WINTER 2001 Vol. 6/1

PIKE REFLECTIONS

Useful Information for Practicing Chemists and Spectroscopists

Pittcon 2001 Surprises

DON'T MISS ANY OF THEM - VISIT US AT BOOTH NUMBER 642 AND BRING THIS NEWSLETTER TO PARTICIPATE

This year, PIKE Technologies will be participating in the Pittsburgh Conference for the 10th time. After eleven years in business and eleven years of systematic growth, we would like to make this year's Pittcon unique and memorable to all of our customers and PIKE personnel as well. We wish this conference to be an extension of our house warming party to which all of you were invited a couple of months ago. We hope that you will continue to celebrate with us our new (totally cool) building, successful relocation and truly exceptional year 2000. For this reason we are planning some changes in appearance and a couple of special events. This year we will be sporting a new booth design, new graphics and product orientation themes. Our new strategy will concentrate more on "solutions" and applications – with edible oil analysis, automation for the pharmaceutical industry, and simplification of FITR analysis, in the forefront. The new key product introductions will include a new version of the Diamond MIRacle - the best selling Single Reflection HATR ever. As a part of this introduction we will be issuing a "sampling challenge" (please see the announcement and the rules on the opposite side). We are also planning on a wide array of gifts and drawings – with all the items listed below. Please keep in mind that some of these items will be available in limited quantities. Some will be available only through participation in the "sampling challenge" and the subsequent drawing. Nevertheless, we hope that you will stop by – not only for material reasons but to say hello, discuss your application needs and see our new products. All of us at PIKE are looking forward to your visit.

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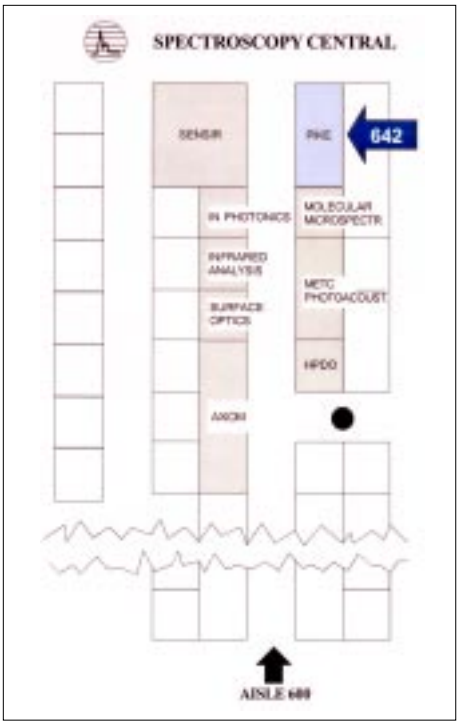
Map to Booth No. 642

As in the past few years, PIKE will be a part of Spectroscopy Central. This year the group members include:

- **Axiom Analytical** -- FTIR, NIR, UV-Vis sample interfacing and multiplexing equipment - booth 631.
- **High Pressure Diamond Optics (HPDO)** -- Diamond anvil cells - booth 634.
- **Infrared Analysis** -- Long path gas cells, reference spectra and gas analysis software - booth 639.
- **InPhotonics** -- Raman fiber optic sampling probes and accessories - booth 641.
- **The Molecular Microspectroscopy Laboratory (MML)** -- Research and educational services - booth 640.
- **MTEC Photoacoustics, Inc.** -- Photoacoustic detectors - booth 636.
- **PIKE Technologies, Inc.** -- Manual and automated accessories for FTIR spectrometers - booth 642.
- **SensIR Technologies** -- FTIR and diamond ATR systems - booth 542.
- **Surface Optics Corporation** -- Hand-held FTIR with a line of surface and powder analysis accessories - booth 637.



Sampling Challenge

Please visit our booth to participate in our sampling challenge. We will have an active FTIR spectrometer with a Diamond, Single Reflection HATR mounted in its sample compartment. *(continued on the next page)*

Spectra will be collected on site and displayed in real time on the screen located in the booth. The results will be available in digital format upon request (saved to a floppy disk). To participate, bring a sample that you believe is impossible to analyze by FTIR and let us try. Listed below are the "rules of the game":

- Samples must be capable (at least theoretically) of producing IR spectra. Inorganics (e.g. bricks, armored plates from Abrams tank and other stainless steel or aluminum, objects) do not qualify. We will be only looking at 4000 cm^{-1} to 650 cm^{-1} spectral range, therefore anything that absorbs in far-IR and NIR is also out.
- Please do not bring explosives. Although some of them produce nice IR spectra, the show management did not give us permission to include such products in this contest.
- Illegal drugs are also excluded (this includes marihuana! - (CA participants please note).
- Poisons - with the exception of raw oysters, cannot be analyzed at this location.
- Smelly things are not allowed - for our own sake and the sake of our neighbors.
- Gas cannot be analyzed with HATR - therefore, no gas please.
- Please be creative - if nothing else comes to mind you can challenge us with parts of your wardrobe, paper samples (US currency is typically difficult to analyze - but we can do it), alcoholic beverages, shoe soles and anything else that you can find in the close proximity of our booth.

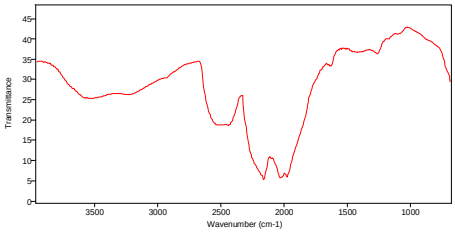
If we cannot collect a decent spectrum of the mutually agreed upon sample, we will give you at least one gift from the list in the center column on page 1. Also, the names of all the participants will be entered in the drawing for the Olympus digital camera (item number II). The drawing will be conducted at 3:00 PM in our booth on Thursday, and the results will be announced immediately afterwards (the participant does not have to be present). The results will also be published on our web site at www.piketech.com and in the next newsletter.

New Products

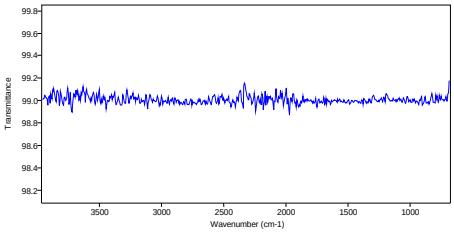
Diamond MIRacle AG™

MIRacle AG is a single reflection Horizontal Attenuated Reflection Accessory (HATR) which utilizes IIa synthetic diamond for the ATR sensing element (crystal plate manufactured under license and covered by patent numbers 5,200,609; 5,552,604 and 5,703,366). The diamond is mounted in the nickel plated, stainless steel base which is also teflon coated for added chemical resistance. Master Bond polymer system with compressive strength in excess of 10,700 PSI, temperature range of -60 deg. F to 300 deg. F and outstanding resistance to acids, bases, chlorinated solvents and alcohols is used to seal the junction between the diamond element and the mounting plate. MIRacle AG is equipped with a high pressure clamping mechanism, calibrated to deliver up to 10,000 pounds per square inch (PSI) of pressure. The clamp features an integrated pressure sensor and battery powered LCD for the real-time display of pressure applied to the sample. This unique design and precise readout allows reliable and reproducible application of pressure to measured samples. The clamp is also fitted with a safety clutch set to the maximum allowable pressure to prevent accidental crystal damage. All components of the MIRacle AG accessory, including the diamond plate and high pressure clamp are backward compatible and fit the existing product base.

The ruggedness of the diamond sampling interface allows for complete flexibility in the analysis of different material types. These include powders, hard/abrasive solids, corrosive materials, oxidizers and other chemically active products. And sampling with the MIRacle AG is extremely simple. The sample is placed directly on the diamond crystal and, if it is in the liquid state, it can be analyzed immediately. Solid samples must be pressed against the crystal for good contact. The clamp, with specialized press tips and digital pressure readout makes this task very easy.



Diamond MIRacle - Energy Throughput



Diamond MIRacle 100% T Line

MIRacle AG is most likely the best performing diamond HATR currently on the market. On average, it delivers a commendable 20% T to 25% T throughput. The minimum energy level in the area of diamond absorption lays between 3% T to 5% T. Such performance produces beautiful, low noise spectra covering the entire mid-IR range with virtually no noise in the critical 2000 cm^{-1} area. (See throughput and 100%T line spectra above). And last, but not least, is the exceptional value of this versatile product - the introductory price for the complete MIRacle AG system is less then $6,000! Stop by to check it out and bring your sample if you need immediate proof…

Edible Oil Analysis Kit

The recommended AOCS Practice Cd 14d-96 for determination of Isolated trans Geometric Isomers by Single Bounce HATR and recent FDA proposal for Trans Fatty Acids in Nutrition Labeling (21 CFR Part 101) created a need for a specialized FTIR accessory to assist in the above procedures.

PIKE Technologies just released the Edible Oil Accessory Kit that provides necessary components for AOCS procedure Cd 14d-96. The Kit includes heated, single reflection Horizontal ATR equipped with a ZnSe crystal (spectral range 4000 - 650 cm^{-1}). Two low voltage cartridge heaters are mounted directly under the crystal base to control the temperature of the crystal plate and the sample. An electronic unit that provides +/- 0.5 deg. C accuracy in the range from ambient to 120 deg. C drives the heaters. The controller features touch panel for data input as well as set point and real-time temperature display. The sample analysis is extremely simple. It requires collection of the background spectrum through the accessory with no sample in place. After that, a small amount of the sample is smeared across the crystal surface and the sample spectrum is collected. Cleaning is done in seconds with a cotton swab soaked in alcohol or other suitable solvent. The Kit is available in all commercial spectrometer configurations.

Automation

Automation of FITR sampling is one of PIKE Technologies' specialties. In the past few years our company developed several unique accessories for automated analysis of various products, including:

- analysis of raw powders with fiber probes (NIR)
- QC of powder samples in glass vials (NIR)
- direct analysis of powders by diffuse reflectance
- transmission measurements of polymer additives
- samples in Microtiter plates
- microscope mapping of semiconductor wafers (motorized microscope stage), and
- NIR analysis of pharmaceutical products in various shapes and sizes

All PIKE designs are based on our efficient R-theta mechanics (with Z motion component available upon request) and the XY stage in case of our 96 well plate (Microtiter plate) autosampler. All these products are driven by PIKE AutoPRO software that can also control the majority of commercial FTIR spectrometers (the slave mode, with the FTIR instrument in full control is also available). Although PIKE offers several standard autosampling configurations, most of the recent sales required certain system modifications to accommodate specific needs of our customers. Some of the changes included different sampling plate layouts, adjustments for sample shapes and sizes, special mapping options, and aperturing, among others. Nevertheless, we were glad to engage in these projects, as it is our philosophy to adjust our products to meet our customers' needs and expectations. A selection of PIKE automated accessories will be shown at our Pittcon booth. We will be thrilled to discuss your automation needs with you - even if displayed products do not exactly meet your requirements.

SciQuest and Fisher, UK

Effective February 1, 2001, majority of our products will be available for direct purchase on the Internet via SciQuest, one of the largest e-commerce sites serving analytical chemistry market. You can find our accessories, complete with pricing information on the SciQuest web site (www.sciquest.com) with the help of their powerful search engine. PIKE part numbers, accessory names and related key words can be used in the search process. Basic information for each product is provided. In addition, SciQuest offers a link to our web site which contains a complete electronic version of the PIKE catalog with images and full description of our products.

In the United Kingdom, our products are currently available through Fisher Scientific catalog and their distribution channels. Fisher Scientific is the leading UK's supplier of laboratory equipment. They distribute products from over 3000 suppliers to 40,000 laboratory customers in the UK and selected overseas markets.

The above companies were added to our distributors' list to increase availability of our products and simplify the buying processs.

Measuring Gases (I)

This issue of PIKE Reflections brings the first installment in the series committed to gas analysis. All articles in this series were written by Dr. Philip L. Hanst, President of Infrared Analysis, Inc, founded in 1985 and committed to promotion of the use of infrared absorption in gas analysis. During a 45 year career in spectroscopy, including positions with NASA and EPA, Dr. Hanst had a major role in the application of the infrared technique to atmospheric analysis. Using infrared spectroscopy, he and Dr. E. R. Stephens discovered the important family of peroxy nitrate air pollutants. He also was the discoverer of carbonyl sulfide in the atmosphere and identified it as the principal sulfur-carrying atmospheric molecule. For a number of years he was involved in pollution measurement from satellites and in the study of the stratospheric ozone depletion.

Back to basics – Absorbance Law

When radiation passes through a thin layer of an absorbing medium, (*dx*), the reduction of intensity, (*-di*), is proportional to the intensity, (*I*), the concentration of the absorber, (*c*), and the transition probability, (*a*):

$$-di = a.c.I.dx \qquad (1)$$

For sample thickness, *L*, and incident radiation intensity, *I(0),* the equation integrates to:

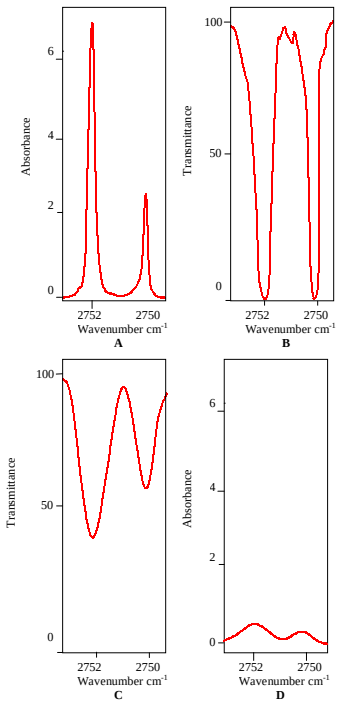
$$-\log I/I(0) = a.c.L \qquad (2)$$

The product *a.c.L* is called the absorbance, and the above equation (2) is the absorbance law. *I/I(0)* is the transmittance. People sometimes call equation (2) Beer's law, or the Lambert-Beer law, or the Beer-Lambert-Bouger law; but as a simple fact of physics, the name absorbance law is more appropriate.

Across a single absorption line, *a* varies with frequency according to the line shape. This shape changes with total pressure, but when *a* is integrated over a whole line, the integral is constant, independent of the total pressure.

Absorbance Law, Failures

If an instrument can reveal the true shape of spectral features, the true absorbance is revealed, and it always obeys the absorbance law (equation 2). Frequently, however, the instrumental resolving power is not high enough to reveal the true spectrum and one sees only an apparent absorbance, which does not obey the absorbance law. Basically, this deviation from the absorbance law happens because the transmittance saturates, or "bottoms out". This is illustrated by the figure here which applies to one of the line pairs of HCl when the concentration-pathlength product is 10,000 PPM-Meters and the total sample pressure is one atmosphere. Diagram A, on the left, shows the line pair with its correct line widths of 0.2 cm^{-1} and the correct absorbance values. (The 3-to-1 ratio between the line intensities results from the 3-to-1 isotopic ratio of Cl-35 to Cl-37.) Diagram B shows the actual transmittance values that would be measured for the sample when using an instrument with a spectral resolution of 0.1 cm^{-1} or better. Diagram C shows the transmittance values that would be measured by an instrument that has a spectral resolution of 1.0 cm^{-1}. Diagram D shows the absorbances calculated from the spectrum of Diagram C. The area under the two lines in Diagram D is much smaller than the area under the two lines in Diagram A. This



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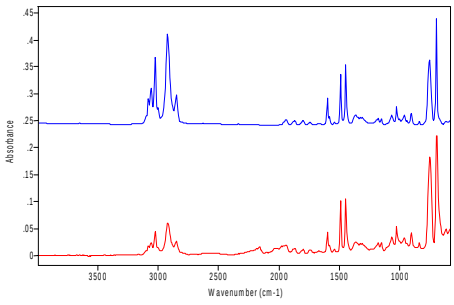
reduction of the area under the lines is the result of the instrumental smoothing over the "bottomed-out" transmittance lines. Those who use the expression Beer's Law would describe the difference in areas between diagrams A and D as a "Beer's law failure". The way to avoid this "Beer's law failure" is to measure only in spectral regions where the true transmittance is high enough so that there is no "bottoming-out" phenomenon. How high the apparent transmittance should be to avoid "bottoming out" depends on the spectral resolution, the total sample pressure and the degree of fine structure in the spectrum. Generally, it is advisable that the half-wavenumber of gas spectra should have apparent absorbances below 0.1 A.U. (log base 10).

$1,000 Answer

In the Q4, 2000 PIKE quiz our readers were presented with the following question:

The figure below shows two infrared spectra of a polystyrene sample collected under identical spectrometer settings. Yet the spectral intensities in both cases are different. To enter this quarter's contest, please explain the reason for this discrepancy and suggest a method for correcting the differences.



Cited below is an excellent answer sent to us by Mr. Joe Van Gompel, Senior Product Specialist at BOC Edwards. Mr. Van Gompel is also the winner of our $1,000 discount towards one of our accessories. **Congratulations!**

The spectra of the polystyrene film in Pike Reflections (Fall 2000) is pretty straightforward. The top spectrum is a transmission sample and the bottom spectrum is an ATR spectrum, probably ZnSe. Definitely not Ge, The trick is that, since the depth of penetration is wave-length dependent, you got less penetration at higher frequencies (3000 cm^{-1}) than you do at lower frequencies (1500 cm^{-1}). The absorbance values for the bottom spectrum are also reasonable for ATR. ATR correction routine available in all FTIR software packages can be used to compensate for this difference.

I must say that you did a nice job of eliminating the fringing in the transmission spectrum, Either that was taken at an angle off perpendicular to the beam, or you used a matte finish polysty-rene film.

The only thing that puzzles me is the distortion at 2100 cm^{-1}. The only place I've seen that before is with a diamond cell, Perhaps this was taken using a diamond press ATR accessory? The refractive indices are about the same between diamond and ZnSe as suggested above... Right on!

$1,000 Question

In the spirit of Pittcon and the never ending PIKE celebration of science and spectroscopy, we would like to present you with the following "off the wall" challenge. There are 10 items listed as awards on the front page of this newsletter. In order to enter our contest (and be eligible for a $1,000 discount drawing), please find a connection between each one of these items and spectroscopy. Entries will be judged for the level of imagination, sense of humor and scientific content (in this order). Entries should not exceed 50 pages of text (short sentence for each object will be sufficient). Good luck!!!

Please mail, fax or e-mail your answers. The addresses and phone numbers are listed below.

The contest continues - all participants who correctly answer the above question will take a part in the drawing of a $1,000 discount towards 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 May 1st, 2001 and the winner will be announced in the next issue of PIKE Reflections and on our web page, at:

http://www.piketech.com

PIKE 2001 Show Calendar

Pittsburgh Conference, Memorial Convention Center, New Orleans, LA
March 5-8, 2001 -- Booth #642:

National ACS Spring Meeting and Exposition, San Diego, CA
April 2-4, 2001 -- Booth #1254

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

Eastern Analytical Symposium and Exhibition (EAS)
Atlantic City Convention Ctr. -- Atlantic City, NJ
Sept. 30 - Oct. 4, 2001 -- Booth # TBA

Visit our web site at: http://www.piketech.com