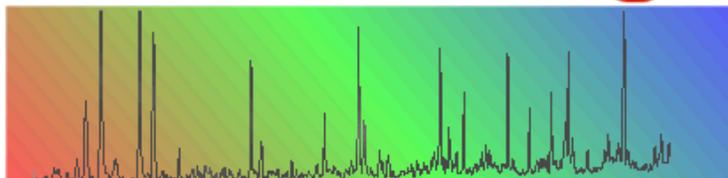




SAS-Chicago



March Meeting

Tuesday, March 11, 2008

The March 2008 meeting will be held at the Cook Hall Lobby at Northwestern University, Evanston IL, 60208. See the map on the last page for directions.

Poster Setup: 5:30 PM

Buffet Dinner: 6:00 PM

Awards: 7:30 PM

Poster Session on Spectroscopy

- 1) **Time-Resolved Ultraviolet Raman Spectroscopy of Coke Formation on H-MFI - Paula M. Allotta (Northwestern University)**
- 2) **Spectroscopic Kinetic and Thermodynamic Studies of Beta-Lactoglobulin with Model Membrane Vesicles - Ning Ge (University of Illinois at Chicago)**
- 3) **Analysis of an Unknown Deposit from an Indy Racing League (IRL) Honda Engine - Ashli Henderson (North Central College)**
- 4) **Comparison of vibrational circular dichroism (VCD) instruments. Development of a new dispersive VCD - Ahmed Lakhani (University of Illinois at Chicago)**
- 5) **Studies of Iron Loading in ZSM-5 Zeolite and Beginning Characterization of Iron-Oxygen Bonds Using Time-Resolved Ultraviolet Raman Spectroscopy and ICP - Dana Sauter (Northwestern University)**
- 6) **Chemistry of in-situ lubricated contacts - Evangeline Su (Northwestern University)**
- 7) **Analysis of Glass Haze from Display Cases at the Field Museum of Natural History - Nick Swanson (North Central College)**
- 8) **Conformational study of different beta-turns coupled to a cyclic disulfide bond to model the turn roles in b-hairpin stability - Ling Wu (University of Illinois at Chicago)**

Please make your dinner reservations for the upcoming meeting by email at sas.chicago@bigfoot.com by using the form on our web page < <http://www.sas-chicago.org/Online%20Registration%20at%20Northwestern.htm> > or by calling Slav Stepanovich at 847-421-2056. Leave your name, affiliation, and a telephone number. Please register by noon Friday, March 7th, so that proper arrangements can be made. If you can't attend, cancel by Monday noon.

Buffet includes:

Assortment of Miniature Deli Style Sandwiches Pasta Salad
Assorted Sweets of the Day Sliced Fresh Fruit
Assorted Soft Drinks & Bottled Water



NORTHWESTERN
UNIVERSITY

Dinner Cost Members: \$25

Students and Unemployed Members: \$10

Non-members: \$30

Time-Resolved Ultraviolet Raman Spectroscopy of Coke Formation on H-MFI

Paula M. Allotta and Peter C. Stair, Northwestern University

H-MFI suffers from deactivation due to carbonaceous buildup (coke) during the methanol-to-gasoline reaction. UV-Raman spectroscopy allows *in-situ* analysis of reaction products during coke formation. Previously heating and cooling was slow (minutes), and coke formation was too fast for real-time UV-Raman measurements. With an infrared laser, heating and cooling are much faster, 10^{-6} to 10^{-3} s. After heating, products and intermediates are trapped inside the zeolite pores and unable to react or desorb. Using this pulse-quench sequence intermediates can be detected by UV-Raman spectroscopy.

Infrared laser heating is a successful approach to forming coke within zeolite pores. With increasing laser power, reactants disappear and products appear. Approximately 90% of the methanol dosed into the pores undergoes some reaction via laser heating. No product desorption is detectable by mass spectrometry. The reaction temperature can be estimated from the beam power and zeolite thermal properties. From this estimation, a 1% increase in laser power is equivalent to a 100°C increase in temperature. These temperatures are consistent with reaction products seen in conventional heating experiments. Methanol and zeolite peaks decrease due to reaction and optical self-absorption, respectively. A peak at 1551 cm^{-1} signals the presence of polyolefins with 6-8 repeat units. Peaks at 1380 and 1624 cm^{-1} show that naphthalene forms initially, but changes in their relative intensity indicate formation of higher aromatics. Spectral changes can be seen by varying the silicon/aluminum ratio. When the aluminum content is decreased relative to the previous discussion, no detectable 1380 cm^{-1} peak is present. This shows that the reaction path is different, and that naphthalene is not formed. Further, under the same reaction conditions much less coke is formed in the low aluminum content zeolite. The absence of product desorption implies polyaromatic formation takes place inside the pores and not on the external surface of the zeolite.

Spectroscopic Kinetic and Thermodynamic Studies of Beta-Lactoglobulin with Model Membrane Vesicles

Ning Ge and Timothy A. Keiderling, University of Illinois at Chicago

Bovine beta-lactoglobulin (β -LG) is a lipocalin protein found in mammalian milk. In the native state, its secondary structure is dominated by β -sheet, though it has the propensity to form α -helices based on secondary structure predictions. Helical formation has been observed during an early stage of β -LG folding from random coil to native state. Our recent studies showed β -LG can adopt a significant fraction of α -helical conformation upon mixing with synthetic phospholipid vesicles. The thermal and chemical stabilities of the protein lipid vesicle complex were followed by far-UV CD and fluorescence for BLG in the presence of anionic lipids with various carbon chain lengths. The orientations of the alpha helix and beta sheets in membrane-bound BLG in the presence of various lipids are studied and compared by use of polarized attenuated total reflection (ATR) FTIR spectra. We also studied the kinetics of the binding process for β -LG and various vesicles using simultaneous stopped-flow CD and fluorescence. Their comparison showed at least two different phases in the kinetic mechanism. Electrostatic, hydrophobic interactions and lipid packing affect the rate constants. Combining the results of these studies, we have developed a model in which β -LG binds and unfolds on the membrane surface and then forms helical components, which insert into the membrane bilayer.

Index of Newsletter Sponsors



For the best in nebulizers, sample introduction components, torches, and spray chambers, call: **1-800-MEINHARD**

Meinhard Glass Products

Geoff Coleman, Ph.D., Vice President

700 Corporate Circle, Suite A
Golden, Colorado 80401-5636 USA
Tel: 303.277.9776 Fax: 303.216.2649
gcoleman@meinhard.com www.meinhard.com



† **Steve Bouffard, Ph.D.**
Technical Sales Specialist
FTIR, UV/Vis/NIR, GC, GC/MS
HPLC, DSC, TGA, TMA and DMA

† **PerkinElmer Life and Analytical Science**
2000 York Road, Suite 132
Oak Brook, IL 60523
Phone: (800) 762-4000
Fax: (203) 944-4914
Steven.Bouffard@perkinelmer.com
www.perkinelmer.com

www.meinhard.com

email

www.perkinelmer.com

email

Analysis of an Unknown Deposit from an Indy Racing League (IRL) Honda Engine

Ashli Henderson and Jeffrey A. Jankowski, North Central College

During a routine engine inspection following an Indy Racing League (IRL) event, Honda and IRL officials discovered a black deposit in the cylinders and on the top surface of the pistons of an engine in an individual race car. This particular type of residue had never before been observed in any previous inspections. Therefore, it was suspected that material from some other component of the fuel or air-intake system had entered the cylinders or some additive had been introduced to the fuel system that resulted in the formation of the deposit. Honda and the IRL have requested an analysis of this residue to determine the chemical nature of the material to identify its source and determine how it entered the engines cylinders.

Fourier Transform-Infrared (FT-IR) spectroscopy was used to analyze the sample leading to the identification of particular functional groups present. This in turn has led to the identification of compounds that are known to be used in the automotive industry. In particular, the identified chemistries are consistent with those used in additives and gasoline and oil formulations to trap dirt and sludge and clean engine deposits from fuel system components. Subsequent analysis of specific samples of these chemistries reveal spectra that are consistent with that of the deposit found in the race car engine. Thus, preliminary results indicate that the deposits found in the race car engine are consistent with known chemistries that are designed to produce such deposits but that are not used in IRL competition.

Comparison of vibrational circular dichroism (VCD) instruments. Development of a new dispersive VCD.

Ahmed Lakhani, Peter Malon, and Timothy A. Keiderling*

University of Illinois at Chicago

We have designed and built a new dispersive vibrational circular dichroism (VCD) instrument, optimized for the measurement of mid IR bands such as the amide I and amide II vibrational modes of peptides and proteins (C=O stretching, and CN stretching – NH bending, respectively) by maximizing sensitivity in the spectral range from 1800 cm^{-1} to 1400 cm^{-1} . The major design factors were to make a compact VCD instrument for biological molecules, to increase signal to noise (S/N) ratio, to simultaneously collect the signals resulting from sample transmission and polarization modulation and to digitally normalize these signals following a design of Diem¹. The design used a 0.3m monochromator, intense carbon-rod light source (~2500K) and a band width limited (< 8 μm) MCT detector. The instrument uses 3 lock-in amplifiers whose outputs monitoring transmission and modulation are digitized and interfaced via RS232 to a personal computer which also controls the monochromator using LabView. The new dispersive VCD spectrometer will be compared with our previously constructed analogue-based dispersive VCD². We have collected spectra for peptides and proteins having different (alpha-helix, beta-sheet, and random coil) dominant secondary structures and for identical samples on several instruments, with comparable resolution and total measurement time.

Index of Newsletter Sponsors

(Click on card or URL to link to Sponsor's web site)

	5225 Verona Road Madison, WI 53711-4495
Andrew M. Haefner, Ph.D. Sales Engineer Molecular Spectroscopy	(800) 648-5456 (815) 479-0127 fax (815) 479 0887 direct
Nicolet FT-IR, Raman FT-NIR, and Microscopy	
andrew.haefner@thermo.com	www.thermo.com

www.thermo.com

[email](mailto:andrew.haefner@thermo.com)

	
Rina K. Dukor, Ph.D. President / CEO	
Bio Tools, Inc. 950 N. Rand Rd. Unit 123 Wauconda, IL 60084 USA	Tel: (847) 487-5500 Fax: (847) 487-5544 Toll Free: (866) BTOOLS1 E-mail: rkdukor@btools.com Website: www.btools.com

www.btools.com

[email](mailto:rkdukor@btools.com)

Studies of Iron Loading in ZSM-5 Zeolite and Beginning Characterization of Iron-Oxygen Bonds Using Time-Resolved Ultraviolet Raman Spectroscopy and ICP.

Dana Sauter and Peter C. Stair, Northwestern University

ZSM-5's 3-D pore structure of straight and sinusoidal channels allows for selectivity of products and structural stability. Transition metal loading into the zeolite pores has been extensively studied, producing results that indicate that iron loaded zeolite exhibits a strong ability to convert methanol to gasoline, even in the presence of water. In this study, differing Si/Al ratios of H-MFI were loaded with iron using the chemical vapor deposition method with the goal of characterizing the iron-oxygen bond within the zeolite pore. Initial studies were concerned with reproducible loadings having an expected Fe/Al= 1/1. ICP was performed on these samples to determine the Fe/Al, and to investigate a correlation between the Si/Al ratio and the amount of iron loaded into a sample. It was found that not only do the Brønsted sites within the zeolite bond with iron, but the silanol groups on the edges of the zeolite, which greatly affect the Si/Al for low alumina containing zeolites. Zeolites with ratios of 33.5 and 128 load with an Fe/Al=1/1, after the excess iron from the silanol groups are taken into account. A Si/Al = 90 does not load uniformly and consistently loads with higher iron content. IR spectroscopy was used to ensure that all the Brønsted sites in these samples were bonding with the iron and ²⁷Al-NMR was used to show that the aluminum was not migrating out of the framework. UV Raman spectroscopy, with an excitation of 244nm, was utilized to characterize the iron-oxygen bonds within the pores and to take an in depth look at structural differences between Si/Al ratios.

Chemistry of *in-situ* lubricated contacts

Evangeline Su, Peter Stair, and Jane Wang, Northwestern University

Our research interest is the non-traditional application of ultra-violet Raman spectroscopy to the study of tribology, which is the study of friction, lubrication, and wear. We have pursued the design of an *in-situ* method to probe lubricant degradation features, such as pressure gradients in a high pressure point contact, and lubricant degradation polymerization chemistry. Tribology, generally considered a mechanical engineering and materials science sub-discipline, has had to turn to theory to satisfy curiosity about what happens *in-situ*, but the limitations of theory, unfortunately, leave many questions open to debate. Some of those questions motivate our work: 1) what temperatures and pressures do lubricant molecules experience in a concentrated contact, 2) are those pressures and temperatures uniform across the ball bearing's surface, and 3) what causes lubricant degradation to occur. In stage 1 of this project, we have built a specialized Raman spectroscopic instrument comprised of an ultra-violet Raman microscope coupled to a tribotesting device. Now in stage 2, we are probing the 3-D and 2-D imaging spaces to compare the average and local chemistry at this fluid film interface between a steel ball bearing and a sapphire window. The instrument design and some early data will be presented.

Index of Newsletter Sponsors

(Click on card or URL to link to Sponsor's web site)

	<p>JERRY SHKOLNIK Executive Sales Representative Chicago, IL</p> <p>TELEDYNE INSTRUMENTS Leeman Labs A Teledyne Technologies Company</p> <p>6 Wentworth Drive Hudson, New Hampshire 03051 Phone: 847.478.0452 Fax: 847.478.0467 Toll Free: 1.800.634.9942 Cell: 603.493.6176 E-Mail: JShkolnik@Teledyne.com www.LeemanLabs.com</p>
---	--

www.leemanlabs.com

[email](#)

<p>Renishaw Inc 5277 Trillium Blvd. Hoffman Estates, IL 60192 Tel (847) 286-9953 Fax (847) 286-9974 www.renishaw.com</p>	 apply innovation
<p>Tim Prusnick Midwest Sales Manager - Spectroscopy Products Division Email timothy.prusnick@renishaw.com</p>	 

www.renishaw.com/spectroscopy

[email](#)

Analysis of Glass Haze from Display Cases at the Field Museum of Natural History

Nick Swanson and Jeffrey A. Jankowski, North Central College

The Field Museum of Natural History in Chicago has thousands of artifacts on exhibit, most of which are contained in glass display cases. The cases themselves can date as far back as the time of the World's Columbian Exposition in 1893, with varying artifacts displayed inside. Over the years, the Conservationists and Exhibitors at the Field Museum have witnessed an unusual problem with the glass of the display cases throughout the Museum. Over time an opaque film, or haze, invariably deposits on the inside surface of the exhibit glass. The rate of development of the haze is as varied as the locations and content of the cases themselves. The goal of this project is to identify the haze and its point of origin in order to assist in maintaining the integrity of the artifacts present and to facilitate the cleaning and maintenance of the cases.

Using FT-IR we have been able to identify the main component of the glass haze from one particular case as glycerin. While the mechanism by which the haze forms is still under investigation, the presence of glycerin is consistent with the construction of the American Alligator diorama that is housed within the display case. Preliminary analysis of the haze from two other display cases has also been conducted. Although the qualitative, macroscopic appearance of the haze in other display cases is similar to that found in the American Alligator case, the physical properties, microscopic appearance, and IR spectra indicate that the chemical composition is different for each of the cases investigated thus far.

Conformational study of different beta-turns coupled to a cyclic disulfide bond to model the turn roles in β -hairpin stability

Ling Wu, Dan McElheny, Ahmed Lakhani and Timothy A. Keiderling, University of Illinois at Chicago

Turns and loops are often classified as the third type of secondary structural element due to their role as the connecting unit between α helices and β sheets in globular proteins. The most common turns connecting adjacent β -sheet strands are β turns. Cyclic peptides are useful structural models for the study of β turn conformation, because they can be easily synthesized and characterized by several spectroscopic methods. We synthesized a selection of cyclic peptides of the general formula $\text{Cyclo}[\text{Ac-Cys-Val-Xxx-Gly-Lys-Cys-NH}_2]$ which were cyclized by a disulfide bond between the two cysteines. Their steric structures were characterized by NMR spectroscopy and restrained MD simulations. Furthermore, we compared their thermal stability using optical (IR, ECD and VCD) spectra and extended the result of this study to aid understanding of the folding energetics of a 12-residue β -hairpin model-Trpzip2. Additional CD and IR studies, based on variation of the Trpzip2 turn sequence from -Asn-Gly- to -^DPro-Gly-, show that the more stable -^DPro-Gly- turn causes the hairpin to follow a different unfolding mechanism for secondary and tertiary structure. Trp residues at non-H-bonded positions on different strands facilitate hydrophobic interactions and consequently formation of the Trpzip β -hairpin and that mutation to residues promoting formation of a more stable turn will additionally stabilize Trp interaction between two strands.

Index of Newsletter Sponsors

(Click on card or URL to link to Sponsor's web site)

<p>Chip McCauslin Sales Representative-Atomic Spectroscopy Analytical Chemist Chicago Area Optical Spectroscopy Instruments</p>  <p>Varian Analytical Instruments 2700 Mitchell Drive Walnut Creek, CA 94598 Phone: 800.926.3000 Ext. 3025 Fax: 925.945.2360 http://www.varianinc.com chip.mccauslin@varianinc.com</p>

www.varianinc.com

[email](mailto:chip.mccauslin@varianinc.com)

 <p>PerkinElmer, Inc. 2000 York Road, Suite 132 Oak Brook, IL 60523 Phone: 800 762-4000 ext. 132 Fax: 630 556-4523 Cell: 630 222-8921 jeff.kukuk@perkinelmer.com www.perkinelmer.com</p>

www.perkinelmer.com

[email](mailto:jeff.kukuk@perkinelmer.com)

Directions and Parking

- For maps and directions to Northwestern University's Evanston Campus go to www.northwestern.edu/visiting/maps.
- After 4 pm, parking is free in the lot across from the Technological Institute on Sheridan Road or behind Cook Hall (accessible by Lincoln Street).
- A bridge connects the Technological Institute and Cook Hall. Posters will be in the Cook Hall Lobby.

