SPRING 2001 Vol. 6/2

PIKE REFLECTIONS

Useful Information for Practicing Chemists and Spectroscopists

Our New Leader

On May 1, 2001, Scott Little became the newest employee of PIKE Technologies, Inc., accepting the position as company President. Scott brings with him over twenty years of experience in the analytical instrumentation industry, most recently as the President of Thermo Spectronic, where he was responsible for merging Spectronic Instruments in Rochester NY with Unicam UV instruments in Cambridge England, forming a new business focused on the UV-Visible Spectrophotometer market. After receiving his Bachelor of Science degree in Mechanical Engineering from Purdue University, Scott began his career in opto/mechanical design at the Harris Corporation in Melbourne Florida working on state of the art acousto-optical instrumentation and laser scanning facsimile machines for commercial and military applications. In 1981, he joined Nicolet Instrument Corporation as a Senior Mechanical Engineer working on FTIR and FT-Mass Spectrometers and accessories, including Michelson interferometers, Gas Chromatography & TGA interfaces, long path gas cells, automated hard disk lube checkers and silicon wafer handling systems for the semiconductor industry. In 1984, he assumed the responsibility of Engineering Manager for FTIR Spectrometers and continued in various management capacities at Nicolet, most recently as the Director of Product Development. He was named the President of the Thermo Optek UV-Vis business in 1999 (now Thermo Spectronic).

Although Scott is a new employee, he is certainly no stranger to PIKE or it's founder, Phil Brierley. Scott was largely responsible for Phil and Irene moving their family to Madison, Wisconsin from England in 1983 to work for Nicolet and after Phil founded PIKE in 1989, Scott worked very closely with him on a number of joint development programs.

We asked Scott to comment on becoming a part of the PIKE team:

"Phil's vision in starting PIKE over ten years ago was to create a different kind of analytical instrument company. That vision of providing "spectroscopic creativity" in the design of optical bench accessories along with "precision and a certain fanatic devotion to detail" has certainly been realized. Just as he had hoped, PIKE customers not only get the personal attention you would expect from a small company, but they also benefit from the use of the latest technology and innovation and best of all, at very reasonable pricing. My goal in joining PIKE is to continue building on his vision and for PIKE to continue to be your favorite spectroscopy company. When you call us, you will not get an operator, or voicemail, but someone who can help you right away. I'm looking forward to hearing from you soon."

Please join us in welcoming Scott to the PIKE team and don't hesitate to give him a call or write him an email at little@piketech.com.

IN THIS ISSUE

PITTCON Reflections

23,086 attendees, including 13,903 exhibitors and 9,083 conferees. A bit lower than Pittcon 2000 (27,670), or Orlando in 1999 (29,893). But we did well. The number of visitors that stopped by our booth was about 10% higher in comparison with the previous show. We are not sure what caused this, but most likely the reasons were multiple:

One: Our wonderful products (of course). This year we introduced a Single Reflection Diamond HATR (MIRacle AG) that generated a lot of interest for many good reasons. To mention a few:

- The best throughput in the single reflection HATR diamond category. An independent, impromptu test, run during the show by one of our clients, confirmed this claim completely. Better throughput means better sensitivity and nicer spectra!
- Unique and sexy clamp design – solid, with digital readout, excellent for reproducible sampling!
- Backwards compatibility – all you current ZnSe, Ge and AMTIR MIRacle users – diamond plates are now available!!! Just pop one in and you would be ready for the toughest samples.
- Price! Price! Price! – no one offers such versatility and performance for less than 6,000 dollars!!!

This year we also concentrated our efforts on promotion of PIKE autosamplers and the new

Edible Oil Analysis Kit. These products generated a lot of interest and brought many of you to our booth.

Two: Location. It is always our goal to make things easier for our customers. For this reason we display our products together with other companies that serve the spectroscopy market. This arrangement allows us to stay close to the "main path", saving you time, effort, and shoe soles.

Three: This year we decided to try something new in the "display department" and we think it worked. Our new booth seemed to attract a number of customers. We have received several positive comments regarding its layout and graphics. The "little angel" poster caught the attention of several visitors and provoked some questions. For these reasons, we would like to clarify the meaning of this image once and for all. The angel, portrayed by Madeline Smith (Stu's daughter) is presenting us with a little miracle (Angel – MIRacle? See the connection?). A bit corny? Possibly, but the angel was beautiful and the plan worked.

Four: All the freebees (see page four of the newsletter). This requires no explanation. Laser balls were clearly the hottest item in our booth (and possibly the entire show).

Five: The contest*. Many of you brought samples to our booth to see how the new Diamond MIRacle would handle them. Most unusual samples? A bundle of hair pulled directly from the customer's head, a couple of coated Si wafers, pieces of black rubber and various polymers, among others. No problem…

Well, regardless of your reasons, it was great to see you all. It was nice to talk about your application needs, run your samples and hopefully provide you with some answers and sampling ideas. If you have any additional questions, or need part numbers or prices for our new products, please call Melissa at 608-274-2721 (or drop her an e-mail at kopp@piketech.com). And please visit us again at the ACS Show in Chicago. Otherwise the Bourbon Street in 2002 would always be an option.

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*By the way, the winner of the digital camera, (which was part of our MIRacle introduction and sampling contest) was Ms. Maria Barker of Raytheon, Tucson, AZ. Congratulations!

Words of Wisdom

Sealed Liquid Cells – Pains and Rewards

Sealed cells have been used for decades for analysis of liquid samples by infrared spectroscopy. The original cell design consisted of two rectangular IR windows (typically KBr or NaCl) united by an amalgamated lead spacer. The cells were mounted between two metal plates, one of them equipped with two injection ports made from hypodermic needles. Amalgamation was achieved by brushing the lead spacer with mercury until complete coverage was obtained. The new generation of FTIR Liquid Cells utilizes the same design principles. The main exceptions include the circular shape of the cell and permanently attached front plate with syringe (Luer) ports. The new FTIR transmission cells are easier to handle. There are several advantages the sealed cell design offers – these include "ready to use" assembly and predetermined, constant pathlength (good for quantification). However, the cells also feature a few inherent disadvantages. One of them is gradual deterioration of the window material (especially the most popular but unfortunately hygroscopic KBr), they do have a tendency to leak, and they are relatively expensive. Window deterioration can be minimized (and/or totally eliminated) by careful selection of the IR material. Liquid cells are now available with ZnSe, AMTIR, Ge and other IR windows, some of which can be used with aqueous or even acidic samples. In most cases, using proper filling procedures can prevent cell leakage. It is important to remember that the amalgamation of the cell offers certain mechanical strength, but the amalgam can break if exposed to excessive pressures (temperature extremes and mechanical shock may also damage the cell). This happens often when a relatively viscous sample is injected to a short pathlength cell. To protect the cell, it is recommended that vacuum, instead of pressure is used for sample introduction. This can be simply done by employing two syringes for cell filling. In such a case, one of the syringes needs to filled-up with a sufficient amount of the sample. The second syringe should remain empty, with the plunger all the way down. The sample is introduced by pulling the plunger of the empty syringe until the cell cavity is completely filled. At this point, a short pause allowing for pressure equilibration is recommended. Then, the cell should be placed in the vertical position, syringes removed and Luer locks plugged with teflon stoppers. With the proper selection of IR windows, gentle cell handling and appropriate filling technique, the sealed cell is still one of the better tools for analyzing liquids by traditional transmission methods.

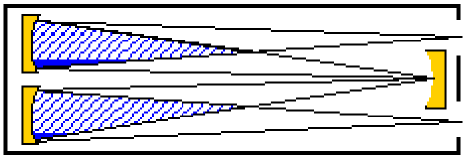
Measuring Gases (II)

This is a second installment in the Gas Analysis Series, written by Dr. Philip L. Hanst. It covers development and use of long path, multiple reflection gas cells.

Gas Sampling - Long Path Gas Cells

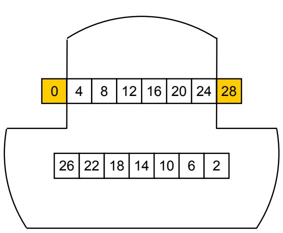
The three-mirror multiple pass cell was first described in a 1942 paper entitled "Long Optical Paths of Large Aperture" by John U. White. Ever since, it has rightly been known as a "White" cell. Although a number of improvements have been developed since then, including some by the original author (White, 1976) the basic three mirror system is still widely used.

The main advantage of the White cell is that it can capture and conserve a major fraction of the energy that can be projected from an optical source. The energy conservation is the result of the re-focusing that occurs on every other pass, as depicted in the first figure, which shows the basic unit of four passes.



The operation of the cell is described by following the light from the source into the cell and through at least four passes, with the aid of the second figure. The light from the source is initially focused into a real image in the entrance aperture of the cell. In the figure, that is designated the zeroth image. After passing through the zeroth image, the beam diverges and is collected by one of the two objective mirrors. This is a spherical mirror situated two focal lengths from the image so that it re-focuses the image, inverted, on the lower part of the opposite single mirror, called the field mirror, which is another spherical mirror of the same focal length. The first image is marked 2 for 2 passes. The field mirror is aimed so that the reflected diverging beam falls entirely of the second objective mirror. This is then aimed to form another image (marked 4) above the center line of the field mirror alongside the zeroth image. If this image falls symmetrically opposite the first image (numbered 2), the beam will be returned to the first objective at the required small angle with the input beam, so that all the energy is again collected and returned, and there will be at least four more passes through the system.

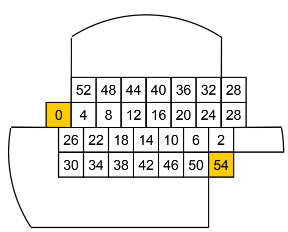
The number of images allowed in the row depends on the placement of the first image in the lower part of the mirror. If it falls exactly on the vertical center line, no more than four passes are possible. The farther to the right it falls, the greater the number of passes allowed. The number of passes is equal to the number of the exit image. In practice, the number of passes is determined by counting the number of images in the bottom row and multiplying by four.



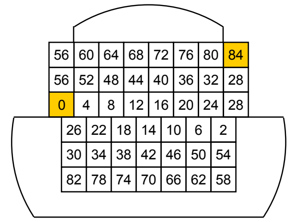
In order to have as much energy as possible going through the system, an enlarged image of the source is usually projected into the entrance aperture, and the exit image is demagnified when the transmitted light is fed into the detection system. Frequently, The only major source of energy loss in the system is the absorption at the mirror surfaces. If R is thereflectivity of the mirrors, R^n is the fractional amount of energy transmitted by the cell after n reflections. Some simple calculations show the magnitude of this loss. The traditional choice for mirror coating has been gold, for which R = 0.98, approximately. In a 52-pass gold-coated mirror system, the fraction of energy transmitted is 0.98^{52}, or 35%. With 104 passes, the fraction of energy transmitted is 0.98^{104}, or 12%. A better choice of mirror coating is protected silver, which can have R as high as 0.995. Then the 52 pass cell will transmit 0.995^{52}, or 77%, and the 104 pass cell will transmit 0.995^{104}, or 59%. The recent availability of the silver coatings considerably enhances the applicability of the multiple-pass cell to trace gas analysis.

A modification of the White system that increases its energy throughput was described by Horn and Pimentel (1971). They created two additional rows of images and doubled the number of passes by means of a retroreflecting pair of mirrors at the normal exit port. This increased the amount of available mirror surface at the in-focus end of cell. This allowed the use of a more collimated beam, thus increasing the energy throughput without enlarging the main collecting mirrors.

The third figure shows the four rows of images and the retroflecting mirror pair. The numbering is carried on from that used in the previous figure, but rather than the last image in a typical White cell exiting the system (image 28) it is redirected by way of the retroreflecting mirror pair so that the pattern of multiple-passing repeats itself and two additional rows of images fall onto the field mirror. If the mirror has an exit cut-out across from the input cut-out as shown in the bottom right-hand corner of the figure, the last pass (number 54) will exit the optical system well separated from the input beam. Using this system, Horn and Pimentel were able to work at 254 reflections at a 10 m base path, for a total path of 2.54 km.



Infrared Analysis, Inc. has carried the Horn and Pimentel concept one step farther by putting a second retroreflecting pair of mirrors on the input side of the field mirror. This creates six rows of images. In this case the field mirror shape can be the “inverted mushroom”, as is used in the simple White cell. The fourth figure here shows the distribution of images in the 6-row case when the total number of passes is 84. For atmospheric studies the image array in the fourth figure will allow an efficient and simple energy transfer between a Fourier transform spectrometer and a large long path cell. If the mirror is cut from a 20 cm. diameter blank, the multiple-pass cell will directly accept nearly all the output energy from the FT-IR instrument. There will be no need for having any transfer optics between the interferometer and the cell.



Low Throughput...

Or Light Limiting Experiments

All PIKE accessories are designed to offer the best possible optical performance (throughput) even under most difficult circumstances. Nevertheless, certain sampling configurations and experimental conditions will inherently limit the amount of infrared energy that reaches the detector after passing through the accessory. This may make performing a meaningful measurement difficult. Examples include wide range of grazing angle specular reflectance experiments (especially when a polarizer needs to be used), multiple reflection ATR measurements and selected, highly absorbing diffuse reflectance applications. From the applications perspective, typically very thin films and molecular orientation studies are affected by this problem. Low throughput causes signal-to-noise ratio (SNR) to worsen, thus limiting the measurement sensitivity.

There are two ways to counter this problem. One is to increase the number of scans collected under light limiting conditions. Coaddition of spectra improves SNR. Specifically, SNR $\propto (N)^{1/2}$, where N is the number of scans at a given resolution added together. Therefore, spectrum consisting of 100 coadded scans would have an SNR 10 times better $(100)^{1/2} = 10$ than a single scan spectrum. The second approach is to equip your FTIR spectrometer with a more sensitive detector. DTGS (deuterated triglicine sulfate) is the most popular detector used in mid-IR. It is simple, inexpensive, robust and excellent for the majority of applications. However, under light limiting conditions (less than 5% throughput) an MCT could be a much better option. MCT (mercury cadmium telluride) detectors are up to 10 times more sensitive than their DTGS counterparts. If considering the switch, please note that MCT detectors are more expensive, need to be cooled down with liquid nitrogen, and would saturate quickly under “normal” sampling conditions.

$1,000 Answer

Our previous quiz required linking of the nine items listed below with spectroscopy. The test proved to be relatively difficult and it did not produce too many correct answers. Nevertheless we have received a few creative entries and one of them is printed below.

PIKE TECHNOLOGIES

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STAMP

Spectroscopy is defined in the Webster's Dictionary in the following way - a: the production and investigation of spectra, b: the process or technique of using a spectroscope (an instrument that produces spectra), c: physics that deals with the theory and interpretation of interactions between matter and radiation. Well, what else do we need to know to determine obvious connections between PIKE stuff and spectroscopy?

I. Tootsie Roll – a good substitute for optical glue (next to the chewing gum). Excellent for re-attaching loose mirrors in your FTIR spectrometer.
II. Olympus Digital Camera - spectroscopy at its best with high quality optical components, including CCD "detector" (Charge-Coupled Device uses a light-sensitive material on a silicon chip to electronically detect photons).
III. "Laser Ball" with diode lasers – HeNe laser is used in FTIR spectrometers as an internal wavelength calibration standard.
IV. PIKE slide chart – invaluable item providing wealth of information on crystal materials used in IR spectroscopy. Connection with spectroscopy – obvious.
V. Beer Mug – a tool useful in astro spectroscopy for inducing stars - both by direct impact and/or (moderate) beer drinking.
VI. Kisses – wrapped in one of the best IR-reflecting materials. Often used for coverage of spectrometer mirrors and your loved ones.
VII. Pen set – basic set of tools often used for writing down new spectrometric theories and formulas.
VIII. Transmission sampling cards – well they are used to mount films and thin polymer samples in the spectrometer's sample compartment. Duuh...
IX. If one considers an eyeball to be a light detector, PIKE shades could be an optical filter for removing UV and part of the visible radiation from entering it.

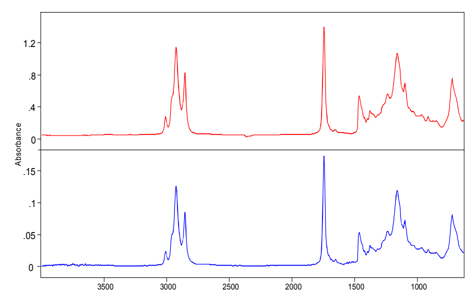
Well there you go. I think this quarter's $1,000 question is much simpler and less time consuming. Give it a shot…

The actual winning entry which was submitted by Joy Chen from Muhlenberg College will be published in its entirety on our web site (unfortunately, we did not have enough room to print it in the newsletter). Congratulations Joy – you've earned a $1,000 discount towards one of our FTIR accessories!

$1,000 Question

We have published close to 20 quiz questions in our newsletters to date. With so many data points, it seems like spectra (and art) related questions generated most of the answers. For these reasons, it is "back to spectra" in this issue of PIKE Reflections. The quiz (suggested by Dr. Kimberly Harris Abramo* of Shimadzu) is a variation on the question published a couple of issues ago. Here it goes: the two infrared spectra of an edible oil sample were collected under identical spectrometer settings. The spectral signatures in both cases were identical, but the absorbance levels were different. To enter, please identify the types of accessories the spectra were collected on and explain the difference in intensity.

*excluded from participation this time :(



The contest continues - all participants who correctly answer the above question will take a part in the drawing of a $1,000 discount toward any PIKE accessory of equal or higher value. Please send your answers by e-mail, mail or fax (please include information regarding the brand and model of your FTIR spectrometer). The drawing will be conducted on August 3rd, 2001 and the winner will be announced in the next issue of PIKE Reflections and on our web page, at:

http://www.piketech.com

Please mail, fax or e-mail your answers.

PIKE 2001 Show Calendar

National ACS Fall Meeting and Exposition, Chicago, IL
August 27-29, 2001 -- Booth #251

Eastern Analytical Symposium and Exhibition (EAS) - TBA