

**A Preview of Our New Products at PITTCON 2005**  
by Scott Little, President

Welcome to the Spring 2005 edition of the PIKE Reflections newsletter!

A few weeks ago, when our Fall 2004 PIKE Reflections was sent to you, we promised our next newsletter would "let the cat out of the bag" regarding some top secret **new product developments** underway at PIKE. So, not to disappoint, we are delighted you are the first to know. On February 28, PIKE will be showing 8 new products and enhancements at the Pittsburgh Conference of Analytical Chemistry and Spectroscopy. We're predicting the biggest splash in Orlando will be the introduction of the PIKE  $\mu$ MAX™ sample compartment FTIR microscope (see article below) including the RotATR™ micro ATR microscope objective and the PIKE Micro Compression Cell. In addition, the FlexIR™ NIR Fiber Optic Accessory, PIKECalc™ sampling computational

**$\mu$ MAX™ - a New Choice for FTIR Microsampling**  
by Ken Kempfert

We are very pleased to offer the  $\mu$ MAX™ - an all new optical design for IR microanalysis - designed for high performance sampling at low-cost coupled with exceptional ease of use. The  $\mu$ MAX is designed to fit into the sample compartment of your FTIR, which minimizes the pathlength of the IR beam and thereby increases IR throughput. Gold-coated reflective optics in the  $\mu$ MAX further enhances IR signal strength and enables its use with the DLaTGS detector in your FTIR.

The  $\mu$ MAX is the first sample compartment IR microscope accessory capable of all **microsampling modes - transmission, reflection and ATR**. The  $\mu$ MAX uses the

software, enhancements to our MIRacle™ & HATR products and a new PIKE catalog (see article on page 4) will be introduced. You can see all of these new products and more when you visit our **booth (#3465) at PITTCON '05** in Orlando, Florida.

As in the past, PIKE will be exhibiting as one of ten organizations in "**Spectroscopy Central**", PITTCON's Spectroscopic Marketplace. The Spectroscopy Central organization offers a wide range of spectroscopy-related products and services. The exhibiting companies include: The Coblenz Society, Control Development, FDM Spectra, FTIR.com, High Pressure Diamond Optics, Infrared Analysis, Infrared Associates, Molecular Microspectroscopy Laboratory, MTEC Photoacoustics and PIKE Technologies.

We wish to thank you for your business and continued trust in us for your spectroscopy needs. We have a small gift for you at our booth - a thank you for stopping to see us. We hope to see you soon in Orlando!

spectrometers detector for convenience and sampling flexibility. For larger micro samples (100 microns and greater) the DLaTGS detector of your FTIR provides excellent performance with the  $\mu$ MAX and enables full mid-IR spectral range coverage to 400 cm<sup>-1</sup>. For smaller micro samples to 20 microns in size, an MCT detector is required.

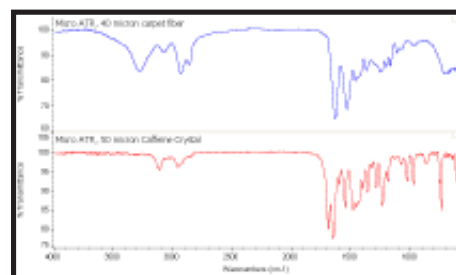
All operations with the  $\mu$ MAX are intuitive and made even easier with standard **Dichroic Optics** which provides full viewing of the sample while collecting IR spectra. The  $\mu$ MAX IR microscope uses a high N.A. **Swartzchild objective and condenser** to focus the IR beam onto the sample and provide excellent sample visualization.

ATR is an excellent sampling option for the  $\mu$ MAX IR microscope. The RotATR™ is a unique, rugged, pivot-designed ATR providing easy and precise operation and



**$\mu$ MAX™ - Sample Compartment Microscope for FTIR**

excellent micro ATR spectra.



**Micro ATR spectra of a 40 micron carpet fiber and a 50 micron caffeine crystal**

Micro ATR works exceptionally well with the  $\mu$ MAX IR microscope. The 100 micron diameter, flat-tipped micro ATR crystal makes intimate contact with the sample to achieve high spectral quality as seen in the data above.

Please contact us for additional product details.

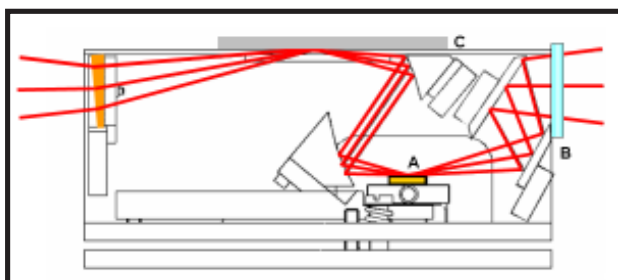
## Measurement of Monomolecular Layers with Specialized FTIR Grazing Angle Accessories

by Gabor Kemeny, PhD.

Monomolecular layer samples have traditionally been a challenge for spectroscopic measurements. Specular reflectance at high “grazing” angles (around 80 degrees from normal to the surface) has been recognized as one of the best spectroscopic techniques to monitor monomolecular layers on reflective surfaces due to the enhancement of the infrared signal (1, 2). The versatility of this sampling technique is further optimized using different specialized grazing angle accessories.

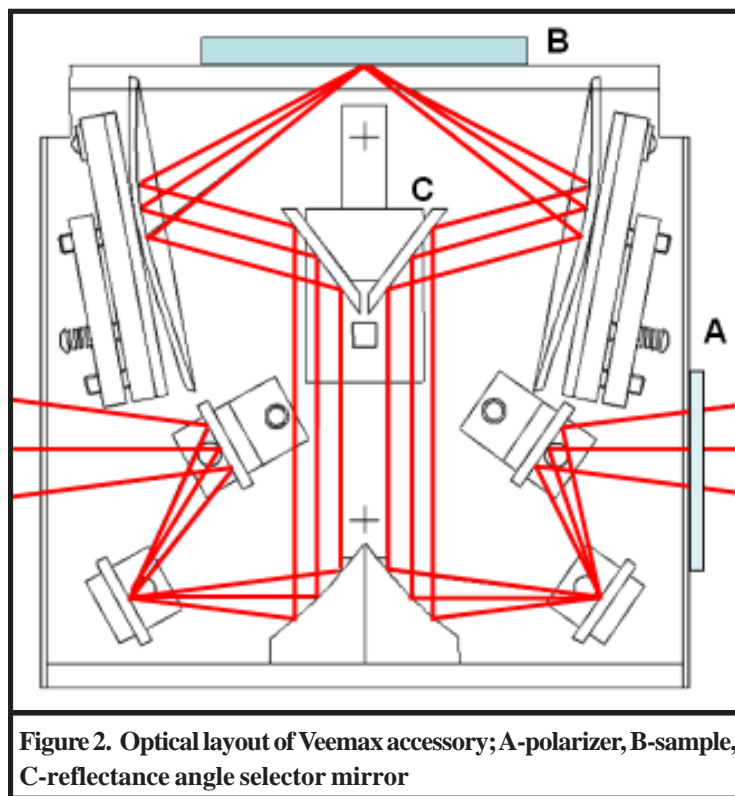
### Experimental

An FTIR spectrometer was equipped with an MCT detector and with three different grazing angle accessories; an 80Spec™ - fixed grazing angle, an AGA™ - variable illumination area and with a VeeMax™ - variable angle reflectance accessory. The fixed angle 80Spec is a dedicated single reflection accessory with simple input and output optics channeling the infrared light with two gold flat mirrors. While this is a powerful tool, its use is limited to larger area samples. When the light from an FTIR instrument is directed to the sample at a high angle, the illuminated area is not as well controlled, its shape is elongated and the IR image size becomes relatively large. When the sample is smaller than the illuminated spot, apertures could be used to limit the size of the infrared beam. In this case, however, the contrast and repeatability of the measurement usually suffers considering the sensitivity required to detect monomolecular quantities of materials. The controlled



**Figure 1. Optical layout of the AGA accessory; A - selector mirror, B - polarizer, C - sample position**

projected image size therefore is a very important advantage of the AGA accessory, which has a special imaging system with a set of variable diameter mirrors. In this unique optical design, the illuminated spot on the sample is the same as the size and shape of the gold mirror at the base of the optical layout (Fig. 1). The selector mirrors in this design are round and are variable in five steps from 3 mm to 12.5 mm diameter.



**Figure 2. Optical layout of VeeMax accessory; A-polarizer, B-sample, C-reflectance angle selector mirror**

The variable angle VeeMax accessory (Fig. 2) on the other hand allows other reflectance and reflectance-absorbance measurements in addition to grazing angle measurements.

All three optical arrangements can be used with additional polarizers to enhance the monomolecular surface species. In this work, a ZnSe wire grid polarizer was used for all samples. All measurements were done using the MCT detector as the IR throughput is relatively low

at grazing angle of incidence in combination with an IR polarizer. All spectra were collected at 8 cm<sup>-1</sup> resolution with a 1 minute data collection time.

### Results and Conclusions

For the comparison of the performance of different optical geometries, monolayer systems used in various biotechnologies were selected. Monolayers are considered initial surface coverages with reactive groups to be modified further for various bio-specific and ligand interactions, fabrication of protein, DNA and carbohydrate chips and biosensors. Gold is known to have high affinity to sulfur containing groups, such as thiols, and generate more or less well-ordered monolayers (4). One of the systems, a self assembled hydroxyl-terminated

alkanethiolate monolayer that was formed on a freshly evaporated gold surface using the method and materials from Platypus Technologies, Inc (Madison, WI, [www.platypustech.com](http://www.platypustech.com)). The monomolecular layer is formed over a timeframe of sev-

eral hours in a dilute solution of HS(CH<sub>2</sub>)<sub>n</sub>(OCH<sub>2</sub>CH<sub>2</sub>)<sub>n</sub>X, where X can represent different functional groups. The ω-functionalized alkanethiolate layer (5) was stable throughout the experiment and was easily measured on all of the accessories.

Figure 3A shows the NH-terminated monolayer measured on the 80Spec with a ZnSe polarizer. The example shown using the AGA is an OH-terminated thiol (Fig.3B). The accessory was adjusted to project a 12.5 mm diameter spot on the gold coated slide. Due to the better definition of the illuminated area, smaller samples can be measured

## Spectroscopic Creativity - our Customer's Perspective

At PIKE Technologies, we speak with our customers on a daily basis about our products, optimizing their use, which one to select for spectroscopy applications – and sometimes....., well we just get a surprise.

That happened recently when the folks at CSI Studios called and asked if we could provide FTIR accessories for their CSI:NY show. Their new episode – “Rain” involved the analysis of a white substance which was examined, compared with suspected materials and was ultimately identified as fake snow.

## Monolayer Analysis - cont.

easily without apertures which in some cases can introduce artifacts. Grazing angle measurements with the VeeMax is shown with a third kind of monolayer, a CH-terminated alkanethiol. –CH terminated alkanes are generally more difficult to identify, due to the lack of characteristic differentiating molecular groups in comparison to organic contamination from ambient air, solvents on the sample or the background slides. For the experiment the VeeMax was set to 78 degrees and produced the characteristic pattern seen in Figure 4.

## Conclusions

Infrared spectroscopic measurements of monomolecular layers can be performed using all of the different grazing angle reflectance accessories discussed in this work and are capable of producing excellent quality spectra. The intensity of the monomolecular signal may be enhanced with polarized light, and the quality of the result is greatly enhanced by using an MCT detector. By controlling the illuminated area with a special optical design of the AGA, one can measure small samples or examine small areas of larger samples. When, in addition to grazing angle analysis, other reflectance measurements are needed; the variable angle VeeMax is an excellent choice.



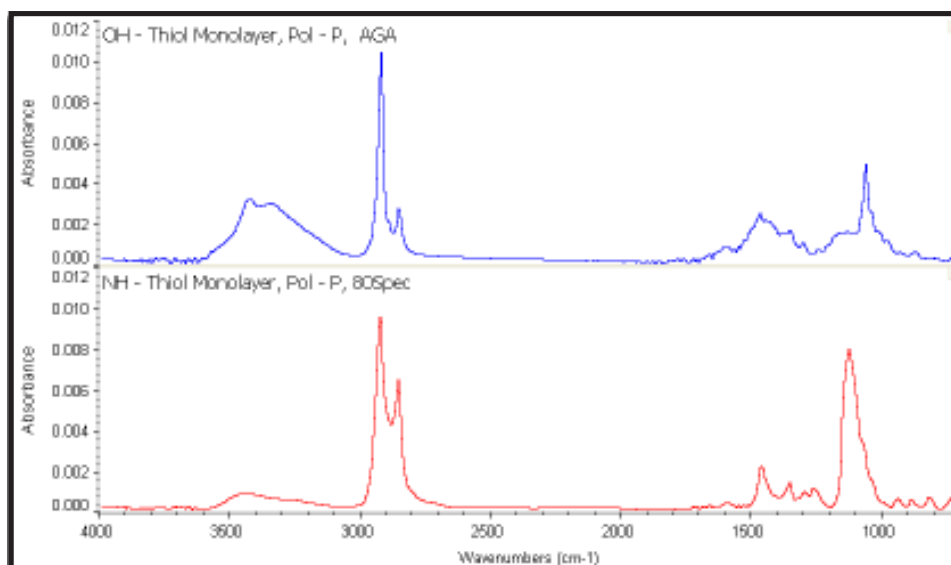
The PIKE Technologies 13 mm pellet press was an essential part of their analysis and helped lead the CSI:NY team toward the culprits, rescue a kidnapped child and bring the criminals to justice.

We think your spectroscopy sampling could become every bit as exciting as that of the CSI:NY team if you use our pellet press. Don't you think so too?

We are honored to have been a part of the CSI:NY show broadcasted last November.

Since PIKE Technologies sampling accessories are an essential sampling tool in the spectroscopy laboratory, we hope to see our products again in future episodes of CSI:NY.

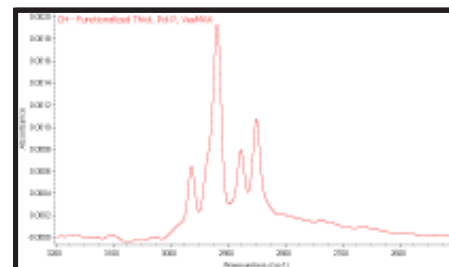
If you would like to contribute to our Customer Perspective articles in the future, please contact us at sales@piketech.com.



**Figure 3. Upper (B), self assembled OH-Functionalized Thiol-Monolayer (measured using the AGA accessory and lower (A), self assembled NH-Functionalized Thiol Monolayer (measured using the 80Spec accessory).**

## References:

1. Francis M. Mirabella: Modern Techniques in Applied Molecular Spectroscopy, John Wiley & Sons 1998
2. R.J. Greenler, *J. Chem. Phys.*, **44**, 10 (1966)
3. Single Adjustment Specular Reflection Accessory for Spectroscopy, US Patent **5,106,196**
4. Pradier, C-M, Salmann, M., Liu, Z., Methivier, C., *Surf. Interface Anal.* **34**, 67-71 (2002)
5. Ulman, A., *Chem. Rev.* **96**, 1533-1554 (1996)



**Figure 4. Grazing angle infrared spectrum of self assembled CH-Functionalized Thiol Monolayer measured using a VeeMAX variable angle reflectance accessory.**

- *Preview of PIKE at PITTCON*
- *New  $\mu$ MAX IR Microscope*
- *Monomolecular Layers - Analysis by Specular Reflectance*
- *Spectroscopic Creativity - Our Customer's Perspective*
- *New Accessories Catalog - Coming Soon!*
- *\$1,000 Question & Answer*
- *Events Calendar*



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## PIKE Technologies Soon to Launch its New Catalog of FTIR Accessories!

With the freshness of the New Year still at hand, PIKE Technologies is proud to present our brand new catalog! Not only are there hundreds of new products; it is also organized in a more efficient style to make it easier to find what you need. The applications content of the catalog has been expanded to provide examples of the type of spectral data you can expect from each PIKE accessory.

Our newest chapter, *Microsampling*, is one of the fastest growing areas of infrared spectroscopy. Given this fact, PIKE is excited

to introduce the  $\mu$ MAX and the *Microsampling* section of the catalog. The new *Integrating Spheres* section offers sampling solutions for a wide range of samples and applications for detailed reflectance analysis. Another new chapter, *Polarization*, offers tools useful for determining sample orientation and enhancing thin surface coatings and can be used with a variety of accessories.

The new PIKE catalog has many automation options available to improve the sampling efficiency of your laboratory.

With all of these new additions to our catalog, we are sure you will find it a valuable resource to help provide sampling solutions for your analysis requirements.



Please contact us at [sales@piketech.com](mailto:sales@piketech.com) to reserve your copy of our new catalog.

## \$1,000 Question & Answer

Wow, we received a lot of responses for our last \$1,000 question and we have a winner! Actually, several of you correctly answered our question, so we did a drawing to pick the winner.

C. R. Chenthamarakshan at the Department of Chemistry at the University of Texas at Arlington, correctly answered the question and won our drawing. Congratulations!

The answer to our question "What is the difference between depth of penetration and effective penetration in ATR?" is highly technical and requires several paragraphs

and numerous equations to describe. A good reference for the detail is *Internal Reflection Spectroscopy, Theory and Applications*, Frances M. Mirabella, Jr. Ed.

Here are our new questions to challenge your historical recollections;

In what year and what city was the first PITTCON held? Who is considered to be the Father of Microscopy?

Send your answers to [sales@piketech.com](mailto:sales@piketech.com) and you could be the winner of a \$1,000 accessory discount and a PIKE gift.

## PIKE Events Calendar

PITTCON 2005  
Feb 27 - Mar 4  
Orlando, FL  
Booth # 3465



ACS West 2005  
March 13 - 17  
San Diego, CA  
Booth #1843



We will be very busy getting out to meet you all at these Spring shows this year. Those of us in these wintry northern climates look forward to the warm weather and sunshine promised at these locations.