with a cuticle thickness of about 2.5 µm, the effective evanescent wave penetration is about $2d_p$. The effective depth of penetration (d_e) has been shown to increase as the ratio of the refractive indices of the sample/IRE approaches 1.0.[5] For example, at a sample/IRE ratio of 0.25, d_e was found to approximate the calculated d_p and at a sample/IRE ratio of 0.66, d_e was shown to approximate $3d_p$.[5,6] This is consistent with d_e for Ge being about $2d_p$.

For ZnSe (with a sample/IRE ratio of 0.64) d_e would be expected to be about $3d_p$. A comparison of a Ge 45° spectrum (cuticle material) with a ZnSe 45° spectrum (cuticle and cortex material) supports this.[4] The cortex of the hair appears to contribute to some degree to the spectrum below 2400 cm^{-1} for a 45° incident light angle with the ZnSe IRE.

This study demonstrates that the FTIR-ATR spectrum of a hair fiber obtained with the Seagull^{TM} variable angle reflection accessory with a ZnSe IRE at an incident light angle between 45° and 40° (the critical angle) appears to enable the detection of spectral changes in cortex materials in the 2000 to 700 cm^{-1} region without physically removing the cuticle from the hair fiber.

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No. 70318

Aperture Design for Near Critical Angle FTIR ATR Spectroscopy

Joseph P. Lucania and Susan L. Berets
Harrick Scientific Products, Inc., P.O. Box 277, Pleasantville, NY 10570

Poster Paper No.2090-5P, 2007 Pittsburgh Conference, February 28, 2007.

Figure 1. Diaphragm aperture installed in the entrance beam side of the Seagull™.

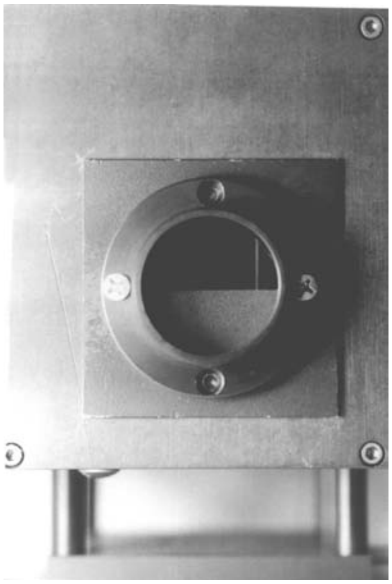

Figure 2. Half-moon aperture installed in the exit beam side of the Seagull™.

OBJECTIVE

The objective of this study is to investigate the effects of geometry changes of apertures on the sensitivity, baseline, and throughput of ATR data acquired near the critical angle.

ABSTRACT

In a previous report,[1] experimental equipment and techniques were discussed that empirically demonstrated the effects of approaching the critical angle in FTIR ATR spectroscopy. Using a variable angle ATR accessory in a modern FTIR spectrometer, it was shown that sensitivity does increase as the critical angle is approached. Contrary to a first order theoretical prediction, however, the increase is limited. Although spectral distortion with certain intense peaks plays a part in this limitation, the chief cause for the apparent discrepancy is the beam spread of the FTIR instrument. This spread is exacerbated by refraction in the ATR element of the accessory. For example, with the reflection angle of the accessory set slightly above the critical angle, almost half of the actual optical rays are below the critical angle. These rays exit the optical system, resulting in an overall increase in the absorbance baseline with no contribution to increased sensitivity. Furthermore, with the same setting, absorbance contributions from rays that are at significantly greater angles than the setting will attenuate those at or near the setting. For this study, a variable aperture in the ATR accessory is used to reduce the beam spread. The effect of decreasing the aperture diameter on sensitivity and throughput will be studied. Similarly, a new "half-moon" aperture, designed to eliminate rays at angles lower than the setting, will be investigated for effects on the baseline and throughput.

EXPERIMENTAL

All spectra were taken with a Thermo/Nicolet Nexus™ 670 FTIR spectrometer equipped with a DTGS detector and a standard mid-IR beamsplitter and using Thermo/Nicolet Omnic™ Version 6.1a software. All spectra were run at 4000cm^{-1} to 400cm^{-1}, 4 cm^{-1} resolution, and a gain equal to 1. Spectra were derived from 64 co-added scans, except where indicated. The spectrometer aperture was set to 100. A background spectrum (air) was taken prior to each sample spectrum, with the exception of the throughput scans taken for all aperture configurations. For these, only one background spectrum (the open beam sample compartment) was used for all sample scans. The purge inputs of the spectrometer and the installed accessory were connected to filtered air (water and CO_2 removed) produced by a Parker Balston Model 75-62 FT-IR Purge Gas Generator at 30 SCFH.

The Harrick Seagull^{TM} variable angle reflection accessory was installed in the sample compartment of the FTIR spectrometer, except where noted. The Seagull^{TM} accessory allows the angle of incidence to be changed continuously from 5^{o} to 85^{o}. The ATR kit was installed in the accessory along with a Luer-compatible liquid cell. All runs were taken using a hemispherical ZnSe ATR element. A Hamilton Model 1002 2.5ml Luer-tipped syringe was used to inject samples into the liquid cell.

All angles were set in the Seagull^{TM} by first moving the angle several degrees lower than the desired angle and then carefully moving the adjustment continuously higher until the desired angle was reached. This was to assure the consistent removal of any backlash.

Spectrophotometric grade Toluene (Alpha Aesar Stock No. 19376) was used as the sample in all tests, except where noted.

Transmission spectra were converted to absorbance spectra using the Omnic^{TM} "Absorb" function. Baseline corrections on the absorbance spectra, where required, were performed using the Omnic^{TM} "Aut Bsln" function. Absorbance peak maxima and associated wavelengths were found using the Omnic^{TM} "Analyze Find Peaks" function. The Harrick CristalCalc^{TM} software was used to calculate some depths of penetration used for the Discussion section.

Two custom apertures were installed in the Seagull^{TM} accessory. An iris diaphragm aperture (Part No. NT41-973; Edmund Optics Inc.; Barrington, NJ) was installed in the entrance beam side of the accessory. See Figure 1. Five precision metal shafts were used to adjust this aperture to exact diameter positions. For the three smaller sizes (4.95mm, 9.93mm, and 14.9mm), the shank ends of three metric drills were used (Part No.'s 029050, 029100, and 016150 Series 2AB Jobber Length Drills; Precision Twist Drill Co.; Crystal Lake, IL). For the two larger sizes (20.0mm and 25.0mm), 7/8" and 1" aluminum rods were machined to the desired sizes. The largest aperture size, with the iris diaphragm fully open, is actually formed by the purge input fitting at 33.0mm.

A half-moon aperture was installed in the exit beam side of the accessory. See Figure 2. The plate used in the half-moon aperture was painted with Sherwin Williams Krylon Semi-Flat Black No. 1613.

RESULTS

In the first experiment, the diaphragm aperture was fully opened (33.0mm diameter). The angle on the Seagull^{TM} was set to 38^{o}. This is the critical angle calculated using 2.4295 as the refractive index of zinc selenide at 2000cm^{-1} and 1.4961 as the refractive index of toluene.[2] Spectra were then taken with and without the half-moon aperture. The results are presented in Figure 3. The spectrum taken with the unapertured equipment has an overall elevation in its baseline of over 0.2 absorbance units. Perhaps more seriously, the same spectrum has a negative distortion preceding the intense peak at 725 cm^{-1} and a positive distortion following the adjacent intense peak at 692 cm^{-1}. These types of distortion are attributed to dispersion. The negative distortion makes baseline correction difficult. Both the negative distortion and the elevated baseline were removed by using the half-moon aperture.

Aperture Design for Near Critical Angle FTIR ATR

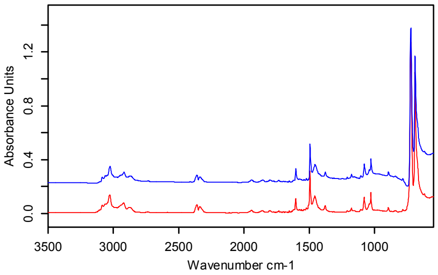

Figure 3. Comparison of toluene spectra at 38° with (red) and without (blue) half-moon aperture.

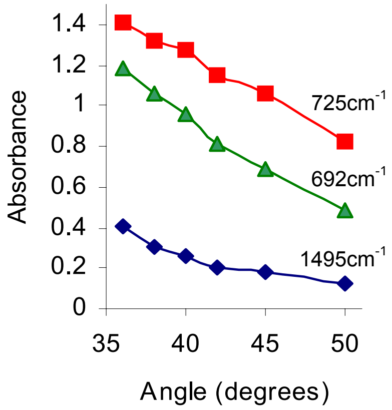

Figure 4. Determination of optimum angle for highest sensitivity.

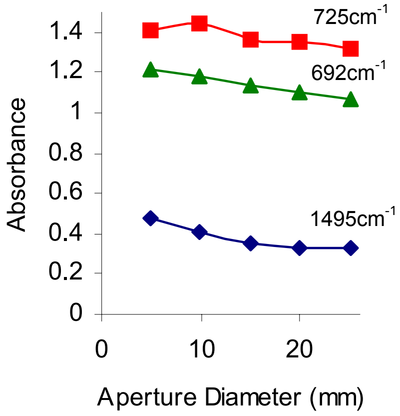

Figure 5. Determination of the optimum aperture for highest sensitivity.

In the second experiment, the half-moon aperture was left in place, the diaphragm aperture was left fully open, and the absorbances at the peaks located at 1495, 725, and 692 cm^{-1} were monitored as the angle was decreased from 50^{o} to 36^{o} (50, 45, 42, 40, 38, and 36 degrees). A summary of the data is presented in Figure 4. A significant increase in sensitivity is observed as the angle is decreased. The spectrum at 36^{o} exhibited negative distortion preceding the 725 cm^{-1} peak, calling the associated baseline corrected absorbance data in question. For this reason, 38^{o} was chosen as the angle for the next experiment.

In the third experiment, the half-moon aperture was left in place, the angle was set to 38^{o}, and the same three peak absorbances were monitored as the diaphragm aperture was decreased from 25.0mm to 4.95mm (25.0, 20.0, 14.9, 9.93, and 4.95mm). Since the noise increased significantly with the 4.95mm aperture, the number of co-added scans was increased from 64 to 256. Figure 5 summarizes the data. A modest increase in sensitivity is observed as the aperture is decreased. The 9.93mm aperture was chosen as the smallest practical size.

DISCUSSION

In ATR spectroscopy, as the angle of incidence is decreased, an increase in sensitivity is expected. However, the beam spread in modern FTIR spectrometers causes some problems as the critical angle is approached and this beam spread is exacerbated by refraction in the high refractive index ATR element material. First, those rays lower than the selected angle that are also lower than the critical angle are lost. This causes an overall decrease in the energy that reaches the detector, resulting in an elevated absorbance baseline. Second, rays higher than the selected angle contribute to a decrease in sensitivity.

Use of the half-moon aperture to eliminate rays lower than the selected angle minimizes absorbance baseline shifts as the selected angle is decreased towards the critical angle. This type of aperture also greatly reduces dispersive negative shifts preceding intense absorbance peaks.

Use of the diaphragm aperture reduces rays higher than the selected angle which contribute to lowered sensitivity. The smaller the diameter of this diaphragm, the higher the sensitivity.

Lowering the angle, as seen in Figure 4, had a significant effect in increasing the sensitivity. Reducing the aperture had a more modest effect. See Figure 5. The most sensitive, practical configuration was with the half-moon aperture in place, the angle set at 38^{o}, and the diaphragm set at 9.93mm. A comparison of the spectrum obtained with these parameters set in the Seagull^{TM} and one obtained using the fixed 45^{o} angle ZnSe ATR Harrick AccessATR^{TM} is given in Figure 6. Table 1 compares the absorbances of peaks obtained with the two setups. The apertured 38^{o} Seagull^{TM} results are 1.3 to 2.9 times higher than those obtained from the 45^{o} AccessATR^{TM} results. Different peaks have different sensitivity increases, probably due to different localized differences in the refractive indices of the sample near the peak maxima. It can be shown that a greater sensitivity increase will take place at any given wavelength for samples of higher refractive indices. In general, the absorbances of weaker peaks were increased more than those of stronger peaks. No correlation can be seen between sensitivity increases and wavelength. Additional work with alternate samples would be required before making further generalizations. Also of interest, three of the peaks detected by the FTIR software for the 38^{o} results were not found by the same software in the 45^{o} results.

One disadvantage of using apertures to increase sensitivity is that the throughput is simultaneously lowered. Figure 7 plots the throughput at 2600 cm^{-1} versus the aperture area for different diaphragm settings with and without the half-moon aperture. (This wavelength was chosen since it lies in an area that is relatively free of spectral artifacts. The open beam sample compartment was run for the background. The sample was the Seagull^{TM} accessory set at 45^{o} with the ZnSe ATR kit and air as the sample.) From aperture openings of 33 to 25mm, there is essentially no change in throughput, indicating that the FTIR beam diameter is smaller than 25mm. Between 25 and 15mm, there is a nonlinear drop in throughput, indicating that the beam is not uniform. Between 15 and 5mm, there is a

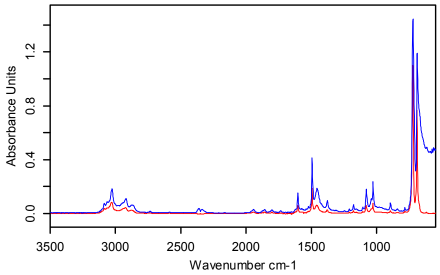

Figure 6. Comparison of toluene ATR spectra taken with a standard 45° accessory (red) and the experimentally apertured Seagull^{TM} at 38° (blue).

Table 1. Comparison of sensitivity of fixed 45° ATR accessory vs. apertured variable angle ATR accessory at 38°.

Peak Wavelength (cm^{-1})	Absorbance		Sensitivity Increase
	45°	38°	
3028	0.0850	0.179	2.11x
2919	------	0.107	------
1604	0.0612	0.149	2.43x
1495	0.187	0.407	2.18x
1457	------	0.179	------
1378	------	0.0891	------
1080	0.0620	0.169	2.73x
1030	0.0781$^{(5)}$	0.227	2.91x
725	1.103	1.444	1.31x
692	0.768	1.185	1.54x

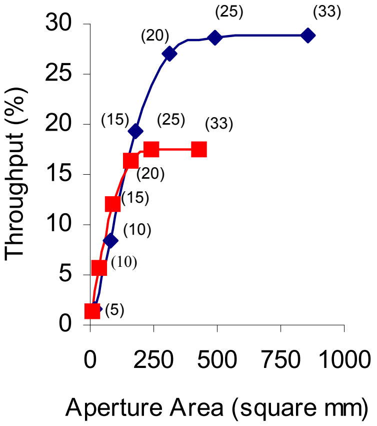

Figure 7. Throughput at 2600cm^{-1} vs. aperture area for configurations with (red) and without (blue) the half-moon aperture. Numbers in parentheses are diaphragm aperture diameters in mm.

steep linear drop in throughput, indicating that the beam is strong and uniform. It is in this latter area, where the most precipitous drop in throughput occurs, that one would expect the best increases in sensitivity to be derived over unapertured equipment. Loss of throughput, of course, will decrease Signal/Noise, so that eventually any benefits of increased sensitivity will be lost. This did in fact occur during the course of this work, at least at the 64 coadded scan level.

Although further work with alternate samples is clearly indicated, one can propose a possible direct method of obtaining the highest possible sensitivity with the equipment here described. First, the critical angle is calculated based on the bulk refractive index of the sample. Then, the Seagull™ is set to this angle. The half-moon and diaphragm apertures are installed with the latter set to 10mm.

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No. 50805

ATR ANALYSIS OF HAIR USING THE SPLITPEA™

Figure 1. The SplitPea™ ATR Microsampler.

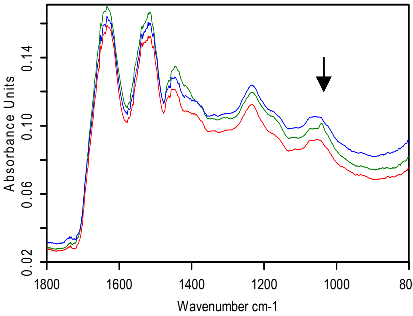

Figure 2: The 1800 to 800 cm^{-1} region of FTIR spectra of a human hair fiber taken at 10 mm (red), 45 mm (blue), and 80 mm (green) from the scalp end of the fiber. The 1040 cm^{-1} absorption (arrow) indicates oxidation of sulfur bonds in the more distal (80 mm) portion of the hair.

INTRODUCTION

The IRS Nanosampler, first described in 1987[1], has evolved into a user friendly, horizontal ATR cell while still maintaining a small sampling area[2]. The SplitPea™ (Figure 1) is available with easily changed germanium, silica, zinc selenide, or diamond Internal Reflection Elements (IRE). The small sampling area (250 micrometer diameter) is of importance in the examination of fibers, including hair fibers, since the fiber will cover a larger portion of the illuminated area of the IRE. For example, a human scalp hair fiber having an average diameter of 50 micrometers will cover about 25% of sampling area of the SplitPea™ IRE. This results in increased spectral absorbances compared to spectra obtained using ATR cells having larger illuminated sampling areas. Thus, one can study a single hair fiber rather than needing to mount multiple hair fibers on a sample card.

EXPERIMENTAL

Spectra were obtained on a FTIR spectrometer equipped with a liquid nitrogen cooled MCT detector using the SplitPea$^{\text{TM}}$ with a ZnSe IRE. A single hair fiber was mounted on a sample card designed to hold the fiber in an alignment parallel to the IR beam. The pressure applicator was lowered until a slight movement of the pressure indicator occurred (pressure was still around zero). The FTIR-ATR absorption spectra were obtained from 4000 to 700 cm^{-1} using 128 scans at a resolution of 4 cm^{-1} and Norton-Beer medium apodization.

RESULTS AND DISCUSSION

While increases in absorptions do occur with increases in applicator pressure, minimum pressure should be applied. The hair fiber has a heterogeneous structure. Both its physical form and spectra can be altered by applying too much pressure.

Since the growth rate of hair fibers is known (averages 0.33 mm/day) and the hair is essentially dead tissue, it can provide a stable "time capsule" of biological events that have occurred in the body[3]. Figure 2 shows the spectra obtained 10, 45, and 80 mm from the scalp end of a single hair fiber. This represents the age of the portion of the hair sampled (going back in time) of about 30, 136, and 242 days, respectively. In this hair sample, one can see that the only major difference in the spectra is the appearance of the band at about 1040 cm^{-1} due to the oxidation of sulfur bonds in the hair forming sulfonic acid groups[4]. Note that the increase in intensity occurs in the portion of the hair more distal from the scalp (and thus more exposed to external factors such as sunlight or other oxidizing media). No changes were noted that were due to internal factors such as breast cancer.[5] However, with the small sampling area of the SplitPea™ and the ease of positioning a single hair fiber on the IRE one could easily determine weekly changes in the hair spectrum by obtaining spectra every 2.3 mm along the fiber shaft.

Thus, the SplitPea**™** horizontal ATR cell should be useful in many types of studies involving hair, including detection of breast cancer[5]. Although this author has only used the SplitPea™ ATR cell without the viewing microscope, I would highly recommend that SplitPea**™** with the viewing microscope or the Video-Meridian**™** for this type of study, since it would make reproducible positioning of the fine hair fiber on the IRE an easier task.

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[2] N.J. Harrick, M. Milosevic, and S.L. Berets, "Advances in Optical Spectroscopy: The Ultra Small Sample Analyzer," *Appl. Spectrosc* **45**, 944-948 (1991).

[3] C.R. Robbins, Chemical and Physical Behavior of Human Hair, Springer-Verlag, New York, 3rd ed. (1994).

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application note

No. 40402

A New Tool for Solid-Phase Analysis in Combinatorial Chemistry

Figure 1. The SplitPea™.

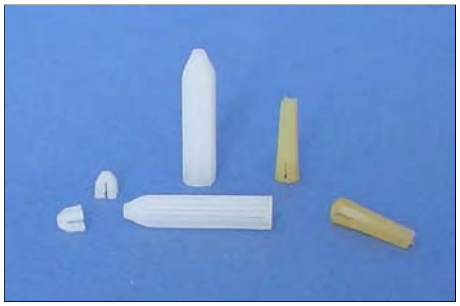

Figure 2. Typical Samples.

Technological advances in the past few years have led to vast changes in the drug discovery paradigm employed by the pharmaceutical industry. One facet of this dramatic change has occurred with the use of combinatorial chemistry on solid-phase supports or resins that allows synthesizing large numbers of compounds very rapidly.

For this technology in general and in the rehearsal phases of combinatorial chemistry synthesis in particular, the ability to follow the progress of multi-step solid-phase reactions is extremely advantageous. Ideally, this should be done directly on the solid-phase thus obviating the tedious cleavage of the synthetic compounds from the solid support. Due to its unique potential to recognize specific functional groups, infrared spectroscopy is not only an excellent tool for following chemical reactions in general, but using HARRICK's SplitPea™ accessory (Figure 1), it also specifically allows the monitoring of chemical reactions directly on the polymeric support.

The small spot size (250 µm) of the SplitPea™ makes it uniquely suited to handle the intricate morphologies of the substrates commonly employed in combinatorial chemistry. These include polymer resin beads, SynPhase™* Crowns or SynPhase™ Lanterns (see Figure 2).

Due to their specific design and high loading capacity (36 µM per item), these substrates are extremely popular for combinatorial chemistry work. However, they are also very problematic for infrared spectroscopic analysis.

The SplitPea™, however, allows straightforward measurements of the SynPhase™ Lanterns. The results of such measurements are shown in Figures 3 and 4.

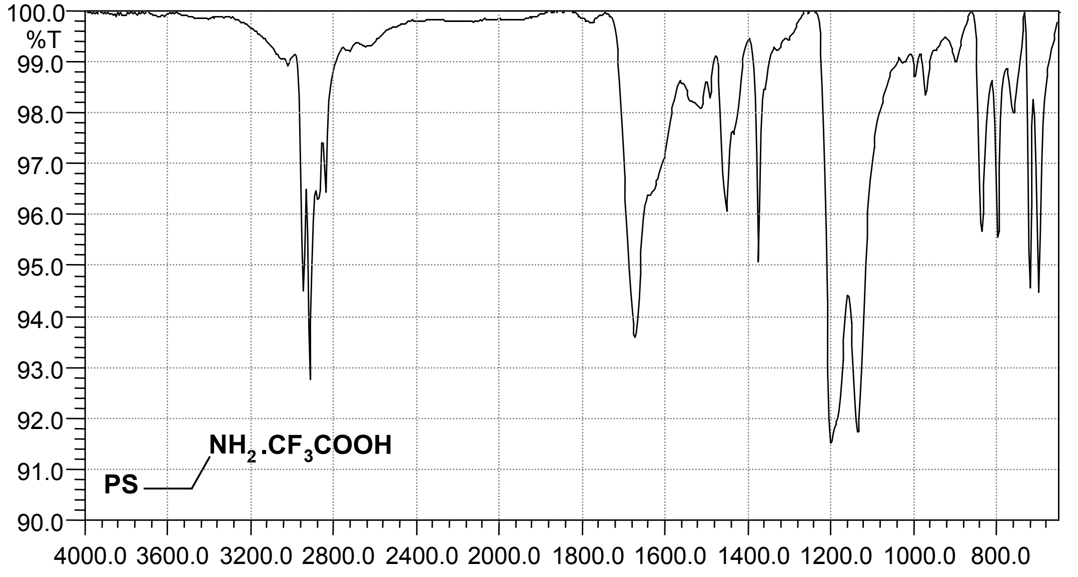

Figure 3. SynPhase™* Lantern Aminomethylated polystyrene, TFA

A New Tool for Solid-Phase Analysis in Combinatorial Chemistry

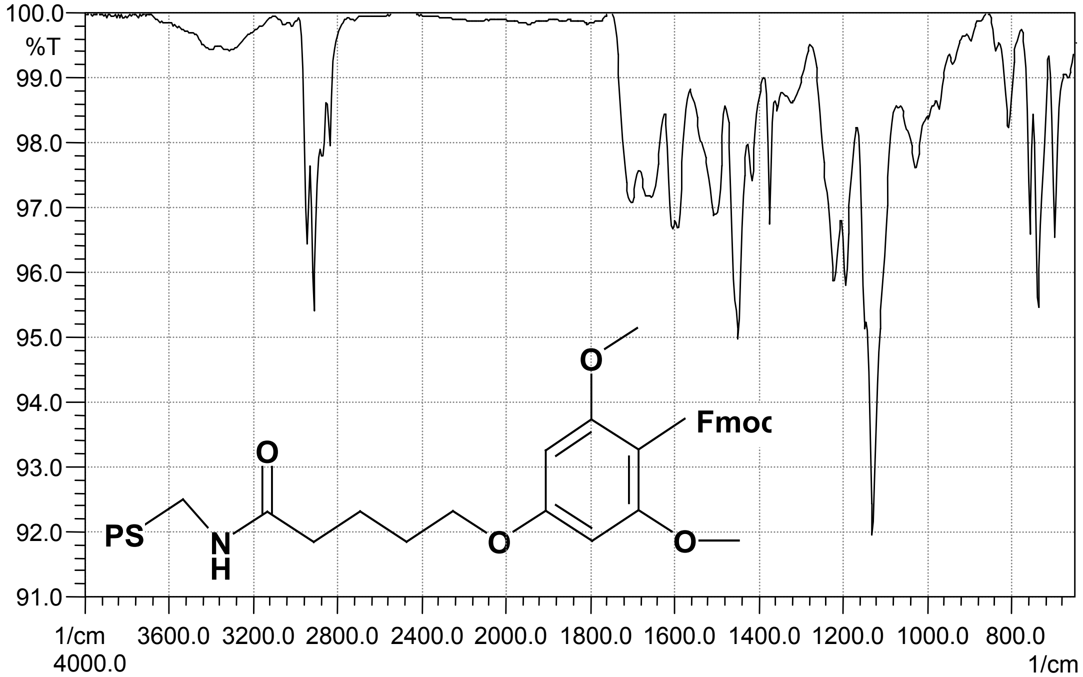

Figure 4: SynPhase™* Lantern polystyrene with PAL-Linker, Fmoc protected.

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[*] Registered trademark of Mimotopes Pty. Ltd., Australia.

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applications note
No. 70307

The Flexible Opto-Mechanical Arm for FTIR Analysis of Samples Outside the Sample Compartment

Appeared in *American Laboratory News Edition* **39(19)**, 22 (2007).

Figure 1. The DaVinci Arm.

Figure 2. Artwork Shown in the Easel Used for Positioning.

ABSTRACT

An articulated opto-mechanical arm, the daVinci Arm, was developed to enable *in-situ* analysis of large samples[1]. The Arm permits the analyses of samples by specular reflection, diffuse reflection and internal (ATR) reflection techniques. It mounts into the sample compartments of most commercial FTIR spectrometers. The ability to use a DTGS detector enables spectra to be recorded below 650 cm-1. Since the arm is articulated, samples can be analyzed within a range of distances in front, below, or above the sample compartment at a wide range of angles. The integral camera provides for magnified viewing and image capture of the sampled spot. The entire optical path of the IR beam is enclosed and integrated into the purge of the host spectrometer.

In specular and diffuse reflection mode, the sampled spot is about 1.25 mm in diameter. In ATR mode, the spot is 0.5 mm in diameter. The live image provided by the integral camera serves as a guide for placing the sample at the focus of the IR beam.

In this work, the daVinci arm is used for the analysis of a large (26” high and 22” wide) oil canvas. The painting is too large to fit into the sample compartment of a spectrometer and cannot be cut or otherwise damaged by the analysis. We demonstrate that the daVinci Arm enables the acquisition of good quality spectra outside of the sample compartment. Since the artwork itself cannot be touched, the only spectroscopic technique that can be utilized was diffuse reflection, which is a non-contact method.

INTRODUCTION

Optically, the daVinci Arm works as follows. The infrared beam is directed by the optics in the sample compartment of the spectrometer through the Arm to the sampling spot some distance away from the sample compartment. There it interacts with the sample either by a non-contact reflection technique or by diamond ATR, a contact sampling method. After interaction of the incident beam with the sample, the light reflected from the sample is returned to the sample compartment and directed to the spectrometer’s detector.

Since the light transfer is accomplished entirely by front-surface aluminum mirrors within purge-containing tubes, there is only a moderate loss of light

The Flexible Opto-Mechanical Arm for FTIR Analysis of Samples Outside the Sample Compartment

intensity due to the transfer optics. Unlike fiber optics, which also can be used for sample analysis outside of the sample compartment, there are no spectral range limitations. The Arm can be used from the UV to the far IR.

Any flat or convex shaped object can be placed in front of the spectrometer for analysis. For sheets of paper, textiles, and similar materials, placing them flat on a table and bringing the Arm in from the top is the easiest configuration for analysis. For other materials, such as the painting studied in this work, placing the sample vertically in front of the spectrometer is the most convenient position. In all cases, it is necessary to have the means to manipulate the sample so that the desired spot on the sample can be analyzed.

The Arm is designed such that, once aligned, it stays aligned within the entire available range of motion. In practice some re-optimization of the optical alignment may be needed when the position of the Arm is changed. It is best to collect the background spectrum in a position that is close to the final sampling position.

When used in the ATR mode, the Arm is totally enclosed and the purge gas within the Arm is fully contained. However, when used in diffuse/specular mode, the light must exit the Arm, interact with the sample and return back. An optically transparent window could be used to enclose the purged environment of the Arm, but that would limit the spectral range, introduce reflectance losses and defocus the beam. To avoid using an optical window, the Arm uses a purge shroud that leaves an aperture for light to exit and return. The size of this opening is as small as possible, making it feasible to maintain a positive purge pressure inside the Arm by slightly elevating the pressure of the purge gas.

EXPERIMENTAL

All spectra were collected at 8 cm^{-1} resolution with 64 scans coadded. For the collection of the background spectrum, a piece of machined stainless steel was used as a reference. The background spectrum was collected at the beginning of the experiment. For the last measurement, the position of the sampling head of the Arm moved significantly, so the alignment was re-optimized by maximizing the signal on the detector.

RESULTS AND DISCUSSION

The painting analyzed is shown in Figure 2. The painting was placed on an easel that

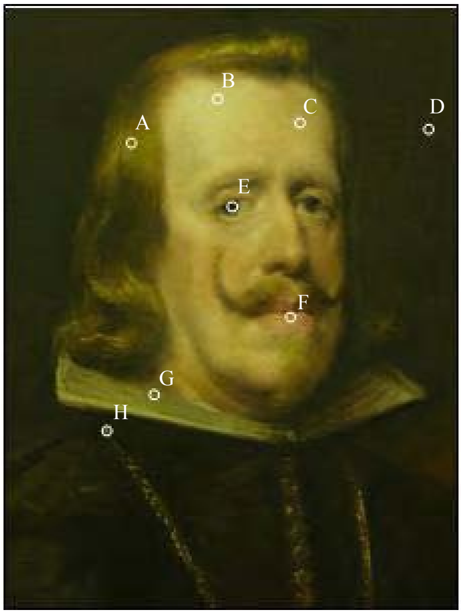

Figure 3. Close-up with Analyzed Points Indicated.

Figure 4. DaVinci Arm Shown in Position Analyzing Point H

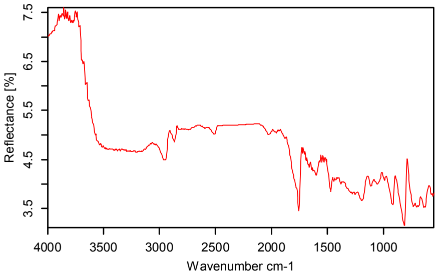

Figure 5. Diffuse Reflectance Spectrum of Point A.

The Flexible Opto-Mechanical Arm for FTIR Analysis of Samples Outside the Sample Compartment

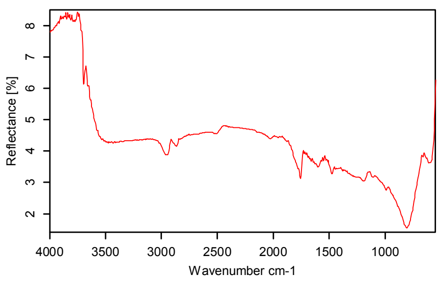

Figure 6. Diffuse Reflectance Spectrum of Point B.

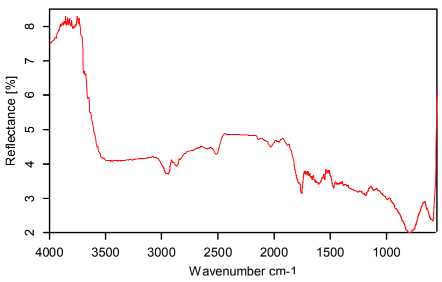

Figure 7. Diffuse Reflectance Spectrum of Point C.

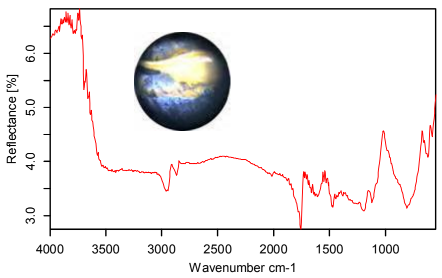

Figure 8. Diffuse Reflectance Spectrum of Point H with a Photograph of the Area Examined.

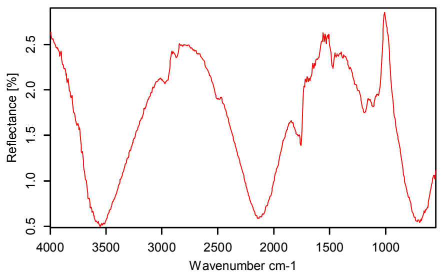

Figure 9. Diffuse Reflectance Spectrum of Point D.

allowed the painting to be tilted, raised, and lowered albeit in coarse steps of about 4”. The tilt of the easel was adjusted to make the canvas vertical and eight points were selected for analysis, as indicated in Figure 3. The choice of points required moving the painting sideways as well as up and down. Since the easel enabled only coarse vertical adjustments, the daVinci Arm was moved vertically to reach the chosen points. Each time the canvas was moved, the sampling head of the Arm was retracted to avoid inadvertently damaging the artwork. After the canvas was brought into the desired position, the sampling head of the Arm was extended towards the canvas into its sampling position.

The positioning of the Arm relative to the sample had to be done very carefully, since the shroud had to be brought very close (1 mm) to the sample without actually touching it. Figure 4 shows the Arm in position (point H) acquiring the spectrum shown in Figure 8.

Significant differences are seen in the diffuse reflectance spectra measured from different locations on the painting, as shown in Figures 5 through 12. Common features are presumably due to the common oil matrix and perhaps the underlying canvas while differences are due to the

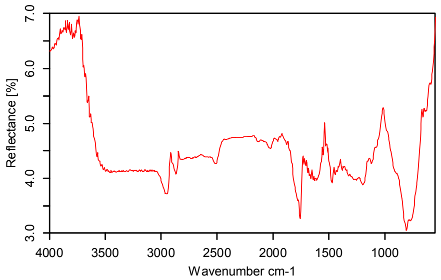

Figure 10. Diffuse Reflectance Spectrum of Point G.

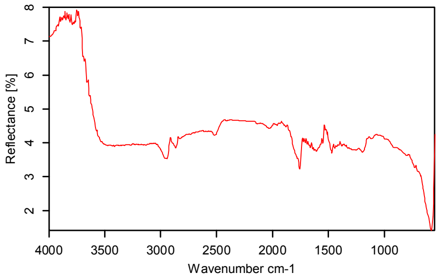

Figure 11. Diffuse Reflectance Spectrum of Point F.

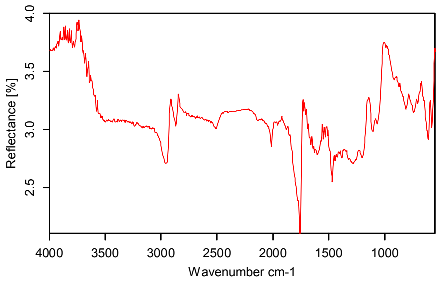

Figure 12. Diffuse Reflectance Spectrum of Point E.

various colored pigments used. However, these spectra are not straightforward to interpret. An oil painting is a complex reflecting system. For a detailed analysis, a careful study could be conducted to analyze and identify spectral features that are due to oil matrix, canvas, varnish, different pigments, etc. Once these samples analyzed by diffuse reflectance are thoroughly characterized, and the effects of the various components identified, the spectra could be more readily interpreted.

Note that although the spectra were recorded outside of the spectrometer sample compartment, the spectral quality is comparable to that of typical in-compartment measurements.

CONCLUSION

We have demonstrated that, using the daVinci Arm, a good quality spectra can be collected from spots on a large flat object that otherwise could not be analyzed by FTIR spectroscopy.

The particular object analyzed in this work was an oil painting that could not be touched while analyzed. Thus we used diffuse reflection, a non-contact technique to conduct the analysis. Presenting the object for analysis required moderate effort and care but was not difficult or time consuming. Less than 5 minutes was needed to get the artwork positioned for each measurement. Virtually any point on the sample could be analyzed.

The diffuse reflectance spectra obtained show considerable detail and thus contain significant information. This information is not easily interpreted, but the spectra do contain valuable information about the sample that could not be obtained otherwise.

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HARRICK SCIENTIFIC PRODUCTS

application note

No. 90112

UV-VIS Diffuse Reflection Spectroscopy of Thermochromic Materials

Figure 1. The Praying Mantis Diffuse Reflectance Accessory.

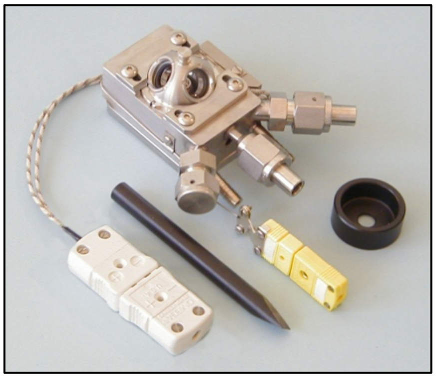

Figure 2. High Temperature Reaction Chamber for the Praying Mantis.

INTRODUCTION

Some materials undergo electronic transitions upon heating. These transitions can readily be studied by UV-VIS spectroscopy. Most samples examined in this manner are liquids. Such liquids are simply poured into commercially available thermostated cuvettes that the liquid is simply poured for analysis by transmission spectroscopy. Analysis of powders and other solids is less straightforward. Many of these samples are simply dissolved in a liquid for easy analysis.

For samples that are not readily soluble or must be analyzed neat, the only feasible UV-VIS analytical methods are specular reflectance and diffuse reflectance. The former is suitable for highly reflective materials; the latter for powders and roughened solids. Both require a mirror assembly to direct the radiation to and from the sample, in addition to a thermostated sampling stage.

This applications note explores the use of diffuse reflectance to probe the color changes of a rough-surfaced solid at various temperatures.

EXPERIMENTAL

The sample investigated herein was a Thermal Liquid Crystal Paint (Edmund Scientific, 3053489) with color changes in the 40-45°C temperature range. The paint was applied to one end of a sandblasted 316 stainless steel disk which fit into the sample cup of the reaction chamber. The paint was allowed to dry thoroughly prior to measurement. An unpainted disk was used as the reference.

The diffuse reflectance measurements were carried out using a commercial UV-VIS spectrometer with the Harrick Praying Mantis™ diffuse reflection accessory (Figure 1) along with its High Temperature Reaction Chamber (Figure 2) installed. Harrick's Automatic Temperature Controller was connected to the K-type thermocouple and the heater of the reaction chamber to regulate and monitor the temperature of the cell.

Prior to data collection, the reference disk was installed in the reaction chamber and the temperature controller was initially auto-tuned for operation at 40°C. The sample stage was then allowed to cool to ambient.

After the temperature stabilized, a reference spectrum was collected. This reference was used for all subsequent measurements.

The reference was then exchanged for the painted disk. Once the reference was in place, the temperature controller set point was selected to be 26°C. The controller was turned on and allowed to reach equilibrium at that temperature and then the sample spectrum was collected. Measurements were taken in this fashion at 39°C, 41°C, 42°C, 43°C, 44°C, 45°C and 46°C.

All the diffuse reflectance spectra were measured with a UV-VIS spectrometer in its double-beam mode with an open slit, a 2-nm SBW and a 5-nm data interval.

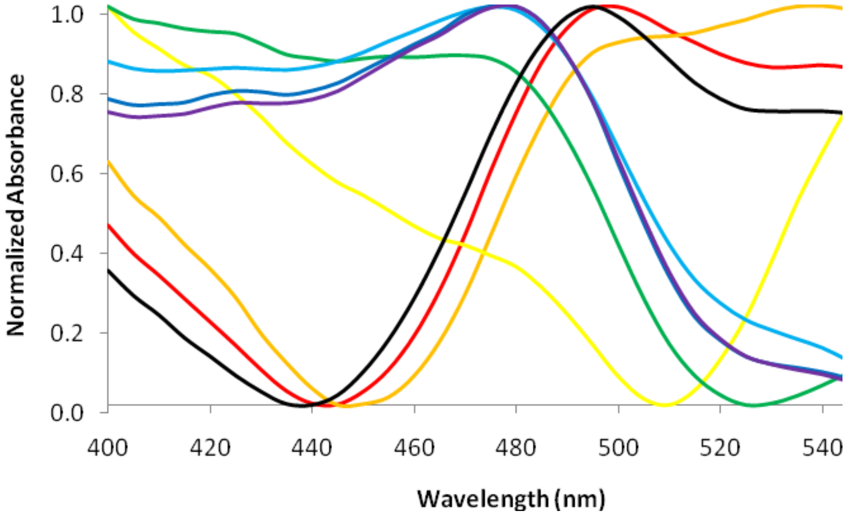

Figure 3. Diffuse Reflectance Spectra of Thermal Liquid Crystal Paint Measured at 26°C (red), 39°C (orange), 41°C (yellow), 42°C (green), 43°C (light blue), 44°C (dark blue), 45°C (purple) and 46°C (black).

RESULTS AND DISCUSSION

Figure 3 shows the spectra of the sample recorded at eight different temperatures. At the lowest temperature, 26°C, the spectrum has a peak at 500nm, in the green region of the visible. As the sample is heated, the peak at 500nm decreases in intensity and another peak arises at 480nm, in the blue. Thus this particular paint changes color in the blue-green region of the visible spectrum as a function of temperature between 26° and 45°. Beyond the working range of the paint, at 46°C, the paint reverts to something akin to its original green wavelength.

CONCLUSION

From the above, it is clear that diffuse reflectance, using the Praying Mantis with its high temperature reaction chamber, is an effective way of measuring temperature-induced wavelength changes, *i.e.* thermochromism, and observing the underlying electronic transitions. The diffuse reflectance sampling technique, combined with a temperature-controlled chamber, is an effective analytical tool for studying temperature-induced changes in solid materials such as powders and rough-surfaced solids. Additional applications include analyzing gas-solid interactions including heterogeneous catalysis.

HARRICK
HARRICK SCIENTIFIC PRODUCTS
applications note
No. 52601

THE DEGREE OF POLYMER CURING, AS MEASURED BY ATR SPECTROSCOPY

SUBMITTED BY HECTOR CASAL, DHC ANALYSIS INC.

Figure 1. The SplitPea™.

INTRODUCTION

The degree of cure is an important QC parameter in many applications. The extent of polymerization determines the physical properties of many materials. Typical examples are inks, varnishes, coatings for electronic components, etc. Many of these materials are cured with UV or electron beam radiation.

In this short example we show how the data can be correlated with process parameters which in turn determine the basic properties of the final product. In this example, the critical parameter is adhesive performance, known to be dependent on the equivalent coating speed.

EXPERIMENTAL

In all cases, it is possible to use the Split Pea accessory to obtain excellent spectra in short time. The data we present here are from a series of UV-cured adhesives. These adhesives all use acrylate chemistry.

The samples were irradiated in a pilot plant with UV light, the samples travel through the UV irradiation chamber at different speeds.

The SplitPea ATR accessory, shown in Figure 1, was used to measure spectra quickly and reliably; the

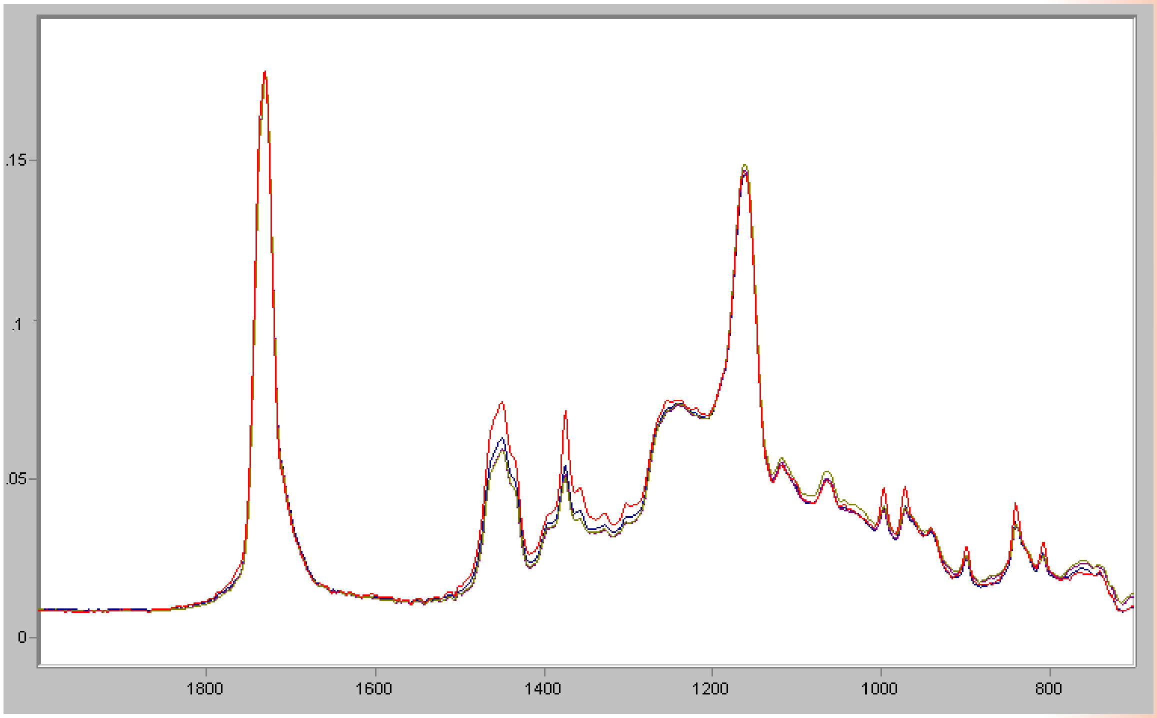

Figure 21: ATR spectra of 4 samples of UV-cured adhesive.

collection time was 30 seconds at 4 cm^{-1} resolution.

RESULTS AND DISCUSSION

Spectra of four samples are shown in Figure 2; these samples were irradiated under different conditions. The differences are clearly evident in the measured spectra. There are some clear trends in band intensities with irradiation time. As a first approximation, the band intensity at ~808 cm^{-1} to provide a quantification of the changes observed in the spectra.

The change in the relative intensity of the signal at ~808 cm^{-1} can be correlated to the known lamp power as seen in Figure 3 and also to the known coating speed as shown in Figure 4.

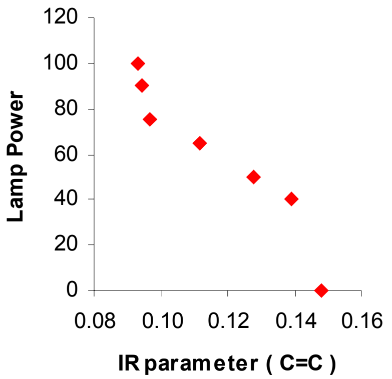

Figure 3: Variation of the relative intensity at 808 cm^{-1} with lamp power. The lamp power is expressed as a percentage of the total possible output.

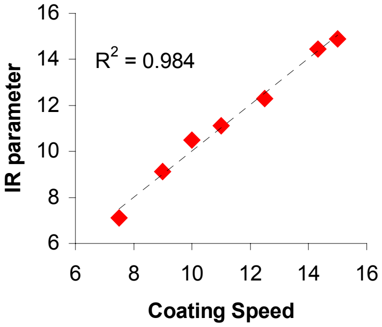

Figure 4: Variation of the relative intensity of the 808 cm^{-1} signal with coating speed in m/min.

CONCLUSION

In some cases, it might not be possible to generate the data for curves of this type. For example, it might not be possible to produce samples at different coating speeds, radiation intensity, etc. However, it is still possible to implement a QC test based on ATR spectra measured with the Split Pea accessory. A group of samples that have passed all the performance tests can be used as the standard. Spectra of production samples are compared to those of the standard group to determine quickly if the production samples meet specification criteria. There are several chemometric and search-style mathematical tools available for comparing spectra reliably

No. 50101

Correlating the Polarizer Indicator to S- and P-Polarization Via ATR

Figure 1. The Horizon ATR Accessory.

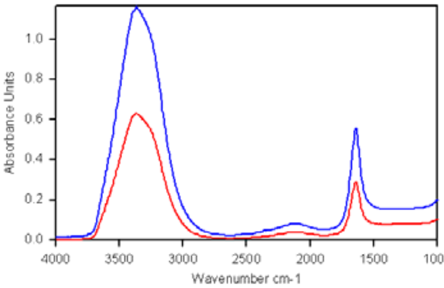

Figure 2. ATR Spectra of Water Recorded with the Polarizer Set to '90' (red) and '0' (blue).

Introduction

Commercial infrared polarizers typically have an indicator showing the orientation of the polarizing element in its mount. The polarizer manuals generally relate the direction of the electric field vector to the indicator. This can then be correlated to the direction of the sample plane to determine which polarization, parallel (p) or perpendicular (s), impinges on the sample.

For transmission, this relationship is straightforward. For reflectance, an accessory is usually used to direct the infrared radiation to and from the sample via mirrors, and this can rotate or even scramble the direction of the electric field. While the desired polarization can be determined theoretically, it is not always easy to do so. If the polarizer manual or the optical layout of the accessory are unavailable, it becomes difficult to use solely theoretical considerations.

However, the orientation of the polarizer can also be determined experimentally. For ATR, it is well-known that the effecting thickness for p-polarized light is nearly twice that for s-polarized light at a 45° incident angle.[1] This factor of two in the effective thickness is directly reflected in the spectral band intensities.

This applications note describes a quick test to experimentally distinguish the two perpendicular positions, generally marked '0' and '90' on the polarizer mount, using ATR spectroscopy.

Experimental

All spectra were collected using the Horizon, a fixed 45° incident angle multiple reflection ATR accessory, and a wire grid polarizer (KRS-5 substrate) installed in a commercial FT-IR spectrometer. The spectrometer was set for 16 scans at 8 cm^{-1} resolution.

The polarizer indicator was set to '90' and the background spectrum was collected. The Horizon trough was filled with water and the sample spectrum was calculated. The polarizer was then rotated 90° to the '0' setting to supply the other polarization and the data collection process was repeated.

Results and Discussion

The resulting spectra are shown in Figure 2. As expected from the relative effective thicknesses at a 45° incident angle, one of the spectra has band intensities roughly twice that of the other. Hence the '0' setting on this polarizer provides p-polarized radiation to the sample.

This method can be used with practically any sample type and any ATR accessory to determine or verify the polarization. The only exception is for those accessories that scramble the incident polarization, such as those that utilize a rod-style ATR crystal.

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HARRICK
SCIENTIFIC
PRODUCTS

No. 70524

application note

DIFFUSE REFLECTANCE SAMPLING METHODS

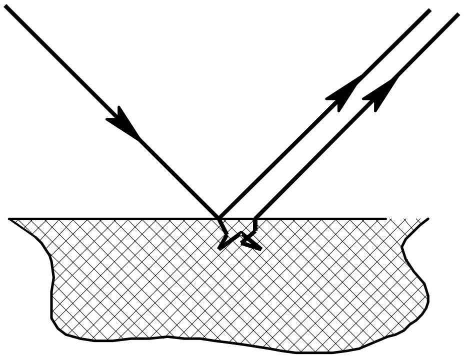

Figure 1. Schematic of the two components of the reflected radiation present in diffuse reflection. Reflected rays are shown in only one direction for clarity.

Figure 2. The Cricket™ Diffuse Reflectance Accessory. Shown here with its TransFlex™ substrates.

INTRODUCTION

Diffuse reflectance is frequently used for quantitative and qualitative infrared analysis of powders and rough surface solids. However, this technique has come to encompass two slightly different methods: true diffuse reflectance and 'in-line diffuse' reflectance. True diffuse reflectance optimizes the collection of the scattered radiation and minimizes the collection of specularly reflected radiation, reducing reststrahlen effects and more closely matching the Kubelka-Munk theoretical requirements. In-line diffuse reflectance, on the other hand, collects much more light, by detecting both the scattered and specular radiation. Although not strictly diffuse reflectance, this technique is frequently used for quickly obtaining spectral information. This paper demonstrates the differences between these two sampling techniques for the common types of samples investigated by diffuse reflectance. These samples include neat solids, solids diluted in a non-absorbing matrix, liquids deposited onto a scattering surface, and solids deposited onto an abrasive.

THEORY

The diffuse reflection spectroscopic technique was developed to facilitate analysis of materials such as papers and powders in their neat state. The theory of diffuse reflection has been investigated in detail by many authors[1, 2, 3].

When an inhomogeneous material is illuminated, some of the impinging radiation penetrates the sample and some is reflected from the surface (see Fig. 1). The portion that penetrates the sample undergoes scattering at a large number of points in its path. The fraction of this radiation that comes back out of the sample is the diffusely reflected component. This component is theoretically described by the widely-used Kubelka-Munk model[4]. The specularly reflected component is just directionally scrambled reflectance (*i.e.* diffused) and hence is described by the Fresnel equations.

In the low absorption limit, the specular component as a function of the refractive index is mainly determined by the real part of the refractive index. Hence it is essentially independent of wavelength and merely causes an easily removable offset in the measured value.

For a strong absorption index, however, the front surface reflectance is a function of both components of the complex refractive index. So the specular component increases with an increase in absorption index. This increased level of reflected radiation counteracts the absorption by the sample. Thus as a function of the absorption index, the in-line diffuse reflectance at first decreases and then reverses direction and starts increasing. As a result, strong absorption peaks degenerate into spurious doublets, also known as reststrahlen bands, and the in-line diffuse reflectance becomes un-interpretable.

Experimental methods that avoid or at least suppress the influence of the specular component collect the true diffuse reflectance spectra. Those that collect a combination of the diffuse and specular components are often also referred to as 'diffuse reflectance,' but are perhaps more correctly named in-line diffuse reflectance.

EXPERIMENTAL

The diffuse reflectance spectra were recorded using a Nicolet Nexus 670 set for 8 cm^{-1} resolution and 32 scans. The Nexus was equipped Harrick Scientific's Cricket™, shown in Figure 1. The Cricket™ was used with both of its modes of operation to collect either in-line diffuse reflectance or diffuse reflectance. For comparison, ATR spectra were collected using the Harrick SplitPea™.

The samples were chosen to reflect the variety of samples that can be analyzed by diffuse reflectance. Specifically, these samples included white paper, spackle (Custom Building Product's Interior Spackling Paste), malachite rock, silicone oil, and a residue.

Several different methods were used to sample these materials, depending on the nature of the sample. The paper was examined neat. The spackle and silicone oil were smeared onto TransFlex™ (roughened aluminum) substrates for analysis. For the residue, a drop of the contaminated toluene was placed on a TransFlex™ substrate and allowed to dry. The malachite rock was abraded by 220-grit silicon carbide sandpaper (Norton Consumer Product's Tukbak Gold T481) and analyzed thereon. The malachite was also pulverized for dilution in ground KBr and for smearing onto a TransFlex™ substrate.

RESULTS AND DISCUSSION

Figures 3 and 4 demonstrate the fundamental difference between diffuse and in-line diffuse reflectance. Figure 3 shows the reflectance of paper, recorded by both diffuse reflectance techniques and by ATR. As seen in the ATR spectrum, paper is highly absorbing around 1040-cm^{-1}. This results in reststrahlen bands in the in-line diffuse reflection spectrum that are

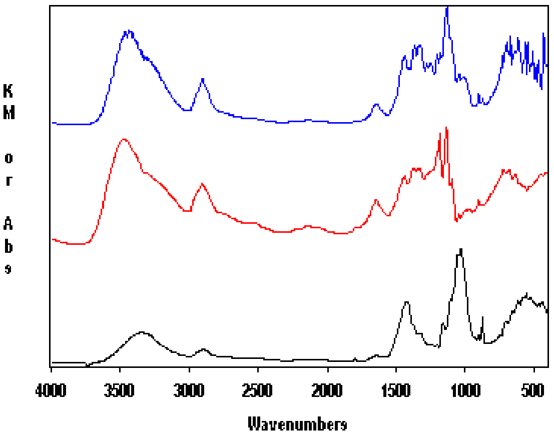

Figure 3. The Reflectance Spectra of Paper Measured by Diffuse Reflectance (top), In-Line Diffuse Reflectance (middle), and ATR (bottom).

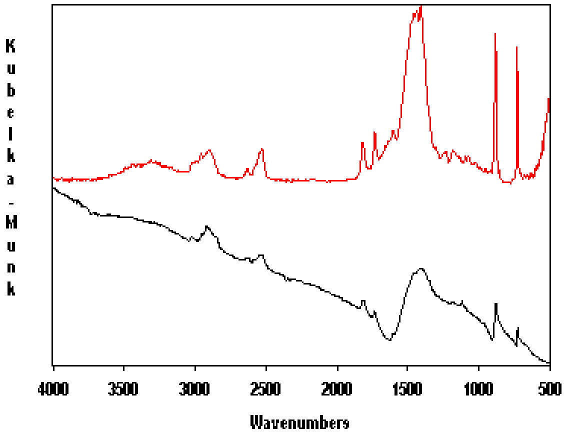

Figure 4. The Reflectance Spectra of Spackle Measured by Diffuse Reflectance (top) and In-Line Diffuse Reflectance (bottom).

absent in the diffuse reflection spectrum.

Figure 4 shows the reflectance of spackle on a TransFlex™ substrate. Here, the in-line diffuse reflection spectrum shows dispersion effects through band inversions and a sloping baseline. These effects are not apparent in the diffuse reflection spectra, although the noise level is higher due to the lower signal levels. This clearly indicates that diffuse reflectance sampling is a better method for strongly absorbing materials, like paper and spackle, than in-line diffuse reflectance.

Figure 5 shows spectra from powdered malachite diluted in KBr. By diluting this sample, it has been made into a weak absorber. Theory predicts that, under these conditions, the diffuse reflection spectrum and the in-line diffuse reflection spectrum will be similar. This is indeed the case. The spectra show only minor differences, particularly in the C-H region.

Figure 6 shows the diffuse reflectance spectra from malachite sampled in three different ways. Dilution in KBR is the traditional method of choice for analysis of solids and powders, despite the fact that it is labor-intensive. The top curve shows the excellent quality of spectra achieved by this method. An alternate approach is to abrade the sample with silicon carbide sandpaper. This is a quick and easy method to use. However, since the sandpaper itself is a poor reflector, this can produce noisy spectra with poorly resolved bands, as shown in the lower spectrum. The result of another quick and easy method is shown in the middle spectrum, where the powdered sample was smeared across the TransFlex™ substrate. Because of the higher total reflectivity of the substrate, better quality spectra are obtained than with the sandpaper.

Figures 7 and 8 demonstrate that these reflectance methods can also be used to examine liquids. Figure 7 shows the spectra of the residue from contaminated toluene evaporated on a TransFlex™ substrate. Here, the diffuse reflection spectrum more clearly resolves the O-H stretch, even though the spectrum is noisier. The comparable band in the in-line diffuse reflection spectrum is obscured by the sloping baseline and other distortions. The contaminant here is, most likely, a type of wax.

Figure 8 is the spectrum of silicone oil smeared on a TransFlex™ substrate, recorded in the diffuse reflectance mode of the Cricket™. As can be seen from the spectrum, this technique, also known as transflectance, can be used effectively to obtain spectral information on liquids.

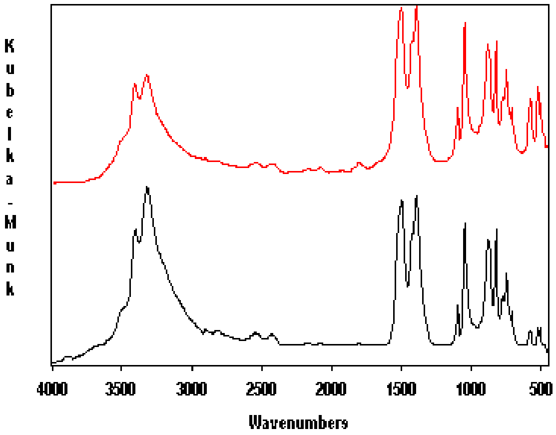

Figure 5. The Reflectance Spectra of Ground Malachite in KBr Measured by Diffuse Reflectance (top) and In-Line Diffuse Reflectance (bottom).

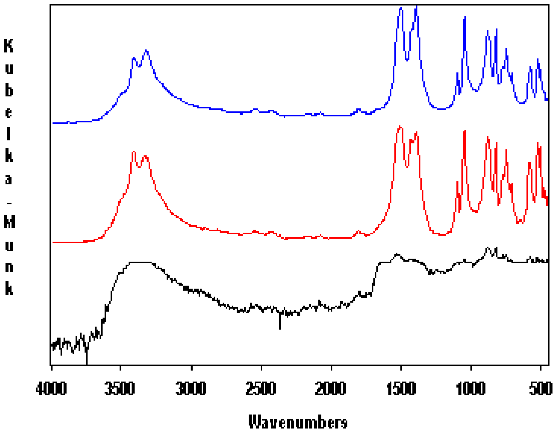

Figure 6. The Reflectance Spectra of Malachite Powder Diluted in KBr (top), Powder Smeared on a TransFlex™ substrate (middle), and Abraded with Silicon Carbide Sandpaper (bottom).

DIFFUSE REFLECTANCE SAMPLING METHODS

CONCLUSION

The diffuse reflectance technique has come to encompass two slightly different methods: diffuse reflectance and in-line diffuse reflectance. This paper demonstrates that diffuse reflectance is superior for obtaining spectra that are free from distortions than the quick in-line diffuse reflectance sampling methods in use today.

Of the different sample preparation methods available for these types of measurements, examining the samples neat or smeared on a TransFlex™ substrate will give the best spectra in the least amount of time. Dilution in KBr, however, remains the best choice for strict applications of the Kubelka-Munk theory.

Furthermore, diffuse reflectance techniques are not just for solids anymore. With the appropriate hardware, information on liquids and pastes and be readily obtained.

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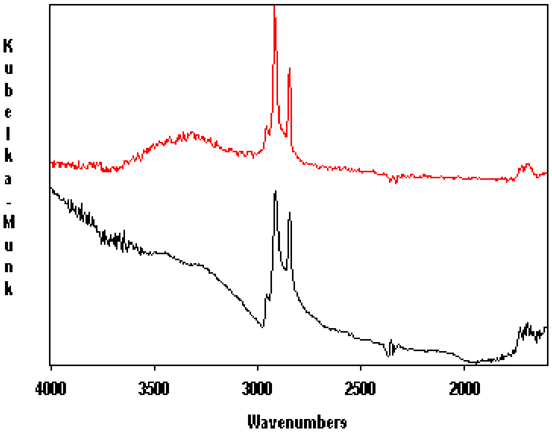

Figure 7. The Reflectance Spectra of the Residue from Evaporated Contaminated Toluene Measured by Diffuse Reflectance (top) and In-Line Diffuse Reflectance (bottom).

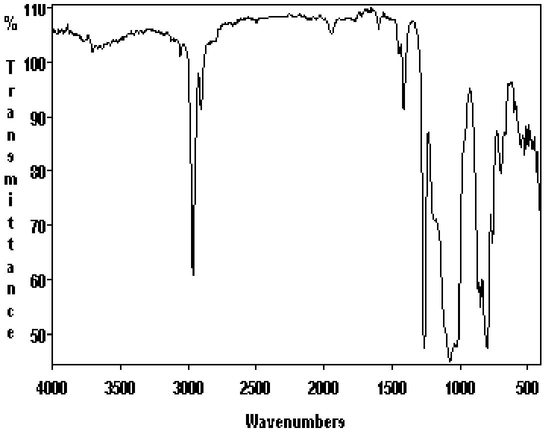

Figure 8. The Transflectance Spectrum of Silicon Oil on a TransFlex™ Substrate.

HARRICK
SCIENTIFIC
PRODUCTS

No. 70521

application note

A FASTIR WAY OF GRINDING OUT SPECTRA

Figure 1. The FastIR™.

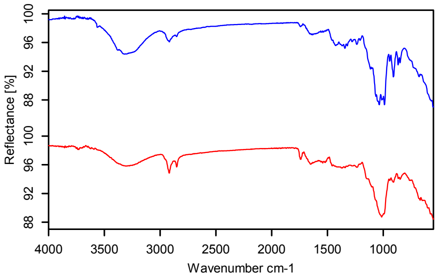

Figure 2. ATR Spectrum of Hot Chocolate Mixes: Carnation (blue) and Swiss Miss (red).

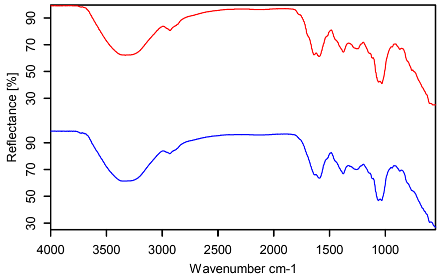

Figure 3. ATR Spectra of Instant Coffee (red) and Decaffeinated Coffee (blue).

INTRODUCTION

Internal reflectance spectroscopy (ATR)[1] is used widely for studying a variety of liquids, pastes, and solids. It is well suited for analyzing optically thick and opaque samples that cannot be examined via conventional transmission spectroscopy. Internal reflectance is straightforward and convenient, resulting in minimal disruption of the sample and requiring minimal sample preparation. For liquids and pastes, the sample is simply placed, as is, in contact with an internal reflectance element (IRE) and the spectrum recorded. For solids, the sample is placed on the IRE and pressure is applied to obtain good contact between the sample and the IRE.

ATR spectra are most commonly recorded using multiple reflection accessories. These accessories allow the beam to repeatedly interact with the sample to provide a greater pathlength. To direct the beam through the crystal, these accessories frequently have multiple mirrors that must be correctly positioned to optimize the performance of the accessory. Given the excellent signal-to-noise ratios of today's spectrometers, many samples do not require multiple interactions to obtain good quality spectra.

Spectra can be easily obtained using single reflection ATR. Single reflection ATR is extremely convenient, both in terms of experimental set-up and sample analysis. The accessories are lower cost than their multiple reflection counter-parts and are very durable.

To illustrate the versatility and power of single reflection ATR, several types of samples have been examined and the results are presented herein.

PERCOLATED RESULTS AND COFFEE HOUSE DISCUSSION

The samples were analyzed using the FastIR™ (Figure 1) in a Mattson Polaris spectrometer with a DTGS detector. The FastIR™ is a horizontal sampling, single internal reflection accessory with fixed 45° incident angle.

The spectrometer collected 16 scans and 8 cm^{-1} resolution with a 30% aperture. Background single beam spectra were recorded with the clean IRE as the reference. All the samples were examined on a flat sampling plate. The powders were compressed against the crystal using the pressure applicator and the films were cast directly onto the crystal.

Figure 2 shows the internal reflectance of two powdered hot chocolate mixes: Swiss Miss® Chocolate Sensation and Carnation® Hot Cocoa Mix. From the spectra, it is clear that the two different blends of hot chocolate use similar ingredients in different proportions.

Figure 3 compares instant coffee and instant decaffeinated coffee. Both types of coffee were cast as films on the crystal. The Taster's Choice® coffees were dissolved in hot water, dripped onto the crystal of the FastIR™, and dried to form films. The coffee spectra express their differences in the 1700 cm^{-1} region, where the decaffeinated coffee lacks some shoulder bands that are present in the non-decaffeinated coffee. These bands are probably due to caffeine or other components that are removed during the caffeine extraction.

And for those who prefer the aroma of fresh ground coffee, Figure 4 compares the spectrum of fresh coffee grounds to the Taster's Choice® instant freeze-dried powder. The instant coffee shows much weaker C-H and carbonyl bands. This is due to the lower concentration of aromatics, cellulose, and other materials that are removed during the freeze-drying process.

SUMMARY

Single, internal reflectance (ATR) measurements can be used for quantitative and/or qualitative analysis. The FastIR™ provides a convenient, quick and reliable method of examining solids and pastes without elaborate sample handling or preparations. The FastIR™ is especially good for the last drop!

REFERENCES

[1]N. J. Harrick, *Internal Reflection Spectroscopy* (Harrick Scientific Corp., Ossining, New York, 1967).

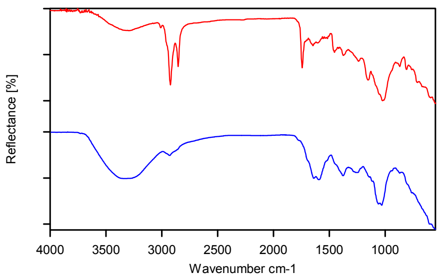

Figure 4. ATR Spectra of Fresh Grounds (red) and Instant Coffee Powder (blue).

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No. 62804

GRAZING ANGLE REFLECTANCE OF SMALL, COATED METALLIC SUBSTRATES

Figure 1. Focus Grazing Angle Accessory.

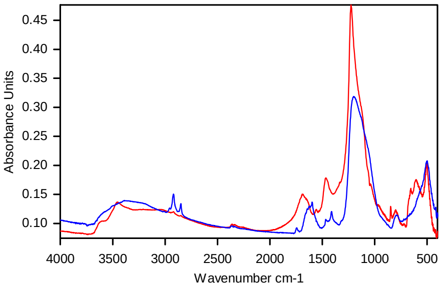

Figure 2. Grazing angle reflectance spectra of two coated steel rivets.

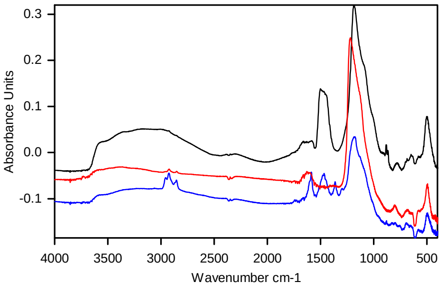

Figure 3. Grazing angle reflectance spectra of three coated copper chips.

INTRODUCTION

Thin films on metal substrates are routinely analyzed by grazing angle specular reflection spectroscopy using accessories such as the Refractor™,[1]. The incident beam is polarized to provide radiation polarized in the plane of incidence since only p-polarized radiation exhibits sensitivity to thin films on metallic surfaces. However, in grazing angle reflectance, the incident beam has a long footprint on the sample and thus, the sample needs to be at least 32 x 15 mm in size. If the sample is smaller, the incoming beam overfills the sample and a portion of the beam is simply not reflected. Analyzing a spot on the sample that is 3 mm in diameter spot is almost impossible using the conventional grazing angle reflectance accessories.

This applications note uses the grazing angle technique to investigate small spots on coated metallic substrates with a specially configured FT-IR accessory.

EXPERIMENTAL

The samples were analyzed using the Focus™ grazing angle reflectance accessory (see Figure 1). This accessory was designed specifically to efficiently analyze small reflective samples and is equipped with a built-in polarizer plate that provides the required p-polarized light. The Focus™ also features PermaPurge™, integrating the accessory into the purge of the FTIR spectrometer. Thus, once the purge of the system is established, the samples can be analyzed rapidly one after the other.

Two types of samples were investigates: small steel rivets and copper chips. The steel rivets were 10 mm in diameter and approximately 3 mm thick. The copper chips were roughly 10 x 10 x 1 mm. Both samples were smooth but not optically polished and were coated with thin films of silicates deposited under different processing conditions.

To examine the samples, a background was collected using a front-surface aluminum mirror placed face-down on the sample stage. Then the mirror was replaced by the sample and the spectrum was collected. All spectra were collected on a commercial FT-IR spectrometer at 4 cm^{-1} resolution with 64 scans co-added. These conditions resulted in a data collection time of just over 1 minute per sample.

GRAZING ANGLE REFLECTANCE OF SMALL, COATED METALLIC SUBSTRATES

RESULTS AND DISCUSSION

The spectra recorded from two of the steel rivets are shown in Figure 2. Both spectra show a band at approximate 1200 cm^{-1}. This is the Si-O band from the depositied silicates. The spectrum shown in blue also has stronger C-H bands, around 2900 cm^{-1}, indicating a higher concentration of hydrocarbons in the coating.

The spectra measured from the copper chips are presented in Figure 3. The spectra of the copper chips show a strong Si-O band around 1200 cm^{-1}, as expected. The three spectra shown indicate differing compositions, particularly with respect to the hydrocarbon and hydrocarbon and hydroxide bands.

Note that some of the copper samples were visibly twisted, as seen in Figure 4. The curvature of the sample defocuses the beam of the spectrometer on the detector. Thus, generally a baseline offset or a curved baseline results from the curvature of the sample.

CONCLUSION

Grazing angle reflectance spectroscopy is the technique to analyze thin coatings on smooth but unpolished metal substrates. Normally, the technique requires relatively large, uniform samples. However, small samples are often all that is available. For these small samples, as well as for analysis of small spots on large samples, the Focus™ represents a unique and important extension to the standard grazing angle reflectance equipment.

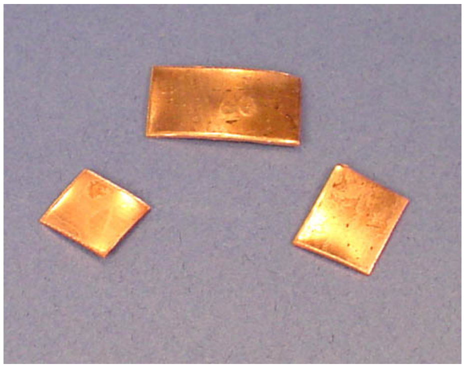

Figure 4. The three coated copper chips.

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[1] N.J. Harrick and M. Milosevic, *Appl. Spectros.*, 44 (3), 519 (1990).

HARRICK
HARRICK SCIENTIFIC PRODUCTS

No. 41109

applications note

IDENTIFICATION OF UNKNOWN FIBERS USING DIAMOND ATR SPECTROSCOPY

Figure 1. The VideoMeridian™ viewing diamond ATR with optional video display.

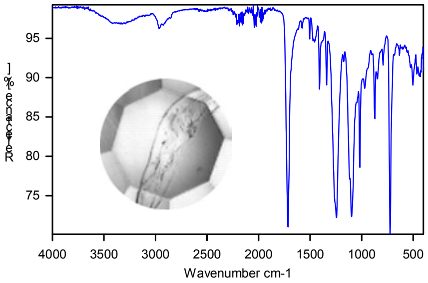

Figure 2. ATR spectrum of a fiber from a neutral-colored flat-weave rug.

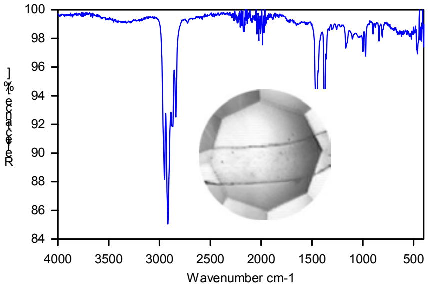

Figure 3. ATR spectrum of a fiber from a neutral-colored tufted rug.

INTRODUCTION

Identification of known fibers and hairs is vital for forensic and crime scene investigations. In these applications, it is critical to have quick, straightforward and accurate methods of analysis with as much documentation as possible. Having the ability to analyze samples in a timely manner can be crutial to a forensics case.

Infrared ATR spectroscopy is an excellent method for identification of such materials. ATR microsamping accessories, combined with the high signal-to-noise ratios of today's FTIR spectrometers, make a powerful tool for examining small samples like fibers and hairs. Furthermore, some of these ATR accessories are now equipped with digital imaging capabilities, simplifying photographic documentation of the samples investigated.

In this applications note, several unknown fibers are examined using Harrick's VideoMeridian™ diamond ATR micro-sampler. Both spectra and digital images are documented. The former are used to identify the composition of the samples, and the latter are exact images of the samples analyzed.

EXPERIMENTAL

Unknown fibers were collected from common household materials. Individual fibers were extracted from rugs and clothes, and an imbedded fiber from a U.S. twenty-dollar bill. These were examined using the VideoMeridian™ with its type IIA diamond ATR crystal (Figure 1) in an FT-IR spectrometer. All spectra were collected with 32 scans at 4 cm^{-1} resolution and a DTGS detector.

Background single beam spectra were collected using the clean diamond ATR crystal. Then the samples were centered on the ATR crystal, using the video display, and the samples were compressed against the diamond with 15 units of pressure on the display. Next, the sample spectra and corresponding digital photographs were collected. Data collection typically took 2.5 minutes, for an experienced operator.

The spectra were then processed through an FT-IR ATR library for identification of the samples. The ATR crystal was cleaned with methyl ethyl ketone between samples.

RESULTS AND DISCUSSION

Figures 2 through 5 show spectra and digital image of the unsupported fibers, as measured with the VideoMeridian.

The spectra were all compared to on-file library spectra and the fibers were identified as follows. The fiber from the neutral-colored flat-weave rug, shown in Figure 2, is a polyester. The tufted rug fiber, whose spectrum appears in Figure 3, is a polyproplyene. The red fiber, shown in Figure 4, is a cotton-polyester blend, and the blue fiber in Figure 5 is made from nylon. Note that all the spectra have an increased noise level around 2100 cm^{-1}. This is due to incomplete compensation of the highly absorbing diamond lattice bands. Very few materials actually absorb in this small range and hence this does not significantly limit the use of this method for identification of materials.

From the digital photographs, it is clear that the diameters of the compressed fibers varied by roughly a factor of three. Since the sampled area on the VideoMeridain diamond is 500µm in diameter, it is possible to estimate the size of the compressed fibers. Figure 4 shows the thinnest fiber examined, and it was compressed to approximately 60µm in diameter.

In addition to free-standing fibers, imbedded fibers can also be examined and identified. Figure 6 shows the ATR spectra from two sections of the bill. One section has a noticeable blue fiber in it that, as deduced from the photograph (Figure 7), is approximately 65 µm in diameter. The other section was more uniform and examined as a reference. The bands at 1715 cm^{-1} and 1250 cm^{-1} appear in the spectrum of the fiber but not in the spectrum of paper. Subtraction results in a spectrum that can be clearly identified as polyester.

SUMMARY

As seen above, the VideoMeridian is a powerful tool for analyzing and identifying unknown fibers. Its video-imaging capabilities are useful both for positioning samples on the diamond ATR crystal and for maintaining photographic documentation.

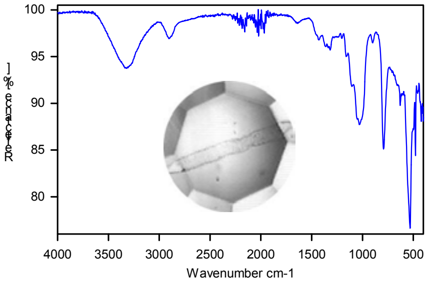

Figure 4. ATR spectrum of a red fiber.

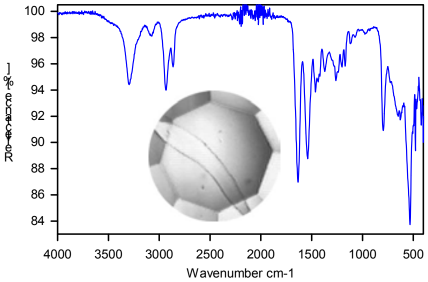

Figure 5. ATR spectrum of a blue fiber.

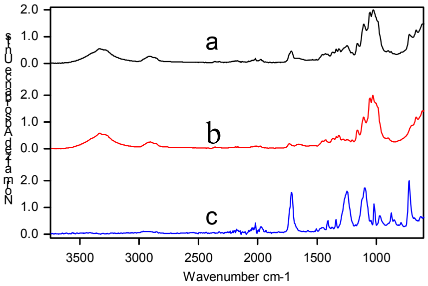

Figure 6. Normalized ATR Spectra of a blue fiber on the greenback (a), an area near that blue fiber (b) and the difference (c).

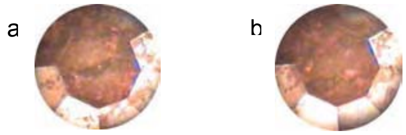

Figure 7. Photographs of the blue fiber on the greenback (a) and an area near that blue fiber (b). Spectra shown in Figure 6.

GRAZING ANGLE REFLECTANCE OF SMALL, COATED METALLIC SUBSTRATES

RESULTS AND DISCUSSION

The spectra recorded from two of the steel rivets are shown in Figure 2. Both spectra show a band at approximate 1200 cm^{-1}. This is the Si-O band from the depositied silicates. The spectrum shown in blue also has stronger C-H bands, around 2900 cm^{-1}, indicating a higher concentration of hydrocarbons in the coating.

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Note that some of the copper samples were visibly twisted, as seen in Figure 4. The curvature of the sample defocuses the beam of the spectrometer on the detector. Thus, generally a baseline offset or a curved baseline results from the curvature of the sample.

CONCLUSION

Grazing angle reflectance spectroscopy is the technique to analyze thin coatings on smooth but unpolished metal substrates. Normally, the technique requires relatively large, uniform samples. However, small samples are often all that is available. For these small samples, as well as for analysis of small spots on large samples, the Focus™ represents a unique and important extension to the standard grazing angle reflectance equipment.

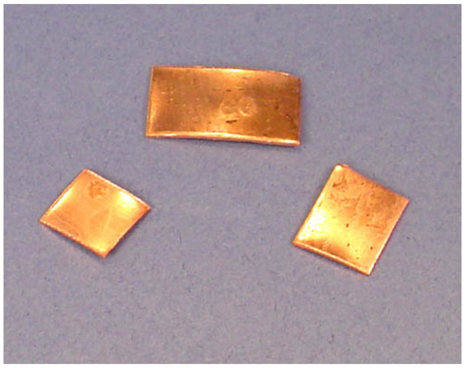

Figure 4. The three coated copper chips.

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[1] N.J. Harrick and M. Milosevic, *Appl. Spectros.*, 44 (3), 519 (1990).

HARRICK

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No. 40302

INFRARED DETECTION OF FILMS ON ROUGH SURFACES: AN ATR STUDY OF HARDWOOD FLOORING

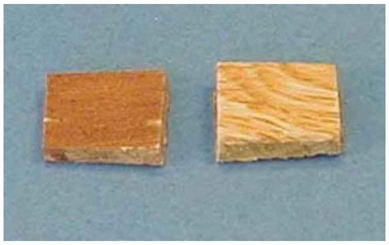


Figure 1. Samples A (left) and B (right).

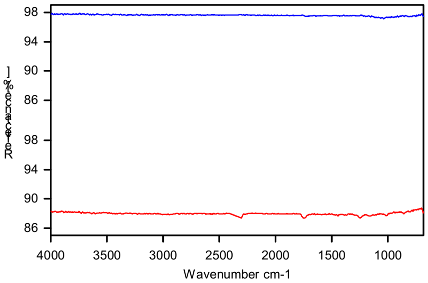


Figure 2. ATR Spectra of Sample A (blue) and B (red) Recorded with the Horizon.

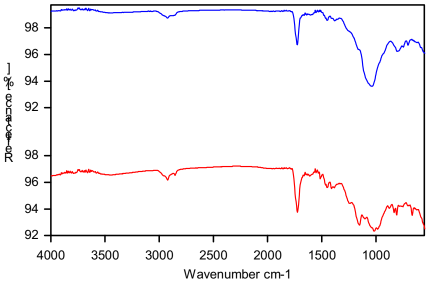


Figure 3. ATR Spectra of Sample A (blue) and B (red) Recorded with the FastIR.

INTRODUCTION

The flooring industry is continually improving flooring materials to make them more durable while limiting the degree of discoloration over time. Multiple reflection ATR infrared spectroscopy is frequently used to examine the man-made flooring materials, like vinyl, but presents problems with the natural flooring materials like the hardwoods. The hardwoods are not flexible enough to obtain good contact over the large area of the ATR crystal, and this makes it difficult to examine the base urethane and additives in the coating.

This applications note investigates the effectiveness of three different ATR methods - multiple reflection ATR, single reflection ATR and micro-ATR for analyzing this type of sample.

EXPERIMENTAL

Two samples of coated hardwood flooring were examined. Both samples were rectangles, approximately 0.5”x0.5”x0.25”, with optically thick coatings. Sample A was stained mahogany and Sample B had natural-colored finish (see Figure 1).

Each sample was examined using three different ATR accessories (see Table 1). All three accessories featured a nominal incident angle of 45º and a crystal with a refractive index of 2.4. The samples were compressed against the crystal using the maximum pressure delivered by the pressure applicator without damaging the crystal. All spectra were collected with the accessory installed in a commercial FT-IR spectrometer. The spectrometer was set for 32 scans at 8 cm^{-1} resolution and used a DTGS detector. Its gain was adjusted to maximize the energy on the detector for each accessory.

RESULTS AND DISCUSSION

Figure 2 shows the multiple reflection ATR spectra of the two pieces of coated wood. The spectrum of sample A shows no bands, while that of sample B shows extremely weak bands. Since the coatings typically used on hardwood flooring are organic-based, infrared transitions are expected. However, the surface of the samples is somewhat rough and the sampling surface is fairly large, so the lack of clear

Table 1: The ATR Accessories Utilized

Type of Accessory	Name	ATR Material	Exposed Sampling Surface (approx. size)
Multiple Reflection ATR	Horizon™	ZnSe	50 x 10 mm
Single Reflection ATR	FastIR™	ZnSe	16 mm diameter
Micro-Single Reflection ATR	Meridian™	Diamond	500 μm diameter

infrared bands may be due to insufficient contact between the sample and the ATR crystal

Figures 3 and 4 show spectra collected using accessories with smaller sampling areas. All the spectra show distinct infrared bands, as expected from the coatings on the flooring. Comparison of these spectra reveals much stronger bands in that obtained using the Meridian, despite the higher signal-to-noise ratio of the FastIR. Hence the Meridian, with its smaller spot size, provides the best overall spectral contrast. Also note the Meridian spectra (Figure 4) contain bands that are better resolved that those in Figure 3. This may simply be an attributed to better contact, but it could also be a localized difference in the sample. Since the sampled area of the Meridian is so much smaller than that of the FastIR, the sampling by the Meridian may show inhomogeneities in the sample that were averaged over into the larger area sampled by the FastIR.

High quality infrared spectra can be collected from these rough surface samples using single reflection ATR. Furthermore, by comparing the two spectra shown in Figure 4, it is clear that the two stains are chemically different. Sample A has a much stronger band at 1070 cm^{-1}. It is likely that this band is due to SiO2 since SiO2 is commonly used as an additive to hardwood flooring finishes to improve their durability.

SUMMARY

Single reflection micro-ATR is by far the most effective method of investigating rough surface samples, such as coatings on hardwood flooring. Some information can be obtained from this type of samples with traditional single reflection ATR, but multiple reflection ATR is ineffective due to inadequate contact between the sample and ATR crystal.

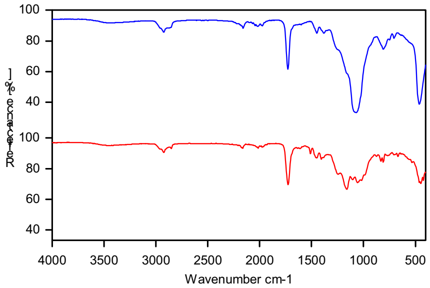


Figure 4. ATR Spectra of Sample A (blue) and B (red) Recorded by the Meridian SplitPea.

No. 40226

Analysis of Fibers by Micro-ATR Spectroscopy

Figure 1. Harrick's Video Meridian ATR

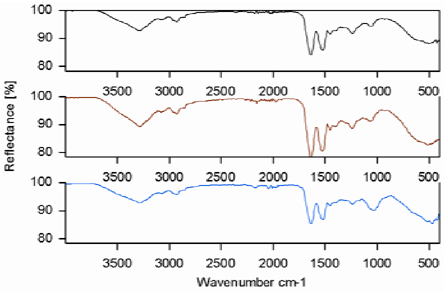
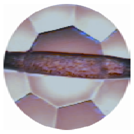


Figure 2. The ATR spectra of a single hair from a human (top), a guinea pig (middle), and a pony (bottom). A sample video output of the guinea pig hair is shown for reference

INTRODUCTION

Identification of organic fibers and other small samples is extremely important to forensics and to quality control for manufacturing facilities. An excellent method for examining these types of samples is FT-IR ATR spectroscopy. However, to obtain the optimum performance from ATR, the ATR accessory must have a small sampling area on the ATR crystal, the ability to accurately position the sample on that area, and a wide wavelength range.

A new accessory has been developed to meet these requirements. This accessory, the Video Meridian™, is an adaptation of Harrick's time-tested SplitPea™[1] with:



- True-color viewing of the sample through the crystal and image capture capabilities
- An unbreakable solid diamond ATR crystal.
- The smallest sampling surface available - 500 µm in 'diameter'.
- An unrestricted spectral range from the UV to the FIR.
- Readily available and interchangeable alternative ATR crystal materials, including Si, Ge and ZnSe.
- A calibrated pressure applicator with digital force display and capable of extremely high contact pressures.

Several examples are shown here.

EXPERIMENTAL

The ATR measurements were recorded using Harrick Scientific's Video Meridian™ on an infrared spectrometer equipped with a DTGS detector. To obtain good contact, the samples were pressed against the ATR crystal. The spectra were collected at 8 cm^{-1} resolution and 32 scans were signal averaged. With each spectrum, an image was also captured.

RESULTS AND DISCUSSION

The ATR spectra and video output for several fibers are shown in Figures 2 and 3. Figure 2 shows the spectra of hair from several different animals. The pony hair shows a stronger band near 1040 cm^{-1}, indicating that it is more oxidized than the others[2], probably due to sun exposure. Along with the spectra is a

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M. Milosevic and S. L. Berets, Appl. Spectros. 45 (6), 944 (1991).

[2] L. Brenner, P.L., M. Garry, and C. S Squires. Tumosa, J. Forsenic Sci. 30(2), 420 (1985).

HARRICK
HARRICK SCIENTIFIC PRODUCTS

No. 41109

application note

IDENTIFICATION OF UNKNOWN FIBERS USING DIAMOND ATR SPECTROSCOPY

Figure 1. The VideoMeridian™ viewing diamond ATR with optional video display.

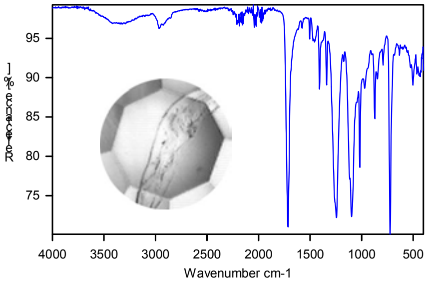

Figure 2. ATR spectrum of a fiber from a neutral-colored flat-weave rug.

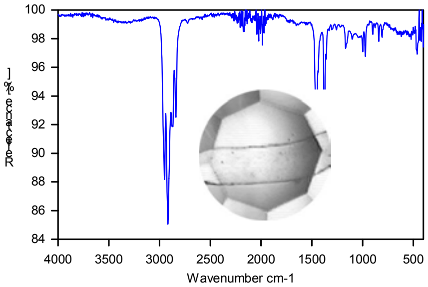

Figure 3. ATR spectrum of a fiber from a neutral-colored tufted rug.

INTRODUCTION

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EXPERIMENTAL

Unknown fibers were collected from common household materials. Individual fibers were extracted from rugs and clothes, and an imbedded fiber from a U.S. twenty-dollar bill. These were examined using the VideoMeridian™ with its type IIA diamond ATR crystal (Figure 1) in an FT-IR spectrometer. All spectra were collected with 32 scans at 4 cm^{-1} resolution and a DTGS detector.

Background single beam spectra were collected using the clean diamond ATR crystal. Then the samples were centered on the ATR crystal, using the video display, and the samples were compressed against the diamond with 15 units of pressure on the display. Next, the sample spectra and corresponding digital photographs were collected. Data collection typically took 2.5 minutes, for an experienced operator.

The spectra were then processed through an FT-IR ATR library for identification of the samples. The ATR crystal was cleaned with methyl ethyl ketone between samples.

RESULTS AND DISCUSSION

Figures 2 through 5 show spectra and digital image of the unsupported fibers, as measured with the VideoMeridian.

The spectra were all compared to on-file library spectra and the fibers were identified as follows. The fiber from the neutral-colored flat-weave rug, shown in Figure 2, is a polyester. The tufted rug fiber, whose spectrum appears in Figure 3, is a polyproplyene. The red fiber, shown in Figure 4, is a cotton-polyester blend, and the blue fiber in Figure 5 is made from nylon. Note that all the spectra have an increased noise level around 2100 cm^{-1}. This is due to incomplete compensation of the highly absorbing diamond lattice bands. Very few materials actually absorb in this small range and hence this does not significantly limit the use of this method for identification of materials.

From the digital photographs, it is clear that the diameters of the compressed fibers varied by roughly a factor of three. Since the sampled area on the VideoMeridain diamond is 500µm in diameter, it is possible to estimate the size of the compressed fibers. Figure 4 shows the thinnest fiber examined, and it was compressed to approximately 60µm in diameter.

In addition to free-standing fibers, imbedded fibers can also be examined and identified. Figure 6 shows the ATR spectra from two sections of the bill. One section has a noticeable blue fiber in it that, as deduced from the photograph (Figure 7), is approximately 65 µm in diameter. The other section was more uniform and examined as a reference. The bands at 1715 cm^{-1} and 1250 cm^{-1} appear in the spectrum of the fiber but not in the spectrum of paper. Subtraction results in a spectrum that can be clearly identified as polyester.

SUMMARY

As seen above, the VideoMeridian is a powerful tool for analyzing and identifying unknown fibers. Its video-imaging capabilities are useful both for positioning samples on the diamond ATR crystal and for maintaining photographic documentation.

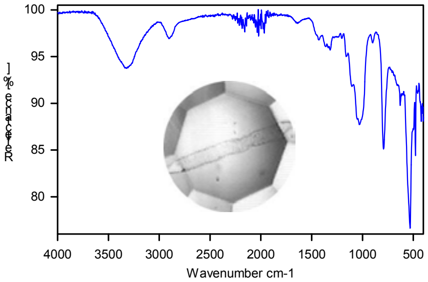

Figure 4. ATR spectrum of a red fiber.

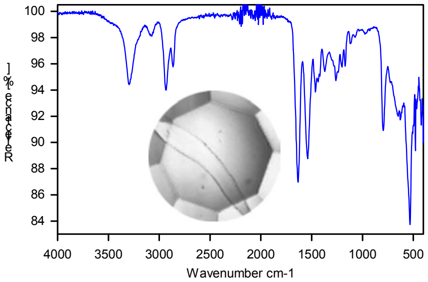

Figure 5. ATR spectrum of a blue fiber.

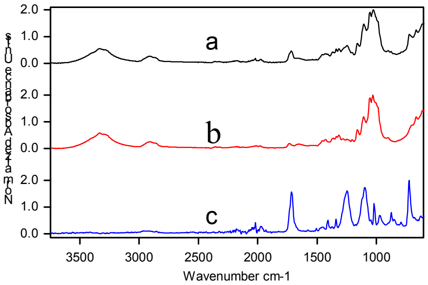

Figure 6. Normalized ATR Spectra of a blue fiber on the greenback (a), an area near that blue fiber (b) and the difference (c).

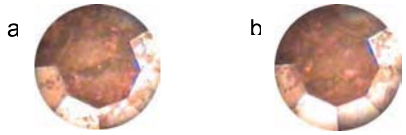

Figure 7. Photographs of the blue fiber on the greenback (a) and an area near that blue fiber (b). Spectra shown in Figure 6.

No. 40303

THE RELATIONSHIP BETWEEN POLARIZATION AND SENSITIVITY FOR GRAZING ANGLE ATR STUDIES OF THIN COATINGS ON SILICON

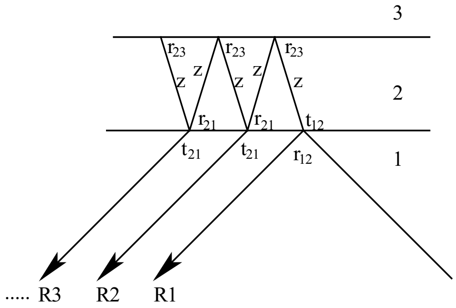

Figure 1. Multiple reflections in the sample layer.

Figure 2. The GATR Ge-ATR Accessory.

INTRODUCTION

There is much interest in examining thin films or monolayers deposited on silicon substrates, particularly in the electronics and semiconductor industries. Since these coatings are so tricky to detect by infrared spectroscopy, most of the work in this area has to do with identification or confirmation of the species deposited on the surface. The development of more sensitive detection methods, like 65º single-reflection Ge-ATR, has spurred investigations into the orientation of these monolayer coatings on Si by using polarized incident radiation. The interaction between polarized light and oriented surface species in ATR is commonly used, so applying this well-known method to 65º Ge-ATR is an obvious extension.

However, little is known in practice about the effect of polarization on the sensitivity enhancement. A better understanding of this phenomenon is vital to optimizing the experimental conditions and interpreting the results obtained. With this in mind, this application note explores the effect of polarization on the sensitivity enhancement for 65º single-reflection Ge-ATR. Both theoretical and experimental considerations are discussed.

THEORY

The theoretical foundation of ATR was developed by Harrick and duPre[1, 2]. They provided formulae for ATR analysis of very thin, weakly absorbing films deposited on a substrate, where the film thickness is much smaller than the wavelength. When these formulae are applied to a thin film on a silicon substrate, using a Ge ATR crystal and an angle of incidence above critical, the result exhibits extraordinary sensitivity to thin films[3, 4, 5].

The expression for the reflectivity of a thin film between two media[6,7] is:

$$\rho^{\mu} = \frac{r_{12}^{\mu} + r_{23}^{\mu} e^{4\pi i k d\sqrt{n_2^2 - n_1^2 \sin^2 \vartheta}}}{1 + r_{12}^{\mu} r_{23}^{\mu} e^{4\pi i k d\sqrt{n_2^2 - n_1^2 \sin^2 \vartheta}}}$$

where the index μ indicates polarization of incident light, r_{12}^{μ} and r_{23}^{μ} are Fresnel amplitude coefficients[6] for the interface between media (1 and 3) and the film (layer 2), *k* is the wavenumber ($k = 1/\lambda$) and ϑ is the incident angle. The refractive indices of the media are complex, permitting this to be applied to both absorbing and non-absorbing media. The above expression is the ratio of reflected and incident electric field amplitudes and it describes the magnitude of the reflected electric field in addition to the phase shift of the reflected field with respect to the incident field. The square of the absolute value of the result of this is the theoretical quantity comparable to the measured reflectance.

When a thin film is sandwiched between a germanium ATR crystal (n_1) and a silicon wafer (n_3), below the critical angle, the electromagnetic wave refracts into the film, propagates to film-silicon interface, partially reflects from that interface (some light is refracted into the silicon), propagates back to germanium ATR element, partially reflects from the germanium-film interface and so on (see Figure 1). Within the film, electric field vectors of all of the partially reflected waves add. The resulting electric field is an infinite sum of the contributions from all of the reflected waves.

The above equation can be expanded using a Taylor series, taking advantage of the identities: $r_{n,n+1}r_{n+1,n} + t_{n,n+1}t_{n+1,n} = 1$ and $r_{n,n+1} = -r_{n+1,n}$. Omitting the polarization index μ and introducing the shorthand $z = e^{2\pi ikd\sqrt{n_2^2 - n_1^2 \sin^2 \vartheta}}$ gives:

$$\rho = \frac{r_{12} + r_{23}z^2}{1 + r_{12}r_{23}z^2} = r_{12} + t_{12}t_{21}z(zr_{23}) + t_{12}t_{21}z(zr_{23})^2(zr_{21}) + t_{12}t_{21}z(zr_{23})^3(zr_{21})^2 + \ldots$$

The terms in the above sum can be related to the components of reflected wave in a direct one-to-one fashion. The first term is just the reflection coefficient of the interface between materials 1 and 2, corresponding to R1 in Figure 1. The second term corresponds to the ray R2 in Figure 1 and all the following terms correspond to the subsequent reflections. Thus there is a strict geometric representation of the Taylor series terms. Since this is just a re-expression of reflectivity, the geometrical representation is applicable for both the subcritical and supercritical incident angle regimes.

Above the critical angle for this system, the electromagnetic wave beyond the totally internally reflecting interface is evanescent. So the wave propagates parallel to the interface and cannot reflect from the film-silicon interface. However, each term in the infinite sum remains meaningful. Since the reflectance amplitude coefficients become of unit amplitude above critical angle, the higher terms in Taylor series expansion become more significant. For very thin films, the phase shifts between the components are negligible and the contributions to the electric field component perpendicular to the interface from all terms become constructive. Similarly, the subsequent contributions to the electric field component parallel to the interface become alternately destructive and constructive. Thus the electric field strength is greatly enhanced within the film for p-polarized radiation and only slightly enhanced for s-polarized radiation. Since absorption is proportional to the square of the electric field amplitude, an enhancement in the electric field strength of ten times results in a hundredfold enhancement in absorption.

EXPERIMENTAL

Two coated Si wafers were examined. One was a 16Å thick photoresist film on a silicon wafer and the other was an optically thick (2-3 μm) coating of the same photoresist. The samples were examined using the GATR single reflection Ge-ATR accessory with a 65º incident angle (Figure 2), in an FT-IR spectrometer. The spectra were collected with 32 scans at 8 cm^{-1} resolution uisng a DTGS detector.

The samples were placed on the ATR crystal and intimate contact obtained using the GATR slip-clutch driven pressure applicator, which delivers 56 in-oz of pressure, and referenced against the clean ATR crystal. Spectra were collected using unpolarized and polarized radiation. The polarization experiments utilized the optional GATR KRS-5 wire grid polarizer.

RESULTS AND DISCUSSION

Figure 3 shows the ATR spectra of an optically thick film of the photoresist measured with two different polarizations of incident radiation as well as with unpolarized incident radiation. As expected, the band intensities are strongest for the p-polarization. The intensities for s-polarization are the weakest and roughly half that of p-polarized light. The peak intensities in the spectrum recorded with unpolarized light falls somewhere between.

The ATR spectra of the 16Å film on Si, shown in Figure 4, has the same general trend, with the p-polarized incident radiation resulting in a spectrum with the most intense bands. Furthermore, the bands at 1500 cm^{-1} and 1250 cm^{-1}, which are clearly visible in the p-polarized spectrum, fall below the detection limit for s-polarization. This indicates a much stronger sensitivity to the p-polarization relative to the s-polarization for thin films on Si substrates than for bulk materials.

Since p-polarized light probes only the component of the bonds perpendicular to the interface, the difference between the spectra shown in Figures 3 and 4 shows the preferential orientation of certain bonds in a very thin film of photoresist.

SUMMARY

As seen above, the high sensitivity of 65° Ge-ATR to thin films on Si substrates is highly dependent on the incident polarization. The p-polarized component is enhanced, while the s-polarized component is not. Thus this method has higher sensitivity to the only the component of the bonds perpendicular to the interface. It would be interesting to see how the bonds orientation evolves with increase in film thickness by recording spectra of films with increasing thickness.

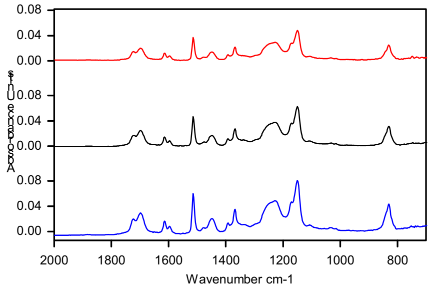

Figure 3. ATR spectra of a 2-3 µm thick photoresist on Si recorded with unpolarized (black), s-polarized (red) and p-polarized (blue) incident radiation.

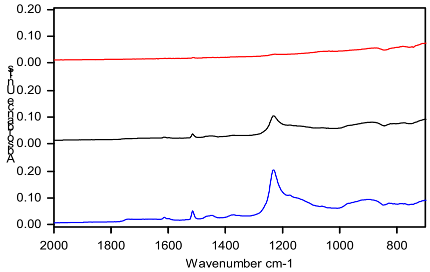

Figure 4. ATR spectra of a 16Å thick photoresist on Si recorded with unpolarized (black), s-polarized (red) and p-polarized (blue) incident radiation.

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No. 40227

ANALYSIS OF THIN FILMS ON SI WAFERS BY FT-IR ATR

Figure 1. The GATR Ge-ATR Accessory.

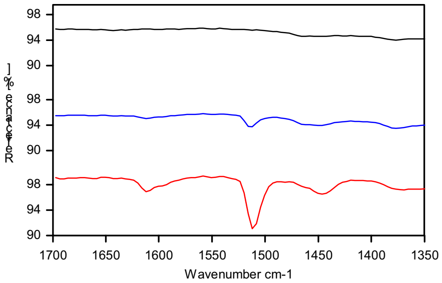

Figure 2. ATR Spectra of Uncoated Si (black), a 16Å Coating on Si (blue), and a 3μm coating on

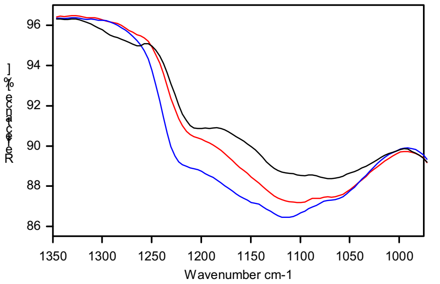

Figure 3. ATR Spectra of a 25Å Coating on Si with 40% (black), 50% (red) and 60% SiO$_2$ (blue).

INTRODUCTION

The semiconductor industry uses infrared spectroscopy to examine photoresist films, typically 20 microns thick or less, on semiconductor substrates. The most common methods of analysis are transmission spectroscopy and multiple reflection ATR. Transmission spectroscopy is limited by the thickness of the coating and the signal-to-noise ratio of the spectrometer. Hence transmission measurements generally require a large number of scans to extract good quality data from the coated wafers. Multiple reflection ATR is more sensitive but it is destructive, since the wafers are generally cut to fit into the apparatus, and is limited by the ability to obtain good contact between the sample and ATR crystal over the entire length of the crystal.

This application note examines extremely thin photoresist films on silicon substrates and changes in such coatings using the GATR, a single reflection Ge-ATR accessory. This method offers high sensitivity, due to thin-film effects, in addition to the potential to examine the wafers *in tact* with good ATR contact.

EXPERIMENTAL

Two sets of Si wafers were examined – an 18Å photoresist on an 8” diameter silicon wafer and several coated 8” diameter wafers with different concentrations of an additive, SiO$_2$, in the 25Å coating. Since contaminants readily adhere to these types of samples, each sample set included at least one standard. Both sets included a clean, uncoated silicon wafer that was exposed to the same environmental conditions as the coated wafers. An optically thick photoresist was also prepared as a standard.

The samples were examined using the GATR (Figure 1), a single reflection Ge-ATR accessory with a 65° incident angle, in an FT-IR spectrometer. The spectra were collected with 32 scans at 8 cm^{-1} resolution using a DTGS detector. The samples were placed on the ATR crystal and intimate contact obtained using 56 in-oz of pressure and referenced against the clean ATR crystal. The spectra of the uncoated reference wafers were measured prior to analysis of the coated samples to ensure that the ATR crystal was clean and that the samples were uncontaminated.

RESULTS AND DISCUSSION

In Figure 2, the ATR spectra of the photoresists and its reference are presented. Both coated wafers (middle and bottom spectra) have a peak at 1550 cm^{-1} that is due to the photoresist and does not appear in the spectrum of silicon (top). Note that the band intensity of the 16 Å film is one third that of the 3 μm film. This sensitivity to the thin film is due to interaction of the evanescent wave with the silicon substrate[1,2,3].

Figure 3 shows the ATR spectra of three 25Å coatings on silicon wafers. The coatings have different concentrations of SiO_2, which can be readily discerned from the band in the 1100 cm^{-1} to 1200 cm^{-1} region. The band intensities increase with greater SiO_2 concentration, as expected.

SUMMARY

Extremely thin photoresists and other coatings on silicon are readily detected by the GATR Ge single reflection ATR accessory. These measurements can be used to extract both quantitative and qualitative information about the coating.

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HARRICK SCIENTIFIC PRODUCTS

application note

No. 40303

TECHNIQUE CONSIDERATIONS FOR THE ANALYSIS OF MINERAL SPECIMENS BY SINGLE REFLECTION DIAMOND ATR

Figure 1. Meridian™ with modified pressure applicator and new powder cup.

Figure 2. Microscope with camera.

ABSTRACT

A previous work[7], using the Harrick Meridian™ accessory, has demonstrated the advantages and potential of single reflection diamond ATR FTIR spectroscopy for the analysis of geological specimens. In that paper it was shown that, in order to obtain reproducible spectra on such samples, it is necessary to reduce the solids to powdered material.

In this report, two analysis technique details will be explored. The first is the size of the particles required for ATR analysis. Both the absorbance of peaks and the reproducibility of measurements increase when the particle size is reduced. The exact nature of these relationships will be investigated. An attempt will be made to determine the optimum practical particle size and a method will be described to reduce samples to that size. The relationship between the ATR sampling size and reproducibility will also be discussed. The second detail is the amount of pressure that must be exerted on the sample to obtain reproducible spectra. The hard samples are essentially incompressible. Hence, the amount of pressure required is small, presumably only that which is required to compress a loosely filled sample holder. In this current work, the exact minimum amount of pressure required will be determined.

For this study, two natural mineral samples, with a wide hardness difference, are chosen: gypsum ($CaSO_4 \cdot 2H_2O$) with a Knoop hardness of 32 psi and milky quartz (SiO_2) with a Knoop hardness of 820 psi. Both samples have several peaks in the 1700 to 400cm^{-1} range, the heights of which will be monitored as the particle size or the pressure is changed.

OBJECTIVE

The purpose of this work is to quantify the effects of particle size and pressure on the sample in the diamond ATR analysis of powdered mineral samples. Two naturally derived samples, of widely different hardness, are used. The effects on sensitivity and reproducibility will be monitored.

EXPERIMENTAL

All spectra were taken with a Nicolet Nexus™ 670 FTIR spectrometer equipped with a DTGS detector and a standard mid-IR beamsplitter and using Nicolet EZ Omnic™ 5.1 software. All spectra were run at 4000cm^{-1} to 400cm^{-1}, 100 scans, and 4 cm^{-1} resolution. The aperture was set to 100 and the gain was set to 8. A background spectrum (air) was taken prior to each sample spectrum in all cases except for the pressure tests, described below. Here, only one initial background spectrum was taken for each sample run. The purge input of the spectrometer was connected to dry nitrogen at 25 SCFH.

The accessory used was a Harrick Meridian™ (diamond SplitPea™). The accessory included a 40º angular mask installed in the diamond ATR cartridge to remove subcritical light rays. The special calibrated pressure applicator and the special powder holder, previously described[7], were installed in the accessory. The pressure applicator had a 4.75mm diameter cylindrical tip (17.72mm^2 area) and had installed a spring (Lee Spring Company P/N LC-050K-5S), which required less compression force than that used in the standard applicator. The force and pressure calibration data for this new applicator are given in Table I. Note that the actual measured force at the "0" marking is not 0 N, since the spring is still slightly compressed at its most relaxed position. The 0.895 x 10^{6}Nm^{-2} pressure setting was used for all samples, except where noted. The volume of the powder holder was 119mm^3. A picture of the Meridian, pressure applicator, and powder holder is given in Figure 1. The purge input of the accessory was connected to the purge fitting inside the sample compartment of the spectrometer. The purge ears of the accessory (PermaPurge™) were extended to the beam ports of the sample compartment. In this manner, samples could be run, one after the other, without breaking purge even with the sample compartment cover of the spectrometer open.

Two natural mineral samples, with widely different hardnesses, were selected for use in this study. The first was selenite, a type of gypsum ($CaSO_4 \cdot 2H_2O$), with a Knoop hardness of 32 psi. With a Mohs hardness of only 2, gypsum is one of the soft minerals. The second was milky quartz, a coarsely crystallized quartz (SiO_2), with a Knoop hardness of 820 psi. With a Mohs hardness of 7, quartz is one of the harder, although not among the hardest, minerals. In addition to their wide hardness difference, these two minerals shared another advantage. For both, corresponding reference materials could be readily obtained from a chemical supplier, to verify their identification. The monolithic (natural type 2A) diamond ATR of the Meridian™, with a Knoop hardness of 7000 psi (Mohs 10), is considerably harder than either of the two selected samples.

Table I. Calibration for pressure applicator with reduced force spring.

Applicator Marking (kg)	Measured Force (N)	Pressure* (10^6 Nm^{-2})
2.5	15.86	0.895
2.0	13.24	0.747
1.5	9.81	0.554
1.0	7.85	0.443
0.5	5.40	0.305
0.0	2.78	0.157

*Tip of pressure applicator has an area of 17.72 mm^2.

The following sample preparation technique was used to quickly reduce over 70g of each solid sample to a powder, with the consistency of fine sand: (a) a hammer was used to produce pieces approximately 12mm on a side or less; (b) these smaller pieces were placed between two stainless steel plates and crushed with a hammer; and (c) the resultant particles were ground with a mortar and pestle until no larger particles were evident. Approximately 0.09g of gypsum and approximately 0.15g of quartz are required to fill the sample holder.

The particle size resulting from the above procedure has been previously estimated to be 420 to 590 microns (-40 to –30 mesh). This size has been shown to be too large for adequate sensitivity and reproducibility with the Meridian diamond equipment. To further refine the prepared samples, type 340 stainless steel 6" x 6" mesh screens were used. Two identical screen sets (McMaster-Carr P/N 92405T17), one for each sample type, were obtained, in order to avoid inter-sample contamination. See Table II. Using these screen sets, one to three grams at each of the listed mesh sizes were obtained for each of the two samples. During sample preparation, it was found that the 250 and 400 mesh sizes were the practical limits for the gypsum and quartz samples, respectively. To obtain sufficient sample at these sizes, a large amount of additional grinding with the mortal and pestle was required. Also with the gypsum sample, the 250 mesh screen became clogged frequently and had to be cleared with compressed air. With finer mesh sizes, it was essentially impossible to accumulate sufficient sample of either type, regardless of the amount of additional grinding.

In order to verify the screen sizes as well as the particle sizes derived from them, the equipment shown in Figure 2 was employed. This consists of a microscope with illuminator and graduated stage (Edmund Scientific #C31147-00). The 10X objective of the microscope was used with its 10X eyepiece, yielding a magnification of 100X. The system was adapted for photomicroscopy by using a microscope tube adapter (Edmund Scientific #C30411-00) and a Nikon camera "T" adapter (Edmund Scientific #C30428-35) to mount the body of a Nikon FG single reflex 35mm camera. The film used for this study was Kodak Elite Chrome 400 color slide film. Dimensional calibration of the system was accomplished by using a stage micrometer (Fisher Scientific 12-561-SM3) with 10 micron divisions.

Table II. Screen opening sizes.

Screen mesh size	Given Screen Opening (µm)	Measured Screen Opening (µm)
80	178	182
100	140	150
150	103	116
250	61	57
400	36	34

Developed slide film allows the use of a projector to view images taken of the stage micrometer and the sample under identical conditions. The greatly enlarged, projected images are readily measured. Microscope pictures of the stage micrometer, one of the screens, and one screened sample are given in Figures 3, 4, and 5, respectively. It can be seen from Figure 5 that screened samples still have a wide particle size distribution, although none of the particles will be larger (at

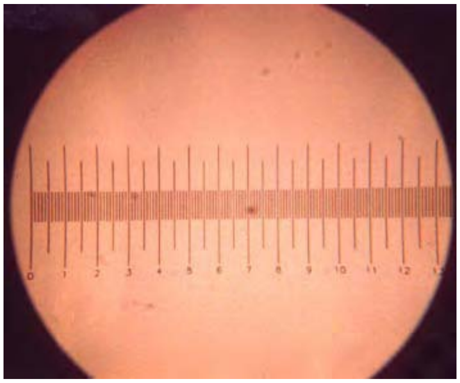

Figure 3. Stage micrometer (photomicrograph at 100X magnification).

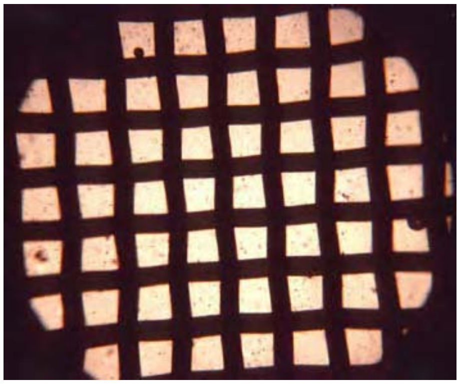

Figure 4. Typical Screen, 150 mesh (photomicrograph at 100X magnification).

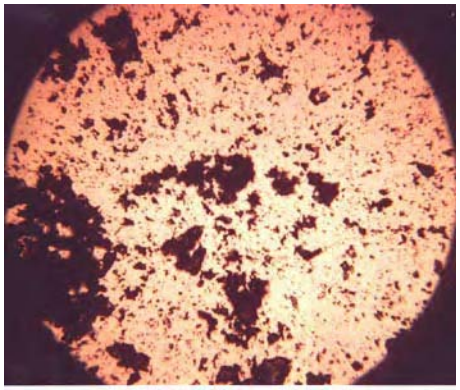

Figure 5. Quartz sample after filtering through 150 mesh screen (photomicro-graph at 100X magnification).

least in two dimensions) than the opening of the screen.

The data in Table II for actual screen sizes were obtained using the above equipment and procedure. The microscope technique proved impractical for measuring particle size directly, due the wide size distribution obtained from the use of only one screen and the tendency of clumping. Distinguishing a clump of small particles from a single large particle is problematic. (Certainly, other, more sophisticated methods[17] for determining particle size and distribution exist, but were not available for this study.)

The gypsum and milky quartz samples were obtained from Minerals Unlimited in Ridgecrest, California. These identifications as gypsum and quartz were confirmed by comparing their spectra, after sample preparation to the -150 and -400 mesh sizes, respectively, with those obtained from the corresponding reference materials. For the gypsum sample, the reference material was calcium sulfate (Alfa Aesar P/N 33301-A1). For the milky quartz sample, the reference material was silicon (IV) oxide (Alfa Aesar P/N 13024-A3). This latter material has a stated mesh size of –400, and an average stated particle size of 2 microns. No sample preparation was performed on either reference material. See Figures 6 and 7. The artifacts observed in the quartz sample spectrum around the Si-O-Si stretch peak (1085-1090cm^{-1}) are attributed to impurities in the natural material.

For this study, three peaks for both gypsum and quartz were monitored for changes as a result of particle size and applied pressure. For gypsum, the peaks at 1113-1117cm^{-1}, 668-669cm^{-1}, and 599-600cm^{-1} were used. For quartz, peaks at 777-779cm^{-1}, 694cm^{-1}, and 452-459cm^{-1} were used.

Full source information for the geological samples, reference materials, and special equipment used in this study is given in Table III.

DISCUSSION

A previous study[7] showed the need to grind solid mineral samples to a fine powder to assure reliable contact with the ATR sampling surface, to obtain good sensitivity and repeatability, and to average out crystal orientation effects, if present. That study also implied that very little pressure applied to the sample was required, since mineral samples were essentially incompressible. In this current study, data have been collected in an attempt to quantitate some of these effects.

The methodology introduced in the previous study and its

Table III. Sources

Alfa Aesar 30 Bond Street Ward Hill, MA 01835 800-343-0660 www.alfa.com (reference materials)	Lee Spring Company 1462 62^{nd} Street Brooklyn, NY 11219 800-426-0272 www.leespring.com (springs)
Edmund Scientific 60 Pearce Ave. Tonawanda, NY 14150 800-728-6999 www.scientificsonline.com (microscope equipment)	McMaster-Carr Supply Co. P.O. Box 440 New Brunswick, NJ 08903 732-329-3200 www.mcmaster.com (screen sets)
Fisher Scientific 2000 Park Lane Drive Pittsburgh, PA 15275 800-766-7000 www.fishersci.com (stage micrometer)	Minerals Unlimited P.O. Box 877 Ridgecrest, CA 93556 760-375-5279 mimi@ridgecrest.ca.us (mineral samples)

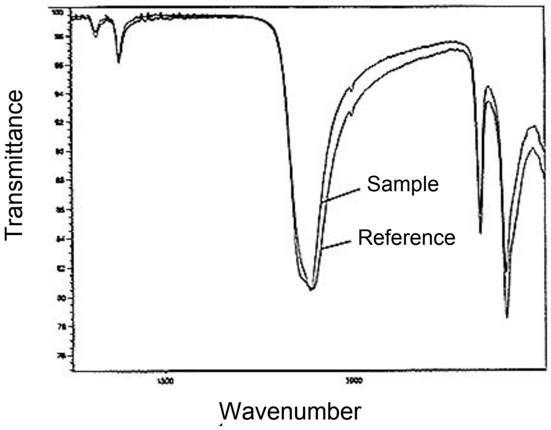

Figure 6. Spectra of gypsum sample (150 mesh) and reference material.

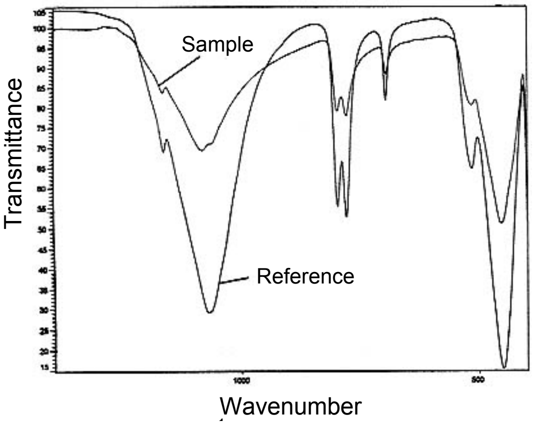

Figure 7. Specta of quartz sample (400 mesh) and reference material.

elaboration here represent an advance in reduced analysis time in the measurement of powdered inorganic substances in the mid-infrared when compared to the technique employed by Miller and Wilkins[9]. Once powdered samples are obtained, they are simply placed on the ATR element, the pressure applicator is applied, and the spectrum is taken. Since the ATR element is diamond, it is not subject to scratching by the relatively hard mineral samples and it is easy to clean for the next sample.

The results shown in Figures 8 and 9 show the expected increase in sensitivity with decreased particle size. Similarly, the data presented in Figures 10 and 11 for reproducibility indicate the expected decrease in relative standard deviation of the measurements with decreased particle size. Some individual deviations from these trends are observed. In particular, a major deviation is noted for the 116 micron gypsum in Figure 10. These deviations are attributed to inadequate sampling (i.e., five samples were not enough to be representative) and to variations in sample size distributions, discussed below.

Unfiltered samples were run for comparison. For such samples, the sensitivities were 0.2 to 0.3 times and the relative standard deviations were 5 to 10 times those obtained for the corresponding -250 mesh samples. Clearly, unfiltered samples should be avoided.

Figures 8 through 11 further indicate that, if smaller particle sizes were obtainable, then these would lead to even further enhancements in sensitivity and reproducibility. A set of data was collected on the quartz reference material. Here the mean particle size is specified as only 2 microns. This material yielded absorbance values that were 2 to 3 times and a relative standard deviation of approximately one-half that of the -400 mesh (34 micron) sample.

Figures 12 and 13 show the relationship between absorbance and applied pressure for gypsum and quartz, respectively. With both samples, the greatest increase in absorbance is obtained with a pressure less than 0.3 x 10^6 Nm^{-2}, the smallest contact setting available with the equipment in use. Figure 13, for quartz, shows the expected curve set for absorbance vs. applied pressure, where, after the initial pressure, a plateau area is reached. For the gypsum, however, there are small, but significant slopes to the curves as greater pressure is applied, as seen in Figure 12. Obviously, the initial assumption that mineral samples are completely incompressible is false. Three things are clear from this data. First, to obtain repeatable results, a calibrated pressure applicator is required. The same pressure should be used to

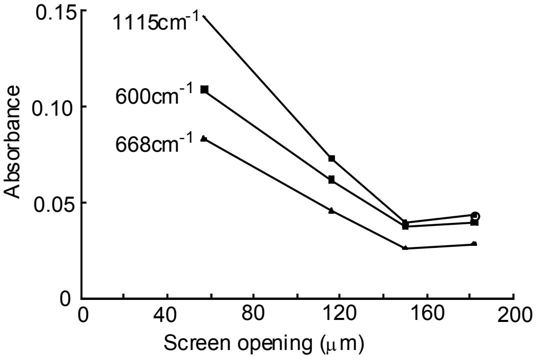

Figure 8. Absorbance vs screen opening for gypsum.

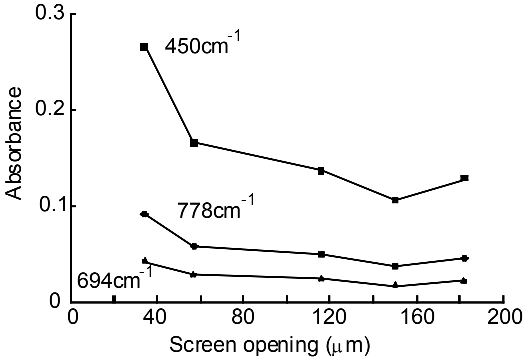

Figure 9. Absorbance vs screen opening for quartz.

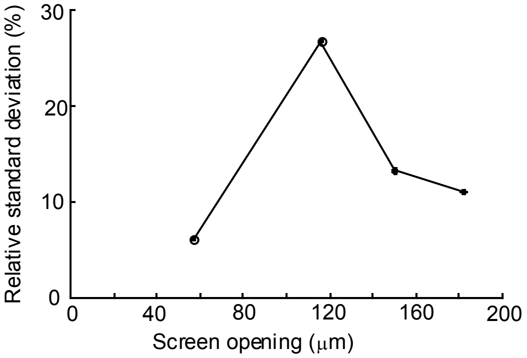

Figure 10. Standard deviation vs screen opening for gypsum.

obtain all data in a given procedure. Second, the applied pressure should be greater than 0.3 x 10^6 Nm^{-2}. Third, the highest pressure calibration setting may be used for all samples. The sensitivities with softer samples will be increased. Although the results with harder samples may not be significantly enhanced, neither will they be harmed.

The screen mesh technique employed only restricts the upper limit of particle size. There can be a wide distribution of particle sizes that go through, say, a 250 mesh screen, and it is this wide distribution that can decrease reproducibility. One method to improve reproducibility would be to use a diamond ATR device with a larger sampling area than that of the Meridian^{TM}, which is approximately 500 microns. The newly released Harrick MVP Star^{TM}, with a larger 1500 micron sampling area, is a candidate. (An additional approach to increasing reproducibility would be to mechanically control particle size distribution by using two screens with different mesh sizes, and capturing the material between the two sizes. The time to obtain sufficient material by this approach could be problematic.)

With the methodology employed here, practical considerations allowed particle sizes no lower than –250 to –400 mesh. Furthermore, the smallest particle size obtainable was sample dependent. The smaller the particle size, the more efficiently packed is the effective pathlength volume above the ATR element sampling surface. More efficient packing of this space will yield higher absorbance values. A small laboratory grinder which would produce sub –400 mesh samples, in small (1-3 gram) amounts after a short time period would be ideal for further work.

RESULTS

To determine the effect of particle size on sensitivity, the absorbances at the previously specified wavelengths were monitored for each of the filtered samples. Five runs on each mesh sample were made. These results are plotted in Figure 8 for gypsum and in Figure 9 for quartz.

To determine the effect of particle size on reproducibility, the same data sets used for the sensitivity results, above, were employed. Here, the average relative standard deviations for all three wavelengths were plotted vs. screen opening size. Figures 10 and 11 present the data for gypsum and quartz, respectively.

For the pressure tests using gypsum and quartz, five runs

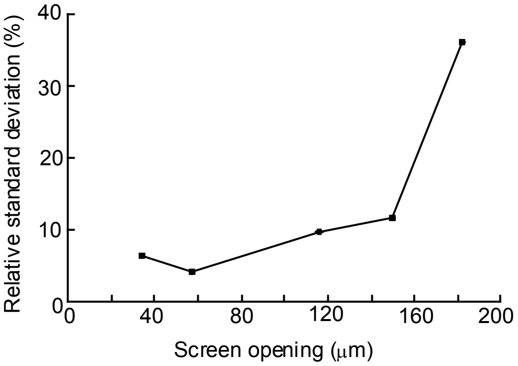

Figure 11. Standard deviation vs screen opening for quartz.

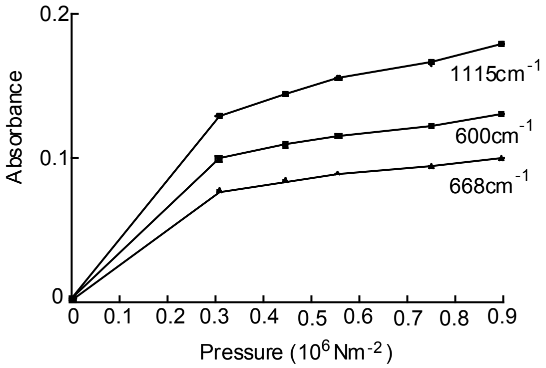

Figure 12. Absorbance vs pressure of –250 mesh gypsum.

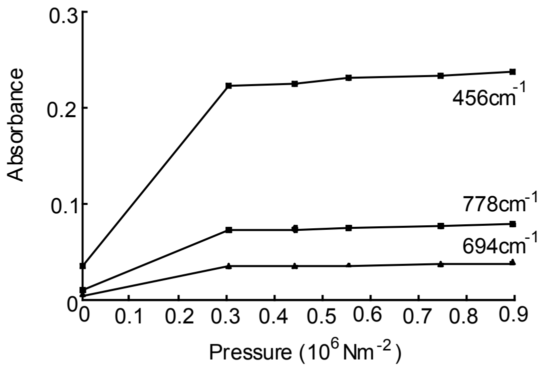

Figure 13. Absorbance vs pressure for –400 mesh quartz.

were made with each sample type. For gypsum, the -250 mesh sample was used. For quartz, the -400 mesh sample was used. Within each run, six pressure settings were employed. These six pressure settings are identical to those listed in Table I, with the following exception: the zero pressure data were collected with no contact of the sample applicator on the sample. At each pressure setting, the absorbances at three wavelengths were monitored. The results of these tests for gypsum are given in Figure 12 and those for quartz are given in Figure 13.

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applications note
No. 60501

NEAR CRITICAL ANGLE FTIR ATR SPECTROSCOPY WITH A VARIABLE ANGLE REFLECTION ACCESSORY

JOSEPH P. LUCANIA AND S. L. BERETS
HARRICK SCIENTIFIC PRODUCTS, INC., BOX 277, PLEASANTBILLE NY 10570

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OBJECTIVE

Variable angle ATR FTIR spectroscopy is used to study the effect on sensitivity of changing the angle of incidence. The angle of incidence can be decreased (moved closer to the critical angle) in order to increase sensitivity. As the critical angle is approached, however, spectral distortion results. Such distortion can be offset by the use of an aperture. Conversely, the angle of incidence can be increased to reduce sensitivity in cases where absorbance values are too high.

ABSTRACT

Although ATR spectroscopy near the critical angle seems to offer the advantage of heightened sensitivity, it is a technique that is generally eschewed by scientists and accessory manufacturers. Most organic compounds have a bulk refractive index of approximately 1.5. For the most common ATR element material, Zinc Selenide, with a refractive index of 2.42, the critical angle is 38 degrees. At smaller angles, total internal reflection no longer occurs. Furthermore, as one approaches the critical angle, spectral distortion results. This is due to fluctuations in the refractive index around absorbance maxima in the analyzed compound and to the beam spread in modern FTIR instrumentation. Most fixed angle ATR accessory designs protect against such phenomena by using an angle of incidence of 45 degrees, safely higher than the critical angle for most samples. In some cases, a larger angle or a higher refractive index ATR material is recommended, especially for use with intensely absorbing samples, to further reduce any distortion. Despite such conderations, there remains the possibility of hypersensitive analysis in the 45 to 38 degree regime. For it is here that the effective pathlength of ATR spectroscopy increases dramatically. To investigate this potential, the Harrick Scientific Products variable angle reflection accessory, the Seagull^{TM}, equipped with a ZnSe ATR element and a liquid cell, is employed. Examples of advantages and pitfalls will be given and the use of apertures will be discussed.

INTRODUCTION

Critical Angle

In an ATR configuration, light traveling within the ATR element reaches the interface between the element and sample. (The refractive index of the ATR element is generally much higher than the sample.) The angle of incidence for which the refracted ray emerges tangent to the surface of the element is called the critical angle, A_c.[1] Derived from Snell's Law, Ac is given by

$$A_c = \sin^{-1} (n_2/n_1) \qquad (1)$$

where n_1 is the refractive index of the ATR element and n_2 is the refractive index of the sample. At angles greater than A_c, light interacts with the sample and is reflected back into the ATR element at the same angle as the incident angle. At angles less than A_c, light exits the ATR element and cannot be collected by the detector of the spectrometer.

Figure 1 plots the critical angle for two common ATR element materials, Zinc Selenide (ZnSe), the material used in this study, and Germanium (Ge) versus the refractive index of the sample. The graph begins at the refractive index of air, 1.00. Most organic and inorganic (e.g., aqueous) substances analyzed with ATR spectroscopy have a refractive index between 1.3 and 1.7. In the calculations for Figure 1, the refractive indices of the two materials at 2000cm^{-1} (5 microns), 2.4295 for ZnSe[2] and 4.0163 for Ge,[3] are used.

Toluene, with a refractive index of 1.4961,[4] is used as the sample in this study. The critical angle, using ZnSe as the ATR material, is 38.010°. The critical angle, using Germanium as the ATR material is 21.870°. Air, used as the sample for the background spectra, has a refractive index of 1.0000. In these cases, the critical angles are 24.306° using ZnSe and 14.417° using Ge, much less than the critical angles with the higher refractive index toluene sample.

Depth of Penetration

The depth of penetration, d_p, defined as the distance required for the electric field amplitude to fall 1/e of its value at the surface,[5] is given by

$$d_p = \frac{W/n_1}{2\pi\left[\sin^2 A - \left(n_2/n_1\right)^2\right]^{1/2}} \qquad (2)$$

where W is the wavelength of light and A is the angle of incidence. The value d_p is an indication of the sensitivity. Using 5 microns as W, and the previously specified refractive indices for ZnSe and toluene, the depth of penetration is plotted versus the angle of incidence in Figure 2. As the critical angle is approached, d_p gets very high. Comparable data for Ge is also shown in the same figure. For any given angle of incidence, the depth of penetration for Ge is much less than that of ZnSe, due to the higher refractive index of Germanium.

EXPERIMENTAL

All spectra were taken with a Thermo/Nicolet Nexus™ 670 spectrometer equipped with a DTGS detector and a standard mid-IR beamsplitter and using Thermo/Nicolet Omnic™ Version 6.1a software. All spectra were run at 4000cm^{-1} to 400cm^{-1}, 64 scans, 4 cm^{-1} resolution, and gain equal to 1. The spectrometer aperture was set to 100 for all runs except where noted. A background spectrum (air) was taken prior to each sample spectrum. The purge inputs of the spectrometer and the installed accessory were connected to filtered air (water and CO_2 removed) produced by a Parker Balston unit at 30 SCFH.

A Harrick Seagull™ variable angle reflection accessory was installed in the FTIR spectrometer. See Figure 3. The Seagull accessory allows the angle of incidence to be changed continuously from 5° to 85°. The ATR kit was installed in the accessory along with a Luer-compatible liquid cell. See Figure 4. All runs were taken using a hemispherical ZnSe ATR element, except where noted. A Hamilton Model 1002 2.5ml Luer-tipped syringe was used to inject samples into the liquid cell.

All angles were set in the Seagull™ by first moving the angle several degrees lower than the desired angle and then carefully moving the adjustment continuously higher until the desired angle was reached. This was to assure the consistent removal of any backlash.

Spectrophotometric grade Toluene (Alpha Aesar Stock No. 19376) was used as the samples in all tests, except where noted. A liquid was chosen to avoid any effects due to inadequate contact that might be introduced with solids.

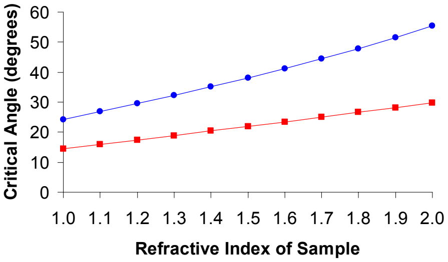

Figure 1. Critical angle vs. refractive index of sample for Zinc Selenide (blue) and Germanium (red).

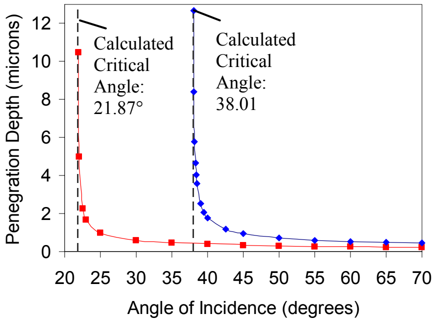

Figure 2. Penetration depth vs. angle of incidence for Zinc Selenide (blue) and Germanium (red).

Figure 3. Seagull™ variable angle reflection accessory with ATR kit and liquid cell installed.

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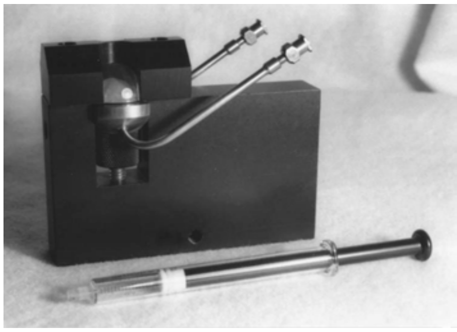

Figure 4. Seagull^{TM} Zinc Selenide ATR kit with liquid cell and Luer-tipped syringe.

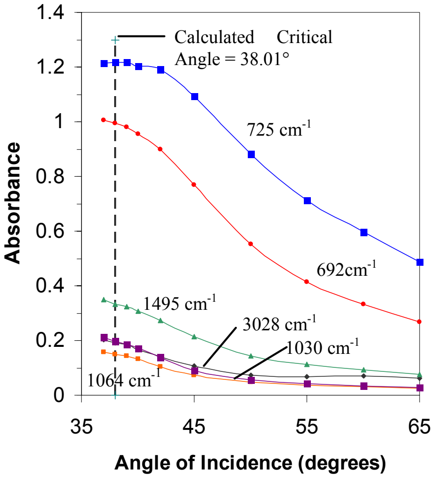

Figure 5. Absorbance vs. angle of incidence for toluene at six absorbance maximum wavelengths without accessory aperture.

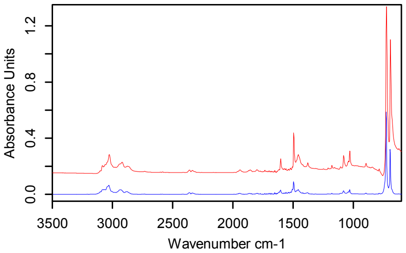

Figure 6. Distorted spectrum of toluene at 39 degrees (top) compared to undistorted spectrum of toluene at 60 degrees (bottom).

Toluene was the liquid chosen due to particular advantages. It has several peaks of varying intensities over the mid-infrared. It has good solubility in the chosen cleaning solvent, MEK. It has sufficient volatility to assure complete removal prior to taking background spectra. It is not hygroscopic.

The Harrick CristalCalc^{TM} software was used to calculate the depths of penetration used in Figure 2. Transmission spectra were converted to absorbance spectra using the Omnic "Absorb" function. Baseline corrections on the absorbance spectra, where indicated, were performed using the Omnic "Aut Bsln" function. Absorbance peak maxima and associated wavelengths were found using the Omnic "Analyze Find Peaks" function.

RESULTS

To study the effect of increasing the angle of incidence, three sets of data were acquired. Each set consisted of toluene spectra at the following angles of incidence: 65, 60, 55, 50, 45, 42, 40, 39, 38, and 37 degrees. The averaged absorbance values at six toluene peak maxima (3028cm^{-1}, 1604cm^{-1}, 1495cm^{-1}, 1030cm^{-1}, 725cm^{-1}, and 692cm^{-1}) versus the angle of incidence are given in Figure 5. Baseline correction was applied to spectra obtained for angles of 45 and less. A very small amount of distortion was observed at 45 degrees; significantly more at smaller angles.

The distortion observed was of two types: first, an overall baseline shift toward higher absorbance values; second, a derivative-like distortion in the area of the two strong peaks at 725cm^{-1} and 692cm^{-1}, termed dispersive distortion. These two types can be clearly seen in the spectrum taken at 39 degrees (near the calculated critical angle), shown in Figure 6. A spectrum taken at 60 degrees, shown in the same figure for comparison, has no diortion. The dispersive distortion causes confusion in any baseline correction.

To study the effect of reducing the aperture size on near critical angle distortions, the angle of incidence of the Seagull^{TM} accessory was set to 39 degrees and the aperture of the spectrometer was set from 100 to 40, in steps of 20. No reduction in spectral distortions was observed. A 3mm thick fixed accessory aperture, with a circular opening of 9.5mm diameter, was installed in the Seagull^{TM} accessory in the slide mount used for the optional polarizer. The mount is located on the left side of the accessory. Figure 7 shows two uncorrected absorbance spectra, one without the accessory aperture and one with the aperture. A significant reduction in baseline shift distortion and a small reduction in dispersive distortion are observed for aperture results.

The runs described for the generation of Figure 5 were repeated, but with the accessory aperture installed. The reslts of these tests are given in Figure 8. For these runs, baseline correction was not required until the angle was reduced to 42 degrees or less. These aperture results show increased absorbance values at the lower angles of incidence

CONCLUSION

The results presented in Figure 5 deviate significantly from expectations based on the theoretical data presented in Figure 2, and may not be accounted for by variations, as a function of wavelength, in the refractive indices of the ATR element, the refractive index of the bulk sample, or the penetration depth.

In Figure 2, the theoretical sensitivity progressively increases as the angle of incidence decreases until, at the critical angle, the sensitivity is infinite. In Figure 5, the actual sensitivity does start to increase with decreasing angle and then levels off near the calculated critical angle. This pattern in actual sensitivity occurs for high and low absorbance value peaks and at all of the various monitored wavelengths. Distortions start to appear prior to reaching the calculated critical angle. Spectra, although distorted, can still be taken at angles less than the calculated critical angle.

The principal reason for the above observations is optical beam spread in the FTIR spectrometer. This spread is increased by the lensing effect of the ZnSe ATR material. Since there is a variation in angle around the central beam, as the critical angle is approached, a certain percentage of the rays will be less than the critical angle. These rays will exit the ATR element and never reach the detector. An upward shift in the entire absorbance spectrum results. With distortion of this type, the sample causes energy to eit the system. As with an aperture, the overall energy loss results in a decrease in the signal to noise ratio over the entire spectrum. At and below the calculated critical angle, there are still rays at higher angles, and it is these which continue to produce spectra, albeit distorted.

Rays that are near the critical angle have a very large penetration depth. These interact with the fluctuating refractive index of strong sample peaks, producing the dispersive distortion. It is this second type of distortion that makes automatic baseline correction especially problematic.

One method of reducing distortions at any given angle of incidence is to substitute Germanium for Zinc Selenide as the ATR element. The much higher refractive index of Ge results in a lower critical angle for any given sample refractive index. See Figure 1. Germanium, however, does have some disadvantages. First, the sensitivity is decreased for any given angle of incidence. See Figure 2. Second, since reflection losses are greater for the higher refractive index material (in the absence of any

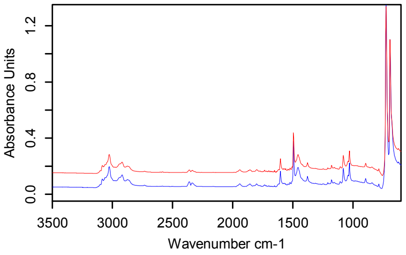

Figure 7. Spectrum of toluene at 39 degrees without (top) and with (bottom) accessory aperture.

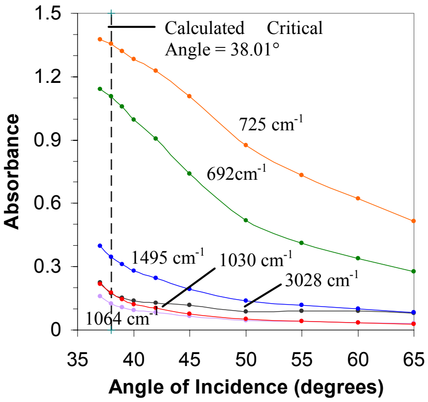

Figure 8. Absorbance vs. angle of incidence for toluene at six absorbance maximum wavelengths with accessory aperture.

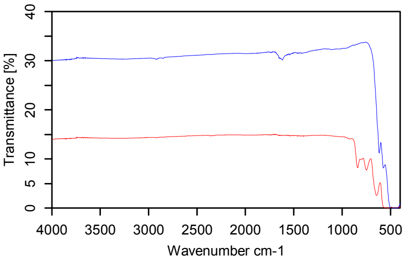

Figure 9. Seagull™ throughput with Zinc Selenide (top) and with Germanium (bottom).

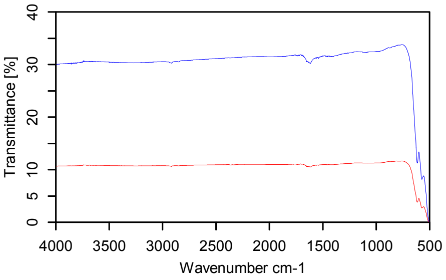

Figure 10. Seagull™ throughput without accessory aperture (top) and with accessory aperture (bottom).

antireflection coatings), an ATR accessory with Germanium has a lower throughput than the same accessory with Zinc Selenide. See Figure 9 for the throughputs of the Seagull™ at 45° for these two ATR materials. Third, the greater lensing effect of Ge, due to its higher refractive index, will exacerbate the instrument beam spread even further, when compared with ZnSe. Finally, the same difficulties with spectral distortion encountered with ZnSe near 38° will be encountered with Ge near 22°.

The use of apertures to limit beam spread allows increases in sensitivity to continue as the critical angle is approached. One cannot rely on the aperture built into the FTIR spectrometer, as this component may not be positioned in the optical train to allow restriction of the beam spread. Rather, one must have an independent aperture fixed within the sample compartment or the accessory. The effectiveness of the accessory aperture is shown in Figure 7 which gives uncorrected absorbance spectra of toluene with and without this aperture near the critical angle. The circular aperture cuts out many of the rays that are less than the critical angle, greatly decreasing the baseline distortion described above. Some effect is also observed in the reduction of the dispersive distortion near the two peaks at 725cm^{-1} and 692cm^{-1}. Corrected absorbance baselines of aperture results, therefore, do come closer to zero. Comparison of the baseline corrected absorbances of the two spectra also indicates a sensitivity enhancement for the aperture results. Figure 8 then shows the results of the same runs done for Figure 5, but this time with the accessory aperture in place. Sensitivity enhancement is indicated at angles near the calculated critical angle, especially for the four smaller peaks at 3028cm^{-1}, 1604cm^{-1}, 1495cm^{-1}, and 1030cm^{-1}. Furthermore, the slopes of the four associated curves promise fairly higher sensitivity at lower angles than those tested.

As with the use of Ge, the use of apertures reduces throughput. See Figure 10 for the throughputs at 45° with and without the accessory aperture installed in the Seagull, using ZnSe as the ATR material. A more efficient aperture could be constructed with a half-moon opening, rather than a full circular opening. This new aperture would block out only the lower angled rays. One can speculate that, with the advent of tunable mid-IR lasers[6] in commercially available spectrometers, true "HyperATR" spectroscopy near the critical angle will be possible. With no beam spread, the critical angle could safely be approached without resort to apertures and their deleterious throughput losses. Mechanical stability would have to be a key feature of such systems, as small changes in the angle of incidence would introduce large absorbance changes.

A pure liquid was chosen as the most convenient sample.

The same conclusions would hold for solids and solutions. In fact, detection of trace solutes may become a major application. The use of solutions, however, can introduce nonlinearities due to the association of dissolved species at higher concentrations.[7]

The major emphasis has been to explore practical means to increase sensitivity for measurements of uniform bulk samples. One practical advantage would be to boost sensitivity in detection of the CH stretch, an important organic marker, occurring around 3000cm^{-1}.[8] As the sensitivity in ATR spectroscopy is proportional to wavelength, the CH stretch peak is typically much weaker than the corresponding peak acquired with transmission spectroscopy. It should be noted however, that there are instances when the sensitivity may be too high. For quantitative analysis, peak maxima should be in the 0.2A to 0.7A range.[9] If peaks have higher absorbances, then the angle of incidence can be increased or Germanium can be substituted for Zinc Selenide.

Alternate means exist for increasing the sensitivity besides variable angle single reflection ATR spectroscopy. By using transmission spectroscopy, the pathlength can be, at least theoretically, increased indefinitely, with an incumbent increase in absorbance values. This approach, however, could prove very problematic for solids and inconvenient for many liquids when compared to the use of ATR spectroscopy. The use of multiple reflection ATR spectroscopy is a common approach to increasing sensitivity. This technique works well for liquids when the sensitivity (proportional to the number of reflections) is well matched to the sample. However, if a number of different samples are to be analyzed, the fixed number of reflections typical of multiple reflection ATR accessories, will not adapt. The use of multiple reflection ATR is limited, when it comes to solids, as hard or curved samples require that the force applied to the sample be exerted over a very small area.

FUTURE WORK

Theory

A clearer explanation for the general nonconformance of the plots shown in Figures 5 and 8 with the expectations of Figure 2 is required. One possible explanation is that, with the beam spread of the FTIR spectrometer, there is, even with the use of an accessory aperture, a significant portion of the rays that are at larger incident angles. These rays serve to reduce the reported absorbance. Rays that are less than the critical angle exit the system and to not add to or subtract from the baseline corrected absorbance values.

A clearer explanation for the S-shaped curves shown in Figure 5 is also required. Detector non-linearity associated with the loss of energy from sub-critical rays is a possible explanation. The fact that without the aperture, many higher angle rays are allowed to contribute, decreasing the overall absorbance, may also be part of the solution. Other effects complicate any explanation. For example, rays closer to the central ray may have a higher intensity than those in the periphery. Also, the beam does not focus to a point, but rather to a spot approximately 6mm in diameter in the open sample compartment.

Equipment and Sample Selection

A variable diameter aperture, with the option of a half-moon opening, is a high priority. The use of an additional pure organic liquid, without the intense peaks of toluene, would shed additional light on the effects of dispersive distortion on baseline corrections and on possible detector non-linearity due to overall high absorbances. The present liquid cell has a depth of 1.3mm. This should be checked and increased, if necessary. The ATR holder bracket should be equipped with captive screws and ATR retainer brackets to facilitate repetitive cleaning in preparation for new samples.

Procedures

The procedure should include angles well below 37°. A method should be developed to change the angle reproducibly in increments less than one degree near the calculated critical angle.

NEAR CRITICAL ANGLE FTIR ATR SPECTROSCOPY WITH A VARIABLE ANGLE REFLECTION ACCESSORY

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applications note

Optimizing Detection of Thin Films on High Refractive Index Substrates using FTIR-ATR Spectroscopy

S. L. Berets, J. Lucania and J. Christenson
Harrick Scientific Products, Inc., Pleasantville New York

M. Milosevic
MeV Photonics, Westport CT

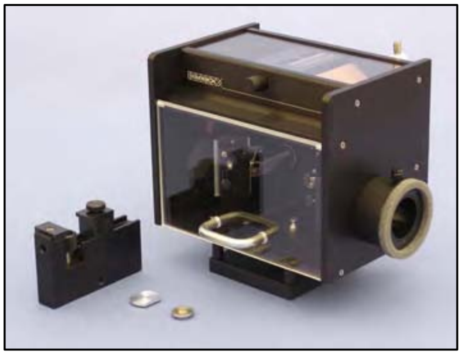

Figure 1. The Seagull™ Variable Angle ATR

Figure 2. The VariGATR™ Grazing Angle ATR Accessory.

INTRODUCTION

Extremely thin films and monolayers on silicon and metal substrates are challenging to measure by infrared spectroscopy. The most sensitive FTIR spectroscopic method for probing these coatings is ATR spectroscopy using a Ge ATR crystal at a relatively high incident angle. Typically these measurements are carried out at an incident angle of 65°. Under these conditions, the evanescent wave is 'trapped' within the coating between the two high refractive index materials, resulting in a signal enhancement.[1]

In principle, the sensitivity of this method depends on the properties of the sample, primarily coating thickness and the substrate refractive index. It also depends on the experimental conditions, specifically the effective incident angle, the incident polarization and the degree of contact between the ATR crystal and the sample.

This paper will explore the role of various experimental parameters to optimize the sensitivity of this method. In particular, the effect of incident angle, the degree of contact, and polarization will be investigated.

THEORY

The theoretical foundations of ATR were developed by Harrick and duPre.[2,3] To summarize, radiation is directed through a prism, typically referred to as the ATR crystal, at an angle higher than the critical angle for internal reflection. The critical angle, θ_c, is defined as:

$$\theta_c = \sin^{-1}\left({}^{n_2}\!/\!{}_{n_1} \right)$$

where n_1 is the refractive index of the ATR crystal and n_2 is the refractive index of the sample. Above the critical angle, an evanescent wave is created at the reflecting surface. When a sample comes in contact with the surface, this evanescent wave interacts with the sample and a spectrum can be recorded to measure this interaction. This interaction is theoretically described by the Fresnel equations, detailed elsewhere.[1,2]

For a silicon wafer, with a refractive index of 3.24 and a Ge ATR crystal, with its refractive index of 4, the critical angle is 58.8°. As the incident angle approaches the critical angle, the evanescent wave penetrates more deeply into the sample and the band intensities increase. In the case of a very thin film on a silicon substrate, this increase in penetration does not change the absorption by the sample. The change in the band absorption results from the change in coupling efficiency of the electric field of the incident light through the interface to the electric field of the evanescent wave.

EXPERIMENTAL

Three sets of FTIR measurements were made: variable angle, variable contact pressure, and two-polarization. For the first two experiments, the composition of the coatings was unknown. For the polarization study, the sample was prepared by the attachment of nitro-benzene diazonium salts to H-terminated <100> Si in HF. All three ATR studies used a Thermo Nexus spectrometer and a torque wrench (Harrick Scientific) for repeatable compression of the sample against the ATR crystal.

For the variable angle studies, the Seagull™ (Figure 1) and the VariGATR™ (Figure 2) were used. The single beam background spectra were collected first and then sample was compressed against the ATR crystal. The single beam sample spectra were recorded at each angle and referenced to the appropriate background. The data were collected at 8cm^{-1} resolution. The Seagull™ was used with a Ge ATR crystal with a specially configured crystal holder and pressure plate, designed to optimize contact between the ATR crystal and the sample. The incident angle was varied from 54° to 68°, in 2° increments, and 128 scans were signal averaged. With the VariGATR™, the angle was varied in 1° increments from 60° to 65° and 32 scans were collected at each angle.

To study the effect of the degree of contact, the GATR™ (Figure 3) was used with its Ge ATR crystal. The degree of contact was varied by changing the torque wrench from its lowest setting, 20 in-oz, to the upper limit of the GATR™, 56 in-oz. These spectra were also recorded at 8cm^{-1} resolution and 128 scans.

For the polarization study, the Harrick GATR™ was used along with a Wire Grid Polarizer on a KRS-5 substrate. The single beam background spectra were recorded at both s- and p-polarization. Then the sample was compressed against the crystal using its 56 in-oz Slip-Clutch. Single beam sample spectra were collected at both polarizations and ratioed to the corresponding background. All spectra were recorded at 2cm^{-1} resolution and 32 scans. The baseline-corrected experimental data was further examined by spectral subtraction and fitting to theoretical simulations using the

Figure 3. The GATR™ Grazing Angle ATR Accessory.

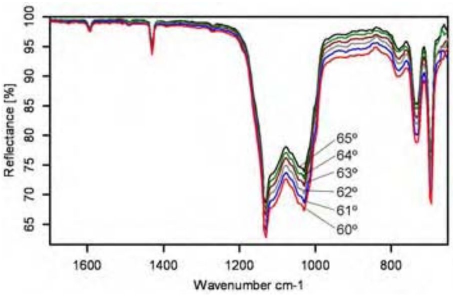

Figure 4. ATR Spectrum of a Thin Film on Si, measured with the VariGATR™ as a function of incident angle.

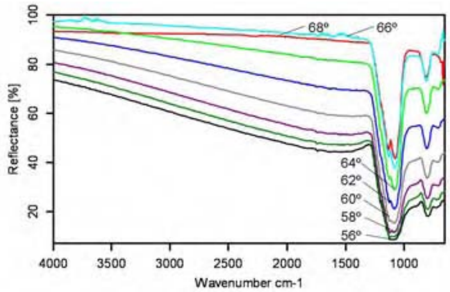

Figure 5. ATR spectrum of a coated Si wafer at various incident angles measured with the Seagull™.

Optimizing Detection of Ultra Thin Films on High Refractive Index Substrates using FTIR-ATR Spectroscopy

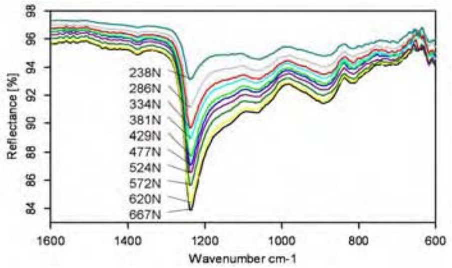

Figure 6. ATR spectra of a thin coating on Si as a function of the force applied to obtain contact.

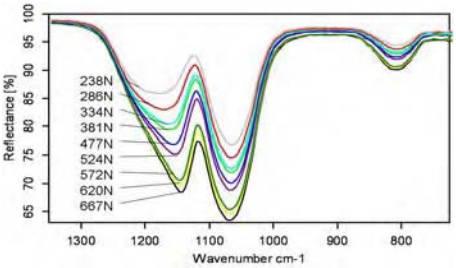

Figure 7. ATR spectra as a function of the force applied to obtain contact.

SOS™ software.[4]

RESULTS AND DISCUSSION

Figures 4 and 5 show the spectra of two different thin films on silicon, measured as a function of the incident angle. Figure 4 shows increasing band intensities as the critical angle is approached. Note that the spectra near critical angle may show a small degree of dispersion, since the baseline appears to be sloping slightly. Figure 5 shows several spectra recorded below critical angle. These spectra show a strongly sloping baseline and an inverted band around 1288cm^{-1}. As the incident angle increases, the baseline straightens out, the inverted band disappears, and two bands in the $1130\text{-}1050\text{cm}^{-1}$ region become better resolved. At an incident angle of 68°, the anomalies are essentially gone and the spectrum looks similar to a transmission spectrum. Thus the ideal angle for measuring this particular sample is significantly higher than the critical angle.

There are several phenomena influencing the experimentally found ideal incident angle. The spectrometer has a focused beam, typically with a ±8° beam spread where the incident angle is defined as that of the central ray. All of these rays interact with the sample, not just the central ray. Both the VariGATR™ and the Seagull ™ use a Ge hemisphere as the ATR crystal. The VariGATR uses the spectrometer's beam as is while the Seagull™ uses a 1:1 imaging. The spectrometer beam refracts at the air-Ge interface, enlarging the beam spread. Note that the hemisphere creates a small illuminated spot, as required for good contact between the ATR crystal and the sample. So there is a trade-off between a smaller beam spread and the degree of contact. The imaging optics of these accessories also differ slightly. The beam spread of the Seagull ™ is probably somewhat larger that of the VariGATR™. If this difference was significant, experimental differences in the dispersion effects would be observed.

Since the sensitivity of an ATR measurement increases approaching the critical angle, it is important to work as close to the critical angle as possible while still collecting transmission-like spectra for ease of interpretation. A variable angle accessory allows optimization of the incident angle for a particular spectrometer and sample. Choice of accessory and possibly the installation of an aperture[5] can be used to reduce the angular spread and further optimize sensitivity.

Another experimental parameter that influences the quality of the spectra is the degree of contact between the ATR crystal and the sample. Figures 6 and 7 show the effect of changing the force applied to the sample on the spectra. Both figures show increasing band intensity with force indicating that better contact is obtained with greater force applied. For ATR measurements of flexible materials, the increase in band intensity eventually saturates as more force is applied. Here, the maximum possible force is applied before the band intensities saturate. Note that the degree of contact is also restricted by the flatness of the sample and ATR crystal. Figure 7 also shows band structure changes around 1200cm^{-1} with increasing applied force. This may indicate a conformational change in the coating. If the coating incorporated an oriented polymer, for example, compressing the sample might alter its orientation.

According to theoretical considerations,[1] the ATR spectra measured at 65° of thin films on Si show sensitivity enhancement for s- and p- polarized light. The spectrum collected with p-polarization is generally enhanced to a greater degree than that collected with s-polarization. In principle, this should provide a clearer distinction between the bonds that are perpendicular to the surface and those that are parallel than other ATR methods. Figure 8 shows the spectra of a thin coating on Si recorded with s- and p-polarization. Bands appear at 1226cm^{-1} and 1112cm^{-1} in the spectrum recorded with p-polarization that are not present in the s-polarized spectrum. The band at 1226cm^{-1} is clearly comparable in strength to the bands at 1526cm^{-1} and 1350cm^{-1} and is likely to be from an oriented species on the surface.

Two other major bands, at 1526cm^{-1} and 1350cm^{-1}, are stronger in the p-polarized data than in the s-polarized data. To determine if this was just due to signal enhancement, the data was further examined by subtraction and simulation. The baseline-corrected s-polarized spectrum was multiplied by four and subtracted from the p-polarized spectrum. The resulting difference is shown in Figure 8. The bands at 1526cm^{-1}, 1350cm^{-1}, 1600cm^{-1} and 1112cm^{-1} all subtract reasonably well. The two stronger bands have a small residual feature from a slight band shift. The band at 1226cm^{-1}, however, is just reduced slightly in intensity, indicating that this band is oriented perpendicular to the surface of the wafer.

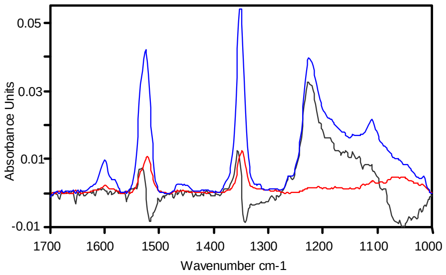

Figure 8. Baseline-corrected ATR spectra of a thin film on <100> Si recorded with s polarization (black) and p polarization (red). The difference is shown in blue.

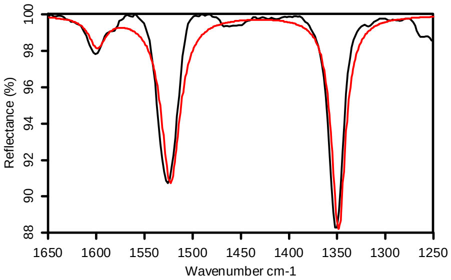

Figure 9. The ATR spectrum of a thin film on <100> Si measured (black) and simulated (red) with p-polarized radiation.

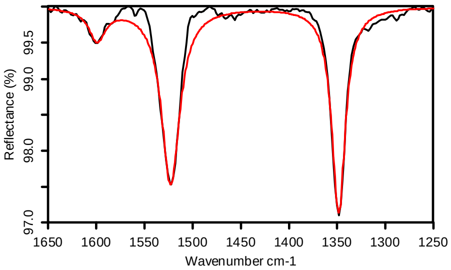

Figure 10. The ATR spectrum of thin film on <100> Si measured (black) and simulated (red) using s-polarized radiation.

Optimizing Detection of Ultra Thin Films on High Refractive Index Substrates using FTIR-ATR Spectroscopy

To further explore whether or not those bands are oriented, the spectra were fitted using the SOS™ software, which calculates the reflectance of a multilayer sample from the Fresnel equations, assuming homogeneous layers. Since the thickness of the coating was unknown, both the peak parameters and the thickness were fit to the s- and p-polarized data. The substrate was assumed to be optically thick with a refractive index of 3.42, and the radiation was perfectly polarized. The resulting coating parameters are shown in Table 1. The simulated spectra are shown in Figures 9 and 10 with the corresponding experimental data. The simulated spectra closely resemble the experimental, supporting the lack of orientation effects in those bands. Note that the coating thickness does influence the appearance of the spectrum and should be measured by independent methods to verify the simulations.

Table 1: Simulation Coating Parameters.

Electronic Polarizability	0.88235
Sample thickness	108nm

Wavenumber	Half-Width	Strength
1349.00	17.00	0.02200
1523.50	23.00	0.02800
1599.00	21.00	0.00480

The band at 1226cm^{-1} is clearly from an oriented species because it only shows spectral features in the p-polarized spectrum. The expected species in the coating, however, do not have characteristic bands at that wavenumber or at 1110cm^{-1}. This, combined with the band location, suggests that it is due to a species that bound to the surface after its preparation, such as SiO_2. SiO_2 naturally forms on Si upon exposure to air, with its dipole moment perpendicular to the surface.[6]

CONCLUSION

The most sensitive FTIR spectroscopic method for probing extremely thin films and monolayers on Si is ATR spectroscopy using a Ge ATR crystal at an incident angle above 58.8°. The readily controllable experimental parameters that control sensitivity include:

○ The incident angle. For best sensitivity, collect the data at an incident angle as close to the experimental critical angle as possible.

○ The degree of contact. For optimal results, use as much contact force as the ATR accessory can deliver without deforming the film.

○ Polarization. P-polarization exhibits enhanced sensitivity relative to s-polarization. The polarization dependence of the method makes it possible to extract information on the orientation of species on the surface using spectral subtraction or spectral simulation methods.

Additional work is in progress to better understand the effect of film thickness in regard to the critical angle and polarization measurements.

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HARRICK SCIENTIFIC PRODUCTS

No. 40929

AN FT-IR STUDY OF PRESSURE-INDUCED SPECTROSCOPIC CHANGES IN A PRESSURE-SENSITIVE ADHESIVE

Figure 1. The VideoMeridian™.

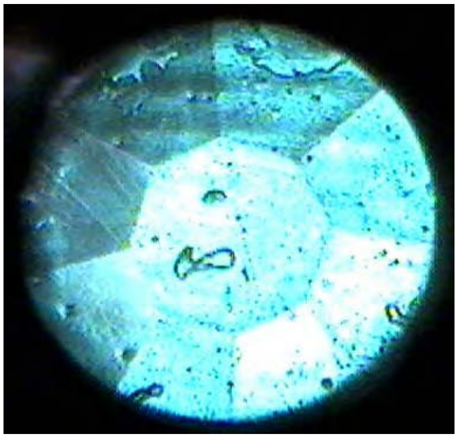


Figure 2. The PSA pressed lightly against the hemi-spherical ATR crystal. Note: The octagonal central area is the area sampled by the VideoMeridian™.

INTRODUCTION

Pressure-sensitive adhesives (PSAs) are self-adhesive materials designed to stick on contact to nearly any surface. First introduced around 1925, the PSA market has expanded from masking tape to include labels, tapes and temporary protective covers for a variety of surfaces. Since PSAs do not rely on solvent evaporation or mixing of chemicals for operation, they are considered safer and more reliable to use in non-structural applications. This, combined with their ease of use, makes PSAs an increasingly attractive alternative to more traditional adhesives.

As the applications of PSAs expand, it becomes increasingly important to understand the chemistry that allows them to function effectively. A better understanding of PSA chemistry can be used to assist in fine-tuning the PSAs for specific applications and also in troubleshooting manufacturing problems.

PSAs are multiphase systems whose adhesive properties are directly related to their chemical architecture. It has been shown that there are visibly observable cavitation effects upon the release of some PSAs. These cavitation effects are thought to be due to the movement of the mobile phases of the PSA. If this is indeed the case, there should be corresponding, observable chemical changes.

This applications note explores the use of FTIR- ATR spectroscopy to study pressure-induced chemical changes in PSAs.

EXPERIMENTAL

A dual-system reactive hot-melt PSA was examined using the VideoMeridian™ diamond ATR accessory (Figure 1) in an FT-IR spectrometer. All spectra were collected with 32 scans at 4 cm^{-1} resolution using a DTGS detector.

A background single beam spectrum was collected using the clean ATR crystal. Then the sample was placed with its adhesive side against the sampling surface of the diamond ATR crystal. The pressure applicator was lowered onto the sample until it just wet against the crystal, as observed with the video imaging system (Figure 2). A spectrum was collected at this contact pressure and then every 2 force units thereafter, as measured on the Video-Meridian™ display, until few additional spectral changes were observed. For reference, a spectrum was also collected

AN FT-IR STUDY OF PRESSURE-INDUCED SPECTROSCOPIC CHANGES IN PRESSURE-SENSITIVE ADHESIVES

from the reverse side of the sample.

RESULTS AND DISCUSSION

The results are presented in Figures 3A through E, where different spectral regions are shown separately. The spectrum shown in red is from the back side of the sample. The spectrum shown in black was recorded at the lowest contact pressure and the highest contact pressure is shown in dark blue.

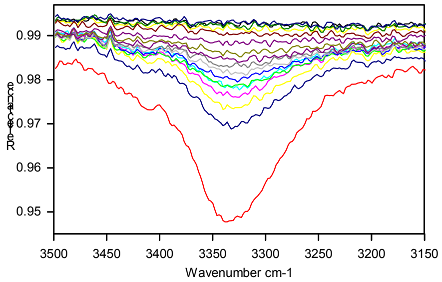
From the spectra, it is clear that it is possible to obtain a spectrum of just the adhesive surface of the PSA when very little pressure is applied.

As the contact pressure increases, some spectra features become more prominent. In particular, the C-O region shows an increase in the 1712 cm^{-1} band and a decrease in the band at 1732 cm^{-1} with increasing pressure. This indicates that the more mobile esters are moving away from the sampled area, and proportionately more of the less mobile esters are detected.

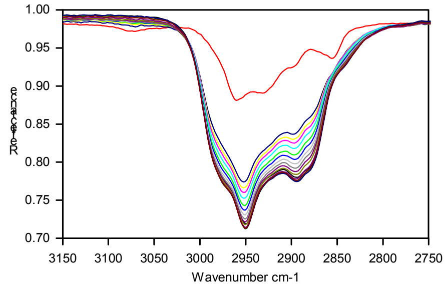
There are a few bands, specifically those at 1388 cm^{-1} and 1470 cm^{-1}, which show no appreciable change in intensity with increasing contact pressure. These bands reflect immobile components of the PSA that are present both in the adhesive surface and bulk material.

SUMMARY

As seen above, PSAs can be readily examined using the VideoMeridian™. The surface of the PSA can easily be distinguished from that of the bulk by applying very little contact pressure between the sample and the ATR crystal of the VideoMeridian™, making this a quick and convenient method of analysis. In addition, the effect of increasing contact pressure on the mobile phases of the PSA can be investigated.



A



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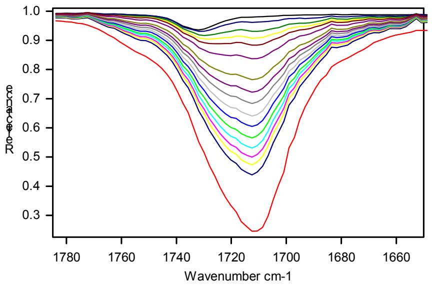

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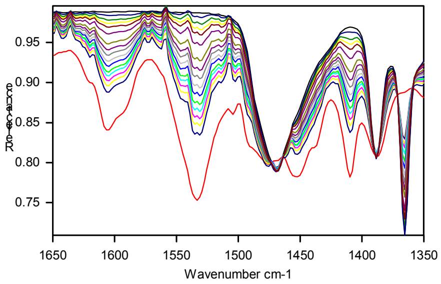

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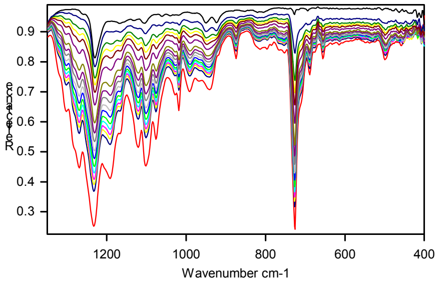

E

Figure 3. The ATR spectra of a PSA as a function of increasing contact pressure (red: substrate; black: lowest contact pressure; dark blue: highest contact pressure).

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PRODUCTS

No. 502xx

application note

DETECTING THIN FILMS ON PLASTIC SUBSTRATES USING MICRO-ATR SPECTROSCOPY

Figure 1. The SplitPea™.

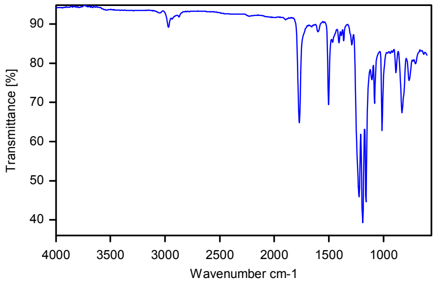

Figure 2. ATR Spectrum of the Cleaned Plastic Substrate.

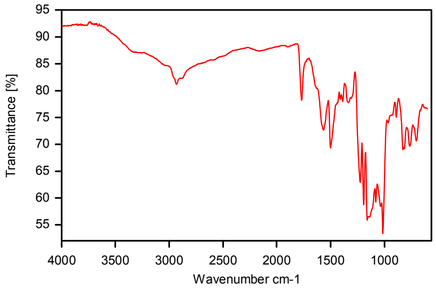

Figure 3. ATR Spectrum of the Coated Plastic.

INTRODUCTION

Plastics are frequently coated on other materials. Some plastics are coated during the shaping process to prevent adhesion to molds. Others are more intentionally coated to change the properties of the surface for particular applications.

Thin coatings on plastics are typically tricky to detect by FT-IR spectroscopy. The common sampling methods, transmission and 45° ATR with a ZnSe or KRS-5 crystal, sample too deeply into the material resulting in spectra that show primarily characteristics of the bulk material rather than the coating. In addition, many of these samples are curved like eyeglass lenses making them less amenable to analysis by these methods.

This applications note explores the analysis of thin films on plastics by micro-ATR spectroscopy. In particular, the SplitPea will be used to detect and identify a coating on plastic. This micro-ATR accessory is uniquely suited to this application since it readily examines irregular and curved samples and can be equipped with a high refractive index ATR crystal for high surface sensitivity.

EXPERIMENTAL

Two slightly curved samples were examined: a coated plastic and a clean, uncoated piece of the sample plastic. The latter was used as a reference to confirm detection of the coating.

The samples were examined using the Harrick SplitPea (see Figure 1) with its Ge hemispherical ATR crystal. The SplitPea was installed in an FT-IR spectrometer. All spectra were collected with 32 scans at 8 cm^{-1} resolution using a DTGS detector.

The background spectra were collected from the clean Ge ATR crystal. Then the sample was placed on the ATR crystal. The sample was compressed against the crystal using the maximum force allowed by the built-in pressure applicator, prior to collection of the sample spectrum.

RESULTS AND DISCUSSION

Figure 2 shows the ATR spectrum of the plastic substrate, while the spectrum of the coated plastic is shown in Figure 3. The substrate itself can be identified as a polycarbonate, by using a standard library spectral search.

From comparison of the two spectra, it is obvious that there are distinct bands in Figure 3 that are due to the coating and not the substrate. The carbonyl band at 1767 cm^{-1} and the 1503 cm^{-1} band are present in both spectra, indicating that there may be a small contribution in the coating spectrum from the substrate.

However, the broad peaks between 3300 and 2500 cm^{-1} show the presence of a carboxylic acid or derivative in the coating which is clearly not present in the substrate.

SUMMARY

In conclusion, thin coatings on plastics can be detected and identified using micro-ATR FT-IR spectroscopy. The SplitPea is an ideal tool for this type of measurement, since it can be equipped with a Ge ATR crystal for low penetration into the sample and can readily accommodate curved or irregularly shaped samples.

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applications note
No. 40608

THE EFFECT OF SURFACE UNIFORMITY ON THE SPECULAR REFLECTANCE FROM CARBON-FILLED COATINGS

Figure 1. The Seagull™.

Figure 2. The Video Meridain™.

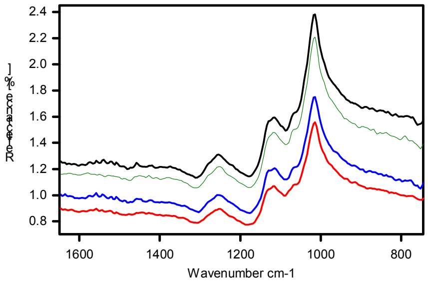

Figure 3. The 60º specular reflectance from two samples recorded with the Seagull.

INTRODUCTION

Carbon-filled coatings are frequently applied to materials to reduce their infrared and visible reflectivity. Specular reflectance is frequently the method of choice for examining the reflectivity of these coatings. However, these coatings are not always smooth and this can produce anomalies that complicate interpretation of the results.

This applications note demonstrates the problem using a routine method used to check the coatings and then explores the effect of coating texture and uniformity on the results.

EXPERIMENTAL

The samples examined were pieces of sheet metal coated by Duracon™ Optical Black Coating from American IMEX Corporation. Several samples were prepared in a similar fashion and then used for this study.

These samples are routinely examined using the Seagull™ variable angle reflectance accessory (Figure 1) that utilizes a 1:1 imaging system. The Seagull™ was set for its specular reflectance mode at an incident angle of 60º.

For a more detailed inspection of the surface, the VideoMeridian™ microsampler (Figure 2) was used. The VideoMeridian™ features a six times beam condenser and a fixed 45º incident angle. It was configured for specular reflectance and its built-in video camera was adjusted so the sample was in focus. The sample was randomly scanned and digital images were acquired, using the MicroSnap™ software, for each position along with the spectra.

Both the Seagull™ and the VideoMeridian™ were used in conjunction with a commercial FT-IR spectrometer and the data was collected over the mid-infrared region at 8 cm^{-1} resolution and signal averaged over 32 scans.

RESULTS AND DISCUSSION

The results from the routine analysis of two samples using the Seagull™ are shown in Figure 3. Two measurements were taken from each sample and the sample was repositioned between measurements. While it is clear that little of the incident radiation is reflected, the reflectance does differ as a function of the sample and its position. This difference is evident both in the base level of reflectivity and in the peak intensity.

The Effect of Surface Uniformity on the Specular Reflectance from Carbon-Filled Coatings

Since the samples were prepared in the same fashion and the samples have some texture, it is possible that the coating is not uniform in either texture or coverage. This non-uniformity could be altering the reflectance from the surface.

To examine this phenomenon in more detail, the surface of one of the samples was randomly scanned and examined using the VideoMeridian™. The resulting spectra are shown in Figure 4 and the corresponding images are presented in Table 1.

Comparison of the band intensities in Figures 3 and 4 shows that the magnitude of the intensity fluctuations is approximately six times smaller with the Seagull than with the VideoMeridian™. This is expected given the differences in the sampled areas between the two accessories.

From Table 1, it is clear that the samples show differences in overall texture. In addition, several sections (d and f in Figure 4) appear to have micro-holes in the coating. The differences in the overall brightness of the visual image are loosely correlated to the band intensity differences shown in Figure 4. It is possible that a direct correlation between the sample texture and the infrared band intensity could be drawn if infrared illuminated portion of the visual image could be distinctly analyzed.

However, the 'holes' in the coating may cause non-linear effects. Since the spectrum is significantly different for a thin film. Figure 5 shows the spectrum of a corner of the sample which is only partly coated. This spectrum shows the transmission-like characteristics of a thin film on a metallic substrate rather than the dispersive characteristics typical of specular reflectance.

CONCLUSION

As seen from the data presented herein, the Video Meridian™ microsampler can be used to examine surface inhomogeneities that are a few millimeters in size both visually and by infrared spectroscopy. Infrared spectroscopy effectively integrates over the illuminated area. However, the effect of surface inhomogeneities can be detected in the resulting spectra for both small and large sampling areas.

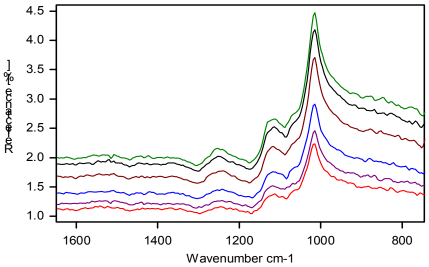

Figure 4. The 45° specular reflectance of sampling positions a through f (top to bottom) recorded with the VideoMeridian™.

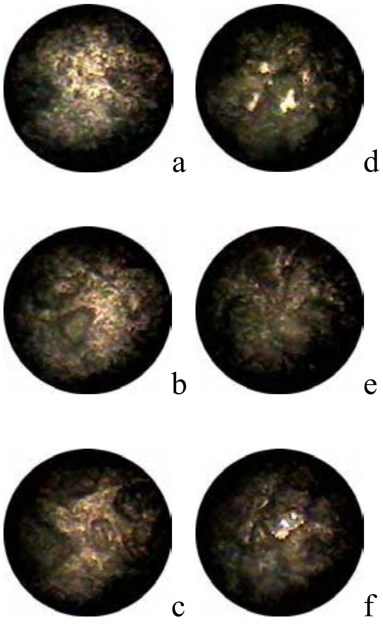

Table 1. Photographs of the sections examined in Figure 4.

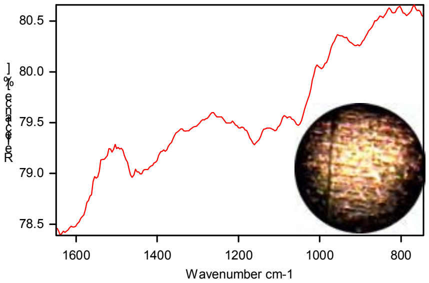

Figure 5. The 45° specular reflectance and photograph of a lightly coated section of the sample.

HARRICK
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No. 601109

application note

IN-SITU ATR-FTIR OF HAFNIUM (IV) TERT-BUTOXIDE ADSORPTION AND DECOMPOSITION ON GE

Shilpa Dubey, Amy M. Herber, Harish Bhandari, and T. M. Klein
Department of Chemical and Biological Engineering, The University of Alabama, Tuscaloosa, AL

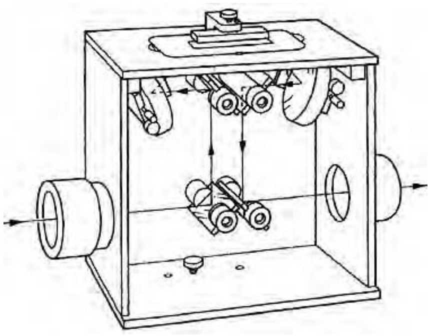

Figure 1. The Horizon™, shown here with its solid sampling plate.

INTRODUCTION

Attenuated Total Reflectance (ATR) FTIR is widely used by researchers to examine a variety of sample types including solids, powders, pastes and liquids for food analysis, biomedical applications, polymers, and thin films. In addition to the identification of functional groups during more routine analysis, ATR-FTIR spectroscopy is useful for mechanistic studies of vapor-solid interactions during chemical vapor deposition or heterogeneous catalysis by *in situ* real time monitoring of surface species[1]. These experiments typically require the IR beam to exit the spectrometer, pass through a vacuum system or pressure reactor containing the ATR crystal and gas phase molecules of interest via IR transparent windows, and finally to return to the spectrometer for detection.

In this note, a Horizon™ accessory in conjunction with a heated trough-plate design with VCO vacuum fittings was used to introduce gaseous reactants to the vicinity of a heated ATR crystal element under low pressure in order to observe absorption and decomposition of Hf (IV) t-butoxide (HTB) on Ge. HTB is a widely used organometallic precursor for HfO_2, and a leading candidate to replace SiO_2 gates in metal oxide semiconductor field effect transistors (MOSFETs).[2,3] With this method, adsorption of hafnium tert-butoxide (HTB), was observed.

EXPERIMENTAL

The Horizon™ accessory (Figure 1) with a Ge crystal element was placed in the sample compartment of an FTIR spectrometer. The inlet of the flow-through cell was connected via VCO fittings with Kalrez gaskets to a heated gas line (50-55ºC) for delivery of the HTB through a gas-liquid bubbler at a temperature of 52ºC. HTB is a clear liquid at room temperature with a vapor pressure of 1torr at 65ºC.[3] The outlet of the flow through cell was connected to a 2.3 ft^{3}min^{-1} rotary vane pump resulting in a pressure of 1 torr in high throughput mode measured by a convection pressure gauge on the outlet side and with a flow rate of 5 sccm of N_2 carrier gas plus 0.67 sccm of HTB when vapor-liquid equilibrium is assumed. Before the experiment the Ge ATR crystal was cleaned with trichloroethylene and dipped in a buffered 1:500 hydrofluoric acid solution to remove the native oxide and to obtain an H-terminated surface.[4] A background spectrum was recorded for an ATR crystal temperature of 100ºC under 0.01 torr pressure without N_2 or HTB flow. Using N_2 as the carrier gas, HTB vapor was delivered to the ATR cell where the crystal was maintained at 100ºC, with a total pressure of 1 torr. During the exposure of precursor on the crystal surface, IR spectra were collected at 5 min intervals. For comparison, liquid HTB was directly applied to the crystal under an inert atmosphere and an IR spectrum was collected at room temperature and atmospheric pressure.

RESULTS AND DISCUSSION

A Ge crystal was chosen instead of the more application relevant Si (100) surface as Ge has a wider range of IR transparency. Figure 2 shows the single beam spectra obtained after 2 min, 9 min, and 9 hrs as well as the applied liquid HTB for comparison. Absorbance peaks between 3000-2800 cm^{-1} are due to *asym* and *sym* C-H stretches of the tert-butyl groups, and are a useful feature for monitoring surface coverage of the precursor.[5]

Figure 3 shows the time dependence of the C-H stretching mode peak area as it increases and then saturates. The saturated absorbance is 5% lower than the applied liquid value indicating that the thickness of a physisorbed layer may have exceeded the depth of penetration of the evanescent wave.

Absorbance peaks in the lower wavenumber region, 1500 to 500 cm^{-1}, are attributed to C-O

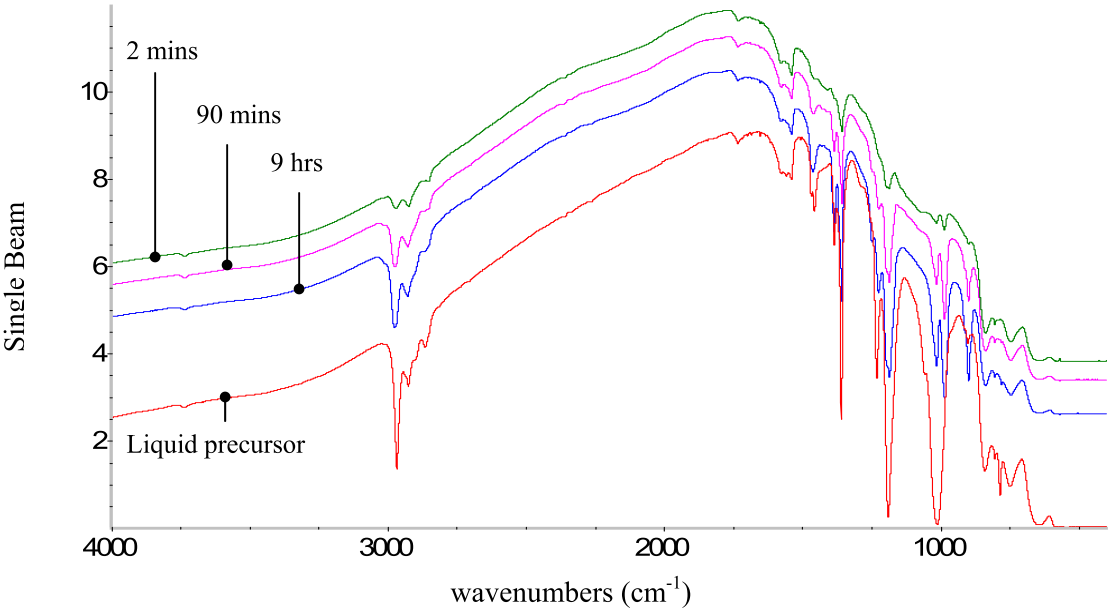

Figure 2 Single beam spectra of adsorbed HTB on Ge at 100ºC and 1 torr and applied liquid precursor at atmospheric pressure and room temperature.

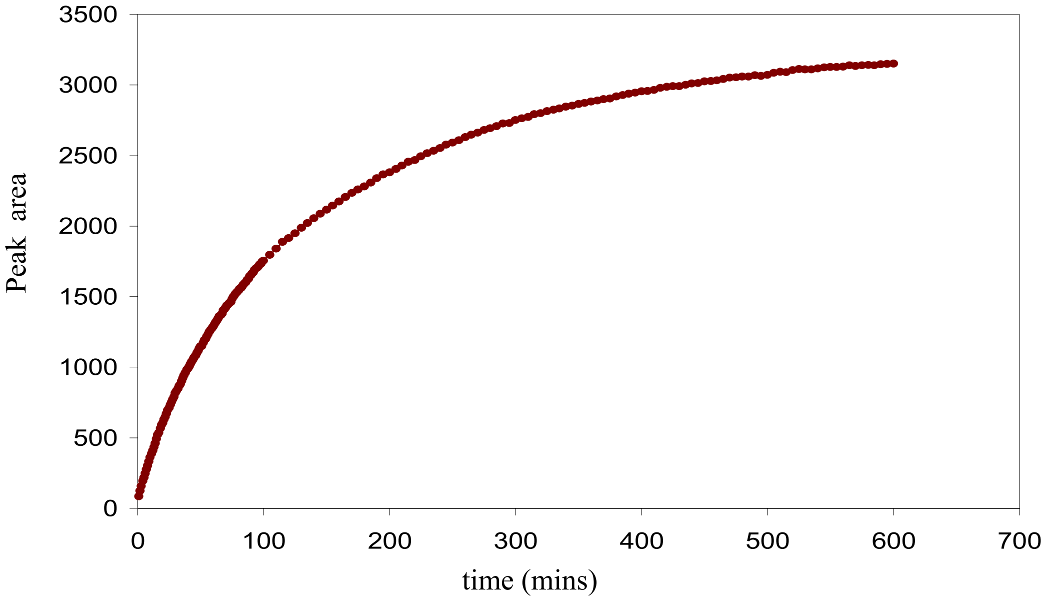

Figure 3 C-H stretch peak area vs. time for HTB absorption on Ge crystal at 100ºC and 1 torr.

(1230cm^{-1}), Hf-O-Ge (1017cm^{-1}), Hf-O-C (985cm^{-1}) and Ge-O (900cm^{-1}) stretching and are shown in Figure 4 with the liquid applied HTB for comparison. Some major differences between the applied liquid and vapor adsorption experiments elucidate the chemical bonding of the precursor to Ge. Specifically, the observed Ge-O and Hf-O-Ge stretches, which are absent in the liquid HTB measurement, show the HTB molecule bonds to the surface through oxygen atoms.

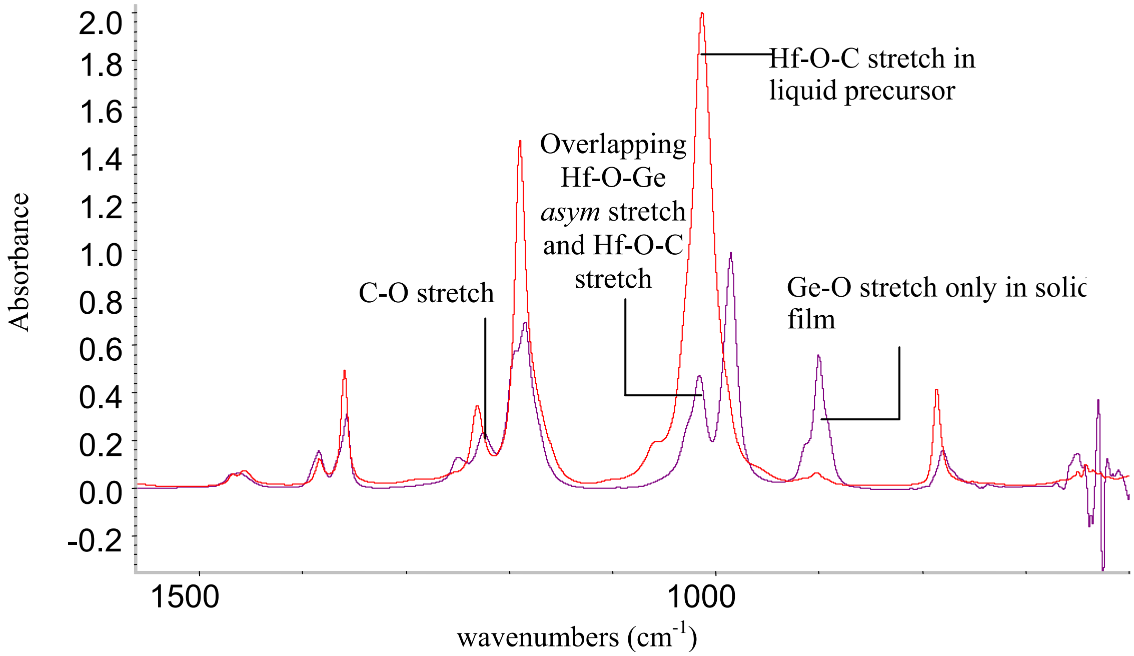

Figure 4 Absorbance spectrum of adsorbed HTB on Ge at 100ºC and 1 torr (purple) and applied liquid precursor at 1 atm and 25°C (red).

CONCLUSION

The Horizon™ ATR accessory was used to observe the adsorption and decomposition of HTB vapor molecules on a heated Ge crystal and the spectra were compared to HTB applied as a liquid drop to the crystal element at room temperature. The spectra indicate attachment of the HTB groups through Hf-O-Ge bonds. The results show that ATR-FTIR is an extremely useful surface sensitive technique to determine functional groups and bonds *in situ* during thin film deposition at moderately elevated temperatures and under vacuum.

ACKNOWLEGDEMENT

This work was supported by the National Science Foundation, Award #0239213.

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HARRICK
HARRICK SCIENTIFIC PRODUCTS

No. 60320

application note

USING FIBER OPTICS FOR MID-INFRARED SPECTROSCOPY WITH A DTGS DETECTOR

Figure 1. The FiberMate.

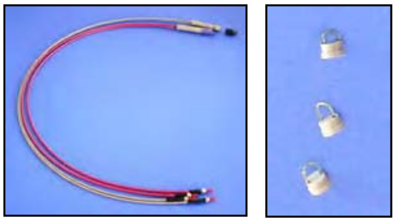

Figure 2. The MultiLoop-MIR fiber probes (left) and sampling tips (right).

Figure 3. AccessATR single reflection ATR accessory.

INTRODUCTION

Mid-infrared fiber optics systems have been commercially available since the early 1990s[1] and are becoming a valuable tool for remote sampling and in-situ studies[2]. Unlike visible and near-IR fiber optic systems, however, these systems are not in common use. The low throughput of the fibers typically required a nitrogen-cooled detector and the fragile nature of the appropriate fibers limit their potential applications.

A new mid-infrared fiber optic system has been recently developed to surmount these limitations. This system includes a high-efficiency FT-IR fiber optic coupler and an ATR fiber optic probe with disposable sampling tips for use over the entire mid-IR.

The paper examines the sensitivity of this new fiber optic system in an FT-IR spectrometer with a DTGS detector. Specifically, the number of reflections for the fiber loop tip will be determined and differences between the tips explored. The sensitivity of the system will also be examined by looking at several samples, including liquids and pastes.

EXPERIMENTAL

All spectra were recorded using a Thermo Nexus 670 spectrometer with a DTGS detector configured for data collection at 8 cm^{-1} resolution.

For the fiber optic measurements, the spectrometer was equipped with Harrick Scientific Products' FiberMate™ fiber optic coupler (Figure 1) and the MultiLoop-MIR™,[3] infrared fiber optic probes (Figure 2). The MultiLoop-MIR™ system consists of two fiber optic probe extensions and disposable loop ATR sampling tips covering the mid-infrared. One of the probes is made from chalogenide (CIR) designed for use from 4000 cm^{-1} to 1700 cm^{-1} and the other is a silver halide composite (PIR) for operation from 2000 cm^{-1} to 600 cm^{-1}. The tips are also made of silver halides. Data collected with the FiberMate™ and MultiLoop-MIR™ installed in the FTIR spectrometer were signal averaged over 64 scans.

For comparison, traditional ATR measurements were recorded using the Harrick AccessATR™, a 45° single reflection ATR accessory with a ZnSe triangular prism ATR crystal (Figure 3). Infrared spectral data collected with the AccessATR™ were averaged over 32 scans.

Background spectra were collected from the clean fiber loop or ATR crystal. Then the samples were brought into intimate contact with the sample. Solid samples were compressed gently against the fiber optic loop. The data collected with the loop were then baseline corrected and joined together to show the full spectral range.

Several different samples were examined: paraffin oil, red grape products and toothpaste. The red grape products included Carlo Rossi Paisano red wine, Progresso Red Wine Vinegar, and Kedem Grape Juice. The toothpastes were Procter & Gamble's Crest® Kid's Sparkle Fun Flavor and Cinnamon Rush Whitening Expressions, Colgate-Palmolive's Colgate® Regular, and Glaxo-SmithKline's Sensodyne®.

THEORETICAL DISCUSSION

In ATR spectroscopy, a material is brought into intimate contact with the optical element, where light is totally internally reflected and the sample interacts with the evanescent wave. This interaction is completely described by the Fresnel equations[4,5]. These equations are complex and not particularly intuitive. Most importantly, they relate the reflectance to the incident angle, the incident polarization, and the complex refractive indices of the sample and ATR element.

For ATR, the reflectivity, R, of a bulk material can be expressed as:

$$R=1-\alpha\, d_e \qquad (1)$$

where α is the absorption coefficient. The d_e term is defined as the effective thickness, analogous to the thickness parameter for transmittance.

For N multiple reflections, the reflectivity becomes:

$$R^N=(1-\alpha\, d_e)^N \qquad (1)$$

For weakly absorbing materials, $\alpha\, d_e \ll 1$ and:

$$R^N=1-N\,\alpha\, d_e \qquad (2)$$

The expression for the effective thickness can be derived from the Fresnel equations. For weakly absorbing isotropic materials, the effective pathlengths[2] for perpendicular and parallel polarizations are:

$$\frac{d_{e\perp}}{\lambda_1}=\frac{n_{21}\cos\theta}{\pi(1-n_{21}^2)(\sin^2\theta-n_{21}^2)^{1/2}} \qquad (3)$$

$$\frac{d_{e\parallel}}{\lambda_1}=\frac{n_{21}\cos\theta(2\sin^2\theta-n_{21}^2)}{\pi(1-n_{21}^2)[(1+n_{21}^2)\sin^2\theta-n_{21}^2](\sin^2\theta-n_{21}^2)^{1/2}} \qquad (4)$$

where λ_1 is the wavelength, θ is the incident angle, and n_{21} is the refractive index of the sample relative to that of the ATR element.

RESULTS AND DISCUSSION

Traditional ATR crystals have well-defined geometries that result in one or more reflections from the sample. A triangular prism, such as used by the AccessATR™ provides a single reflection. Multiple reflection ATR crystals are typically parallelepipeds or trapezoids in which the number of reflections can be easily calculated the length and thickness of the crystal, in addition to the incident angle. For the fiber loop probe used herein, it is extremely difficult to ascertain the effective incident angle and the number of reflections from geometrical considerations. However, the number of reflections can be determined experimentally.

Spectra were recorded from paraffin oil using four different

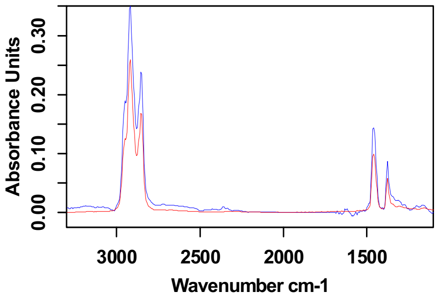

Figure 4. ATR Spectra of parafin oil, recorded by single reflection ATR (red) and fiber optic loop 3 (blue).

Loop ID	Effective Number of Reflections CIR Probe 3100-2850 cm^{-1}	 PIR Probe 1530-1230 cm^{-1}
1	2.2	1.8
2	2.1	1.6
3	2.0	1.8
4	2.0	1.7

Table 1. Effective number of reflections for the fiber loop probe as compared to 45° single reflection ATR on ZnSe.

sampling tips and the two fiber probes. These spectra were then compared to the single reflection ATR spectrum (see Figure 4). For all eight combinations, the differences between these spectra were calculated to determine the effective number of reflections, which are presented in Table 1.

With respect to band intensities, the fiber loop sampling tips provides the sensitivities equivalent to slightly less than two ATR reflections when compared to ATR with a 45° incident angle on a ZnSe triangular prism. Note that the effective number of reflections appears to be a function of wavelength. This is due to the fact that the infrared bands around 3000 cm^{-1} technically do not meet the criteria for a weak absorption and thus eq. 3 does not pertain.

The measured number of reflections is not surprising. The 'incident angle' for the fiber loop is not well defined and is likely to be much higher than 45°, since it is the grazing or high angle rays that propagate down the length of the fiber.

Note that there are also slight differences among the fiber loops. This most likely results from the small differences in the overall length and curvature of the loop. These slight differences indicate that for quantitative measurements it would be advisable to either use an internal standard or calibrate each loop prior to use.

Figures 5 through 9 show spectra of several liquids and pastes recorded using this fiber optic system. The first three figures show spectra recorded from different liquids, all derived from red grapes. Figure 5 shows the spectrum of grape juice, where distinct characteristics of the organic component of the grapes are clearly visible over the water bands. Figure 6 shows the spectrum of wine. There are distinct bands at 2984 cm^{-1} and 1046 cm^{-1} that are not present in juice. These are the C-H and C-O bands respectively from the alcohol in the wine. Figure 7 shows the spectrum of red wine vinegar. Bands are present in the 1712 cm^{-1} and around 1300 cm^{-1} that are characteristic of the carboxylic acids in vinegar.

Figure 8 shows spectra from two gel-like toothpastes. These spectra are very similar, indicating that their composition is nearly identical. This is confirmed by the package, which indicates very similar primary ingredients (see Table 2).

In Figure 9, two additional spectra of white toothpastes are shown. The spectrum of Sensodyne has some similarities to that of glycerin[6] and it has distinct bands around 830 cm^{-1},

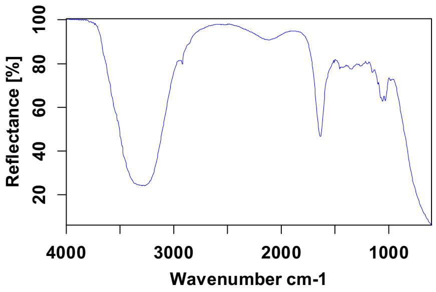

Figure 5. ATR spectrum of red grape juice.

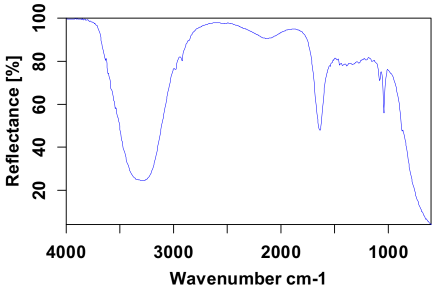

Figure 6. ATR spectrum of red wine.

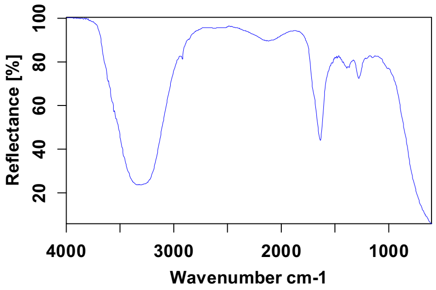

Figure 7. ATR Spectrum of red vinegar.

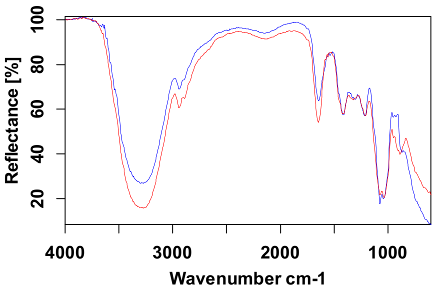

Figure 8. ATR spectra of colored toothpastes: Cinnamon Rush (red) and Kid's Crest (blue).

indicating the presence of a peroxide. From the spectra, the Colgate paste appears to have a lower concentration of glycerin and somewhat more water.

CONCLUSION

This mid-infrared fiber optic system, consisting of the FiberMate coupler and MultiLoop-MIR, has sufficiently high sensitivity for use with commercial FTIR spectrometers and their DTGS detectors. These fiber optic probes provide approximately two reflections from the sample compared to single reflection 45° ATR on ZnSe. As demonstrated here, this system is suitable for use in qualitative studies and has sufficient performance for quantitative work. Additional work is in progress to determine the limits of this system for quantitative studies.

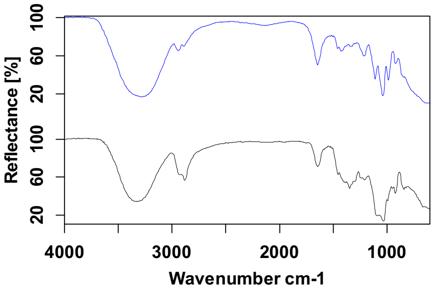

Figure 9. ATR spectra of white toothpastes: Colgate Regular (blue) and Sensodyne (black).

Toothpaste	Primary Ingredients Listed
Crest Cinnamon Rush	sodium fluoride, sorbitol, water, hydrated silica, glycerin, Tetrasodium pyrophosphate
Kid's Crest	sodium fluoride, sorbitol, water, hydrated silica, trisodium phosphate, xanthan gum
Sensodyne	potassium nitrate, sodium monofluorophosphate, calcium peroxide, flavor, glycerin, hydrated silica, PEG-12
Colgate Regular	sodium monofluorophosphate, dicalcium phosphate dehydrate, water, glycerine, sodium lauryl sulfate, cellulose gum

Table 2. Primary ingredients of the toothpastes examined.

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[6] See www.chemexper.com

No. 60201

DISTINGUISHING RELEASE AGENTS ON O-RINGS USING ATR SPECTROSCOPY

Figure 1. The MVP2™.

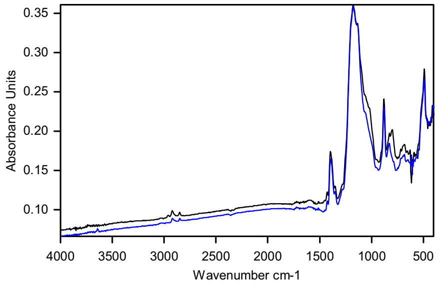

Figure 2. ATR Spectra of a Viton O-Ring. Outside Surface: Black; Inside: Blue.

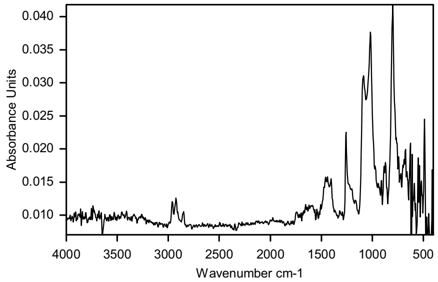

Figure 3. The difference between the inner and outer surfaces of the Viton® O-Ring.

INTRODUCTION

O-rings are typically coated with a release agent during manufacturing. These release agents are used to coat the o-ring molds or are imbedded in the mold to facilitate the removal of the o-ring from the mold. The composition of the release agents varies, depending on the o-ring material.

For the o-ring manufacturer, it is important to be able to detect the release agent on the o-ring if the o-rings unexpectedly begin to stick in the molds. For the o-ring user, it can be important to know about the composition of the release agent used. An o-ring chosen for its chemical inertness may cause minor but critical contamination due to the residual release agent.

There are several potential problems for detecting the release agents on o-rings by infrared spectroscopy. The release agent forms a very thin layer on the o-ring surface. The o-rings are typically small and frequently carbon-filled. Thus analysis of these samples requires a surface sampling technique with a relatively small sampling area, such as single reflection ATR.

This applications note demonstrates the use of ATR to distinguish o-ring release agents from the underlying o-ring material.

EXPERIMENTAL

Three different samples were examined: a black Viton®[1] o-ring, a reddish brown Buna o-ring and another black o-ring of unknown origin. The Buna o-ring appears to have a whitish coating on the surface, while the other two o-rings have black outer layers. The samples were examined as is for the measurements of their exterior surface. For inspection of the interior, a small slice was taken from each o-ring.

The samples were examined using the Harrick MVP-Pro™ in conjunction with a commercial FT-IR spectrometer. The MVP-Pro™, shown in Figure 1, is an ATR accessory with a 3-mm diameter hemispherical Si crystal at a fixed 45° incident angle. The MVP-Pro™ was equipped with a 40° mask to block some of the undesirable low-angle rays impinging on the ATR crystal.

All infrared spectra were collected over the mid-infrared region at an 8 cm^{-1} resolution with the signal averaged over 32 scans.

RESULTS AND DISCUSSION

Figure 2 shows the ATR spectra of both the outer surface and the inner substrate of a Viton® o-ring. Both spectra show characteristics typical of a poly(aliphatic) fluoride, as

Distinguishing Release Agents on O-Rings by ATR Spectroscopy

expected from this fluoroelastomer. The strong similarity between the spectra indicates that the release agent coating the o-ring is extremely thin. Slight spectral differences can be seen around 1020 cm^{-1} and 800 cm^{-1}. Subtracting the two spectra results in the spectrum shown in Figure 3, which is clearly identifiable as silicone oil. Silicones are one of the materials frequently used as a release agent.

In Figure 4, the ATR spectra from the outside and inside of a Buna o-ring are presented. Both spectra have a curving baseline, indicating that the sampled areas have a high refractive index. This is probably due to the degree of carbon-filling in the substrate and the thickness of the release agent coating. The coating is thin enough so that the ATR spectrum of the outside of the o-ring is also sampling the o-ring material. However, there are distinct bands around 3515 cm^{-1}, 1532 cm^{-1}, 1456 cm^{-1}, and 1404 cm^{-1} that are due to the release agent. These bands are not present in the spectrum of the inner o-ring material.

Figure 5 shows spectra from a black o-ring of known origin. The release agent is clearly distinguishable from the o-ring substrate material. The spectrum of the inside of the o-ring has a curving baseline, indicating that it is carbon-filled. Very little of this baseline effect is seen in the spectrum of the coating, indicating that the coating is not extremely thin, as it is with the Buna o-ring (Figure 4).

CONCLUSION

From the infrared spectra presented here, it is clear that the o-ring release agent can be distinguished from the bulk o-ring material using Si micro-ATR methods and the MVP-Pro™. Additional work is in progress to try to further reduce the effects of the carbon-filling on the spectra of o-rings.

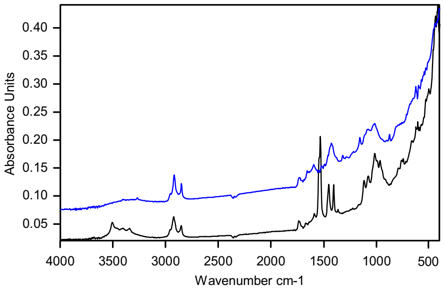

Figure 4. ATR Spectra of a Buna O-Ring. Outside Surface: Black; Inside: Blue.

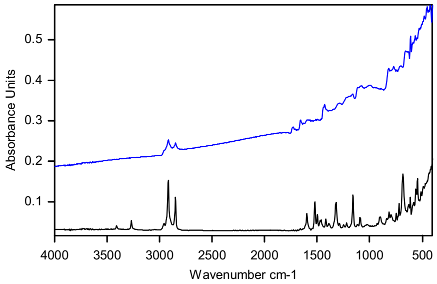

Figure 5. ATR Spectra of a Black O-Ring. Outside Surface: Black; Inside: Blue.

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[1] Registered trademark of Dupont Dow Elastomers.

HARRICK SCIENTIFIC PRODUCTS

No. 91201

application note

Coupled XAS-Raman *Operando* analysis of TiO_2 supported oxomolybdate catalysts in methanol conversion

Camille La Fontaine, Françoise Villain, Valérie Briois
Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin, BP48, 91192 Gif-sur-Yvette, France

Xavier Sécordel, Anthony Yoboué, Elise Berrier, Sylvain Cristol
UCCS, Unité de Catalyse et de Chimie du Solide, Bât C3 Université des Sciences et Technologies de Lille, 59655 Villeneuve d'Ascq Cedex, France

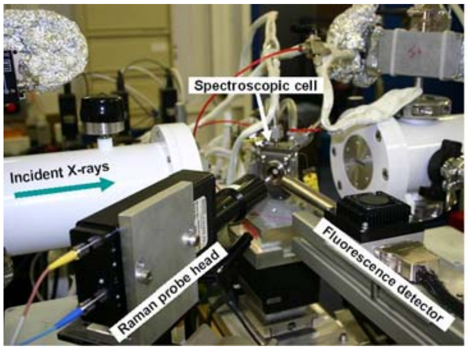

Figure 1. *Operando* Raman-XAS coupling setup.

INTRODUCTION

Although heterogeneous catalysts are essential in numerous chemical processes, the exact structure of the active phase remains unsolved in many cases. In order to understand the structure-reactivity relationships, a relevant approach consists in examining the catalyst while operating. This so-called *operando* approach has recently attracted a fast growing attention and was applied to various spectroscopic techniques: IR, Raman, Electron Paramagnetic Resonance (EPR), UV-visible and X-ray Absorption Spectroscopy (XAS). However, it generally appears that one single spectroscopic technique does not make it possible to spell out the complexity of the catalytic process. Therefore, the direct coupling of complementary *in situ* or *operando* techniques offers a multiple and simultaneous characterization of a working catalyst for a much better understanding of the catalytic process. Such studies request the use of a single spectroscopic reactor fitting the combined peculiarities of catalysis and the involved spectroscopic techniques. The coupling of XAS and Raman spectroscopy was successfully performed in a commercially available *operando* chamber to supplement our previous Raman studies on TiO_2 supported oxomolybdate catalysts.[1] The investigated selective oxidation of methanol is both a major economic reaction for vaporization of biomass by-products and a probe reaction broadly used to study redox and acido-basic properties of bulk and supported oxides.

EXPERIMENTAL

The catalysts were prepared by incipient wetness impregnation of an ammonium heptamolybdate solution on pure anatase TiO_2 commercially available by Sachtleben™. The specific area of the support was 93.5 $m^2.g^{-1}$ after a pre-treatment (rinse with distilled water and calcination 5h at 500°C). The concentration of the impregnation solution was adjusted in order to reach a molybdenum

Coupled XAS-Raman *Operando* analysis of TiO_2 supported oxomolybdate catalysts in methanol conversion

loading corresponding to 7.5%$_{wt}$ MoO_3/TiO_2.

Catalyst *in situ* and *operando* characterization by combined XAS and Raman spectroscopies was performed at the French Synchrotron Radiation Facility SOLEIL on the SAMBA beamline. X-ray Absorption Near Edge Structure (XANES) spectra were recorded at the Mo K-edge (20000 eV) using a Si(220) sagitally focusing double crystal monochromator. The monochromatic beam was focused at the sample position onto a spot of 200 × 300 μm^2 and the fluorescence signal was recorded by a Silicon drift detector. Using a fast scan of the step by step monochromator, only 4 minutes were needed to collect one XANES spectrum (19900-20200 eV). Therefore, a rather good monitoring of the catalyst was allowed in working conditions at different temperatures and under selected atmospheres.

The *in situ* and *operando* analyses were allowed by using an environmental spectroscopic chamber developed by Harrick with a special planar dome designed for Raman measurements[1]. The silica window was replaced by a thin mica plate (200 µm thick) to improve its transmission towards X-rays without any dramatic alteration of the Raman signal. Due to its implementation on the beamline, the spectroscopic cell was vertically set up and oriented at 45° with respect to X-rays incident beam and fluorescence detector[2,3], as shown in Figure 1. The gas flow through the powdered catalyst was reversed compared to usual operation in order to help maintaining the powder inside the sample holder in vertical position. The methanol was introduced in the spectroscopic chamber by means of a He flow bubbling in a saturator equipped with a condenser whose temperature was set to 0°C to get a methanol partial pressure of 3.91 kPa.

Simultaneously to XANES data, Raman spectra were recorded in working conditions using a Kaiser HL5R Raman spectrometer. A Raman probe head equipped with a long working distance objective (×10) facing the sample was used to focus the 514.5 nm excitation beam and collect the scattered light (Figure 1). Optical fibres were used for light guiding and the scattered light was collected by an air-cooled CCD.

RESULTS AND DISCUSSION

The 7.5%$_{wt}$ MoO_3/TiO_2 catalyst was first activated by a gradual increase in temperature

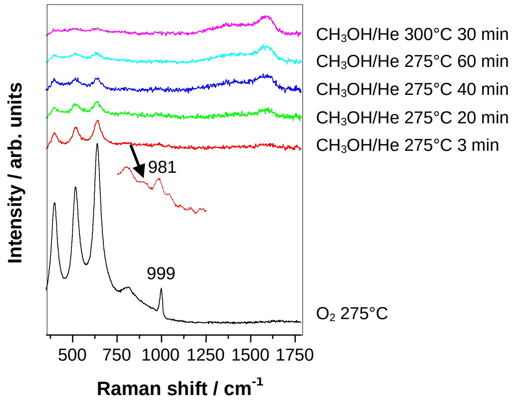

Figure 2. Raman spectra evolution along *operando* experiment.

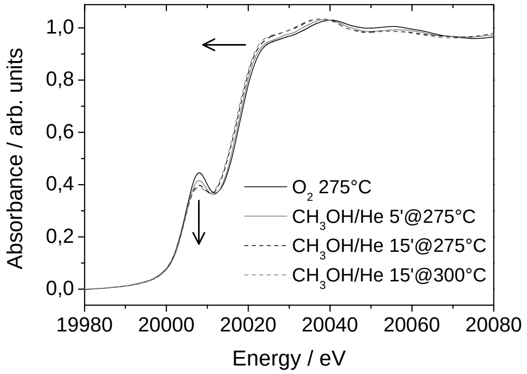

Figure 3. Evolution of the XANES spectra along *operando* experiment.

(5°C.min^{-1}) up to 350°C under pure oxygen flow. The structural evolution of the active phase was then examined while flowing CH_3OH/He at 275°C. No oxygen was introduced in the feed to allow the characterization of the reduced species although a CH_3OH/O_2/He mixture is generally used for a continuous regeneration of the active phase along the catalytic process.

The *operando* Raman and XANES spectra recorded under CH_3OH/He flow at 275°C are reported in Figures 2 and 3. The Raman spectrum of the activated catalyst (Figure 2, black curve) exhibits three lines at 396, 513 and 618 cm^{-1} characteristic of well crystallized anatase, and a sharp line at 999 cm^{-1} assigned to terminal Mo=O stretch vibration, $\nu(Mo{=}O_t)$. Upon CH_3OH/He flow, the intensity of the Raman spectrum rapidly and markedly decreases. The $\nu(Mo{=}O_t)$ mode is still detected (981 cm^{-1}) after 3 minutes of exposure, which is not the case anymore after 20 minutes on stream. In parallel, broad lines are detected around 1300 and 1600 cm^{-1} indicating surface poisoning by heavy carbon species in absence of oxygen. On the contrary, the 2800-2950 cm^{-1} range remains blank, indicating that the methanol and product fragments do not last durably at the surface of the active phase.

Changes along exposure to CH_3OH/He flow were also pointed out on the XANES profiles (Figure 3). The intensity of the pre-edge feature is rapidly and noticeably affected. Moreover, a visible shift of the absorption edge to lower energies is observed after 15 minutes at 275°C, which is relevantly amplified upon increasing the temperature to 300°C. The shape of the near-edge spectrum is obviously modified, suggesting a true change in the coordination of the molybdenum with a partial reduction of the molybdenum active sites under methanol flow[4].

Both spectroscopic techniques are indicative of rapid structural evolutions during the catalytic process and give complementary information. These results confirm the relevancy of this time-resolved, coupled *operando* analysis of catalysts. A comparison with other catalysts of different structures (e.g. TiO_2 supported oxomolybdate catalysts with a low molybdenum loading) should allow going one step further in the understanding of the structure-reactivity connections.

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Real-Time Monitoring of Thermally Induced *Trans* Fats in Corn Oil Using the FatIR™ Oil Analysis System

Figure 1. The FatIR™.

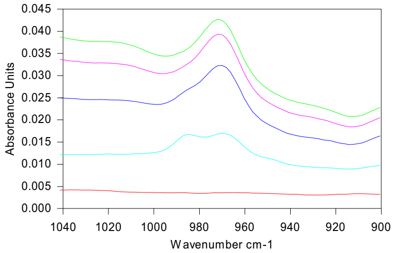

Figure 2. Real-time spectra in the *trans* double bond region of corn oil at 102°C after 5 min (red), 30 min (light blue), 70 min (blue), 115 min (pink), and 155 min (green).

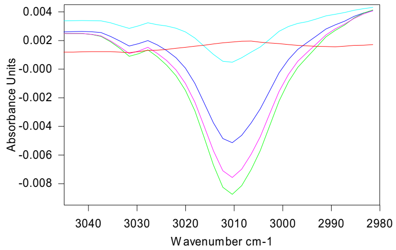

Figure 3. Real-time spectra in the *cis* double bond region of corn oil at 102°C after 5 min (red), 30 min (light blue), 70 min (blue), 115 min (pink), and 155 min (green).

INTRODUCTION

Trans fats (TFA) are unsaturated fatty acids whose carbon chains contain double bonds in the *trans* configuration rather than the more common *cis* configuration. Edible oils have only negligible amounts of TFA[1]. However, when exposed to heat the *cis* double bonds in oil may be isomerized to their *trans* configuration. The amount of thermal isomerization is a function of temperature, exposure time and degree of unsaturation of the oil[2]. In this application we monitored in real-time *in situ* the thermal isomerization of corn oil at a moderate temperature (102°C).

EXPERIMENTAL CONDITIONS

The spectra of the heated corn oil were collected *in situ* using the FatIR™ in an infrared spectrometer with a DTGS detector and conditions set for 32 scans at 4 cm^{-1} resolution. Corn oil was placed on the slightly preheated (30°C) crystal covering the entire sampling surface. The spectrum of corn oil at 30°C was collected. The temperature of the crystal was slowly raised from 30°C to 102°C over a 15 minute period. Spectra of the corn oil at 102°C were collected after 0, 5, 30, 70, 115 and 155 minutes. The crystal was allowed to cool to 30°C and the spectrum of the cooled corn oil was collected. The spectrum of corn oil at 102°C recorded at t = 0 minutes was subtracted from the spectra recorded after t = 5, 30, 70, 115 and 155 minutes.

RESULTS

Figure 2 illustrates the changes that take place in the *trans* double bond region (1050-900 cm^{-1}) of the spectrum due to prolonged exposure to moderate heat. There is a marked progressive rise in the 966 cm^{-1} *trans* band with time. In addition to the rise of the 966 cm^{-1} peak there is also an initial increase in the 985 cm^{-1} peak that disappears later on.

The increase of the *trans* band with time is paralleled with a decrease of the band at 3010 cm^{-1} associated with the CH stretching vibration of *cis* double bonds (Figure 3). By comparing the spectra of corn oil before heating (T = 30°C) and after cooling back to 30°C, in the regions of interest (Figures 4 and 5), we see that there are heat induced changes in the *trans* and *cis* regions. Specifically, there is an increase in the concentration of the *trans* isomers and simultaneous decrease in the concentration of the *cis* isomers.

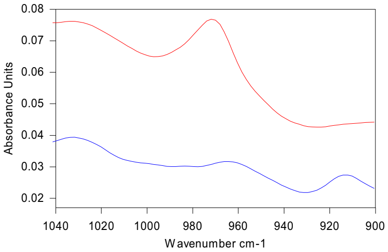

Figure 4. Real-time spectra in the *trans* double bond region of corn oil at 30°C before heating (blue), after cooling (red).

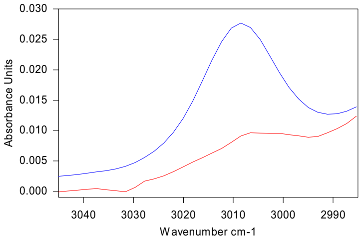

Figure 5. Real-time spectra in the *cis* bond region of corn oil at 30°C before heating (blue), after cooling (red).

CONCLUSION

The new FDA requirement that *trans* fats be included in the Nutrition Facts panel has created a need for the routine determination of the total *trans* fat content of edible oils and fats and for the monitoring of the formation of *trans* isomers during processing of vegetable oils.

Harrick Scientific's FatIR™ Oil Analysis System not only provides a fast and easy way for the routine determination of the total *trans* fat content in edible oils and fats, but also enables real-time *in situ* monitoring of the formation of heat induced TFA.

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[1] Masanari Semma, *Journal of Health Science*, **48(1)**, 7-13 (2002).

[2] F. R. van de Voort, A. A. Ismail, J. Sedman and G. Emo, *J. Am. Oil Chem. Soc.*, **71**, 243-253 (1994).

No. 31201

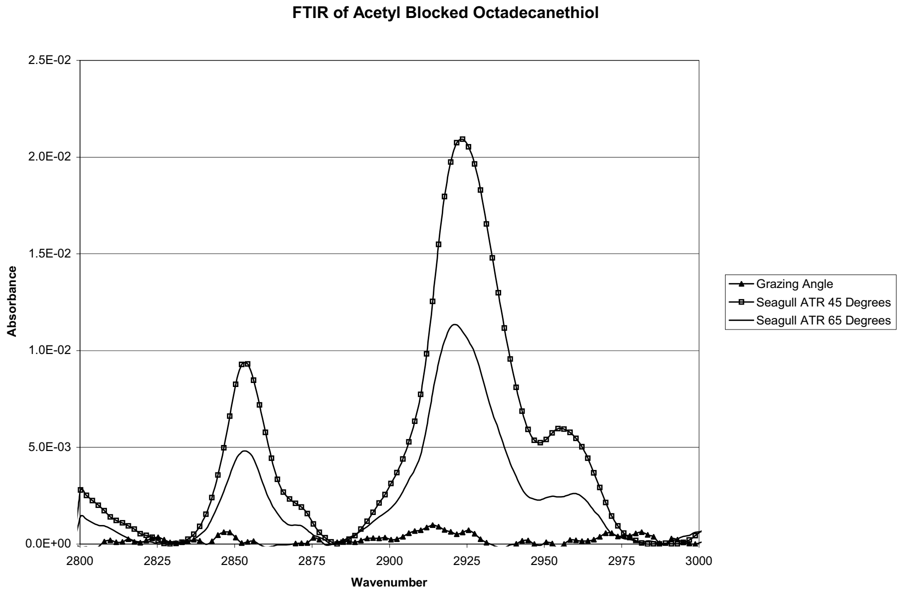
SELECTIVE ATTACHMENT OF MOLECULAR WIRES

The advancement of nanoscale technology depends on the ability to link nanostructures to the outside world in a precise and stable manner. Different techniques have been used to achieve this including bonding dithiolated molecules into break junctions,[1] contacting two monolayers together with a mercury drop electrode,[2] and most recently, chemically bonding metal contacts to the ends of a symmetric dithiol molecule.[3] As soon as more complicated molecular electronic components are created, for example a single-molecule field effect transistor, selective attachment of molecular wires to specific electrodes will be necessary. Since thiols have such a high affinity for gold, they are a good choice for chemically bonding molecular electronic components to a gold conductor. A blocking group mounted on the thiol head that could prevent its adsorption onto gold until the group is intentionally removed would be extremely valuable and could enable selective attachment.

The Seagull Variable Angle Reflectance Accessory.

Using octadecanethiol as a model, acetyl and benzoyl blocking groups have been found to inhibit adsorption of dialkyl thiols, but not completely prevent it. The general procedure for creating the alkanethiol self assembled monolayers (SAM) is straightforward, a gold substrate is submerged into a 1 mM solution of the desired thiol in THF overnight. The sample is then rinsed with excess THF and dried under argon before the quality of the SAM formed is assessed through ellipsometry and FTIR measurements.

Below are examples of FTIR measurements of SAM's formed from acetyl and benzoyl blocked octadecanethiol using two different techniques. The collection parameters for all three spectra are the same and it can be noted that the SAM's are barely visible using typical grazing angle reflectance. Using the Seagull with a Ge crystal though, the SAM's are easily detected with peaks around 2917 and 2850 cm^{-1} attributed to the CH_2 groups in the alkyl chain and peaks at 2965 and 2877 cm^{-1} attributed to the CH_3 group. The variable angle allows the optimization of the angle for the best sensitivity of the films that in this case is 65 degrees.



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[1] Reed, M. A. ; Zhou, C. ; Muller,C. J.; Burgin, T. P. ; Tour,J. M. *Science*, **1997**, 278, 252.

[2] Holmlin, R. et al., *J. Am. Chem. Soc.,* **2001**, 123, 5075.

[3] X. D. Cui, et at., *Science*, **2001**, 294, 571.

No. 40401

SPATIAL RESOLUTION OF MICRO-ATR INFRARED SPECTROSCOPY

Figure 1. The Video Meridain™.

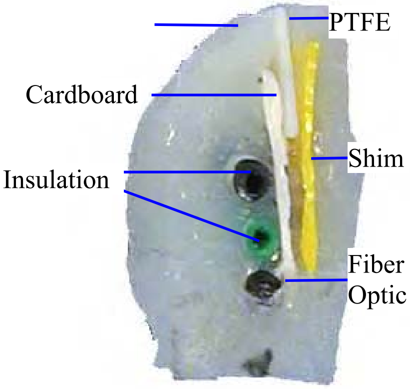

Figure 2. Epoxy Imbedding.

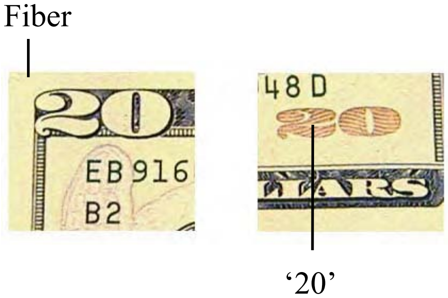

Figure 3. The two sections examined from the face-side of the new twenty-dollar bill.

INTRODUCTION

With the recent growth in single reflection ATR infrared spectroscopy, there has been a push to examine ever smaller samples or segments thereon. This method is limited by the ability to position the sample on the active sampling area of the ATR crystal and by the relative size of the sample to the sampling area. Two categories of ATR micro-samplers are currently available: microscope ATR objectives and in-compartment accessories. While the microscope ATR objectives offer higher spatial resolution, the required MCT detector restricts the spectral range. The in-compartment accessories offer greater spectral range.

This paper explores the spatial resolution of this technique, using a new micro-ATR in-compartment accessory with imaging capabilities. Several samples will be examined, including imbedded materials, inks on paper and fibers.

EXPERIMENTAL

The ATR spectra were recorded using an FT-IR spectrometer, configured for data collection at 8 cm^{-1} resolution and 32 scans. The spectrometer was equipped with Harrick Scientific's Video Meridian™ diamond ATR (Figure 1). Background spectra were collected from the clean ATR crystal and then the samples were compressed against the horizontal diamond ATR. The calibrated pressure applicator with digital read-out was used to apply 100 units of force. The spectra were then collected and the sample photographed using the built-in camera.

Several sections of two different samples were examined: an epoxy imbedding (Figure 2) and a new twenty-dollar bill (Figure 3). The imbedding was prepared by inserting several materials parallel to each other in Devcon® (ITW) 5-minute epoxy and then allowing the epoxy to dry thoroughly. The imbedding was then sliced to expose a clean surface of the materials. The materials imbedded included: 500 µm thick PTFE, 500 µm thick shim stock, cardboard (approx. 500µm thick), black and green insulation from 18 gauge multi-strand wire insulation and a fiber optic (Edmund Scientific P/N 2504).

RESULTS AND DISCUSSION

First, several samples of known thickness were examined to determined if they could be spatially separated from the background material in the embedding. For reference, the

Spatial Resolution of Micro-ATR Infrared Spectroscopy

spectrum of the epoxy imbedding material is shown in Figure 4. The spectrum has increased noise in the 2100 cm^{-1} region due to the strong absorption bands of the diamond ATR crystal.

Figure 5 demonstrates the ability to resolve 500 µm thick samples from the surrounding material. No features due to the epoxy are present in either spectrum. Figure 5 shows the spectrum of PTFE, as expected, and the shim is readily identifiable as polyester. The photographs in 5 also clearly delineate the edges of the samples, reasserting the fact that the octagonal sampling area of the ATR crystal is 500 µm in diameter.

Several materials of unknown thickness and/or composition were then investigated within the imbedding. Figure 6 is the ATR spectrum of a piece of thin cardboard. It is interesting to note that the cardboard spectrum is clearly that of cellulose. There is no observable contribution from the epoxy, despite the fact that cardboard is somewhat porous.

Figure 7 shows the spectra of two different colors of wire insulation. The photograph of the green insulation shows the curved shape of the sample. The edges of this sample are also well defined, indicating that the wall thickness of this insulation is roughly that of the sampling area. Both spectra are distinct from that of the imbedding material.

Figure 8 contains spectra and photographs of a fiber optic. From this data, the black sheathing has a wall thickness of roughly 500 µm and is composed of a low density polyethylene, while the individual fibers are approx. 250µm in diameter and made from poly(methyl methacrylate).

Having determined that it is possible to spatially resolve a carefully formulated sample, a readily available sample was also examined – a new twenty-dollar bill. Figure 9 shows the spectra of two sections of the golden ‘2’ on the face side of the bill. The photographs show that this section is actually composed of red and gold stripes, roughly 500 µm in width. The spectra of both stripes are similar, but the bands in the ATR spectrum of the golden stripe are lower in intensity. This would be expected if the golden coating is actually a thin layer of gold, since the gold would mask the spectrum of the underlying paper.

Figure 10 show the ATR spectra from two sections of the bill. One section has a noticeable blue fiber in it that, from the photograph, is approximately 65 µm in diameter. The other was more uniform and examined as a

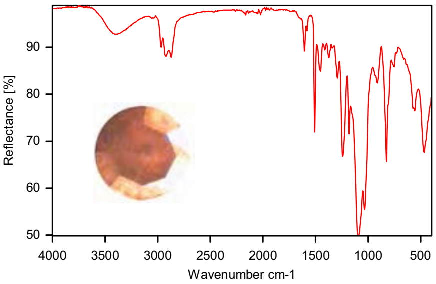

Figure 4. ATR Spectrum of Epoxy.

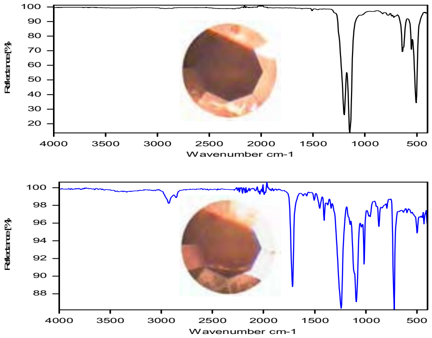

Figure 5. ATR Spectrum of 500 µm thick PTFE (upper) and Shim (lower).

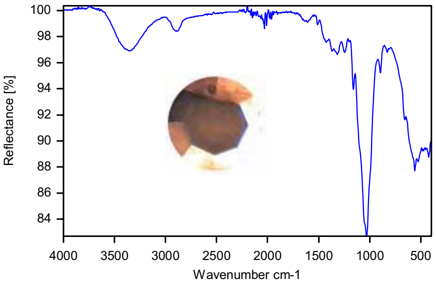

Figure 6. ATR Spectra of ~500µm thick Cardboard.

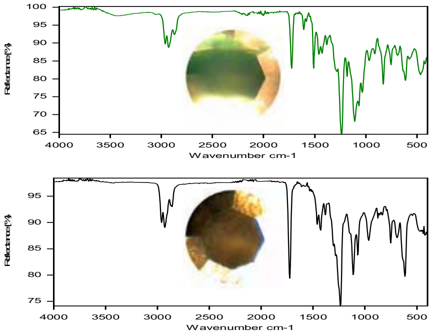

Figure 7. ATR Spectra of Green (upper) and Black (lower) Insulation.

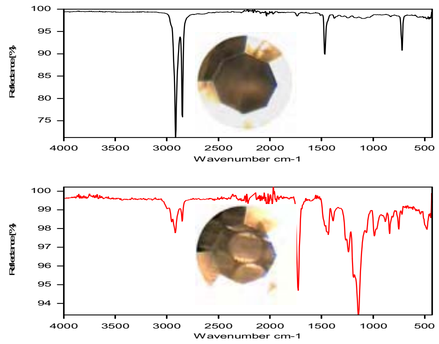

Figure 8. ATR Spectra of a Fiber Optic: Sheathing (upper) and Interior (lower).

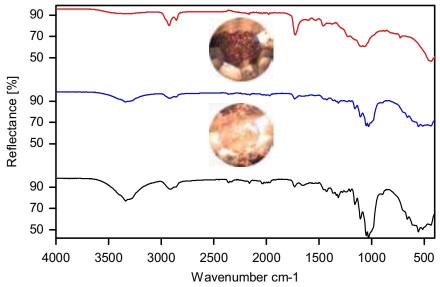

Figure 9. ATR Spectra of the shiny “20” on the face side of the bill: red section (upper), gold section (middle) and an unprinted section (lower).

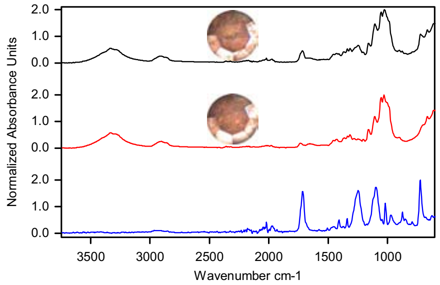

Figure 10. Normalized ATR Spectra of a blue fiber (top) on the greenback, an area near that blue fiber (middle) and the difference (bottom).

reference. The bands at 1715 cm^{-1} and 1250 cm^{-1} appear in the spectrum of the fiber but not in the spectrum of paper. Subtraction results in a spectrum that is clearly identifiable as polyester.

CONCLUSION

With the use of the Video Meridian™ micro-ATR accessory, it is straightforward to spatially resolve samples such as imbedded materials, inks on paper and fibers. In fact:

- 500µm-wide sections of materials can be readily resolved spatially from bulk substrates.

- Smaller segments can also be resolved through judicious use of spectral subtraction methods.

No. 40608

THE EFFECT OF SURFACE UNIFORMITY ON THE SPECULAR REFLECTANCE FROM CARBON-FILLED COATINGS

Figure 1. The Seagull™.

Figure 2. The Video Meridain™.

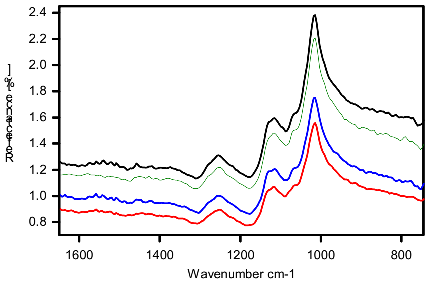

Figure 3. The 60º specular reflectance from two samples recorded with the Seagull.

INTRODUCTION

Carbon-filled coatings are frequently applied to materials to reduce their infrared and visible reflectivity. Specular reflectance is frequently the method of choice for examining the reflectivity of these coatings. However, these coatings are not always smooth and this can produce anomalies that complicate interpretation of the results.

This applications note demonstrates the problem using a routine method used to check the coatings and then explores the effect of coating texture and uniformity on the results.

EXPERIMENTAL

The samples examined were pieces of sheet metal coated by Duracon™ Optical Black Coating from American IMEX Corporation. Several samples were prepared in a similar fashion and then used for this study.

These samples are routinely examined using the Seagull™ variable angle reflectance accessory (Figure 1) that utilizes a 1:1 imaging system. The Seagull™ was set for its specular reflectance mode at an incident angle of 60º.

For a more detailed inspection of the surface, the VideoMeridian™ microsampler (Figure 2) was used. The VideoMeridian™ features a six times beam condenser and a fixed 45º incident angle. It was configured for specular reflectance and its built-in video camera was adjusted so the sample was in focus. The sample was randomly scanned and digital images were acquired, using the MicroSnap™ software, for each position along with the spectra.

Both the Seagull™ and the VideoMeridian™ were used in conjunction with a commercial FT-IR spectrometer and the data was collected over the mid-infrared region at 8 cm^{-1} resolution and signal averaged over 32 scans.

RESULTS AND DISCUSSION

The results from the routine analysis of two samples using the Seagull™ are shown in Figure 3. Two measurements were taken from each sample and the sample was repositioned between measurements. While it is clear that little of the incident radiation is reflected, the reflectance does differ as a function of the sample and its position. This difference is evident both in the base level of reflectivity and in the peak intensity.

The Effect of Surface Uniformity on the Specular Reflectance from Carbon-Filled Coatings

Since the samples were prepared in the same fashion and the samples have some texture, it is possible that the coating is not uniform in either texture or coverage. This non-uniformity could be altering the reflectance from the surface.

To examine this phenomenon in more detail, the surface of one of the samples was randomly scanned and examined using the VideoMeridian™. The resulting spectra are shown in Figure 4 and the corresponding images are presented in Table 1.

Comparison of the band intensities in Figures 3 and 4 shows that the magnitude of the intensity fluctuations is approximately six times smaller with the Seagull than with the VideoMeridian™. This is expected given the differences in the sampled areas between the two accessories.

From Table 1, it is clear that the samples show differences in overall texture. In addition, several sections (d and f in Figure 4) appear to have micro-holes in the coating. The differences in the overall brightness of the visual image are loosely correlated to the band intensity differences shown in Figure 4. It is possible that a direct correlation between the sample texture and the infrared band intensity could be drawn if infrared illuminated portion of the visual image could be distinctly analyzed.

However, the 'holes' in the coating may cause non-linear effects. Since the spectrum is significantly different for a thin film. Figure 5 shows the spectrum of a corner of the sample which is only partly coated. This spectrum shows the transmission-like characteristics of a thin film on a metallic substrate rather than the dispersive characteristics typical of specular reflectance.

CONCLUSION

As seen from the data presented herein, the Video Meridian™ microsampler can be used to examine surface inhomogeneities that are a few millimeters in size both visually and by infrared spectroscopy. Infrared spectroscopy effectively integrates over the illuminated area. However, the effect of surface inhomogeneities can be detected in the resulting spectra for both small and large sampling areas.

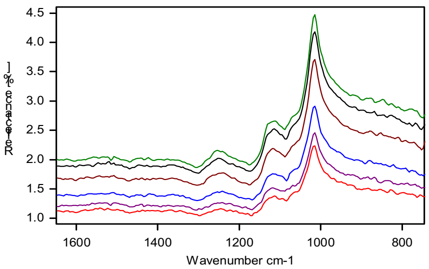

Figure 4. The 45° specular reflectance of sampling positions a through f (top to bottom) recorded with the VideoMeridian™.

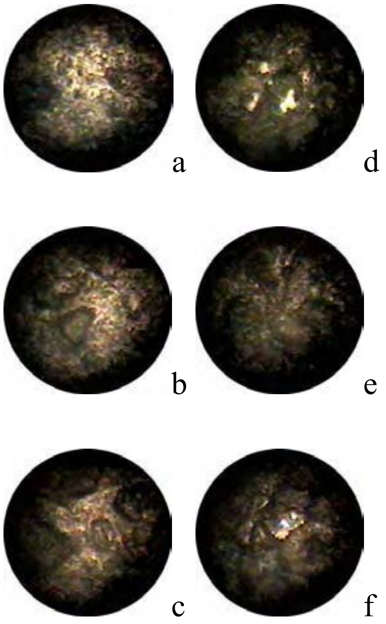

Table 1. Photographs of the sections examined in Figure 4.

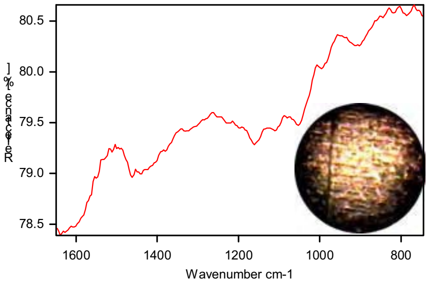

Figure 5. The 45° specular reflectance and photograph of a lightly coated section of the sample.

No. 50107

THE COLOR OF MONEY: A DIAMOND ATR STUDY OF THE NEW TWENTY DOLLAR BILL

Figure 1. The Video Meridian Diamond ATR Accessory.

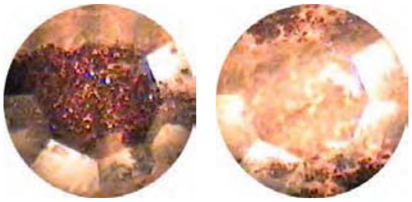

Figure 2. Photographs of the red (left) and gold (right) sections of the bill.

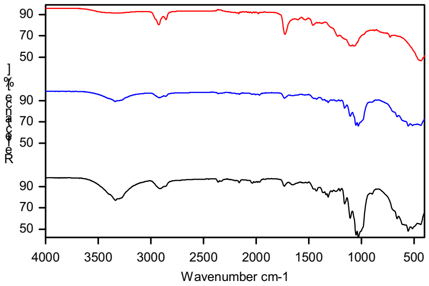

Figure 3. ATR spectra of the shiny "20" on the face side of the bill: red section (upper), gold section (middle) and an unprinted section (lower).

INTRODUCTION

The year 2003 presented big changes for the greenback - the United States Mint introduced a new, multicolored twenty-dollar bill. This new design is intended to make forgery more difficult and detection of illegal bills easier by incorporating a greater variety of colored inks and textual details.

Detection of inks and dyes on substrates, like the greenback, is vital for forensics investigations and for the textiles and printing industries. For forensics, matching inks on papers and cloth from crime scenes to those used by suspects is vital in the investigation process. For the textiles and printing industries, colors and coating uniformity must be reasonably consistent from one lot to the next.

This applications note explores the use of video-imaging micro-ATR (µATR) to distinguish differences in chemical composition of various portions of the new twenty-dollar bill.

EXPERIMENTAL

The sample twenty-dollar bill was obtained directly from a local bank, in an attempt to obtain one with as few surface contaminants as possible. The bill selected was crisp and lacked winkles or folds, indicating that it was recently placed in circulation.

The sample was examined using the Video Meridian, a single reflection µATR accessory using a type IIA diamond, installed in the sample compartment of a commercial FT-IR spectrometer. All spectra were collected with 32 scans at 8 cm^{-1} resolution using a DTGS detector. The sample was compressed against the 500µm diameter sampling area of the diamond ATR crystal using the same force and then referenced against the clean ATR crystal. The sample was positioned using the digital image displayed on the computer screen and the image was captured for each section of the bill examined.

RESULTS AND DISCUSSION

Figures 2 and 3 show the spectra recorded from two different sections of the golden "20" on Jackson's side of the bill. Figure 2 shows that there are two visually different parts of the "20" – one which is red and the other golden. The golden area just fills the octagonal sampling area of the ATR crystal, indicating that it is approximately 500 µm wide. The red section to its left appears somewhat wider, since its edges are not apparent. The spectra from these two sections are shown in Figure 3, along with the spectrum of an unprinted section of the bill. All three spectra show a band at 1100 cm^{-1}. This band is primarily due to the underlying paper since the ink coating does not completely cover the paper. The red portion has a significantly stronger carbonyl band at 1730 cm^{-1}, in addition to stronger bands in the C-H region. The spectrum of the golden section is nearly identical to that of the underlying paper, with significantly less intense bands. This masking effect is typically seen with extremely thin metallic coatings (<<10Å). Hence it is likely that the golden color on the bill is, indeed, a very thin gold coating.

Figures 4 and 5 demonstrate the ability to distinguish different colors of green. The top two spectra show bands at 1000 cm^{-1}, probably due to the paper substrate. All three spectra are slightly different, indicating the possibility that three different kinds of green ink or dye were used. The spectrum of the serial number has bands around 1400 cm^{-1}, indicating that the ink may contain an aromatic component, while that of the shield has a band near 1170 cm^{-1}, indicating a C-O bond. The spectrum of the zero shows both components, so it may be different ink entirely or a mixture of the two.

In addition to inks and coatings of various kinds, the paper itself has discernible fibers in it. Figures 6 and 7 show two sections from the edge of the greenback. One section has a noticeable blue fiber in it, which is approximately 65µm in diameter. The other was more uniform and was examined for a reference. The bands at 1715 cm^{-1} and 1250 cm^{-1} appear in the spectrum of the fiber but not in the spectrum of paper. Subtraction of the paper background spectrum from the fiber spectrum results in a spectrum that is clearly identifiable as polyester.

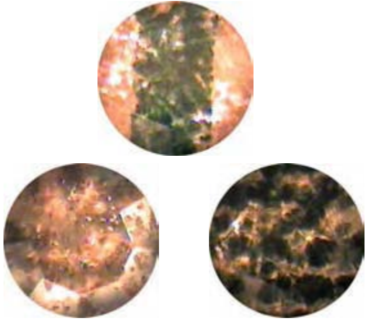

Figure 4. Photographs of the three green sections sampled: the serial number (top), the shield (lower left), and the zero (lower right).

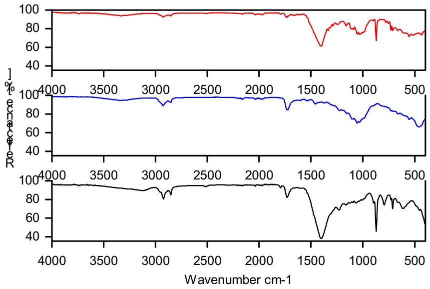

Figure 5. ATR spectra of green printing on the new twenty-dollar bill taken from the serial number near Jackson (top), the shield next to Jackson (middle), and the large zero on the lower left below the White House (bottom).

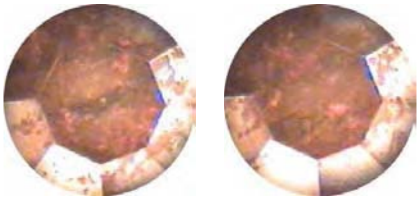

Figure 6. Photographs of the blue fiber (left) and substrate (right).

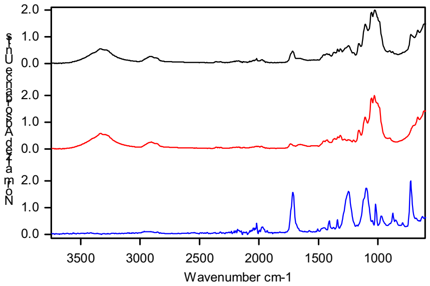

Figure 7. Normalized ATR Spectra of a blue fiber (top) on the greenback, an area near that blue fiber (middle), and the difference (bottom).

SUMMARY

As shown above, µATR, combined with video imaging, is a powerful tool for examining differences in chemical composition of fine details on substrates. Such samples are routinely examined in the forensics and textiles forensics industries; and the Video Meridian provides the tool to obtain detailed information quickly and easily.

No. 50309

DETECTION OF THIN FILMS ON POLYCARBONATE AND OTHER PLASTIC SUBSTRATES

Figure 1. The GATR™ Accessory: 65º ATR with Ge crystal.

Figure 2. The FastIR™ Accessory: 45º ATR with ZnSe crystal.

INTRODUCTION

Thin organic coatings or monolayers on semiconductor and metallic substrates are straightforward to detect by FTIR spectroscopy, using either ATR methods[1-5] or grazing angle specular reflectance[6-10]. It is much more difficult to detect such coatings on plastics, because the refractive indices of the two materials are so closely matched. However, detecting these films on plastics, such as polycarbonates, is becoming increasingly important as the industry continues to expand.

Polycarbonates and other plastics are widely used as an alternative for glass in products such as optical lenses, medical devices and electronics equipment. Plastics offer many advantages. They are lighter than glass and do not shatter, in addition to being easy to color, mold and recycle. However, polycarbonates and other plastics weather poorly, scratch easily and are permeable to some gases. To circumvent these limitations, polycarbonate and plastic substrates frequently are coated. These coatings include anti-reflection, anti-abrasion and chemically resistant films. Some of these coatings are extremely thin and not readily detected by standard ATR spectroscopy methods.

This paper explores the use of high incident-angle Ge-ATR to detect these thin coatings on polycarbonates and other plastic substrates.

THEORY

In ATR, the sample must be brought into intimate contact with the optical element, where light is totally internally reflected and the sample interacts with the evanescent wave. This interaction is completely described by the Fresnel equations[11,12]. These equations are complex and not particularly insightful. But they do derive the reflectance as a function of the incident angle, incident polarization, and complex refractive indices of the sample and ATR crystal.

In the low absorption limit, the degree of interaction can be measured by an effective pathlength (d_e). This pathlength is defined as the sample thickness which will gives the same absorption as in transmission. For thick films, the effective pathlengths[11] for the two polarizations are:

$$\frac{d_{e\perp}}{\lambda_1} = \frac{n_{21}\cos\theta}{\pi(1-n_{21}^2)(\sin^2\theta - n_{21}^2)^{1/2}}$$

$$\frac{d_{e\parallel}}{\lambda_1} = \frac{n_{21}\cos\theta(2\sin^2\theta - n_{21}^2)}{\pi(1-n_{21}^2)[(1+n_{21}^2)\sin^2\theta - n_{21}^2](\sin^2\theta - n_{21}^2)^{1/2}}$$

where λ_1 is the wavelength, θ is the incident angle, and n_{21} is the refractive index of the sample relative to that of the ATR crystal.

Since the effective thickness is a function of the incident angle and refractive index of the ATR crystal, by using a high refractive index crystal, like Ge, and a high incident angle, it should be possible to distinguish more of the surface characteristics of the sample.

Table 1 shows the effective thicknesses calculated[13] for the GATR™, in comparison to a typical ATR accessory.

	Incident Angle	ATR Crystal	$d_{e\perp}$ (μm)	$d_{e\parallel}$ (μm)
GATR	65°	Ge	0.090	0.168
FastIR	45°	ZnSe	0.205	0.410

Table 1. Differences in Effective Thicknesses at 2000 cm^{-1} for the Two Accessories Used Herein.

EXPERIMENTAL

The ATR spectra were recorded using a Nicolet Nexus 670 spectrometer, configured for data collection at 8 cm^{-1} resolution and 32 scans. The Nexus was equipped with Harrick Scientific Products' GATR™ Thin Film Analyzer (Figure 1) or the FastIR™ (Figure 2) ATR accessories. The GATR™, with its 65° incident angle and Ge ATR crystal, has a lower effective pathlength than traditional horizontal ATR accessories, like the FastIR™, with its 45° incident angle and ZnSe crystal. This gives the GATR™ higher sensitivity to species on the surface of a material.

Background spectra were collected from the clean ATR crystal. Then the samples were compressed against the crystal and the sample spectra collected.

Two different sets of samples were examined: polycarbonate samples and eyeglass lenses. The polycarbonate samples included an uncoated reference and a sample with an unknown coating. The eyeglass lenses were all plastic lenses, possibly also polycarbonates, with an abrasion-resistant coating. An additional antireflective coating may also have been present on some of the lenses.

RESULTS AND DISCUSSION

The two known polycarbonate samples were examined first, to determine if coatings on plastics could be distinguished from the substrate using high incident-angle Ge ATR under ideal circumstances. Figures 3 and 4 show the spectra of the uncoated and coated samples, respectively. Figure 3 is readily identifiable as a polycarbonate, as expected. Figure 4 shows bands that are clearly not from the polycarbonate. In particular, the broad band from 3600 cm^{-1} to 2500 cm^{-1} in the coating does not appear in the substrate and it indicates that the coating is likely to be a carboxylic acid or derivative.

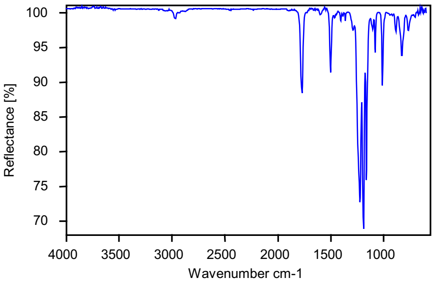

Figure 3. ATR Spectrum of Uncoated Polycarbonate, Recorded with the GATR™.

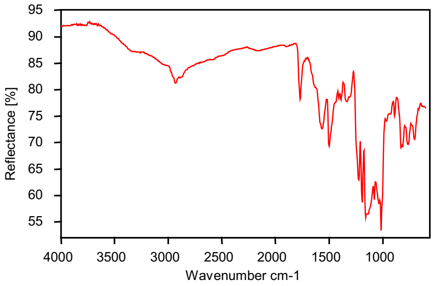

Figure 4. ATR Spectrum of Coated Polycarbonate, Recorded with the GATR™.

Next, the eyeglass lenses were examined. Since the lens substrates were unknown, spectra were recorded using both the FastIR™ and the GATR™. Since, for the same sample, the GATR™ has a lower effective thickness than the FastIR™ (see Table 1), the GATR™ should sample a greater proportion of the coating relative to the substrate. Comparison of the two spectra should reveal differences between the coating and the substrate.

Figure 5 shows a much narrower band shape for the band around 1100 cm^{-1} in the spectrum recorded with the GATR. In addition, this spectrum shows significantly reduced band intensities in the –OH and –CH region. This would be consistent with a thin metal oxide surface coating. The –OH and –CH bands seen in the spectrum of the lens are probably from the substrate or underlying layer, where their band intensity would be masked by the metal oxide overcoat.

Figures 6 and 7 show spectra from two other lenses. Both Figures show only small differences between the bulk (FastIR) and surface (GATR) spectra, making it more difficult to access whether the differences are due to the coating or to the variations in response between the two sampling methods.

Figure 6 shows a slightly stronger band at 1111 cm^{-1} in the lower spectrum relative to the band at 1020 cm^{-1}. In addition, the bands in the –OH and –CH region are also less intense in the lower spectrum. This may indicate that the band at 1111 cm^{-1} is from the coating and the –OH and –CH bands are in the substrate. The spectra here are similar to that from Figure 5. Both samples are coated with the same material, but the coating is thinner for sample B.

Figure 7 includes spectra from yet a different lens. Here, the lens substrate or under-coating is distinctly different from that shown previously. There is a slight difference in relative band intensities in the fingerprint region for the two spectra recorded, possibly indicating differences in composition between the coating and bulk material.

SUMMARY

With the use of high incident-angle Ge-ATR, thin coatings on polycarbonates and other plastic substrates can be detected. The effectiveness of this method depends on the thickness of the coating. For coatings with thicknesses significantly smaller than the effective thickness, it may be necessary to make measurements at two incident angles, one high and one near critical, to extract information regarding the coating composition. Such measurements could be carried out using a variable angle ATR accessory like the Seagull™.

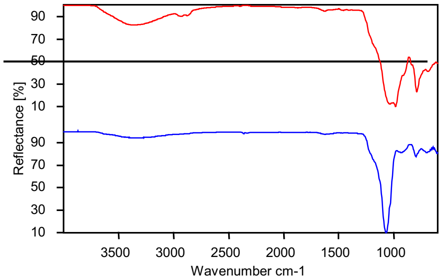

Figure 5. The ATR spectra of Eyeglass Lens A, Recorded with the FastIR™ (top) and GATR™ (bottom).

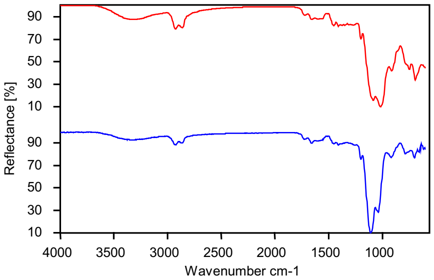

Figure 6. The Normalized ATR spectra of Eyeglass Lens B, Recorded with the FastIR™ (top) and GATR™ (bottom).

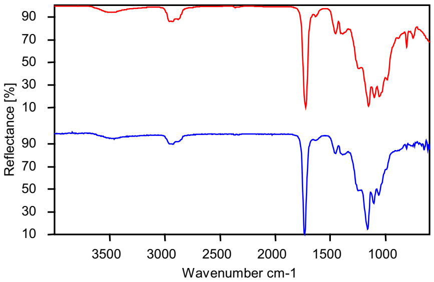

Figure 7. The ATR spectra of an Eyeglass Lens C, Recorded with the FastIR™ (top) and GATR™ (bottom).

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[13] Using Harrick Scientific's CristalCalc™.

No. 070101

ANALYSIS OF POLYMER BRUSH FORMATION ON SI WAFERS BY GATR-FTIR

Misty D. Konopacki and Stephen G. Boyes
Department of Chemistry and Geochemistry, Colorado School of Mines, Golden, CO 80401

Figure 1. GATR Ge-ATR Accessory.

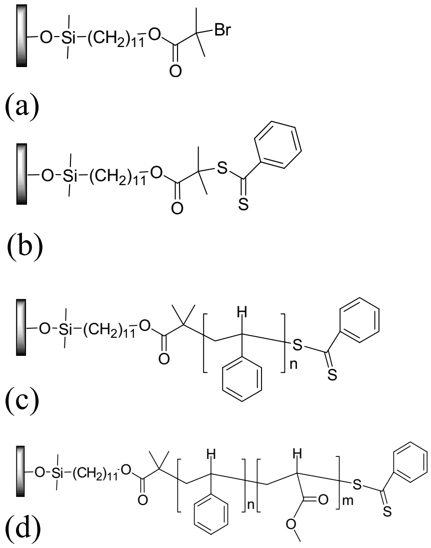

Figure 2. Reaction Sequence for Formation of a Diblock Copolymer Brush on Flat Si Wafers.

INTRODUCTION

Polymer brush systems consist of ordered assemblies of polymeric chains that are terminally grafted or absorbed onto a surface or interface at one or more tethering points.[1] Interest in the formation of these surface immobilized thin films has increased over the last few years due to their potential use in a wide variety of applications including biomedical devices, microelectronics, tailoring of surface properties, nano-patterning, thermo-responsive adhesives, and controlled gene/drug delivery.[1-3] The formation of polymer brushes from flat silica surfaces, often requires multiple reactions at the surface to effectively functionalize the silicon dioxide layer with active sites and for the subsequent polymerization from these active sites.

Grazing angle attenuated total reflectance (GATR)-Fourier transform infrared (FTIR) spectroscopy is especially advantageous over other FTIR techniques in that it allows for direct spectra collection from silica wafer substrates without destruction of the surface functionality or need for an ideal substrate, such as a silicon ATR crystal (Figure 1). This application note examines the utilization of GATR-FTIR to distinguish surface modifications of silica wafers with thin films and polymer brushes. This method requires optical contact between the sample and the ATR crystal for good sensitivity.

EXPERIMENTAL

To remove any surface contaminates from the Czochraclaki-grown <111> Si wafers, all substrates were first cleaned in a 30:70 *v/v* hydrogen peroxide and concentrated sulfuric acid solution (*Caution, caustic!*). Next, an 11-carbon tertiary bromo-isobutyrate initiator was deposited in an anhydrous toluene solution onto the cleaned Si wafers (Figure 2a). Wafers were then cleaned sequentially with toluene, methanol, and methylene chloride. GATR- FTIR spectra were collected at a resolution of 8 cm^{-1}, with 64 background scans, and 256 sample scans. Samples with previously deposited bromo-initiator were then subjected to a modified atom transfer addition (ATA) reaction to convert the terminal bromine to a dithioester end-group (Figure 2b)[4]. Wafers were cleaned and spectra were collected, as before. Finally, homopolymer and diblock copolymer brushes of poly(styrene) (PSty) and PSty-*b*-poly(methyl acrylate) PMA were formed from the dithioester moieties (Figure 2c and 2d, respectively). GATR-FTIR spectra were collected, as before, after samples were cleaned.

RESULTS AND DISCUSSION

The GATR-FTIR spectrum of the immobilized bromo-initiator (Figure 3a) shows peaks at approximately 2850 and 2930 cm^{-1} which are assigned to the CH$_2$ stretching and the C-H stretching vibrations, respectively, and at approximately 1740 cm^{-1}, which is assigned to the carbonyl stretching vibration of the ester group. Once the bromo silane initiator was deposited and characterized, a modified ATA reaction was carried out to convert the terminal bromine to a dithioester end group (Figure 2b). The GATR-FTIR spectrum of the sample after reaction with a dithioester containing compound (Figure 3b) indicates few discernable differences to that of the immobilized bromo-silane initiator spectrum. We attribute this to the relatively weak intensity of aromatic C-H and C-C stretches, especially when only one aromatic ring is present per immobilized molecule, and to the fact that the C=S stretching vibration appears in the finger print region. This spectral region is subject to large variation in the GATR-FTIR of silicon wafers due to the strong absorbance of the native silicon dioxide and lattice bands.

To determine the efficiency of the immobilized dithioester surface towards surface initiated polymerizations, a PSty homopolymer brush was synthesized (Figure 2c). The GATR-FTIR spectra for the PSty homopolymer brush (Figure 4) confirmed the presence of PSty due to the expected appearance of aromatic C-H stretching around 3100 cm^{-1} and C=C aromatic doublets at 1420-1480 cm^{-1}. Using a homopolymer brush of PSty, a PSty-*b*-PMA diblock copolymer brush was formed (Figure 2d). The GATR-FTIR spectra confirmed the addition of MA in the formation of the PSty-*b*-PMA diblock copolymer brush (Figure 5) due to the appearance of a carbonyl stretch at 1720 cm^{-1} and an increase in the CH$_2$ stretch at approximately 2920 cm^{-1}.

Along with GATR-FTIR spectroscopy, each Si wafer system was characterized by ellipsometry, goniometry, and x-ray photoelectron spectroscopy. Ellipsometry was used to determine thicknesses, before and after GATR-FTIR collection, given that some degree of force is required for good contact between the ATR crystal and the treated wafer.

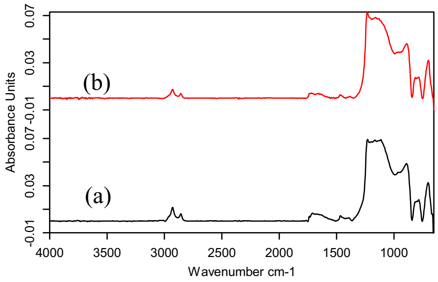

Figure 3. GATR-FTIR of surface immobilized (a) 11-C tertiary initiator and (b) dithioester surface structure.

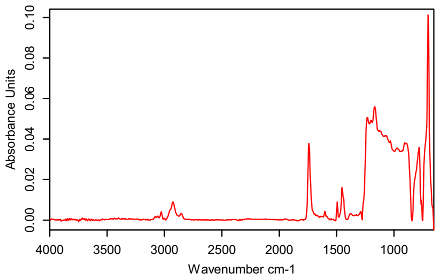

Figure 5. GATR-FTIR of PSty-b-PMA diblock copolymer brush.

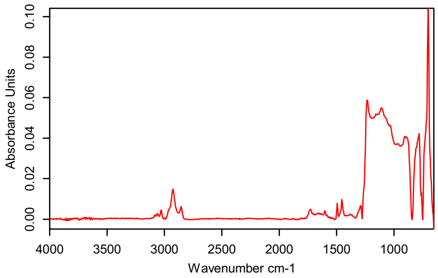

Figure 4. GATR-FTIR of PSty homopolymer brush.

ANALYSIS OF POLYMER BRUSH FORMATION ON SI WAFERS BY GATR-FTIR

Since changes in thickness were apparent for these samples, a sample wafer at each modification step was kept strictly for spectral collection and not for thickness measurements.

CONCLUSIONS

Overall the GATR Ge-FTIR accessory has proven useful in spectral elucidation of films whose characteristic stretches are apparent in the 1300-3300cm^{-1} spectral range. However, the native silicon oxide stretches of the Si wafer mask other peaks in the fingerprint region, which could be avoided by the use of float-zone Si wafers. The GATR-FTIR method is essentially non-destructive, but the high contact force required does influence the thickness of these polymer brush structures.

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HARRICK SCIENTIFIC PRODUCTS

No. 70124

MID-INFRARED FIBER OPTICS FOR IN-SITU STUDIES OF HUMAN SKIN

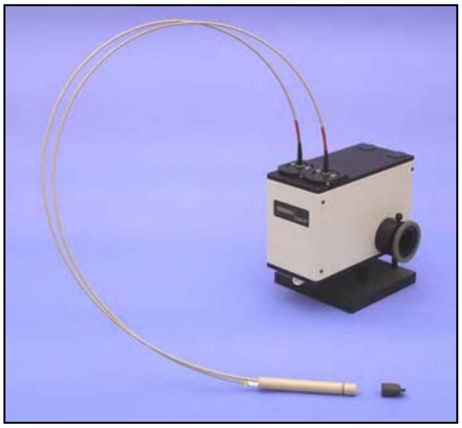

Figure 1. The FiberMate fiber optic coupler shown with the MultiLoop-MIR fiber optic probe.

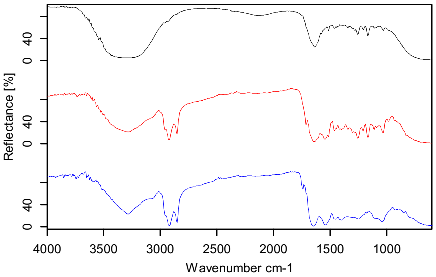

Figure 2. Fiber loop ATR spectra of NoAd sunscreen (black), NoAd sunscreen on skin (red) and clean skin (blue).

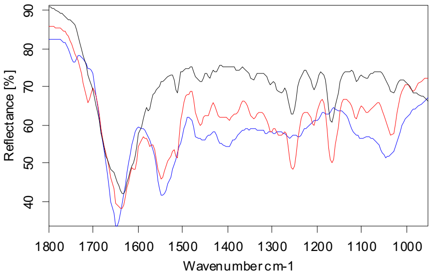

Figure 3. Fiber Loop ATR spectra of NoAd sunscreen RFD 15 (black), NoAd sunscreen on skin (red) and clean skin (blue).

INTRODUCTION

Mid-infrared ATR spectroscopy is widely used to study human skin. Applications include detection of cancerous tissue and, drug absorption studies. These studies are generally done with in-compartment ATR accessories and the sample must be brought in contact with the ATR crystal within the spectrometer sample compartment. More recently, *in-situ* skin analysis by ATR has been extended outside the box using mid-infrared fiber optics; however, the fiber optics used required a high sensitivity MCT detector.

A new high-efficiency fiber optic coupler and state-of-the-art fiber optics now enable out-of-compartment *in-situ* skin studies using a standard DTGS detector.

EXPERIMENTAL

The ATR measurements were recorded using Harrick Scientific's MultiLoop MIR probes with FiberMate™ fiber optic coupler installed in an FTIR spectrometer with a DTGS detector. The probe tips were pressed gently into the skin of a human subject during data collection. Spectra were collected at 8 cm^{-1} resolution with 32 scans signal averaged. Note that the Multiloop-MIR™ includes both a chalcogenide fiber for operation from 6500 to 1700 cm^{-1} and a silver halide fiber for operation from 2000 to 600 cm^{-1}. Spectra were recorded using both fibers and then merged to produce the full-range mid-infrared spectra as shown here.

RESULTS AND DISCUSSION

The ATR spectra of clean skin, NoAd Suncreen®[1] (SFP15), and sunscreen rubbed into the skin are shown in Figure 2. It is clear that the spectrum of the sunscreen-coated skin shows characteristics from both the underlying skin and the sunscreen. These changes are evident in the OH stretching and fingerprint regions (see Figure 3). The width of the OH band for the sunscreen-coated skin is slightly wider than that of skin and slightly narrower than that of the sunscreen alone.

The sunscreen-coated skin spectrum in Figure 3 clearly resolves bands at 1513, 1255, 1166, and 1031 cm^{-1} that are present in the sunscreen but not in skin.

CONCLUSION

The FiberMate fiber optic coupler and Multiloop-MIR

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[1] NoAd is a registered trademark of Solar Cosmetic Labs, Inc.

fiber optic probes provide a highly efficient and sensitive fiber optic accessory, enabling *in-situ* out-of-compartment studies of samples such as human skin using a standard DTGS detector.

USING FIBER OPTICS FOR MID-INFRARED SPECTROSCOPY WITH A DTGS DETECTOR

Figure 1. The FiberMate.

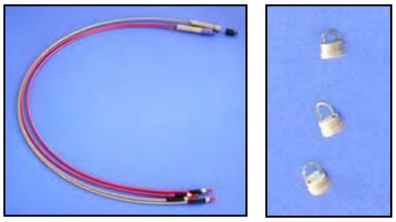

Figure 2. The MultiLoop-MIR fiber probes (left) and sampling tips (right).

Figure 3. AccessATR single reflection ATR accessory.

INTRODUCTION

Mid-infrared fiber optics systems have been commercially available since the early 1990s[1] and are becoming a valuable tool for remote sampling and in-situ studies[2]. Unlike visible and near-IR fiber optic systems, however, these systems are not in common use. The low throughput of the fibers typically required a nitrogen-cooled detector and the fragile nature of the appropriate fibers limit their potential applications.

A new mid-infrared fiber optic system has been recently developed to surmount these limitations. This system includes a high-efficiency FT-IR fiber optic coupler and an ATR fiber optic probe with disposable sampling tips for use over the entire mid-IR.

The paper examines the sensitivity of this new fiber optic system in an FT-IR spectrometer with a DTGS detector. Specifically, the number of reflections for the fiber loop tip will be determined and differences between the tips explored. The sensitivity of the system will also be examined by looking at several samples, including liquids and pastes.

EXPERIMENTAL

All spectra were recorded using a Thermo Nexus 670 spectrometer with a DTGS detector configured for data collection at 8 cm^{-1} resolution.

For the fiber optic measurements, the spectrometer was equipped with Harrick Scientific Products' FiberMate™ fiber optic coupler (Figure 1) and the MultiLoop-MIR™,[3] infrared fiber optic probes (Figure 2). The MultiLoop-MIR™ system consists of two fiber optic probe extensions and disposable loop ATR sampling tips covering the mid-infrared. One of the probes is made from chalogenide (CIR) designed for use from 4000 cm^{-1} to 1700 cm^{-1} and the other is a silver halide composite (PIR) for operation from 2000 cm^{-1} to 600 cm^{-1}. The tips are also made of silver halides. Data collected with the FiberMate™ and MultiLoop-MIR™ installed in the FTIR spectrometer were signal averaged over 64 scans.

For comparison, traditional ATR measurements were recorded using the Harrick AccessATR™, a 45° single reflection ATR accessory with a ZnSe triangular prism ATR crystal (Figure 3). Infrared spectral data collected with the AccessATR™ were averaged over 32 scans.

Background spectra were collected from the clean fiber loop or ATR crystal. Then the samples were brought into intimate contact with the sample. Solid samples were compressed gently against the fiber optic loop. The data collected with the loop were then baseline corrected and joined together to show the full spectral range.

Several different samples were examined: paraffin oil, red grape products and toothpaste. The red grape products included Carlo Rossi Paisano red wine, Progresso Red Wine Vinegar, and Kedem Grape Juice. The toothpastes were Procter & Gamble's Crest® Kid's Sparkle Fun Flavor and Cinnamon Rush Whitening Expressions, Colgate-Palmolive's Colgate® Regular, and Glaxo-SmithKline's Sensodyne®.

THEORETICAL DISCUSSION

In ATR spectroscopy, a material is brought into intimate contact with the optical element, where light is totally internally reflected and the sample interacts with the evanescent wave. This interaction is completely described by the Fresnel equations[4,5]. These equations are complex and not particularly intuitive. Most importantly, they relate the reflectance to the incident angle, the incident polarization, and the complex refractive indices of the sample and ATR element.

For ATR, the reflectivity, R, of a bulk material can be expressed as:

$$R=1-\alpha\, d_e \qquad (1)$$

where α is the absorption coefficient. The d_e term is defined as the effective thickness, analogous to the thickness parameter for transmittance.

For N multiple reflections, the reflectivity becomes:

$$R^N=(1-\alpha\, d_e)^N \qquad (1)$$

For weakly absorbing materials, $\alpha\, d_e \ll 1$ and:

$$R^N=1-N\,\alpha\, d_e \qquad (2)$$

The expression for the effective thickness can be derived from the Fresnel equations. For weakly absorbing isotropic materials, the effective pathlengths[2] for perpendicular and parallel polarizations are:

$$\frac{d_{e\perp}}{\lambda_1}=\frac{n_{21}\cos\theta}{\pi(1-n_{21}^2)(\sin^2\theta-n_{21}^2)^{1/2}} \qquad (3)$$

$$\frac{d_{e\parallel}}{\lambda_1}=\frac{n_{21}\cos\theta(2\sin^2\theta-n_{21}^2)}{\pi(1-n_{21}^2)[(1+n_{21}^2)\sin^2\theta-n_{21}^2](\sin^2\theta-n_{21}^2)^{1/2}} \qquad (4)$$

where λ_1 is the wavelength, θ is the incident angle, and n_{21} is the refractive index of the sample relative to that of the ATR element.

RESULTS AND DISCUSSION

Traditional ATR crystals have well-defined geometries that result in one or more reflections from the sample. A triangular prism, such as used by the AccessATR™ provides a single reflection. Multiple reflection ATR crystals are typically parallelepipeds or trapezoids in which the number of reflections can be easily calculated the length and thickness of the crystal, in addition to the incident angle. (2) For the fiber loop probe used herein, it is extremely difficult to ascertain the effective incident angle and the number of reflections from geometrical (3) considerations. However, the number of reflections can be determined experimentally.

Spectra were recorded from paraffin oil using four different

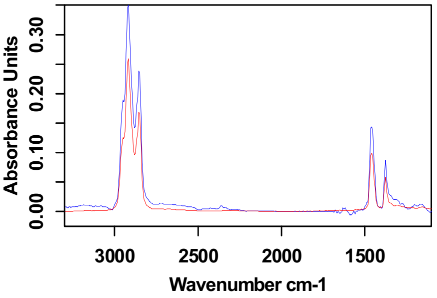

Figure 4. ATR Spectra of parafin oil, recorded by single reflection ATR (red) and fiber optic loop 3 (blue).

Loop ID	Effective Number of Reflections	
	CIR Probe 3100-2850 cm^{-1}	PIR Probe 1530-1230 cm^{-1}
1	2.2	1.8
2	2.1	1.6
3	2.0	1.8
4	2.0	1.7

Table 1. Effective number of reflections for the fiber loop probe as compared to 45° single reflection ATR on ZnSe.

sampling tips and the two fiber probes. These spectra were then compared to the single reflection ATR spectrum (see Figure 4). For all eight combinations, the differences between these spectra were calculated to determine the effective number of reflections, which are presented in Table 1.

With respect to band intensities, the fiber loop sampling tips provides the sensitivities equivalent to slightly less than two ATR reflections when compared to ATR with a 45° incident angle on a ZnSe triangular prism. Note that the effective number of reflections appears to be a function of wavelength. This is due to the fact that the infrared bands around 3000 cm^{-1} technically do not meet the criteria for a weak absorption and thus eq. 3 does not pertain.

The measured number of reflections is not surprising. The ‘incident angle’ for the fiber loop is not well defined and is likely to be much higher than 45°, since it is the grazing or high angle rays that propagate down the length of the fiber.

Note that there are also slight differences among the fiber loops. This most likely results from the small differences in the overall length and curvature of the loop. These slight differences indicate that for quantitative measurements it would be advisable to either use an internal standard or calibrate each loop prior to use.

Figures 5 through 9 show spectra of several liquids and pastes recorded using this fiber optic system. The first three figures show spectra recorded from different liquids, all derived from red grapes. Figure 5 shows the spectrum of grape juice, where distinct characteristics of the organic component of the grapes are clearly visible over the water bands. Figure 6 shows the spectrum of wine. There are distinct bands at 2984 cm^{-1} and 1046 cm^{-1} that are not present in juice. These are the C-H and C-O bands respectively from the alcohol in the wine. Figure 7 shows the spectrum of red wine vinegar. Bands are present in the 1712 cm^{-1} and around 1300 cm^{-1} that are characteristic of the carboxylic acids in vinegar.

Figure 8 shows spectra from two gel-like toothpastes. These spectra are very similar, indicating that their composition is nearly identical. This is confirmed by the package, which indicates very similar primary ingredients (see Table 2).

In Figure 9, two additional spectra of white toothpastes are shown. The spectrum of Sensodyne has some similarities to that of glycerin[6] and it has distinct bands around 830 cm^{-1},

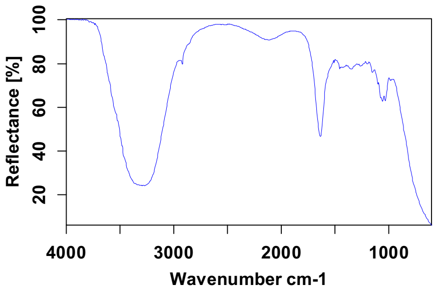

Figure 5. ATR spectrum of red grape juice.

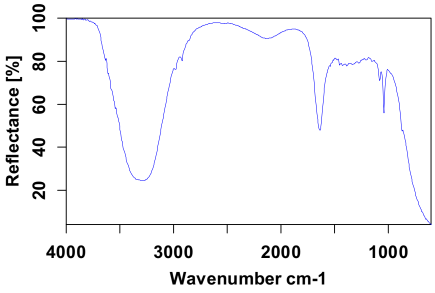

Figure 6. ATR spectrum of red wine.

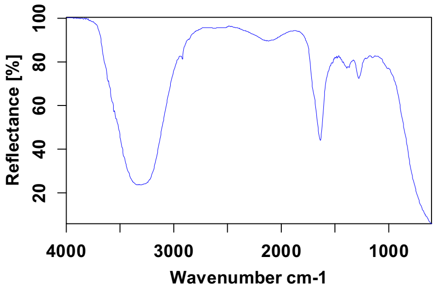

Figure 7. ATR Spectrum of red vinegar.

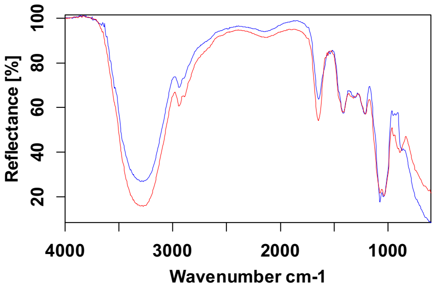

Figure 8. ATR spectra of colored toothpastes: Cinnamon Rush (red) and Kid’s Crest (blue).

indicating the presence of a peroxide. From the spectra, the Colgate paste appears to have a lower concentration of glycerin and somewhat more water.

CONCLUSION

This mid-infrared fiber optic system, consisting of the FiberMate coupler and MultiLoop-MIR, has sufficiently high sensitivity for use with commercial FTIR spectrometers and their DTGS detectors. These fiber optic probes provide approximately two reflections from the sample compared to single reflection 45° ATR on ZnSe. As demonstrated here, this system is suitable for use in qualitative studies and has sufficient performance for quantitative work. Additional work is in progress to determine the limits of this system for quantitative studies.

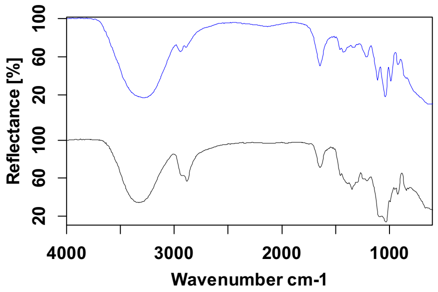

Figure 9. ATR spectra of white toothpastes: Colgate Regular (blue) and Sensodyne (black).

Toothpaste	Primary Ingredients Listed
Crest Cinnamon Rush	sodium fluoride, sorbitol, water, hydrated silica, glycerin, Tetrasodium pyrophosphate
Kid's Crest	sodium fluoride, sorbitol, water, hydrated silica, trisodium phosphate, xanthan gum
Sensodyne	potassium nitrate, sodium monofluorophosphate, calcium peroxide, flavor, glycerin, hydrated silica, PEG-12
Colgate Regular	sodium monofluorophosphate, dicalcium phosphate dehydrate, water, glycerine, sodium lauryl sulfate, cellulose gum

Table 2. Primary ingredients of the toothpastes examined.

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[6] See www.chemexper.com

Characterization of "Skin" Orientation of Thermotropic Liquid Crystalline Copolyester Moldings

Robert A. Bubeck, Lowell S. Thomas, and Kathey Robertson,
Michigan Molecular Institute, 1910 W. Saint Andrews Road, Midland MI 48640;
S. L. Berets, Harrick Scientific, 141 Tompkins Ave, Pleasantville NY 10570

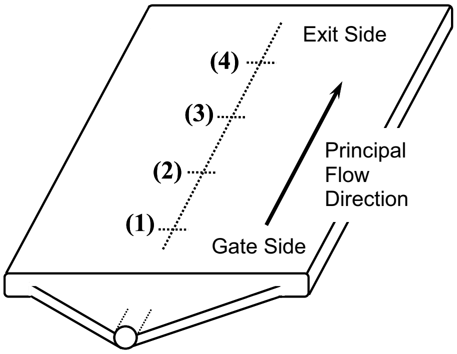

Figure 1. 76 mm by 76 mm injection-molded plaque configuration with coat hanger gate shown at the bottom. The numbered positions are 15 mm apart.

Figure 2. The Seagull.

Figure 3. The Seagull ATR Rotator.

INTRODUCTION

Thermotropic liquid crystalline polymers (TLCPs) combine the virtues of superior tensile properties with the ability to injection mold with very easy flow through the spontaneous ordering of molecules. A critical processing issue is the development of high anisotropy during TLCP processing and its effects on the physical properties of injection molded parts. Although the concurrence of high tensile properties and high directional orientation is of great benefit in fiber spinning, severe anisotropy can be a plague in obtaining balanced properties in many injection molded parts. The rigid nature of the mesogenic segments in TLCP molecules usually leads directly to a high orientation bias favoring the direction of flow with injection molding and other directional processing of thermotropes.[1,2] A "skin-core" morphology usually develops on the surface and the interior, respectively, of the article during melt processing by extrusion, fiber spinning, injection molding, etc.[3]

Layer orientation in injection molded plaques of 6-hydroxy-2-naphthoic acid/6-hydroxybenzoic acid (HBA/HNA) (58 mol % HBA and 42 mol % HNA) random type copolyesters was determined by Pirnia and Sung[4] using Fourier transform Infrared (FT-IR) attenuated total reflection (ATR) dichroism. This technique enabled these researchers to determine relative orientation encompassing a depth of material 5 μm below the sample surface. Using measured dichotic ratios to calculate the orientation function, the skin, intermediate layers and core were characterized for a series of samples cut from positions along injection-molded plaques by progressively removing material by milling. Generally, Pirnia and Sung reported that: (1) orientation was highest for the skin and progressively decreased as one proceeded from the intermediate layers to the core; and (2) orientation was lowest in the gate region of the moldings. The highest orientation was usually observed in the region beyond the immediate gate area, with orientation parameters on the average of 0.77 to 0.88 being reported for the skin layer. Pirnia and Sung characterized the "skin" that had encompassed a depth of 5µm in sampling surface orientation using their FT-IR technique.[4]

Similar measurements were performed on our samples to determine if the magnitude of the "skin" orientation is similar to what Pirnia and Sung found for their injection-molded samples.

EXPERIMENTAL

Materials and Fabrication

The TLCP primarily utilized in this study is a copolyester containing 4,4'-dihydroxy-α-methylstilbene (DHαMS) as the mesogen and a terephthalate/isophthalate/2,6-naphthalenedicarboxylate molar ratio of 65/10/25.[5,6] Sample plaques 76 mm by 76 mm were fabricated using a Boy 30T2 injection molding machine with which both melt and mold temperatures were readily controlled. A mold with an insert with polished faces was utilized permitting the fabrication of plaques of various thicknesses from 0.8 mm to 3.2 mm.

Polymers with molecular weights of about 35,000 g/mol were evaluated. Fill times of 1 s were utilized.

It was ascertained that the samples required surface cleaning to remove contaminants before examination. The cleaning was performed using a 1% solution in de-ionized water of Alconox Liqui-Nox® cleaning agent with a Bransonic 220 ultrasonic cleaner for duration of 10 min. Both the initial presence of contaminants and their effective removal were verified for selected samples by atomic force microscopy using a Topometrix 2000 AFM.

Surface Orientation

Samples were cut from an area down the centerline of a 0.8 mm thick plaque molded with a melt temperature of 290ºC and a mold temperature of 45ºC. The positions are shown in Figure 1. The direction of melt flow during injection-molding progresses from position 1 towards position 4.

ATR Spectroscopy

The samples were analyzed by infrared spectroscopy using the Seagull Variable Angle Reflectance Accessory (see Figure 2) in a Nicolet Nexus 670 FTIR spectrometer. The Seagull was equipped with its ATR Rotator (see Figure 3), a ZnSe ATR crystal and a Wire Grid Polarizer. The polarizer was set for s-polarization and the Seagull was set for an incident angle of 55°. This incident angle was selected since it was slightly above the experimental critical angle for the samples examined.

Each sample was oriented on the sampling stage of the ATR Rotator so its cut edges corresponded to the 0° and 90° degree settings. A background spectrum was recorded using the clean ATR crystal and then sample spectra were collected with the ATR Rotator set to 0°, 45°, and 90°. The spectra collected were signal averaged over 32 scans at 8 cm^{-1} resolution, over a spectral range from 4000 cm^{-1} to 650 cm^{-1}.

For comparison to the results of Pirnia and Sung, the spectra were converted to absorbance units and baseline corrected. Then the dichroic ratio and orientation parameter were calculated as described by Pirnia and Sung.

RESULTS AND DISCUSSION

Figure 4 shows the unprocessed fingerprint region of the spectra collected from the from the position 4 sample. These spectra show distinct differences as a function of the position of the sample in the sampling plane. In particular, the band at 1500 cm^{-1} is much weaker at the 0° position than at the 90° position. This is expected, since this band is associated with the skeletal vibration of the benzene and naphthalene rings in the copolymer. These rings are oriented roughly parallel to the polymer chain axis. The band at 1730 cm^{-1}, on the other hand, shows very little change in intensity due to the rotation of the sample in the sampling plane, indicating that it is not strongly associated with an oriented species within the copolymer.

The orientation differences in these particular samples are extremely easy to observe by FTIR spectroscopy. The rotation of the sample could be tuned to maximize and/or minimize the intensity of the band at 1500 cm^{-1}, to position the sample more precisely along its oriented axis than the mechanical alignment used herein.

The spectra were processed to compare the orientation function to that published in the literature. The orientation function for the band at 1500 cm^{-1} is shown in Figure 5. The orientation function is approximately the same as that reported by Pirnia and Sung. In addition, the orientation is lowest in the gate region as expected.

SUMMARY

The orientation of injection-molded polymers can be analyzed in a straightforward manner by ATR dichroism. Using a variable angle accessory and sample holder, like the Seagull and its ATR Rotator, allows the incident angle to be optimized for a given sample and also makes it possible to determine if the orientation axis is along the axis predicted.

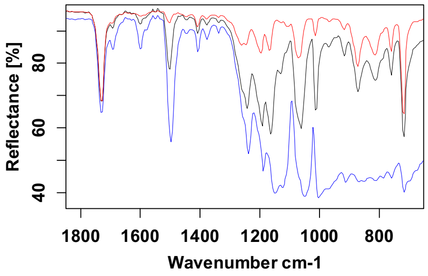

Figure 4. ATR spectra of the sample from Position 4 recorded at 0° (red), 45° (black), and 90° (blue).

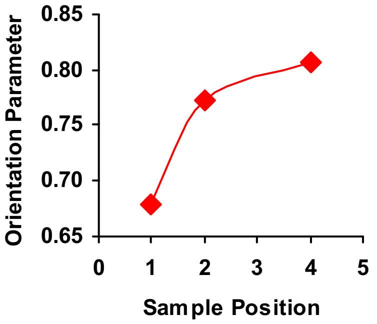

Figure 5. The orientation functions at various positions from the Gate.

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