Summer 2004 Vol. 9/2

PIKE TECHNOLOGIES
Spectroscopic Creativity
Reflections
Newsletter for Practicing Chemists and Spectroscopists

What's New at PIKE Technologies?

by Scott Little, President

A whole lot! With many thanks to you, the PIKE Technologies family is growing at an ever increasing rate. The start of our 15th year is proving to be our best yet as orders have reached an all time high. To ensure that product delivery times remain at the responsive level you've become accustomed to, we have hired two new manufacturing product specialists, **Charlene Porter** and **Dawn Streuly**. Charlie specializes in the production of MIRacle ATR systems and die kit polishing operations. Dawn will specialize in Multi-reflection HATR, Specular and Diffuse Reflectance as well as transmission cells and holders. Charlie and Dawn have extensive experience in mechanical and electronics assembly, so we are very excited they have joined the PIKE team!

In addition, PIKE expanded its engineering design and product development capacity with the addition of **Steve Vogel** to the PIKE engineering group. Steve has a degree in Mechanical Engineering and a Masters in Business Administration along with many years of optical design experience on FTIR accessories and IR microscopes. Steve comes to us from Zygo Corporation and was previously a senior engineer with Spectra-Tech, Inc. in Shelton, Connecticut.

PIKE Technologies - seated left to right, Scott Little, Irene Brierley, Krista Garcia, Charlene Porter, Dawn Streuly, and Steve Vogel. Standing left to right, Ken Kempfert, Dave Engerman, Dennis Shanks, Dennis Maier, Gabor Kemeny, Ryan Shiroma, Kent Gundlach, Patrick Little. On vacation - Stuart Smith and Shawn Gardner.

Also a result of our recent growth, our manufacturing area has gone through a complete re-design and layout. The new layout makes room for our new Fryer MD-20 CNC machining center and a new Hyprez lapping system. At the same time, we have co-located our previously centralized stock room area into each of our five production work centers for improved efficiency and to assist in the effort to keep our delivery times the most responsive in the industry.

To help get to know the newest PIKE people, check out the group photo. Perhaps the only complaint about our recent growth comes from our company dog (Annie), who lost her favorite bathroom spot to expanded parking! Although the company is getting larger, our pro-customer orientation remains the same. ***We want PIKE Technologies to be your favorite spectroscopy company!***

New Products from PIKE Technologies

If you attended PITTCON 2004 and visited our booth, we hope you got a chance to see these new products. This article is a review for those of you who did and new information for everyone else.

Catalytic chambers for diffuse reflectance. Two versions available for 500 or 900 C maximum temperature. Chambers may be used with vacuum or pressurized and reactive gases may also be flowed into the chamber. ***Applications:*** Study of catalytic reactions and chemical reactions.

Specular reflectance plate for the MIRacle ATR accessory - 45 degree angle of incidence and easily changeable with ATR crystal plates. Samples are placed over the 7 mm beam port for analysis. ***Applications:*** Coatings on reflective materials.

Transmission sampling plate for Microtiter Plate Accessory - utilizes a specially bonded polymer mask to define the sample area providing an easily cleaned surface during sample exchange. ***Applications:*** Micro-chemistry formulation and biotechnology research.

If you would like additional information about any of these new products, please contact us by phone, e-mail or through our web site.

Integrating Spheres - Introduction and Theory

by Gabor Kemeny, PhD.

Measuring Sample Reflectance

Reflectance sampling accessories focus the beam coming from the spectrometer onto the sample. In order to achieve the best signal-to-noise ratio (SNR), the smaller the focus is, the easier it is to refocus the illuminated sample spot back onto the detector. This arrangement serves well if the sample is microscopically homogeneous, but will result in spectral differneces when the sample is exchanged. The focused beam sees a different portion of the sample resulting in increassed measurement-to-measurement differences. This is called insertion error because the spectrum will be slightly different each time the sample is inserted.

Some industrial or natural samples are inhomogeneous either because they are mixtures of different substances or because they have a particle size comparable to the probing beam diameter. Clearly, if the probing beam could be larger and the reflected light could all be collected, a more representative spectrum could be measured.

Some other samples develop a directional scattering. Fibers wound on a mandrel are highly oriented not just macroscopically as parallel, unidirectional filaments, but also in many cases the molecules of the drawn fibers are oriented within the fiber itself. When placed in a reflectance accessory this will generate different results depending on the angle from which the detector is "viewing" the sample. When the overall reflectance needs to be measured reproducibly, for example to measure the concentration of a minor ingredient in the sample, only isotropic optical systems, insensitive to such directionalities could be utilized.

In some cases, not just the reflectance in a small solid angle but the reflectance in all angles is sought. Most reflectance accessories measure at fixed or variable angles, narrower or wider collection angles, but there is a need for a device that uniformly collects all reflected light from a sample, in other words it measures the total reflectance of the sample.

All of the above concerns are addressed with integrating sphere based reflectometers.

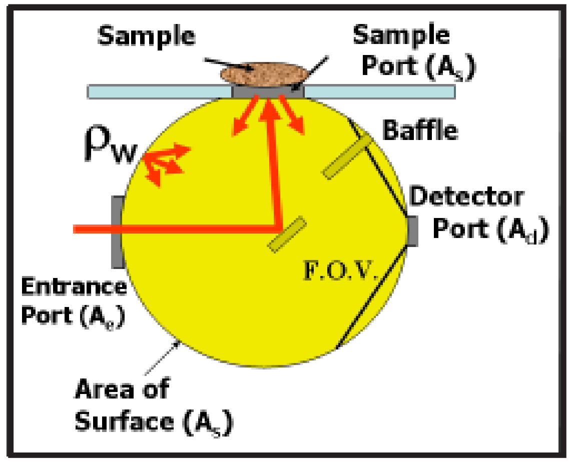
The main reasons for using integrating spheres for the measurement of sample reflectance therefore are the following:

- Efficient measurement of combined diffuse and specular reflectance
- Uniform detection of reflectance even when sample is inhomogeneous
- Isotropic detection of reflectance even on samples that reflect in preferred directions
- Reduction of polarization effects from the illuminating beam and the sample
- Measurement of absolute reflectance (with special integrating spheres)

Integrating Sphere Optics

Integrating spheres are highly reflective enclosures that are placed in close proximity to the sample, such that the reflected light enters the sphere, bounces around the highly reflective diffuse surface of the sphere wall and finally impinges upon the detector - usually part of the integrating sphere assembly. The name, integrating sphere, refers to one of the main functions of the device, namely that it spatially integrates the light flux, in our application the light reflected from a sample. In spite of the long history of engineering and development of the sphere, the applications and further developments continue to this day. Advances in the theory, in detector and electronics development and most of all, new applications drive the progress.

As the name implies, the main part of the device is a sphere with a very highly reflecting inner surface. The surface should approach the ideal Lambertian scatterer, which means that the light falling on the surface is evenly scattered in all directions and the scattered light intensity is proportional to the cosine of the angle of observation. The infrared beam from the interferometer is directed through an entrance port onto the sample placed behind the sample port



(shown above). Samples can be directly touching the sphere or separated from the sphere by a thin, infrared transparent window. The detector is placed close to the sphere, so that it can view the integrating sphere with a large solid angle. In order to improve the isotropy (non-directionality), the detector must not be directly in the line of sight of the sample. A small, also highly reflective and scattering baffle is placed in the sphere such that it blocks the first reflection of the sample from the detector.

A well-designed sphere has the sample close to the sphere geometry so that the sphere will collect close to the full available hemispherical reflectance (2π steradians). A window to separate the sphere and sample may be important in some cases but it will place the sample a small distance from the sphere, thereby somewhat reducing the collected high-angle reflectance. The PIKE Technologies integrating spheres are coated with the highest possible reflective surface for the desired wavelength region. The coating of the surface of the sphere has to be uniform and close to being a perfect Lambertian scatterer. These characteristics allow the light falling in the sphere to be uniformly distributed over its entire surface.

Sphere Throughput

The amount of light actually collected on the detector surface is important. The throughput of the sphere is defined as the ratio of the incoming light impinging on

Spectroscopic Creativity - *our customer's perspective*

Will Costa of Fiveash Data Management, Inc. (FDM) in Madison, WI is a true believer in the entrepreneurial spirit and spectroscopic creativity. Will created his spectral data bases venture over 10 years ago and today has grown it to be a successful company providing library search spectra for FTIR, Raman, NIR, MS and NMR to laboratories around the world.

Today FDM has available over 100,000 spectra in formats for all spectroscopy software packages.

Many of FDM's databases have been purchased or licensed from other companies, governments or universities around the world. Will is always interested in finding new collaborators. FDM also serves as a reseller for well known databases like the Hummel Polymers.

Will offers several unique products to his customers including the new Comprehensive Organics and the new ATR/FTIR Spectra of Cosmetics and Personal Care Products, all at especially competitive pricing.

Will views spectra in his new FTIR data bases

Will recently opened his company's first lab and is currently working on a new adhesives ATR/FTIR spectral data base. His connection with PIKE Technologies is that he uses a MIRacle™ ATR accessory to collect the data.

You can learn more about FDM's databases by calling Will at 608-236-9145 or visit the FDM web site at www.fdmspectra.com. Free trial data bases can be downloaded from the his web site.

Integrating Spheres - *continued*

the detector. The closer the sphere surface is to ideal reflectance, the higher the throughput. The detector, the sample and the illumination require that a portion of the wall of the complete sphere be removed. The smaller the cutouts - the better the throughput. Due to other considerations, such as reduction of light scatter from the edges of the sphere cutouts, called ports, these have to be optimized and cannot be too small.

The throughput can be expressed with these sphere design parameters:

$$\tau = \frac{A_d}{A_s} \times \frac{\rho_w}{1-\rho_{w,\,avg}}$$

Where A_d is the detector area, A_s is the sphere area, ρ_w is the sphere wall hemispherical reflectance, $\rho_{w,avg}$ is the average sphere wall reflectance.

The sphere throughput is higher if the light falling on the detector is increased by the multiple reflections of the light. Another way of looking at the integrating sphere is that it enhances the detector signal by collecting the light, and if the wall surface is reflective enough, bounce it around until it hits the detector. The factor that is used to express this gain is called the sphere multiplier (M), which is a function of the wall reflectance (ρ_w), the proportion of the total area of ports to the surface of the sphere (*f*).

$$M = \frac{\rho_w}{1-\rho_w(1-f)}$$

The brightness of the sphere, using the same amount of input light flux is dependent opon the wall reflectivity, the port-to-sphere surface ratio and the size of the sphere surface.

$$L_s = \frac{\Phi_i}{\pi A_s}\frac{\rho_w}{1-\rho_w(1-f)}$$

where Φ_i is the input light flux and A_s is the area of the sphere wall surface.

For the sphere the area of the sphere obviously depends on the sphere diameter, and thus the formula shows that a smaller sphere is brighter than a larger diameter one.

$$L_s \sim \frac{M}{D^2}$$

The sphere diameter cannot be reduced too far however, because the sample diameter will also have to be decreased proportionally when the sphere is smaller. For typical spectroscopic applications the optimum sphere diameter is influenced by the beam size coming from the FTIR spectrometer and the typical sample size of 3-25 mm. Most integrating sphere modules use a 2-4 inch diameter sphere to accommodate the above design parameters. In a practical design the openings of the sphere need to be kept around 5% for optimum throughput. Wall reflectance is usually between 95-99% and results in a sphere gain of 10-30.

Integrating Spheres for Mid-IR and NIR

Integrating spheres, although much more efficient than an optical system with an equivalent detector position, still have lower throughput than the direct imaging optics. In the visible and NIR, where very good sources and excellent, high-speed detectors are readily available, the SNR is usually not limited by the reduced light level. In the mid-IR, in order to utilize the above discussed advantages and benefits of integrating spheres, the reduced throughput needs to be offset by the use of high sensitivity cooled detectors, such as the liquid nitrogen cooled MCT detector utilized by PIKE Technologies. The near-infrared and mid-infrared measurements using integrating sphere optics usually have different analytical and measurement goals as well as different features, thus the two spectral regions, specific applications and the associated optimized devices will be treated separately in future technical notes.

Editors Note: *This note is condensed to fit our publication. If you would like our complete article, please contact us.*

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PIKE Celebrates 15th Anniversary

PIKE Technologies is a fun place to work. As part of our celebration of serving our customers for 15 years, PIKE employees attended an Aerosmith concert in Madison.

Charlie and Kent joined Aerosmith for a special rendition of "Dream On".

We will have an open house to celebrate our 15th at PIKE on September 17th. Please join us!

Reflections on PITTCON 2004

PITTCON 2004 was busy for PIKE Technologies. Overall attendance was up 10% from last year at 24,905. PIKE showed 7 new products in our booth. Since PITTCON was located in Chicago, everyone at PIKE was able to spend time at the booth introducing our new products, answering questions, and renewing old acquaintances. This was Krista's first opportunity to attend PITTCON. She enjoyed meeting the people she talks to on a daily basis and looks forward to speaking with more customers at future trade shows.

Congratulations to the winner of our drawing for a Dell Axim X3 Handheld PC, Allan Knerr of Phelps Dodge Magnet Wire Company!

Thank you to all of our visitors for stopping by to chat with PIKE. We look forward to seeing you next year in Orlando!

$1,000 Question

It's back! Send your answers to sales@piketech.com and you could be the winner of a $1,000 accessory discount and a PIKE gift!

- *Since it's introduction in 1997, how many PIKE MIRacle ATR systems have been shipped?*
- *In PIKE's original "Bedtime Stories", what musical instrument was used as an analogy to explain FTIR?*

Closest guess wins - good luck!

PIKE Events Calendar

ACS East 2004
August 23 -25
Philadelphia, PA

PIKE Open House
September 17th
Madison, WI

We hope to see you at these locations. Please stop and see what's new and let us know how we can help solve your spectroscopy sampling challenges.