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Attention Presenters: Check this final program to verify the schedule of your talk or poster. Changes may have occurred since the preliminary program.

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FACSS International Office
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WELCOME TO FACSS 2010

On behalf of the Governing Board and Executive Committee, it is my pleasure to welcome you to Raleigh, North Carolina for the 37th annual meeting of the Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) and the national meeting for both the Society for Applied Spectroscopy (SAS) and the Coblentz Society.

FACSS 2010 is jam-packed with opportunities to network with fellow scientists and learn about the latest developments and trends in analytical chemistry and spectroscopy. Plenary speakers include seven leading scientists receiving national and international awards presented at FACSS: Christy Haynes (Arthur F. Findeis Award), Richard McCreery (Charles Mann Award), Marc Porter (ANACHEM Award), Paul Gemperline (William F. Meggers Award), Kay Niemax (Lester W. Strock Award), Boris Mizaikoff (Coblentz Clara Craver Award), and Martin Moskovits (Ellis R. Lippincott Award). Uli Hacksell from ACADIA Pharmaceuticals will give a plenary presentation on analytical chemistry in drug discovery, and Robert Houk from Iowa State will talk about 30 years of ICP-MS. In addition to the awards symposia, the technical program includes topics ranging from dielectrophoretic separations to atomic spectroscopy. Our goal is for FACSS to continue to be a leading venue for leading analytical scientists to debut their most innovative new discoveries.

The Exhibits and Workshops at FACSS complement the main technical program, providing information about commercial instrumentation, publications, societies and services as well as practical training for professional scientists. FACSS also provides an Employment Bureau as well as several events and awards specifically for student attendees. Social events at FACSS start with SAS events on Sunday and end with the FACSS Wednesday Evening Event – "Analytical Chemistry….It's All Fun and Games at FACSS."

Raleigh is great city to enjoy when the technical sessions and conference receptions wind down for the evening or week. The Raleigh Convention Center and conference hotels are located in the center of the city within a short walk of numerous restaurants, parks, museums and nightspots. The free R-Line bus service stops at the Convention Center and conference hotels and serves the downtown area.

The continued success of FACSS depends on the many volunteers who generously give their time to the meeting and organization. The 2010 team has worked hard to assemble an outstanding technical program and exhibit and to keep the FACSS organization running smoothly and quietly behind the scenes. Please take a moment to thank these volunteers when you see a ribbon attached to their name tag.

Even as we begin FACSS 2010 in Raleigh, we are looking forward to future meetings and planning how to improve and grow the conference. FACSS exists to serve its seven member societies. In order to meet that central mission, the Governing Board and Executive Committee need feedback and ideas from you, the people who make up the meeting. We are constantly searching for new ideas to improve the FACSS conference, new technical areas to add to the program, and especially new people to take on leadership positions organizing the technical program and running FACSS. Please take a few moments during the week to discuss your ideas and interests with the current FACSS officers so the conference can better serve you.

Doug Gilman
2010-2011 Governing Board Chair

GENERAL INFORMATION

LOCATION: All conference symposia and exhibits will be held at the Raleigh Convention Center. Workshop locations are indicated on your workshop name badge.

PROGRAM. This printed program contains titles and abstracts as submitted by the authors. It is not possible to edit these submissions.

SPEAKERS. There will be an LCD projector for each symposium. Speakers must supply their own computer with their presentation. Please show up 30 minutes before your session begins. Each speaker should adhere to the time allotted for the talk.

POSTER SESSIONS.

Sunday SAS Sponsored Student Poster Session – *Ballroom Lobby, Convention Center*

- 7:00 – 9:00 pm SAS Poster Session & FACSS Welcome Mixer

Monday Poster Session – *Ballroom Lobby, Convention Ctr.*

- 9:00 – 10:30 am - Poster Session. Set up posters between 7:30 – 8:00 am and remove by 5:00 pm. Odd numbered poster boards present 9:00 – 9:45 am; even numbered poster boards present 9:45 – 10:30 am
- 4:00 pm – Poster viewing and break

Tuesday and Wednesday Poster Session – *Exhibit Hall – Ballroom B*

Posters remain up all day on their designated day. Set up posters between 7:30 – 8:00 am and remove by 5:00 pm on Tuesday and Wednesday

- 9:00 – 10:30 am – Poster Session. Odd numbered poster boards present 9:00 – 9:45 am; even numbered poster boards present 9:45 – 10:30 am
- 1:30 pm – Poster viewing and dessert break

Thursday Poster Session – *Ballroom Lobby, Convention Ctr.* Posters remain up all day. Set up posters between 7:30 – 8:00 am and remove at 4:00 pm.

- 9:00 – 10:30 am – Poster Session. Odd numbered poster boards present 9:00 – 9:45 am; even numbered poster boards present 9:45 – 10:30 am
- 1:30 pm – Poster viewing and break

FACSS WORKSHOPS. A list of workshops, descriptions, and the locations begin on page 36. You must register for a FACSS workshop at the conference registration desk.

EMPLOYMENT BUREAU. The bureau is located in Room 201 of the convention center. The hours are Monday through Wednesday, 9:00 am to 5:00 pm and Thursday 9:00 am – 3:00 pm. See page 4 for additional information.

EXHIBITS. The exhibition is located in Ballroom B and will be open as follows: See page 28 for details.

Monday (Opening Reception)	5:30 – 7:30 pm
Tuesday	9:00 am – 4:30 pm
Wednesday	9:00 am – 3:00 pm

BREAKS.

Monday

- 9:00 – 10:30 am & 4:00 – 4:20 pm – *Ballroom Lobby*

Tuesday

- 9:00 – 10:30 am – *Exhibit Hall*
- 1:30 pm – Dessert Break – *Exhibit Hall*
- 4:00 pm – *Exhibit Hall*

Wednesday

- 9:00 – 10:30 am – *Exhibit Hall*
- 1:30 pm – Dessert Break – *Exhibit Hall*

Thursday

- 9:00 – 10:30 am & 1:30 – 2:00 pm – *Ballroom Lobby*

INTERNET ACCESS. Complimentary wireless internet access will be available in the Exhibit Hall and Ballroom Lobby.

REGULATIONS. The following regulations are in the best interest of the conference.

1. There is no smoking in any conference area.
2. An official name badge is required at all times.
3. No advertising may be placed in the conference area.
4. Only official exhibitors may display in the Exhibit Hall.
5. No distribution of product/meeting literature in sessions.

SPECIAL EVENTS.

SUNDAY

4:30 – 7:00 pm **"What's Hot" Exhibitor Presentations**, *Ballroom A*

7:00 – 9:00 pm **Welcome Mixer and SAS Sponsored Student Poster Session**, SAS and Coblentz Student Award Presentations, *Ballroom Lobby*

MONDAY

8:00 am **ACS Division of Analytical Chemistry Arthur F. Findeis Award Plenary Lecture**, **Christy L. Haynes,** University of Minnesota**,** *Ballroom A*

12:30 pm **Free Lunch and Employment Discussion for Students, sponsored by SABIC Innovative Plastics.** *Room 307*

4:20 pm **Plenary Lecture**: The Role of Analytical Chemistry and Spectroscopy in Drug Discovery and Development: Challenges and Opportunities**, Uli Hacksell,** ACADIA Pharmaceuticals, *Ballroom A*

5:30 – 7:30 pm **Reception for Exhibit Opening** (wine, beer, light hors d'ouvres) *Ballroom B*

TUESDAY

8:00 am **Charles Mann Award for Applied Spectroscopy**; **Richard McCreery**, National Institute for Nanotechnology, Canada, *Ballroom A*

8:30 am **ANACHEM Award, Marc D. Porter**, Nano Institute of Utah, *Ballroom A*

4:20 pm **Plenary Lecture:** Celebration of 30 Years of ICP-MS, **Robert S. Houk**, Iowa State University*, Ballroom A*

WEDNESDAY

8:00 am **SAS Applied Spectroscopy William F. Meggers Award**; **Paul Gemperline**, East Carolina University, *Ballroom A*

8:30 am **SAS Applied Spectroscopy Lester W. Strock Award; Kay Niemax**, BAM*, Ballroom A*

4:30 pm **FACSS Awards Presentation,** *Room 305A*

4:40 pm **Plenary Lecture:** Distinguished Service: Becoming an Oxymoron? **Alex Scheeline**, University of Illinois at Urbana-Champaign, *Room 305A*

6:00 pm **Wednesday Evening All Inclusive Event,** complimentary for all conferees**.** *Ballroom A, page 27*

THURSDAY

8:00 am **Coblentz Clara Craver Award**; **Boris Mizaikoff,** University of Ulm, Germany, *Ballroom A*

8:30 am **Ellis R. Lippincott Award; Martin Moskovits,** University of Carolina, Santa Barbara, *Ballroom A*

COMPANION REGISTRATION. Does not include access to symposia or exhibit hall other than for special events. Cost is $55 and includes the following: **Sun.** Evening Welcome Mixer*,* **Mon.** coffee/pastries 9:00 AM *and* Exhibit Hall Opening Reception*,* **Wed.** Evening Event.

EVENTS OF SPECIAL INTEREST TO STUDENTS

Sunday Evening, *Raleigh Convention Center, Ballroom Lobby*

- Welcome Mixer; 7:00 – 9:00 PM
- SAS Sponsored Student Poster Session; 7:00 – 9:00 PM
    - SAS and Coblentz Student Award presentations.

Monday through Thursday

- FACSS Student Poster Awards will be presented daily.

Monday through Thursday

- Employment Bureau, *Room 201*
  Monday – Wednesday 9:00 AM – 5:00 PM; Thursday 9:00 AM – 3:00 PM

SPECIAL INVITATION TO STUDENT ATTENDEES

- **Monday 12:30 pm - Free Lunch and Employment Discussion for Students.**
  Hosted by SABIC Innovative Plastics
  Eat lunch and chat with professionals from a wide range of professional fields (academic, government, chemical industry, pharmaceuticals, goods and services, etc.) It's a unique opportunity to ask questions, get helpful tips, and discuss topics that relate to your specific career-seeking situation within the current job market. *Room 307*. ***Sign up at conference registration desk***.

FACSS EMPLOYMENT BUREAU

The FACSS Employment Bureau is now online so you can manage your employment efforts anywhere you can connect to the internet! The Employment Bureau is a free service to both job seekers and employers that provides job and applicant listings, message boards, and interviewing booths.

How to register: From the FACSS website (www.facss.org), click on Employment in the top menu.
You can create a Job Target account to manage resumes, search employment opportunities and set up personal job alerts. Post your resume online, anonymously if desired, and create a job alert to email new postings directly to your in-box.

At the conference:
Location: The employment bureau is located in Room 201 in Convention Center
Hours: 9:00 AM – 5:00 PM, Monday – Wednesday and 9:00 AM – 3:00 PM on Thursday

Check your Job Target in-box to follow-up on your employment leads. Internet access will be available in the Employment Bureau, the Exhibit Hall, and the Ballroom Lobby. Two desktop computers and a printer will also be available in the Employment Bureau to help you in your job/candidate search.

FACSS ORGANIZATION

Member Organizations of FACSS
American Chemical Society, Analytical Division
American Society for Mass Spectrometry
ANACHEM
International Society of Automation – Analysis Division
Coblentz Society
Royal Society of Chemistry
Society for Applied Spectroscopy

FACSS is the National Meeting for the Society for Applied Spectroscopy and the Coblentz Society

2010 Chair Persons and Executive Committee

Governing Board Chair	**S. Douglass Gilman**, *Louisiana State University* E-mail: sdgilman@lsu.edu
Governing Board Chair Elect	**Ian Lewis,** *Kaiser Optical Systems, Inc.*
Past Governing Board Chair	**Becky Dittmar,** *3M*
Second Past Governing Board Chair	**Gary Brewer,** *ABB*
Secretary	**Christopher Palmer,** *University of Montana*
Treasurer	**Scott McGeorge,** *Transition Technologies, Inc.*
Exhibit Chair	**Mike Carrabba,** *The Hach Company* E-mail: mcarrabba@hach.com
General Chair	**David Butcher,** *Western Carolina University* E-mail: butcher@email.wcu.edu
Program Chair	**André Sommer,** *Miami University* E-mail: sommeraj@muohio.edu
Workshop Chair	**Brandye Smith-Goettler,** *Merck*
Employment Chair	**Matthew Schulmerich,** *University of Illinois*

2010 Program Section Chairs

Awards	**Pavel Matousek,** *Rutherford Appleton Lab*
Atomic Spectroscopy	**Steven Ray**, *Indiana University*
Bioanalytical	**Neil Lewis**, *Malvern Instruments*
Chemometrics	**Barry Lavine**, *Oklahoma State University*
Chromatography	**Neil Danielson,** *Miami University*
Mass Spectrometry	**David Muddiman**, *North Carolina State University*
Molecular	**Linda Kidder***, Malvern Instruments*
Nanotechnology	**Shouzhong Zou,** *Miami University* and **Frank Zamborini,** *University of Louisville*
Process Analytical	**James Rydzak,** *GlaxoSmithKline*
Raman	**Ian R. Lewis,** *Kaiser Optical Systems, Inc.*
Royal Society of Chemistry	**John M. Chalmers,** *VS Consulting*
Surface Plasmon Resonance	**Jean-Francois Masson**, *University of Montreal*
Terahertz	**Gil Pacey,** *Miami University*
SAS Student Poster Session	**Bonnie Saylor** and **Victor Hutcherson**, *Society for Applied Spectroscopy*

FACSS CHAIRS

GOVERNING BOARD CHAIR

Doug Gilman

Louisiana State University

Doug first attended FACSS in Philadelphia in 1992. He gave the first talk of his career at FACSS as a student in Detroit in 1993. Since 2000 when FACSS was in Nashville, Doug has given many talks and organized and chaired several symposia for the meeting. He was the Bioanalytical Section Co-Chair for FACSS 2002 in Providence with Chuck Henry, and he was Awards Chair in 2005 in Quebec City. Doug was the Program Chair for FACSS 2006 in Lake Buena Vista, FL. He is a member of SAS and the Analytical Division of ACS.

Doug grew up in Exeter, California in the San Joaquin Valley just below Sequoia National Park. Doug started his path towards becoming a scientist at Harvey Mudd College in Claremont, CA. He carried out undergraduate research in organic synthesis under the direction of Bill Daub, and he completed a B.S. degree in Chemistry in 1989. Doug then headed east for graduate school at Penn State University. At Penn State, he figured out that he liked bioanalytical chemistry better than organic synthesis and was lucky enough to land in Andy Ewing's group.

Doug left State College, PA to take a postdoctoral position with Bruce Hammock where he was an NIH postdoctoral fellow in the Departments of Entomology and Environmental Toxicology at UC Davis. In 1997, Doug headed to the Southeast to start as an Assistant Professor at the University of Tennessee in Knoxville. While at the University of Tennessee he received an NSF CAREER Award (2001) and was promoted to Associate Professor (2003). In 2004 Doug moved further south to Louisiana State University in Baton Rouge where he is currently a faculty member in Chemistry. Doug's research program at LSU focuses on the development of new bioanalytical techniques to study enzyme inhibition and protein aggregation. He also studies electroosmotic flow dynamics and magnetic bead aggregation in capillaries and microfluidic devices.

Most of Doug's free time today is devoted to activities of interest to his children, Rohin (10) and Priya (9). Doug's wife, Indu, also an analytical chemist, is an Assistant Professor and Director of the Proteomics and Metabolomics Facility at the Pennington Biomedical Research Center in Baton Rouge. Doug and his wife are still loyal Penn State fans, and they enjoyed Penn State's bowl victory over LSU in January. Also in 2010, Doug and Indu finally published their first paper together after meeting in the Ewing laboratory at Penn State almost 20 years ago.

GENERAL CHAIR

David J. Butcher

Western Carolina University

David J. Butcher is currently Professor of Chemistry and Associate Dean of the College of Arts and Sciences at Western Carolina University (WCU) in Cullowhee, NC. He is married to Dr. Karen Butcher and has two children, Emily 19 and Neil 17, with whom he enjoys leisure time. He received his bachelor's degree in 1982 from the University of Vermont. After three years of employment at Pfizer and Bowdoin College, he received his Ph.D. from the University of Connecticut in 1990. His graduate work, conducted under the direction of Robert G. Michel, involved the development of instrumentation for laser excited atomic fluorescence and ionization spectroscopies. He joined the faculty at WCU in 1990 as an Assistant Professor of Chemistry, was promoted to Associate Professor in 1997, was promoted to Professor in 2001, and became Department Head in 2002. Prof. Butcher became Associate Dean in April, 2004. Prof. Butcher has more than 50 publications in a variety of areas of analytical chemistry, including graphite furnace atomic absorption spectrometry, diode laser atomic absorption spectrometry, and ion trap mass spectrometry. Along with Prof. Joseph Sneddon, he is co-author of the volume "A Practical Guide to Graphite Furnace Atomic Absorption Analysis." His current research interests include environmental analytical chemistry; currently he is involved in a phytoremediation project to remove lead and arsenic from the soil at a housing development in Western North Carolina. He has also been involved in a number of novel teaching innovations in general and analytical chemistry. He serves as Associate Editor for Book Reviews of the *Microchemical Journal*, and Associate Editor for Education for *Spectroscopy Letters*. He received the 1998 WCU University Scholar Award as the outstanding researcher. He serves on the Editorial Boards of Microchemical Journal, Spectroscopy Letters and Applied Spectroscopy Reviews. He served as Chair of the American Microchemical Society Undergraduate Award Committee and is currently Chair of the A.A. Benedetti-Pichler Award Committee. In 2001, he served as Program Chair for 28th FACSS meeting held in Detroit, MI. In 2009, he became Editor-in-Chief of *Analytical Letters*. In 2010, he became Editor-in-Chief of *Instrumentation Science and Technology.* He is the General Chair for the 37th FACSS meeting to be held in Raleigh, NC in 2010.

FACSS CHAIRS

PROGRAM CHAIR

Andre' J. Sommer

Miami University

Andre' J. Sommer (Andy) earned an M.S. degree in Physical Chemistry from Lehigh University in 1981. His research was conducted with Dr. Roland Lovejoy on the infrared spectroscopy of atmospheric gases for planetary modeling. In 1985 he earned a Ph.D. in Analytical Chemistry from Lehigh University, under the direction of Dr. Henry Leidheiser. His doctoral research focused on the study of corrosion mechanisms using Raman microspectroscopy. Upon leaving Lehigh, he served a one year postdoctoral appointment with IBM in their Systems Technology Division. In 1986 he joined the Molecular Microspectroscopy Laboratory (MML), under the direction of Dr. Jack E. Katon, as the Assistant Director to the laboratory and was promoted to the facility's Director and Assistant Professor, within the Department of Chemistry, in 1995. More recently, he was promoted to Associate Professor in 2001 and Professor in 2005. During his tenure, as the Assistant Director to the MML, he helped build the facility into an internationally recognized laboratory that specializes in the molecular analysis of microscopic particles, or microscopic spatial domains on large samples. Highlights of his research include the development of an FT-Raman microprobe and fundamental studies of spatial resolution in infrared microspectroscopy. From 1995 to the present, his group pioneered the development of infrared ATR microspectroscopic imaging and demonstrated the method's potential for biomedical research. Andy's research interests are focused on the development and applications of molecular microspectroscopy for chemical analysis and materials characterization. Specific areas involve the design, construction and characterization of novel Raman, luminescence and infrared microscopes with the goal of overcoming the wavelength limitation. Current goals are to employ the techniques in a quantitative capacity, specifically for biological imaging. Andy has authored and/or coauthored 72 papers, 7 book chapters, and has given 180 presentations. Through his association with the MML, he has collaborated with more than three hundred fifty scientists employing molecular microspectroscopy to solve problems related to fundamental research and/or industrial processing. He has 29 years experience in molecular spectroscopy and microspectroscopy. In addition, he served on the faculty of the Annual Workshop on Molecular Microspectroscopy at Miami University for sixteen years, and directed this workshop for six years. Andy has been an active member of the Society for Applied Spectroscopy and the Coblentz Society for the past sixteen years. He currently is the Treasurer of the Coblentz Society and has served as a board member and President. He served as an organizer and presider of Molecular Microspectroscopy Symposia for the Federation of Analytical Chemisty and Spectroscopy Societies over the years of 1993-1995 and 2000-2003.

EXHIBITS CHAIR

Mike Carrabba

The Hach Company

Mike Carrabba is currently the Global Director of Open Innovation for the Hach-Lange family of companies. He received his B.S. in Chemistry from Salem State College in 1981 and his Ph.D. from Tufts University in 1985. Mike's graduate work was conducted under the tutelage of Dr. Jonathan Kenny and focused on the utilization of laser-induced fluorescence to examine ultra-cooled gas phase molecules in a supersonic jet molecular beam. After graduate school, Mike joined EIC Laboratories where he eventually became Vice-President for the Spectroscopy Division. He conducted a variety of research programs, including photoelectrochemical etching of semiconductors, fiber optic chemical sensors and state-of-the-art Raman spectroscopy. During this time, he introduced the use of holographic filters for Raman spectroscopy and developed numerous types of Raman instrumentation and techniques, several of which resulted in U.S. patents. After leaving EIC, Mike joined Chromex, Inc., a manufacturer of Raman spectroscopy systems, as Marketing Manager and most recently was the OEM Division Manager at Jobin Yvon, Inc. Mike has been very active in FACSS over the years serving as Governing Board Chair (2002), Program Chair (2000), Exhibits Chair (2006 -2011), Program Section Chair for Raman (1992-1999, 2001), Chairperson of the Long Range Planning Committee (1999 – 2008) and as a member of the Governing Board. In 2003 he received the ASTM Award of Merit for his 12 years of service as the Chairman of the ASTM Subcommittee on Raman spectroscopy. In 2004, he received the FACSS Charles Mann Award for Applied Raman Spectroscopy and in 2007 the Williams Wright Award for Applied Spectroscopy. He is also a member of the Society for Applied Spectroscopy (SAS) and Coblentz Society. On the home front, his wife, Professor Mary Widmark Carrabba of Southern Oregon University, a highly skilled Infrared microscopist and the former treasurer for SAS, complements Mike's Raman background.

PROGRAM and CONFERENCE SPONSORS

FACSS greatly appreciates the support it receives from its sponsors.

ANALYTICAL METHODS/INSTRUMENTATION

Innovative Scientific Solutions
Spectral Energies
Spectra-Physics

ATOMIC SPECTROSCOPY

Agilent Technologies
Bruker AXS
Elemental Scientific
Glass Expansion
Innovative Scientific Solutions
IonSense
Meinhard Glass Products
PerkinElmer Life & Analytical Sciences
Prosolia
RSC, Journal of Atomic Analytical Spectrometry
Thermo Fisher Scientific

EXHIBIT OPENING

Agilent Technologies

FINDEIS AWARD

Nikon Instruments

GENERAL

B&W Tek
Bruker Daltonics
Cobalt Light Systems
Taylor & Francis
WITec Instruments

CHARLES MANN AWARD

Dorothy Mann, on behalf of the Mann Family

MASS SPECTROMETRY

Bruker Daltonics
Eksigent Technologies
LEAP Technologies
Thermo Fischer Scientific

MOLECULAR SPECTROSCOPY

Council for Near Infrared Spectroscopy

NANOSCIENCE

CH Instruments
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PROCESS

Mettler Toledo AutoChem
Society for Applied Spectroscopy

RAMAN

BioTools
HORIBA Scientific
Kaiser Optical Systems
NIST
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Spectroscopy Magazine / Advanstar
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STUDENT EMPLOYMENT LUNCH AND PANEL DISCUSSION

SABIC Innovative Plastics

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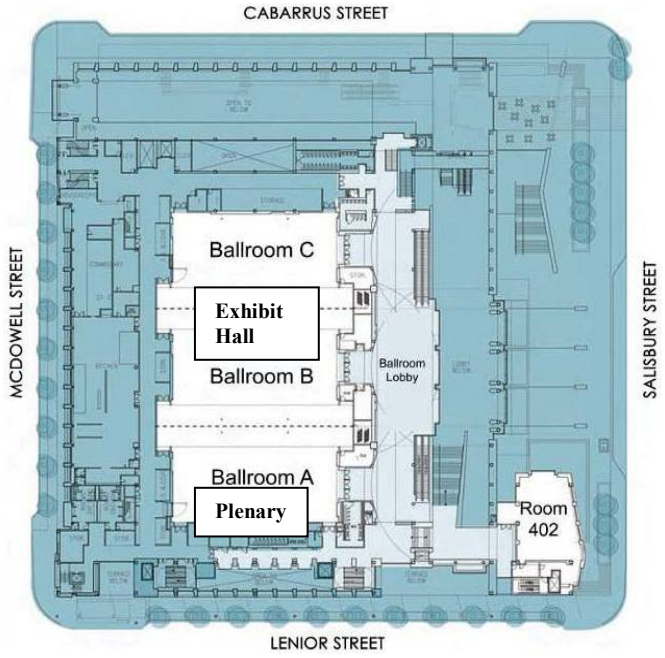
FLOOR PLANS



Level 4

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Plenaries
Poster Sessions
Exhibit Hall

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Symposia Rooms
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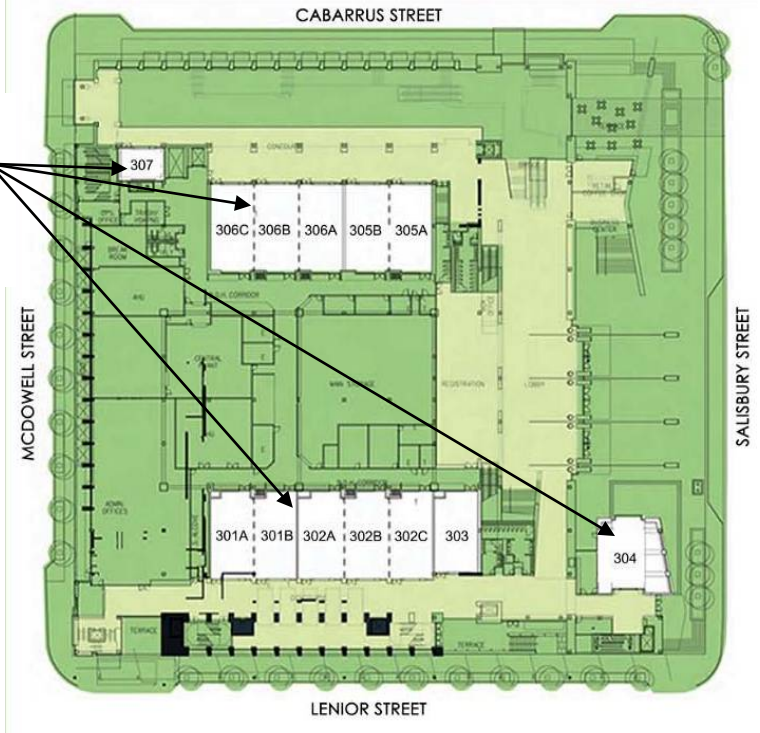
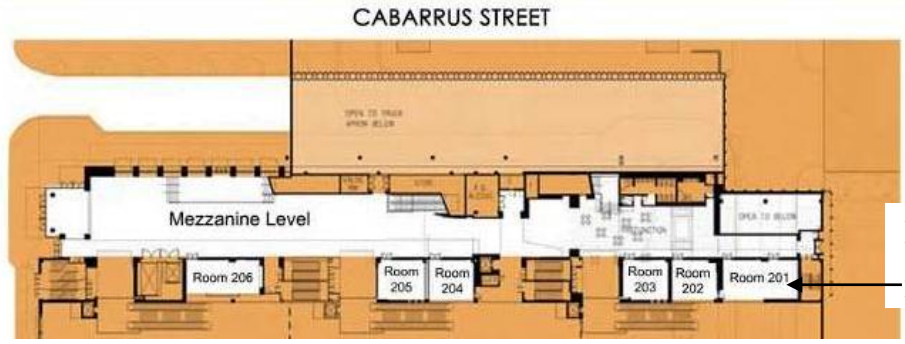
304,305A, 305B, 306A,
306B, 306C, 307





Level 2

Employment Bureau

FACSS AWARDS

DISTINGUISHED SERVICE AWARDS

Awarded to an individual(s) for recognition of exceptional, long-term service to the FACSS organization. The 2010 recipients have served with excellence in many different capacities and contributed to the continuing success of FACSS through consistent dedication and sacrifice.

Awards will be presented Wednesday, 4:30 pm, *Room 305A*

Scott W. McGeorge

Transition Technologies, Inc.

Scott received his B.Sc. degree in chemistry from the University of Waterloo with a minor in computer science in 1980. He was able to combine aspects of these disciplines at McGill University in Montreal where he studied with Dr. Eric D. Salin. He received his Ph.D. in 1985 for applications of image sensor technology for atomic spectroscopy. After graduate school, he worked for an instrumentation company with funding from an Industrial Postdoctoral Research Fellowship. He later managed a corporate group assigned to the development of multiple dispersion ICP spectrometer systems employing echelle optics and photodiode array detection. He has authored or co-authored 13 publications. He served as Exhibit Chair for the Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) conference from 1996 to 2005. He is currently the FACSS Treasurer. Scott founded Transition Technologies, Inc. in 1994 and has developed a business model geared to providing quality scientific support for the products that the company distributes across Canada. The Company's analytical product line was initially dedicated to productivity enhancements for ICP optical and mass spectrometry. Strategic technology selections have expanded the analytical portfolio to include LIBS and biochemistry analyzers , to name a few. In 1999 TTI expanded into the life science arena and currently expends a significant effort servicing the genetics community providing solutions for mutation detection, genotyping, and nucleic acid analysis using denaturing HPLC, High Resolution Melt technology and/or sophisticated software tools for DNA sequence analysis. In 2005 the Company added automatic bio-reactor sampling systems with integration support for a variety of downstream technologies. Very recently TTI has begun to develop technical enclosure solutions to enhance sustainability, safety, and sample/equipment protection. Scott is the father of three engaging children and his interests and accomplishments include playing snare drum for the Streetsville Pipes and Drums, sailing, a 2nd degree black belt in Goju-Jitsu Ryu, a 1st degree black belt in Jiu-jitsu, collecting rare and unusual corkscrews, scuba diving (advanced and Nitrox certified), soccer, and tropical fish husbandry (cichlids – hapllochromine and mbuna). He loves working with the FACSS group, has made many wonderful friends, and looks forward to watching the conference grow, prosper and make a real impact on science.

Alexander Scheeline

University of Illinois

Alexander Scheeline is Professor of Chemistry at the University of Illinois at Urbana-Champaign. He is a native of Hollidaysburg, PA. He attended Michigan State University, where he did research with S. R. Crouch in heteropolymolybdate kinetics. He received his Ph.D. from the University of Wisconsin-Madison under the direction of J. P. Walters, focusing on plasma diagnostics of bipolar pulse spark discharges. Following a National Research Council post-doctoral fellowship at the National Institute for Standards and Technology, he was assistant professor at the University of Iowa before moving to Illinois. He served as Program Officer in Analytical and Surface Chemistry at the National Science Foundation for one year. Current research interests are in instrument development (ultrasonically-levitated drops as microreactors), reactive oxygen species, oscillatory reactions, nonlinear dynamics, and noise-induced hearing loss. Scheeline first attended FACSS II in Indianapolis as a graduate student. With only 4 exceptions, he has attended every meeting since. With Richard Sacks and Joel Goldberg, he sparked the annual "Rodney Danger(ous)field Symposium" on high voltage, high current discharges for elemental analysis. He was assistant program chair (assisting Matt Klee) for the last of a string of meetings in suburban Philadelphia, and then program chair for the 1986 meeting in St. Louis. In response to Tomas Hirschfeld's premature demise, he was instrumental in establishing an award for inventive graduate students in Hirschfeld's honor and memory. Together with Syd Fleming, he participated in the beginnings of electronic publishing in *Applied Spectroscopy* by submitting the FACSS Preliminary Program to the Journal that year. He and Fleming helped negotiate the joint meeting the Pacific Conference in 1991. He chaired the governing board in 1989. In subsequent years, he served on the board, representing ACS or SAS, served on the long range planning committee, and continued to arrange symposia. In addition to his activity in support of FACSS, Scheeline is a member of the American Chemical Society, The Electrochemical Society, Society of Electroanalytical Chemists, Society for Free Radical Biology and Medicine, Association for Research in Otolaryngology, Coblentz Society, Optical Society of America, the EPR Society, Sigma Xi, Phi Kappa Phi, Alpha Chi Sigma, and a fellow of the Society for Applied Spectroscopy. He is on the editorial board of *Biophysical Chemistry* and editor of the *Journal of the Analytical Sciences Digital Library*. He is one of six members of the Chemistry faculty at Illinois to teach the standard undergraduate curriculum at the Faculty of Chemistry, Hanoi University of Science. In his "copious" free time, he is a history buff, playing popular piano and vocal music from the early 20th century and the occasional ditty by Tom Lehrer. A notable character flaw is a tendency to punning.

PREVIOUS AWARDEES:

1994 L. Felix Schneider
1993 Edward Brame and Syd Fleming
2001 David Coleman
2003 Jeanette Grasselli Brown
2009 Paul Bourassa and Mike Carrabba

FACSS AWARDS

The Tomas Hirschfeld Scholar and the FACSS Student Awards recognize outstanding contributions by individuals who are Ph.D. and M.Sc. candidates.

FACSS STUDENT AWARD

Jacob Shelley

Indiana University

Presentation: Tuesday, 10:30 am
Room 302B

Jake Shelley was born in Albuquerque, NM in 1984. He earned his B.S. in Chemistry from Northern Arizona University in Flagstaff, AZ in 2005. Jake's research at NAU, while working under Dr. Diane Stearns, was focused on finding metal-DNA adducts in Chinese hamster ovary cells using ICP-AES. Jake worked at Los Alamos National Laboratory for four summers on a wide range of projects including metallomics using X-ray fluorescence, using nanoporous silica as a matrix-free MALDI substrate, and method development for detecting a wide range of radioactive materials. Jake joined Prof. Gary Hieftje's group in 2005 where his current research focus has been in the development, characterization, and application of novel plasma ionization sources for ambient, molecular mass spectrometry. His particular focus has been on the Flowing Atmospheric-Pressure Afterglow (FAPA) source. In 2009, Jake received the Center for Analytical Instrumentation Development (CAID) Fellowship from Purdue University to study in Aston Labs under Prof. R. Graham Cooks. This collaborative effort has been focused on directly comparing these novel plasma sources and coupling them with a miniature mass spectrometer for portable, direct, and sensitive analyses of sample surfaces. In addition, Jake has given 8 invited presentations at both national and international conferences, as well as developed a short course on Ambient Desorption/Ionization Mass Spectrometry. He is also an author of 12 research publications and 2 patents.

TOMAS HIRSCHFELD SCHOLARS

Ishan Barman

Massachusetts Institute of Technology

Presentation: Wednesday, 2:50 pm
Room 305A

Ishan Barman is pursuing his Ph.D. in Mechanical Engineering and is the Lester Wolfe Fellow at the George R. Harrison Spectroscopy Laboratory, Massachusetts Institute of Technology (MIT) in Cambridge, USA. His interests are in disease diagnosis, laser spectroscopy and chemometrics. In particular, his doctoral work involves developing new methodologies for blood disorder diagnostics, especially diabetes and malaria. Previously, he obtained his B.Tech and S.M. in Mechanical Engineering from Indian Institute of Technology (IIT) Kharagpur and MIT, respectively. He has so far published 5 papers with 5 more under review. His work has also led to the filing of a total of 5 patents in USA and India.

Anil Kumar Kodali

University of Illinois

Presentation: Wednesday, 2:30 pm
Room 305A

Anil Kodali is a Mechanical Engineering Ph.D. student in Chemical Imaging and Structures Laboratory headed by Dr. Rohit Bhargava at University of Illinois, Urbana Champaign. He has obtained a B.Tech degree from Jawaharlal Nehru Technological University, India and M.S from University of Illinois in mechanical engineering programs. His research interests are in design of novel optics and instrumentation to facilitate massively multiplexed biomolecular sensing. His current research focus is in two major paths: 1) rational design and fabrication of surface-enhanced Raman scattering-based probes using metal-dielectric nanoparticle constructs and 2) Narrow band mid-IR reflection filters to design a discrete frequency IR imaging instrument. In his doctoral work, to date he has authored 5 research publications and is in the process of filing for 2 patents. He is also a regular attendee and presenter at FACSS and other conferences in USA. Earlier, he has won MechSE alumni award for excellence in teaching and Colgate-Palmolive award for excellence in research at University of Illinois.

TOMAS HIRSCHFELD AND FACSS STUDENT AWARDS - Call for Applications for 2011

The Tomas Hirschfeld Scholar(s) and the FACSS Student Awards recognize the most outstanding papers submitted to FACSS by a graduate student. Recipients will receive financial support to help them attend the 2011 FACSS meeting in Reno, NV (October 2 - 6). In 2010 one FACSS Student Award and two Tomas Hirschfeld Scholars are being presented. In order to have your presentation considered for a Tomas Hirschfeld Scholar Award or FACSS Student Award, students should submit their abstract using the FACSS web site submission form and indicate on the dropdown menu on the form their interest in these awards.

The submission process involves submitting an abstract, completing the web site submission form, and submitting three sets of the following:

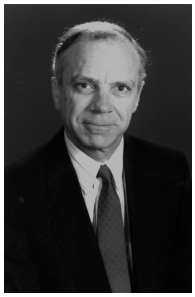
a) the form, available on the FACSS web site
b) a 250 word abstract of the work to be reported
c) two letters of nomination, one by the student's mentor. An explanation of the inventive contributions by the student to the work should be given. Creativity was a primary characteristic of Tomas's work, and thus should be a characteristic of the awardee
d) a copy of the candidates resumé
e) a copy of the candidate's graduate transcript
f) copies of reprints and/or preprints of research accomplished.

The recipients will be included in a session highlighting young scientists and their work.

The FACSS Web site will begin accepting abstracts and applications for FACSS student awards in January 2011. Go to www.facss.org to submit an application.

CHARLES MANN AWARD

For Achievements in the Field of Applied Raman Spectroscopy



Richard L. McCreery

University of Alberta

Presentation: Tuesday, 8:00 am
Ballroom A

Richard L. McCreery is currently Professor of Chemistry at the University of Alberta, with a joint appointment as a Senior Research Officer at the National Institute for nanotechnology. Until 2006, he was Dow Professor of Chemistry at the Ohio State University. He received his B.S. in chemistry from the University of California, Riverside, in 1970, and Ph.D. under Ralph Adams at the University of Kansas in 1974. His research involves spectroscopic probes of electrochemical processes, the electronic and electrochemical properties of carbon materials, and molecular electronics. Much of the research involves collaborations with materials scientists and engineers, as well as surface scientists and electrochemists. Current grant support includes projects funded by the National Science Foundation (US), an Alberta Ingenuity Scholar Award, an NSERC Discovery grant, and a CFI/SEGP funded Hybrid Device Facility in the NINT clean room. He leads an effort at NINT and UofA to investigate hybrid devices for molecular electronics, which combine existing CMOS technology with new electronic and optoelectronic devices containing active molecular components. McCreery has written over 200 refereed publications, including a book entitled "Raman Spectroscopy for Chemical Analysis" and eight U.S. Patents, with two of those extended to Europe (PCT) and Japan. Six of the patents are licensed by ZettaCore, Inc (Denver) to commercialize molecular memory devices having higher data density, longer retention, and lower cost than conventional microelectronic memory.

ANACHEM AWARD

The ANACHEM Award is presented annually to an outstanding analytical chemist based on activities in teaching, research, administration or other activity, which has advanced the art and science of the field.

Marc D. Porter

Nano Institute of Utah

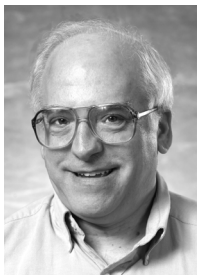
Presentation: Tuesday, 8:30 am
Ballroom A

Marc Porter, USTAR Professor of Chemistry, Chemical Engineering, Bioengineering, and Pathology, is the Director of the Nano Institute of Utah. He has been the director of the Microanalytical Instrumentation Center at Iowa State University (1993-2003); the Institute for Combinatorial Science at Iowa State University (2002-2006); and the Center for Combinatorial Sciences, Biodesign Institute at Arizona State University (2006-2007). He has been involved in several entrepreneurial endeavors: Advanced Analytical Technologies, Inc. (Ames, IA); CombiSep, Inc. (Ames, IA); and Concurrent Analytical, Inc. (Honolulu, HI), which has two subsidiaries – Nanopartz, Inc., a world leader in the innovation and manufacturing of spherical gold nanoparticles and gold nanorods, and Directed Bioflux, Inc., which focuses on newly patented or proprietary technologies that reduce incubation times in heterogeneous immunoassays. Research in his laboratory is focused on fundamental and technological issues in the movement of nanotechnology to clinical diagnostics.

SOCIETY FOR APPLIED SPECTROSCOPY AWARDS

DISTINGUISHED SERVICE AWARD

Recognizing members for their long-time service to the society



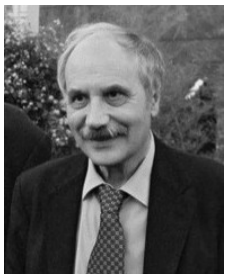
Alexander Scheeline

University of Illinois at Urbana-Champaign

Alexander Scheeline is Professor of Chemistry at the University of Illinois at Urbana-Champaign. He is a native of Hollidaysburg, PA. He attended Michigan State University, where he did research with S. R. Crouch in heteropolymolybdate kinetics. He received his Ph.D. from the University of Wisconsin-Madison under the direction of J. P. Walters, focusing on plasma diagnostics of bipolar pulse spark discharges. Following a National Research Council post-doctoral fellowship at the National Institute for Standards and Technology, he was assistant professor at the University of Iowa before moving to Illinois. He served as Program Officer in Analytical and Surface Chemistry at the National Science Foundation for one year. Current research interests are in instrument development (ultrasonically-levitated drops as microreactors), reactive oxygen species, oscillatory reactions, nonlinear dynamics, and noise-induced hearing loss. Scheeline joined SAS as a student in 1975. He has served the Society in numerous roles including tour speaker, representative to the FACSS governing board, member of the nominating committee, member (and chair) of the Strock Award committee, member of the publications committee, member (and chair) of the tour speaker committee, frequent member of the team judging student posters at FACSS, Book Review Editor for *Applied Spectroscopy*, and Secretary. In collaboration with mentors and students, he has twice been recipient of the W. F. Meggers Award. Together with Syd Fleming, he participated in the beginnings of electronic publishing in *Applied Spectroscopy* by submitting the FACSS Preliminary Program to the Journal electronically in 1986. He is proud to have been a friend of Bill Fateley's, and enjoyed service on the search committee that hired SAS Executive Director Bonnie Saylor. He became a Fellow of the Society in 2008. In addition to membership in SAS, Scheeline is a member of the American Chemical Society, The Electrochemical Society, Society of Electroanalytical Chemists, Society for Free Radical Biology and Medicine, Association for Research in Otolaryngology, Coblentz Society, Optical Society of America, the EPR Society, Sigma Xi, Phi Kappa Phi, and Alpha Chi Sigma. He is on the editorial board of *Biophysical Chemistry* and editor of the *Journal of the Analytical Sciences Digital Library*. He has been program chair and governing board chair of FACSS. He is one of six members of the Chemistry faculty at Illinois to teach the standard undergraduate curriculum at the Faculty of Chemistry, Hanoi University of Science. In his "copious" free time, he is a history buff, playing popular piano and vocal music from the early 20th century and the occasional ditty by Tom Lehrer. A notable character flaw is a tendency to punning.

HONORARY MEMBERSHIP AWARD

Recognizing those individuals who have made exceptional contributions to spectroscopy.



Nicolo' Omenetto

University of Florida

Nicolo' Omenetto joined the University of Florida in Gainesville at the end of 2001 and is currently Professor in the Department of Chemistry. After earning his *Laurea* in Chemistry from the University of Padova (Italy) in 1964, he was appointed as Assistant Professor at the University of Pavia (Italy) in 1969, and received the *Libera Docenza* in Spectrochemistry in 1971. From 1979 until 2001, he was appointed at the Joint Research Centre of the European Commission in Ispra (Italy). He did postdoctoral work with Jim Winefordner from 1971-73 and 1978-79. The research interests of Dr. Omenetto have been directed towards the theory and applications of atomic and molecular spectroscopic methods of analysis, with particular emphasis to the use of tunable lasers and to the development of techniques such as atomic and molecular fluorescence, atomic ionization, photo-thermal, photo-fragmentation, and laser induced breakdown spectroscopy (LIBS). In addition to these developments, fundamental diagnostic studies flames and plasmas have been pursued, improving the understanding of the interaction between the laser and the atomic/molecular systems investigated. One publication on LIBS modeling was recognized with the Spectrochimica Acta Atomic Spectroscopy Award in 2001. Dr. Omenetto has delivered many keynote and plenary lectures, and has published over 240 scientific papers in peer-reviewed journals, including 5 book chapters and 2 Monographs (with J.D. Winefordner). He has edited two books: "Analytical Laser Spectroscopy", Wiley, N.Y. (1979) and (together with J. Parks) the book "Resonance Ionization Spectroscopy 1990", Institute of Physics Conference Series No. 114, IOP Publishing Ltd., Bristol, U.K. (1991). Since 1994, he is one of the editors of Spectrochimica Acta Part B, Atomic Spectroscopy. In September 2006, he has been elected *Fellow of the Society of Applied Spectroscopy*. Dr. Omenetto has been the 2009 recipient of the Lester W. Strock Award.

SOCIETY FOR APPLIED SPECTROSCOPY AWARDS

LIPPINCOTT AWARD

Given to honor the memory of Ellis R. Lippincott for significant contributions to vibrational spectroscopy. The medal is sponsored jointly by the Society for Applied Spectroscopy, the Coblentz Society, and the Optical Society of America.

Martin Moskovits

University of California, Santa Barbara

Presentation: Thursday, 8:30 am, Ballroom A

Martin Moskovits is Professor of Chemistry at the University of California, Santa Barbara where he also served as Susan and Bruce Worster Dean of Science from 2000 to 2007. From 2007 to 2010 he served as Chief Technology Officer of API Technologies Corp. (ATNY.OB), a publicly traded company (OTCBB:APIA) specializing in advanced electronics, magnetics and nanoptics for defense and communications applications, and President of its NanoOpto subsidiary. He is also a founder of Spectra Fluidics, a startup company dedicating to developing sensors based on microfluidics. He has degrees in Physics and Chemistry from the University of Toronto, where he received his PhD in 1971. In 1968 he founded an electronics company in Toronto, which was sold in 1970. From 1971-73 he was employed as a materials scientist by Alcan International, Kingston Ontario. Returning to the University of Toronto in 1973, he eventually attaining the rank of Professor of Chemical Physics in 1982. From 1993-1999 he was Chair of the Department of Chemistry at U of T. He was a member of the University of Toronto Governing Council. He was also the founding Director of the program in Nanoelectronics for the Canadian Institute for Advanced Research. In research he is best known for his work in plasmonics, and especially its application to surface-enhanced Raman and for developing porous aluminum oxide as a nanotemplate for metal and semiconductor nanowires. He is the author or co-author of over 270 technical papers and inventor on 18 patents. He has delivered over 280 invited talks at national and international meetings and conferences. He has supervised the research of over 100 graduate students and postdoctoral fellows. In research he is known for his pioneering work in nanotechnology, developing nanofabrication techniques in anodic alumina templates, single-nanowire-based field-effect-transistor sensors, and enhanced optical and plasmonic effects in gold and silver nanostructures. He is a Fellow of the American Association for the Advancement of Science, the Optical Society of America and the Royal Society of Canada, and Vice Chair of the US Department of Energy's Basic Energy Sciences Advisory Committee. He was a Guggenheim Fellow in 1987, winner of the 1993 Gerhard Herzberg Award of the Spectroscopy Society of Canada, 1993 Royal Society of Chemistry (London) award in Surface and Colloid Science, 1995 Johannes Marcus Marci Medal of the Czech Spectroscopy Society, and the 2008 NanoTech Briefs Nano 50 Innovator award.

LESTER W. STROCK AWARD

Established by the SAS New England section to recognize an author(s) of an outstanding paper or series of papers.

Kay Niemax

Federal Institute of Materials Research & Testing (BAM)

Presentation: Wednesday, 8:30 am, Ballroom A

Prof. Dr. Kay Niemax is Wilhelm-Ostwald-Fellow at the Department I (Analytical Chemistry and Reference Materials) of the Federal Institute for Materials Research and Testing (BAM) in Berlin (Germany). His current fields of research are plasma and laser spectrochemistry. He studied physics, chemistry and mathematics at University of Kiel (Germany) and received his Diploma and Ph.D. in physics from the Institute of Experimental Physics at Kiel University in 1970 and 1972, respectively. 1979 he became a lecturer in physics (Habilitation) and in 1984 professor of physics in Kiel. 1979-1980 he was Visiting Fellow at JILA in Boulder (Colorado), one of the worldwide leading research institutes in atomic and molecular physics, home of three recent Nobel Price winners in Physics. 1985 he became head of the Elemental Analysis Department at Institute of Spectrochemistry and Applied Spectroscopy in Dortmund and moved to Stuttgart (Germany) in 1993 where he received the Chair in Physics of the University of Stuttgart-Hohenheim. 1997-2010 he was director at ISAS-Institute for Analytical Sciences in Dortmund (Germany) and professor for Physical-Chemical Analysis at the Faculty of Physics of the Technical University of Dortmund. From 1970 to 1985 his major research interest were in the field of plasma and laser physics, laser spectroscopy of atoms and small molecules, spectral line broadening and atomic collisions processes at thermal energy. In particular, the laser spectroscopic technique to derive level isotope shifts of atoms from highly resolved Rydberg series lines and the investigations of pressure broadening and shift of Rydberg levels are closely connected with his name. Since 1985 Prof. Niemax is working in the field of spectrochemistry and applied spectroscopy. He is developing laser based techniques for chemical analysis of solid, liquid and gaseous samples. Very recently he succeeded to quantify the chemical composition of nanoparticles by plasma spectrometry. In the future this new technique will be of high importance in those fields where the elemental composition and mass either of airborne nanoparticles or nanoparticles in liquids have to be controlled and measured. Prof. Niemax has published over 230 papers in peer reviewed international journals and made numerous scientific contributions to proceedings and books. He gave more than 120 invited plenary and keynote talks at international conferences and numerous invited talks at universities and research institutes in Germany and abroad. He is serving as reviewer for the major scientific journals and many funding agencies in Germany and abroad, such as DFG, National Science Foundation (USA) or Department of Energy (USA). He is member of several review panels evaluating scientific institutions in Germany and abroad. Furthermore, he is a Fellow of the Society of Applied Spectroscopy (USA).

SOCIETY FOR APPLIED SPECTROSCOPY

WILLIAM F. MEGGERS AWARD

Recognizing the author(s) of an outstanding paper appearing in Applied Spectroscopy

Presented for "Methods for Kinetic Modeling of Temporally Resolved Hyperspectral Confocal Fluorescence Images" Authors: Cutler, Patrick J.; Haaland, David M.; Andries, Erik; Gemperline, Paul J. Volume 63, Issue 2, (February 2009), pp. 153-163 and "Systematic Method for the Kinetic Modeling of Temporally Resolved Hyperspectral Microscope Images of Fluorescently Labeled Cells" Authors: Cutler, Patrick J.; Haaland, David M.; Gemperline, Paul J. Volume 63, Issue 3, (March 2009), pp. 261-270.

Presentation: Wednesday, 8:00 am, Ballroom A

Paul J. Gemperline

Dr. Paul J. Gemperline, ECU Distinguished Professor of Chemistry, has more than 27 years of experience in chemometrics, a sub-discipline of analytical chemistry that utilizes multivariate statistical and numerical analysis of chemical measurements to provide information for industrial process understanding, modeling and control. The main theme of Dr. Gemperline's research is the development of new algorithms and software tools for analysis of multivariate spectroscopic measurements using chemometrics. His work has led to advances in self-modeling curve resolution for characterization of evolving chemical systems, pharmaceutical application of chemometrics, cluster analysis and classification, and multivariate calibration. Dr. Gemperline's current research interests lie in developing chemometric methods for monitoring, understanding, and controlling batch chemical reactions and processes. Recent work has focused on the developed novel algorithms for fitting comprehensive models to batch reactions with calorimetry and in-situ spectroscopic measurements. Professor Gemperline's research achievements include more than 60 publications in the field of chemometrics and $1.8M in external grant funds from government and industrial sources. Professor Gemperline has directed over 40 undergraduate research projects, 19 M.S. theses, hosted nine visiting Ph.D. students, and supervised five post-doctoral research assistants. Dr. Gemperline is a sought-after speaker on research topics in chemometrics, having presented more than 30 invited lectures at international venues and universities in Europe and Asia. Dr. Gemperline is currently Editor-in-Chief of the Journal of Chemometrics. In 2003, he received the Eastern Analytical Symposium Award in Chemometrics, the highest award in the sub-discipline.

David M. Haaland

David M. Haaland received his Ph.D. in physical chemistry from the University of Rochester and was employed by Sandia National Laboratories from 1972 until 2008. He is currently a contract employee with Sandia, the sole proprietor of Spectral Resolutions Consulting, and North American Editor of the Journal of Chemometrics. Haaland has an appointment with the University of New Mexico Department of Molecular Genetics and Microbiology. Haaland's research interests have been the application of chemometric methods to the quantitative and qualitative analysis of spectral data. The work of Haaland and his collaborators has included development of classical least squares (CLS) methods for quantitative spectral analysis, automation of factor selection for PLS and PCR, and invention of new augmented classical least squares (ACLS) methods that are ideally suited to updating quantitative multivariate calibration models. Haaland recently led a team in the development of a new 3D hyperspectral confocal fluorescence microscope with associated MCR software to investigate cell signaling in eukaryotic cells and the photosynthetic processes in bacteria and plants. His current research interests involve the application of multivariate curve resolution (MCR) to hyperspectral imaging from fluorescence spectrometers and the analysis of magnetic resonance spectral images of human brains. Haaland has published 130 journal articles and conference proceedings, presented 150 invited talks, and has 13 issued U.S. patents licensed to U.S. industry. He has received the Chemistry in Statistics Award with E. V. Thomas (1991), the Bomem-Michelson Award (2004), the EAS Award for Achievements in Chemometrics (2005) and an R&D 100 Award (2009).

Erik Andries

Since August of 2008, Erik Andries has been a full-time mathematics faculty at Central New Mexico Community College (CNM) in Albuquerque, NM. He is also a visiting research scientist at the Center for Advanced Research Computing (CARC) at the University of New Mexico (UNM) where he has an ongoing collaboration with John Kalivas at Idaho State University (calibration maintenance and transfer). In addition, he is also a member of the UNM Cancer Center Shared Resource for Bioinformatics and Computational Biology. Prior to CNM, he spent two years as a research scientist at InLight Solutions where he worked on spectroscopic data analysis algorithms for non-invasive glucose sensing. Before that he was a postdoctoral fellow at the UNM Department of Pathology (developing spatio-temporal models of cell-signaling) and Sandia National Laboratories (extracting kinetic information from hyperspectral fluorescence image data using multivariate curve resolution procedures). In 2004, Erik Andries received a Ph.D. in applied mathematics from UNM. His interest in "bio/chemo-informatics/metrics" was galvanized by a year-long industrial co-op at GlaxoSmithKline in 1999. He is originally from New Orleans, LA.

Patrick J. Cutler

Patrick J. Cutler is currently a PhD student in the Boimedical Sciences Program at the University of New Mexico working in the Pathology Department under the advisement of Dr. Diane Lidke. Cutler began his research career working with Dr. Paul J. Gemperline at East Carolina University (ECU) while acquiring his BS in chemistry with a double major in math (2001-2006). He continued his work with Dr. Gemperline while attaining an MS in chemistry from ECU (2006-2008). During his work with Dr. Gemperline, Cutler worked on several chemometrics related projects including the kinetic modeling of hyperspectral fluorescence microscope images. In his current work, Cutler is working on single particle tracking in hyperspectral fluorescence microscope images, and the application of this technique to investigate membrane protein dynamics. His focus in this research is on algorithm development and the biological system of interest. The high affinity IgE receptor, FceRI, is being investigated in these studies. This receptor is involved in the allergic response and is an excellent system for investigating membrane protein dynamics along with signal initiation and downstream signaling effects. Cutler is a current fellow for the NSF Integrative Graduate Education and Research Traineeship in Integrating Nanotechnology with Cell Biology and Neuroscience.

GRADUATE STUDENT AWARDS

Barbara Stull Graduate Student Award
Recognizing a graduate student for outstanding research in spectroscopy and presented in honor of our longtime colleague Barbara L. Stull

Karolin Kroening

University of Cincinnati

Poster Presentation: Tuesday, Exhibit Hall

Karolin Kroening is a 4th year graduate student in Dr. Joe Caruso's research group (http://www.uc.edu/plasmachem/). She received her master's degree in 2006 from the University of Bologna, Italy basing her research on hydroxyapatite/chitosan composites for bone substitution. Karolin's research in the Caruso group has focused on the identification and cytotoxicity of chemical warfare agent degradation products and on protein phosphorylation studies in cerebral spinal fluid, a study that may help drug development for patients diseased with a hemorrhagic stroke.

Recognizing a graduate student for outstanding research in spectroscopy

Olivier R. Bolduc

University of Montreal

Presentation: Thursday, 11:50 am, Room 301B

Olivier R. Bolduc is currently a Ph. D student at University of Montreal in the group of Prof. Jean-François Masson. He studied mechanical engineering at l'École Polytechnique de Montréal before joining the Chemistry program in 2005 at University of Montreal. Olivier started in the Masson group as an undergraduate intern. His research focuses on the development of surface plasmon resonance (SPR) instrumentation and the investigation of amino acid based self-assembled monolayers (SAM) as nonfouling elements for SPR biosensors. In particular, Olivier developed a small and portable SPR instrument. The development of the peptide monolayer has allowed detection of biomolecules directly in crude biological solution. Peptides significantly prevent nonspecific adsorption of serum and allow immobilization of antibodies and enzymes to the SPR sensor. These bioanalytical spectroscopic techniques are currently applied for drug efficiency screening in collaboration with the pharmacy department of the University of Montreal. Olivier published five peer-reviewed papers, four as primary author and one conference proceeding. The results of his research lead to two patent applications showing his interest for developing applied analytical technologies based on spectroscopy. His research was also presented in 14 international and national events. Olivier holds scholarships from the University of Montreal graduate school (FESP) and Quebec's natural science and technology agency (FQRNT). Olivier R. Bolduc is a SAS student member since 2008 and a Coblentz Society student member since 2009. He is involved in the Department of Chemistry graduate student association. He served in the Canadian reserve between 2004 and 2009 as a member of the armored reconnaissance corps and intensively practices kickboxing and dragon boating.

FELLOWS AWARD

Recognizes individual members for their outstanding service to the field of spectroscopy

Mark Arnold

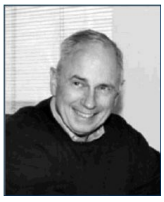
Mark A. Arnold is the Edwin B. Green Chair Professor in Laser Chemistry, the former Director of the Optical Science and Technology Center, and the current Chair of Chemistry at the University of Iowa. He earned a Bachelor of Science degree in Chemistry at Indiana University Purdue University at Indianapolis and his doctorate degree in Analytical Chemistry at the University of Delaware. He joined the faculty in the Department of Chemistry as an Assistant Professor in 1982. Recent honors include a Collegiate Fellow Award from the College of Liberal Arts and Sciences and a Regents Award for Faculty Excellence from the Iowa State Board of Regents. He is a co-founder of ASL Analytical, Inc. - a spinoff company located within the BioVentures Center at the University of Iowa Research Park. Professor Arnold's research interests center on the development of near infrared spectroscopy for chemical sensing in biosciences, biotechnology, and clinical medicine. Near infrared sensing offers the means for nondestructive, *in situ* analytical measurements performed in real-time. The resulting analytical information can enable optimization and control of critical processes. Current research efforts focus on sensors to enhance: 1) the management of diabetes, 2) hemodialysis treatments for people with end-stage renal failure, 3) treatment of critically ill neonatal, pediatric and adult patients undergoing intensive care, and 4) the control of bioreactor systems for cell expansion and protein expression.

FrankV. Bright

Frank earned his B.S. degree in chemistry from the University of Redlands (1982). He completed his Ph.D. in 1985 at Oklahoma State University in Professor Linda B. McGown's laboratory. He conducted postdoctoral research with Professor Gary M. Hieftje at Indiana University (1985-'87). In 1987, Frank joined the University at Buffalo, The State University of New York Department of Chemistry as an Assistant Professor; he is currently SUNY & UB Distinguished Professor and the A. Conger Goodyear Professor of Chemistry. Frank's research program centers on: [i] hybrid materials for chemical sensing and anti-fouling coatings; [ii] molecular-level interactions in supercritical fluids and ionic liquids; and [iii] biodegradable platforms for accelerated wound restitution. Frank has co-authored more than 250 peer-reviewed publications on these subjects along with eight issued US Patents. He has also served on numerous journal and corporate advisory boards. He has been awarded the 3 M Non-Tenured Faculty Award (1988-'91), the Eastern New York American Chemical Society Buck-Whitney Medal (1999), the SUNY Chancellor's Award for Excellence in Teaching (2000), the New York Section of the Society for Applied Spectroscopy Gold Medal (2003), the Akron Section Award of the American Chemical Society (2003), the A.A. Benedetti-Pichler Award in Microchemistry from the American Microchemical Society (2005), and the Jacob F. Schoellkopf Medal of the Western New York American Chemical Society (2006). In spare moments, Frank enjoys wood working, hanging around the pool (summer only), traveling, hiking, snow shoeing (winter only), fishing, and handgun hunting.

Joseph A. Caruso

Joe Caruso holds a Ph.D. from Michigan State University. After a one-year postdoctoral fellowship at The University of Texas - Austin, he joined the University of Cincinnati Chemistry faculty and since then he has authored or co-authored 380 scientific publications and presented more than 325 invited lectures at universities, scientific meetings, government and industry labs. His current research interests are in metallomics studies involving transgenic plants and their phytoremediation mechanisms or enhancements; evaluating cell signaling changes through phospho- or metallo- proteomes as biomarkers in the CSF of certain stroke patients; investigating the metalloproteomes associated with viruses and their affect on viral capsid stability; and the effects on cell signaling changes when arsenic toxified cells are given selenium species as part of the nutrient mix. Caruso is a member of the American Chemical Society, Society for Applied Spectroscopy and a Fellow of the Royal Society of Chemistry. He has been honored by Eastern Michigan University with its 1990 Distinguished Alumni Award, by the American Chemical Society with the 1992 Cincinnati Chemist of the Year Award, the Federation of Analytical Chemistry and Spectroscopy Society with the 1994 Anachem Award, and with the 2000 Spectrochemical Analysis Award given by the Analytical Division of the American Chemical Society. As one of their most cited authors in the 2001 to 2005 period – he was recognized by J. Chrom. A at the ACS, Sept 2006 meeting. In 2006 he also was presented the Society of Applied Spectroscopy, Distinguished Service Award. In that year he received the University of Cincinnati - Excellence in Doctoral Student Mentoring Award and in 2007 he received the 2007 Rieveschl Award for Distinguished Scientific Research. He states that the true awardees should be his wife, Judy, his three children seven grandchildren and his graduate students, postdocs, collaborators, and visitors, who, in many ways, made any and all achievements possible and of whom he is very proud.



Theodore Eyring

Activities & Awards include: ACS Utah Award, 1976; NATO Senior Fellowship, 1977; Dept of the Army Outstanding Civilian Service Medal, 1977; Indo-American Fellowship, 1978; J.S. Guggenheim Fellow, 1982; University Distinguished Research Award, 1991; Willard Gardner Prize of the Utah Academy of Sciences; Arts and Letters, 1993; ASUU Student Choice Award for Excellence in Teaching, 1997; Robert W. Parry Teaching Award, 1998

Eyring's first refereed publication in 1956 involved infrared spectroscopy applied to thiols adsorbed on zinc minerals. Over the intervening half century he has published more than 300 papers many of which describe applications of a variety of spectroscopies. One of his current studies is the following: He and his co-workers make and use metal nanoparticles in the 2 nm to about 60 nm diameter range. In one project, nanoparticles of silver metal are incorporated into the pores of a naturally occurring zeolite called chabazite. The resulting material is a very effective trap for capturing xenon gas molecules present in truly trace amounts in air. This phenomenon has a potential practical application in a hospital operating amphitheater where it would be advantageous to recycle xenon which is an especially effective but expensive anesthetic. How silver metal nanoparticles capture xenon was addressed by a computational chemistry graduate student, Hoa Nguyen, in a paper published jointly with the Truong group. The capture of other

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permanent gases from complex gas mixtures continues to be a well funded experimental gas chromatography/materials project in the Eyring group.

Joseph Gardella

Joseph A. Gardella, Jr. is Professor and Larkin Chair of Chemistry at the University at Buffalo, State University of New York (aka UB). He also serves as the Director of the UB/Buffalo Public Schools Interdisciplinary Science and Engineering Partnership. He has been on the faculty at UB since 1982. Joe was born and raised in Detroit Michigan, and completed a dual degree program in Chemistry (B.S.) and Philosophy (B.A.) from Oakland University in Rochester Michigan., a Ph.D. in Analytical Chemistry at the University of Pittsburgh and postdoctoral research in Physical Chemistry at the University of Utah. He served as a visiting scientist/program officer at the National Science Foundation Chemistry Division in 1989-90. From 1999-2005, he was Associate Dean for External Affairs in the College of Arts and Sciences and he was responsible for coordinating and leading the College's programs in working with industry, community, government and elementary and secondary schools. From 1996-2006, he was the Director of the UB Materials Research Instrumentation Facility, managing ca. $9M of shared research instrumentation. As a Faculty Fellow in the Institute for Local Governance and Regional Growth from 2005-2006, he pursued policy studies in regional science and environmental policy and public participation. More info is at: www.acsu.buffalo.edu/~gardella. Professor Gardella's research interests are in quantitative analysis and surface chemistry, broadly applied to the study of environmental effects at polymer surfaces and tissue engineering with synthetic biomaterials. His work and that of his Ph.D. students has resulted in some 240 publications and a similar number of invited talks worldwide. His work is funded by the National Science Foundation, Office of Naval Research, National Institutes of Health and industry. Besides his research interests, he has long standing interests in curriculum development for scientists and non- scientists. He was a senior member of UB's Undergraduate College, a group of faculty that developed a new general education program with a major emphasis on innovative science and laboratory courses for non-science majors. He served as Chair of the Undergraduate College Curriculum Committee for three years, which was responsible for faculty input on all phases of development of the curriculum. He is presently a member of the UB Honors College Council. At UB, he has been involved in environmental programs of all types. As the Chair of the UB Environmental Task Force in the mid 1990's he was involved in a variety of public service and policy projects, including student environmental auditing of Buffalo City Hall, and a funded project in developing field environmental analysis studies for access for community groups and local governments. He was part of the planning for the Environment and Society Institute, and served on the founding Steering Committee. Professor Gardella has been active in program development in undergraduate research, interdisciplinary studies, service learning and other academic reform areas. He is the UB representative to the Western New York Service Learning Coalition (WNYSLC, www.wnyslc.org). Gardella was co-PI on the Community Linked Interdisciplinary Research (CLIR) program, funded by the Hewlett Foundation at UB, to develop and sustain course based public service research as a means to increase the participation of undergraduates in integrative research or scholarly activity. He is also the Co-PI on the Professional Science Masters program in CAS, funded by the Alfred P. Sloan Foundation, developing innovative masters programs in the sciences. He served as Program Director and Principal Investigator of the NIH funded Research Institute in Biomedical Materials Science and Engineering (RIBSE) (www.ribse.buffalo.edu), a summer interdisciplinary undergraduate research program He has been recognized locally and nationally for his work in all areas of academic endeavor. Most recently, he was named a fellow of the American Association for the Advancement of Science (2007) and the AVS, the Technology Society (American Vacuum Society) (2004), for his research accomplishments. He has been awarded the National Science Foundation Award for Special Creativity twice (2009-2011 and 1991-1993). He has been awarded a 2005 National Science Foundation Presidential Award for Excellence in Science, Mathematics and Engineering Mentoring (PAESMEM), the 72nd Jacob Schoellkopf Medal of the Western New York American Chemical Society (2002), the 2003 Ernest Lynton Award for Faculty Public Service, three SUNY Chancellor's Medals for Excellence in Teaching (1996), Faculty Service (2004) and Scholarly and Creative Work (2005), and has been a fellow of the Exxon Education Foundation (1989-91) and Lawrence M. Gelb Foundation (1986-89). He was the awarded the second Distinguished Chemistry Alumni Award at Oakland University in 1998. He has a real life besides this stuff, which includes his wife, Carol Kizis, his daughter, Claire Seung Hee, and son, Joseph Jee Yoon. They all enjoy traveling, reading, gardening and other important pursuits which do not involve academic politics. They reside in North Buffalo, where they enjoy the weather.

Timothy Keidering

Tim Keiderling obtained a PhD in 1974 from Princeton doing research on structural as well as electronic and vibrational spectroscopic studies under the direction of Elliot Bernstein. As a postdoc, 1973-76, at the University of Southern California with Philip Stephens he participated in developing vibrational circular dichroism (VCD). In 1976 he moved to the University of Illinois at Chicago as an Assistant Professor and began a program developing VCD applications, instrumentation and theoretical modeling, as well as spectral studies of transition metal complexes in host crystals. Most of the VCD work was initially on small molecules made chiral through isotopic or very simple substitution for ease of theoretical modeling. As he moved through the ranks, both instrumentation and theoretical tools were developed for broad band VCD spectral acquisition and interpretation, and the technique moved to study of polypeptides, polynucleotides and proteins and correlation to secondary structure. Magnetic VCD spectra were also obtained and related to Zeeman splitting of degenerate normal modes. In the last decade, his research has shifted from wider applications of VCD to emphasize protein folding and related model studies using designed peptides with isotopic labeling and T-jump dynamics studies, utilizing extensive theoretical modeling and a variety of spectral techniques, as well as protein-membrane interactions studied under equilibrium and dynamic conditions with several physical and spectral methods.

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Katrin Kneipp

Technical University of Denmark, Department of Physics Charité - Universitätsmedizin Berlin, Institut für Molekularbiologie und Bioinformatik Harvard University, Medical School Massachusetts Institute of Technology

Education:

Diploma thesis in physics, Friedrich-Schiller-University Jena

Ph. D. Thesis in physics, Friedrich-Schiller-University Jena

Habil. Thesis in chemistry, Friedrich-Schiller-University JenaFacultas docendi in experimental physics, Humboldt-University Berlin

Awards:

Heisenberg Fellowship of the Deutsche Forschungsgemeinschaft DFG (1992-1995)

Meggers Award of the Society for Applied Spectroscopy (1999)

Rockefeller-Mauze visiting chair at Massachusetts Institute of Technology (2000/2001)

Fellow of the American Physical Society (2004)

Topics in research:

Ultrasensitive and nanoscale spectroscopy exploiting local optical fields of nanostructures, single molecule methodologies, surface-enhanced Raman scattering (SERS), spectroscopy of innovative nanomaterials, DNA characterization, linear and non-linear Raman spectroscopy, biomedical spectroscopy, trace detection and analysis in medicine, pharmacy, and environmental analysis

Curtis Marcott

Curtis Marcott is currently a Senior Partner and Spectroscopic Consultant at Light Light Solutions, LLC (Athens, GA). He received his B. A. degree from Concordia College (Moorhead, MN) in 1974 and his Ph.D in Physical Chemistry from the University of Minnesota in 1979. In 1979 he joined the Corporate Research Division of Procter & Gamble's Miami Valley Laboratories in Cincinnati, Ohio, as a staff infrared spectroscopist. He was appointed Research Fellow in 1996 and was a member of the Analytical Discovery Department in the Global Analytical Capability at the time of his retirement from P&G in December 2007. Curt is a member of the Editorial Advisory Board of *Applied Spectroscopy,* and is a past member of the Editorial Advisory Boards of *Analytical Chemistry* and *Vibrational Spectroscopy,* the A-page Advisory Panel of *Analytical Chemistry,* and the Board of Managers of the Coblentz Society. He is Program Committee Chairman for the 2009 Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) meeting, Chairman of the Program Committee for the 6th International Conference on Advanced Vibrational Spectroscopy (2011), Chairman of the Publications Committee for the Society for Applied Spectroscopy (2006-2008), and Infrared Short Courses Instructor (2008-). Dr. Marcott received the 1993 Williams-Wright Award from the Coblentz Society for achievement in industrial vibrational spectroscopy, was named the 2001 Cincinnati Chemist of the Year, and is an Adjunct Professor of Chemistry at Miami University in Oxford, OH. His research interests include infrared spectroscopy of adsorbed species, timeresolved infrared linear dichroism spectroscopy of polymers under small-amplitude strain, vibrational circular dichroism, applications of IR spectroscopy in phase science, GC-IR, photoacoustic spectroscopy, near-IR spectroscopy, Raman spectroscopy, spectroscopic imaging, and chemometrics.

Oliver C. Mullins

Dr. Oliver C. Mullins is a chemist and Scientific Advisor in Schlumberger Wireline Headquarters. Building on existing technology, he is the primary originator of Downhole Fluid Analysis, a significant new product line in the oil industry, for which he was awarded three Gold Medals. He has authored the book "The Physics of Reservoir Fluids; Discovery through Downhole Fluid Analysis," which the Award of Excellence from the Society of Technical Communication. He has been Distinguished Lecture for both the SPWLA (twice) and SPE on this topic. The corresponding tools exploit visible and near-infrared spectroscopy and fluorescence and are being used to uncover compartmentalization, connectivity and hydrocarbon fluid complexities in subsurface formations. Dr. Mullins also leads an active research group in asphaltene and petroleum science which has resolved several important controversies. His research focuses on the molecular and colloidal structures of asphaltenes and in particular asphaltene dispersion in reservoir crude oil. He has co-edited 3 books and coauthored 9 chapters on asphaltenes. He has coauthored 150 publications with 2000 literature citations in refereed journals to these publications. He has coinvented 56 allowed US patents. He is also Adjunct Professor of Petroleum Engineering at Texas A&M University. His hobbies include skiing, scuba, biking and blues saxophone.

Yukihiro Ozaki

Yukihiro (Yuki) Ozaki was born in Sakai, Osaka, Japan in 1949. Yuki graduated from Osaka University in 1973 with B.S degree in chemistry. Yuki also obtained his M.S (1975) and Ph.D. (1978) in chemistry from Osaka University. After he spent for two years and a half at National Research Council, Canada as a research associate, he joined the Jikei University School of Medicine in Tokyo in 1981. In 1989 he moved to Kwansei Gakuin University as an associate professor of Chemistry Department. Currently, he holds a position of professor in the Department of Chemistry, School of Science and Technology. Since April 2006, Yuki has been Dean of School of Science and Technology, Kwansei Gakuin University. He is also Director of Research Center for Single Molecule Vibrational Spectroscopy and Director of Research Center for Environment Friendly Polymers of the university. Yuki has been internationally very active in research and education of physical chemistry and spectroscopy for the last three decades. He was a senior research fellow of Princeton University in 1993. Currently, he is an honorary professor of Jilin University and Changchung Institute of Applied Chemistry, Chinese Academy of Science, and a guest professor of Peking University. Yuki is an associate Editor of *Applied Spectroscopy* and a member of the editorial board of *Journal of Raman Spectroscopy*, *Journal of Molecular Structure*, and *Vibrational Spectroscopy*. Since 2009, Yuki has been the President of Asian NIR Consortium. Yuki wrote many spectroscopy books; for example: "Near-Infrared Spectroscopy" with Heinz W. Siesler, Satoshi Kawata and Michael Heise in 2002 (Wiley-VCH), "Two-Dimensional Correlation Spectroscopy" with Isao Noda in 2004 (John Wiley & Sons), "Near-Infrared Spectroscopy in Food Science and Technology" with W. Fred McClure, and Alfred A. Christy in 2006 (Wiley-Interscience),

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and "Raman, Infrared, and Near-Infrared Chemical Imaging" with Slobodan Sasic in 2010 (Wiley). He was also an Associate Editor of the "Handbook of Vibrational Spectroscopy" from Wiley in 2001, co-edited with John Chalmers and Peter Griffiths. Yuki's research program has been concerned with basic studies and applications of far ultraviolet (FUV), infrared (IR), Raman, and near-infrared (NIR) spectroscopy. His spectroscopy research covers from the development of new type of instruments such as an ATR-FUV spectrometer, basic studies of physical phenomena like a study on mechanism of surface-enhanced Raman scattering to applications involving those to polymers, nano materials, and biological samples. His research interests also involve the developments spectral analysis methods such as two-dimensional correlation spectroscopy and chemometrics. Yuki received many awards including the 1998 Tomas Hirschfeld Award, the 2001 EAS Award for Achievements in Near Infrared Spectroscopy, the Spectroscopical Society of Japan Award (2002), the 2005 Science and Technology Award of Japanese Government (Ministry of Education, Culture, Sports, Science and Technology), and Gerald Birth Award of International Conference Diffuse Reflectance Spectroscopy, the Japan Society for Analytical Chemistry Award (2008), and Gold Medal Award of Wroclaw University, Poland (2009).

Steven Soper

Prof. Steven A. Soper received his Ph.D. in Bioanalytical Chemistry from the University of Kansas in 1989, which was followed by a Postdoctoral Fellowship at Los Alamos National Laboratory, where he worked on single-molecule detection methods for the high speed sequencing of the human genome. Prof. Soper is currently a chaired professor in Chemistry at Louisiana State University with joint appointments in Mechanical Engineering and Biological Sciences. He is also the director of an interdisciplinary research center, Center for BioModular Multi-Scale Systems, which was founded in 2004 and is funded by the NSF. Prof. Soper also holds a joint appointment at Ulsan National Institute of Science and Technology in Ulsan, South Korea, where he is the World Class University Professor. His research interests include micro- and nanofabrication of integrated systems for biomedicine, ultra-sensitive fluorescence spectroscopy, high-resolution electrophoresis, sample preparation methods for clinical analyses, and nanofluidics. As a result of his efforts, Prof. Soper has secured extramural funding from a variety of agencies ($32M USD) and has published over 230 manuscripts in peer-reviewed publications and is the author of six patents. He is also the founder of a startup company, BioFluidica, which is marketing devices for the isolation and enumeration of rare cells from environmental and clinical samples. His list of awards includes the Benedetti-Pichler Award, Fellow of the AAAS, Fellow of the Royal Society of Chemistry, R&D 100 Award, Distinguished Masters Award at LSU and Outstanding Scientist/Engineer of Louisiana in 2001. Prof. Soper is currently the Editor of the Americas for the *Analyst* and on the Editorial Board for *Journal of Fluorescence* and *Micro- and Nanosystems*. Finally, Prof. Soper has graduated 30 Ph.D.s from his research group and currently has 12 graduate students working under his direction.

Isiah Warner

Boyd Professor of LSU System, Philip W. West Professor of Analytical and Environmental Chemistry, Howard Hughes Medical Institute Professor. Professor Warner is an analytical chemist with close to 300 refereed publications in a variety of journals relevant to his area of research. He has particular expertise in the area of fluorescence spectroscopy, where his research has focused for more than 35 years. He is considered one of the world's experts in this area. For example, he is the corresponding author in the highly cited biannual reviews on "Molecular Fluorescence, Phosphorescence, and Chemiluminescence Spectrometry", for the journal, *Analytical Chemistry*. Over the past 20 years, he has also maintained a strong research effort in the areas of organized media and separation science. Professor Warner has been performing research in the more specific area of analytical measurements using ionic liquids for several years. It is this research on ionic liquids which has lead to the recent conceptualization and implementation of agroup of uniform materials based on organic salts (GUMBOS) as novel materials which can be exploited for a variety of analytical measurements. Novel nanoparticles (nanoGUMBOS) have been developed from these materials which can primarily be classified as frozen ionic liquids. However, some GUMBOS are not ionic liquids since they do not fit the traditional definition. Several publications in key chemistry journals (e.g. Nano Letters, ACS Nano, and Langmuir) and a pending patent have resulted from this new area of research.

EMERITUS AWARD

Awarded for longtime, tireless dedication to the field of spectroscopy and to the society

Theodore Rains

Dr. Theodore C. Rains has been a member of the SAS since 1968. He served as president of the society in 1982 and held numerous other positions in SAS and FACSS. Dr. Rains was born in 1925. He received his B.S. degree from Eastern Kentucky University in 1950 and a Ph.D. from University of Tennessee in 1965. His chemical career began in 1951 at Kentucky Synthetic Rubber Co. A year later he was offered a position at the Oak Ridge National Laboratory, where he embarked upon a career that gave him the opportunity to be involved in numerous research activities. Most notably of these were his work related to atomic absorption and emission spectroscopy. By the time he had completed his professional experience of 12-plus years at Oak Ridge National Laboratory and 25 years at National Institute of Standards and Technology, he had over 170 publications, including 3 books. Since retiring from NIST, Ted has taken his experience he gained with his involvement in the development of NIST Standard Reference Materials and began a new hobby. In 1990 Ted started High-Purity Standards in Charleston, SC, a manufacturer of certified reference material and quality control solutions. His hobby has grown into a successful company of over 35 employees. Ted is still fascinated with chemistry. You can still find him working in the lab at High-Purity Standards 5 days a week.

ACS DIVISION OF ANALYTICAL CHEMISTRY
ARTHUR F. FINDEIS AWARD FOR ACHIEVEMENTS BY A YOUNG ANALYTICAL SCIENTIST

Christy L. Haynes

University of Minnesota

Presentation: Monday, 8:00 am, Ballroom A

The Analytical Division of the American Chemical Society is proud to announce that Dr. Christy L. Haynes will receive the 2010 Arthur F. Findeis Award to recognize and encourage outstanding contributions to the fields of analytical chemistry by a young analytical scientist. Dr. Haynes is a recently tenured Associate Professor of Chemistry at the University of Minnesota. Prof. Haynes began her academic career as an undergraduate at Macalester College. She performed her doctoral work with Prof. Richard P. Van Duyne at Northwestern University, finishing her thesis on nanoparticle optics and surface-enhanced Raman spectroscopy in 2003. Her thesis work was recognized with the ACS Nobel Laureat Signature Award in Graduate Education. Prof. Haynes performed postdoctoral work as a NIH NRSA fellow with Prof. R. Mark Wightman at UNC, Chapel Hill, focusing on single cell measurements of exocytosis. She began her faculty appointment at the University of Minnesota in Fall 2005, and her group focuses on a wide array of analytical studies in both biological and environmental contexts. Prof. Haynes has been recognized with multiple prestigious awards in addition to the 2010 Findeis Award, including a Searle Scholars Award, a 3M Non-Tenured Faculty Award, an NIH New Innovator Award, a Sloan Foundation Award, a Dreyfus Teacher- Scholar Award, and the SEAC Young Investigator Award.

Call for Nominations
ACS Division of Analytical Chemistry Awards 2011

Deadline: November 1, 2010

- ACS Division of Analytical Chemistry Award in Electrochemistry
- ACS Division of Analytical Chemistry Award in Spectrochemical Analysis
- ACS Division of Analytical Chemistry J. Calvin Giddings Award for Excellence in Education Sponsored by the Division of Analytical Chemistry
- ACS Division of Analytical Chemistry Arthur F. Findeis Award for Achievements by a Young Analytical Scientist Sponsored by Philip Morris, USA
- ACS Division of Analytical Chemistry Award in Chemical Instrumentation Sponsored by the Dow Chemical Company
- ACS Division of Analytical Chemistry Award for Distinguished Service in the Advancement of Analytical Chemistry Sponsored by Waters Corporation

For more information, visit www.analyticalsciences.org

COBLENTZ SOCIETY CLARA CRAVER AWARD

The Craver Award is presented annually to an outstanding young molecular spectroscopist whose efforts are in the area of applied analytical vibrational spectroscopy.

Recognizing a young individual under the age of 45, who has made significant contributions in applied analytical vibrational spectroscopy.

Boris Mizaikoff

University of Ulm, Germany

Presentation, Thursday, 8:00 am
Ballroom A

The Coblentz Society is pleased to announce that Dr. Boris Mizaikoff, Chaired Professor of the Institute of Analytical and Bioanalytical Chemistry, University of Ulm, Germany has been selected as the recipient of the 2010 Craver Award. In 2006, The Coblentz Society created an award to recognize the efforts of young professional spectroscopists that have made significant contributions in applied analytical vibrational spectroscopy. The Society has named this award for Clara D. Craver in recognition of her pioneering efforts in promoting the practice of infrared vibrational spectroscopy and her many years of service to the Coblentz Society. Further, the Craver Award is the Society's complement of its prestigious 'Coblentz Award' that recognizes young spectroscopists for efforts in fundamental aspects of vibrational spectroscopy. This award is presented to Professor Mizaikoff in recognition of his recent work in the development, miniaturization, and application of mid-infrared optical chemo-biosensors, and for opening up new frontiers in nano-sensing technologies. Dr. Boris Mizaikoff received his Ph.D. in Analytical Chemistry at the Vienna University of Technology in 1996. Heading the Chemical Sensor Laboratory (CSL) he has been responsible for numerous research projects in the field of chemical IR sensors, including 4 multinational projects funded by the European Union. In 1997, he has been with the University of Texas, Austin/USA as a postdoctoral fellow. In October 2000 he finalized his Habilitation (Assoc. Prof. for Analytical Chemistry) at the Vienna University of Technology. Since Fall 2000 he was faculty member at the Georgia Institute of Technology, School of Chemistry and Biochemistry, heading the Applied Sensors Laboratory (ASL). Since 2004 he was Director of the Focused Ion Beam Center (FIB2 Center) at Georgia Tech, and since 2005 member of the Center for Cell and Molecular Signaling at Emory University, School of Physiology. In Fall 2007, he has joined the faculty at the University of Ulm, Germany, as a Chaired Professor heading the Institute of Analytical and Bioanalytical Chemistry. Today, his research interests focus on optical sensors, biosensors, and biomimetic sensors operating in the mid-infrared spectral range, applications of novel IR light sources (e.g., quantum cascade lasers), system miniaturization and integration based on micro- and nanofabrication, multifunctional scanning nanoprobes (e.g., combination AFM-IR and AFM-SECM-IR), scanning probe tip integrated nano(bio)sensors, focused ion beam (FIB) microscopy, development of chemical recognition interfaces for separation and sensing applications (e.g., molecularly templated materials), chemometric data evaluation, advanced vibrational spectroscopic techniques (e.g., SEIRA), environmental analytical chemistry, process analytical chemistry, and biomedical diagnostics. Dr. Mizaikoff is author/co-author of over 110 peer-reviewed publications, 14 patents, and numerous invited contributions at scientific conferences.

COBLENTZ SOCIETY WILLIAM G. FATELEY STUDENT AWARD

William G. Fateley Student Award Established, Inaugural Award presented at FACSS 2010

The family and former group members of William G. Fateley, in conjunction with The Coblentz Society and The Society for Applied Spectroscopy, wish to announce the formation of a Student Award to honor the career and life of William G. Fateley. The yearly award will consist of a $1000 prize to the selected student(s). All winners would be recognized, and invited to present their research results, at an appropriate professional conference (e.g., FACSS, Pittcon).

Although Bill Fateley passed away in 2009, he is still remembered as a larger-than-life figure in the spectroscopy community. Bill was highly regarded for his scientific contributions, but also loved by many people for his humor, his generosity, and for never taking himself too seriously (and some may have thought he was a bit of an ornery cuss, and Bill was ok with that too). At Bill's memorial service at Kansas State University, many speakers spoke of Bill's quick wit and his unmistakable laugh. Nearly everyone had examples of how he had provided personal support. Whether it was a material gift (a beautiful handmade clock) or a simple positive word when one was needed, Bill was always helping and encouraging people throughout his life. Perhaps Bill's biggest impact was his contributions to the social fraternity of international Spectroscopy in the pre-LinkedIn, pre-Facebook world. His long years of service to Pittcon, serving as Conference President in 1971, and to the Society for Applied Spectroscopy, serving as Editor of *Applied Spectroscopy* for 20 years, were a benefit to us all.

Another lasting component of Bill's legacy was his encouragement for students to attend professional conferences and meet their peers. He made it a goal to introduce young scientists to the "people" in the field and to get them personally involved. Fostering this interaction was important to Bill; perhaps as much as it was for the Science. His efforts included sending his students to many international conferences. If need be, he even went as far as to bring the social interaction center (disguised as a mobile spectroscopy lab) directly to the conference. Bill's commitment to encouraging students to attend conferences, meet and interact with their colleagues and contribute to the field of Spectroscopy, has produced a whole new generation of spectroscopists. And enriched the groups and societies that Bill championed so strongly. This is the legacy that we hope to continue with the establishment of this award.

The William G. Fateley Student Award will be administered by The Coblentz Society. Bill was a 1965 Coblentz Award Winner and thus the Coblentz Society is well suited to remember Bill's scientific contributions. The Society will accept nominations for the Student award, review the candidates, and forward the top three nominees on to the Fateley Representative Committee. This committee will select the student that most closely embodies the spirit of Bill's desire to promote the science and society of spectroscopy.

The inaugural award will be presented during one of two special sessions at FACSS 2010 honoring Bill Fateley. These sessions will consist of a mixture of presentations from Bill's colleagues and former group members.

Please consider contributing to the formation of this award, and continuing the positive impact Bill Fateley had on the spectroscopic community.

Due to his worldwide impact on the field of Spectroscopy, tax deductible donations to the award fund are encouraged from all of "Wild" Bill's friends, colleagues and professional societies.

Funds may be sent to:
"The William G. Fateley Award / The Coblentz Society"
First Financial Bank
335 South College Avenue
Oxford OH 45056

Thank you.
The Family, former research group members and colleagues of William G. Fateley.

WILLIAM G. FATELEY STUDENT AWARD

Presentation: Tuesday, 11:10 am, Room 306A

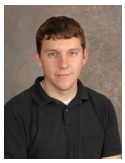
Ali Eftekhari-Bafrooei received his M.Sc. degree in Physical Chemistry from Sharif University in Tehran, Iran where he studied surface reaction mechanisms. He traveled to the USA to join Professor Borguet's group at Temple University in 2005 and is currently pursuing his Ph.D, which he expects to defend in the summer of 2010. He has been studying the vibrational spectroscopy and dynamics of interfacial water at hydrophilic and hydrophobic with surface specific technique of vibrational sum-frequency generation (SFG). His experiments are the first to reveal how the ultrafast vibrational dynamics of water at a solid interface (H2O/SiO2) can be slowed down, a consequence of the reduced number of neighbors. Furthermore, he used dilute solutions of HDO in D2O to decouple the O-H stretch and observed a frequency dependent vibrational relaxation of this mode. The redshift of the O-H stretch in SFG spectra with increasing charge at the silica surface suggested a correlation with the strength of the hydrogen bond network. This provided a connection between the spectroscopy and dynamics of water at a charged interface, and existing theoretical models for vibrational dynamics of bulk hydrogen bonded systems. In addition, he has contributed to a variety of studies where vibrational SFG has been critical in determining the structure of self assembled monolayers and their effect on processes as diverse as neuronal cell growth and electronic device fabrication.

COBLENTZ SOCIETY STUDENT AWARDS

For many years, the Coblentz Society has encouraged young scientists to pursue studies in spectroscopy by seeking nominations of outstanding students for the Coblentz Student Awards. The awardees receive a copy of the Society's Deskbook, a certificate, and a year's membership in the Society. Their names, the names of their faculty advisors, their institution, and their anticipated graduation date appear in the Society's Newsletter in the August issue of Applied Spectroscopy.

Ram Bhatta is a doctoral student in the Department of Chemistry at the University of Akron (UA). He received a M. Sc. from Tribhuvan University, Nepal in 2002 and has been associated with UA since 2008. Prior to joining the UA, he was a lecturer of chemistry in Nepal. He served as a lecturer of chemistry at Acme Engineering College, REHDON College and Amrit Campus form 2002 to 2007. Ram's research in the area of computational spectroscopy and vibrational dynamics focuses on large amplitude vibrations in small molecules and on the simulation of sum-frequency vibrational spectra at buried interfaces. He is a member of American Physical Society, Golden Key International Honor Society and Nepal Chemical Society.

Praveenkumar Boopalachandran was born in 1976 in India. He received his Chemical Engineering degree from University of Madras, India, in July 1999. In January of 2002 he received his Master of Science degree in Chemistry from Texas A&M University, Commerce. In August 2003 Praveenkumar enrolled in graduate school in the Department of Chemistry of Texas A&M University, College Station under the guidance of Prof. Wayne Goodman. He was awarded his Master of Science degree in Physical Chemistry in May 2006. Currently he is pursuing the PhD in physical chemistry under the guidance of Prof. Jaan Laane. His graduate research concentrates on studying the electronic excited state of pyridine and determining the torsional potential function of butadiene. He is expected to graduate in the fall of 2010.



Bryon Herbert is a third year Ph.D. student at the University of Delaware under the guidance of Dr. Karl S. Booksh. He obtained his B.S. in chemistry from Drexel University in Philadelphia (PA) during which he obtained an internship within the Process Analytical Technology group at GlaxoSmithKline. From his PAT experiences, Bryon gained an interest in the application of vibrational spectroscopic methods to explore the dynamic chemical systems found within the development of new pharmaceutical compounds. His graduate research has stemmed from this experience to include the collection of vibrational spectra, infrared and Raman, and the chemometric modeling of spectral variations to explore the underlying information. This includes the exploration and the application of a discrete wavelet transform on midinfrared spectroscopy to isolate and refine the spectral attributes and to increase the accuracy of the calibration model. This work has extended into the ability to probe prediction set spectra for uncalibrated chemical features not captured within the calibration model and adapt the model space to omit the interfering elements prior to model application. Bryon's work has been presented at several conferences and as contributions to manuscripts presently in the submission process to peer-reviewed journals as well as others currently in draft.

Yuliya Luzinova graduated from Clemson University in 2006 with a BS in Chemistry. She is presently a 4th year PhD candidate in the Department of Chemistry and Biochemistry, Georgia Institute of Technology, majoring in Analytical Chemistry. Her research focuses on development of mid-infrared sensors for environmental detection/monitoring in harsh environments. The major part of her thesis focuses on investigating natural gas hydrate formation and dissociation processes utilizing fiberoptic mid-infrared evanescent field spectroscopy and studying carbonate minerals and hydrocarbons within cold seep ecosystems using infrared attenuated total reflection (IR-ATR) spectroscopy. She has one research article published in Organic Geochemistry Journal and two additional research articles ready for submission on the topic of her doctoral research. In addition she has five articles/proceedings published as a collaborator. She has presented at several conferences through out her graduate studies.

Hajime Okajima is currently a graduate student in the Department of Chemistry, School of Science, the University of Tokyo, under supervision of Professor Hiro-o Hamaguchi.

PREVIOUS FACSS BOARD AND MEETING CHAIRS

1973	
Jeannette Grasselli	Governing Board Chair
1974 – Atlantic City	
James White	Governing Board Chair
George Heinz	General
James White	Program
Edward Ruffing	Exhibit
1975 - Indianapolis	
James Holcombe	Governing Board Chair
Gerald Wallace	General
James Holcomb	Program
Edward Ruffing	Exhibit
1976 - Philadelphia	
Edward Brame	Governing Board Chair
Edward Brame	General
Edward Dunlap	Program
Douglas Robinson	Arrangements
Edward Ruffing	Exhibit
1977 - Detroit	
Edgar Peck	Governing Board Chair
Mitch Kapron and James Burns	General
Jeannette Grasselli	Program
L. Felix Schneider	Arrangements
Edward Ruffing	Exhibit
1978 - Boston	
James Williamson	Governing Board Chair
Paul Lublin	General
James Cosgrove	Program
James Cornwell	Arrangements
Edward Ruffing	Exhibit
1979 - Philadelphia	
Peter Keliher	Governing Board Chair
Douglas Robinson	General
Philip LeFleur	Program
Sydney Fleming	Arrangements
Edward Ruffing	Exhibit
1980 - Philadelphia	
L. Felix Schneider	Governing Board Chair
Sydney Fleming	General
Theodore Rains	Program
Robert Barford	Arrangements
Edward Ruffing	Exhibit
1981 - Philadelphia	
Jack Katon	Governing Board Chair
Robert Barford	General
Mary Kaiser	Program
James Cavanaugh	Arrangements
Peter Keliher	Exhibit
1982 – Philadelphia	
Sydney Fleming	Governing Board Chair
James Cavanaugh	General
Andrew Zander	Program
Matthew O'Brien	Arrangements
Peter Keliher	Exhibit
1983 - Philadelphia	
Mary Kaiser	Governing Board Chair
Matthew O'Brien	General
John Lephardt	Program
D. Bruce Chase	Arrangements
Peter Keliher	Exhibit
1984 - Philadelphia	
Theodore Rains	Governing Board Chair
D. Bruce Chase	General
Patricia Rouse Coleman	Program
Fred Corcoran	Arrangements
Peter Keliher	Exhibit
1985 - Philadelphia	
Robert Barford	Governing Board Chair
Fred Corcoran	General
Matthew Klee	Program
Marshall Fishman	Arrangements
Peter Keliher	Exhibit
1986 - St. Louis	
Ronald Schroeder	Governing Board Chair
Marshall Fishman	General
Alexander Scheeline	Program
Terry Hunter	Arrangements
Edward Brame	Exhibit
1987 - Detroit	
Patricia Rouse Coleman	Governing Board Chair
David Coleman and L. Felix Schneider	General
John S. Beaty	Program
Edward Brame	Exhibit
1988 - Boston	
James Cavanaugh	Governing Board Chair
Frank Plankey and John S. Beaty	General
Roger Gilpin	Program
Edward Brame	Exhibit
1989 - Chicago	
Alexander Scheeline	Governing Board Chair
Paul Bourassa	General
Robert Michel	Program
Edward Brame	Exhibit
1990 - Cleveland	
Nancy Miller-Ihli	Governing Board Chair
Charles Belle	General
Steven Hughes	Program
Edward Brame	Exhibit
1991 - Anaheim	
David Coleman	Governing Board Chair
Richard Deming and Constance Sobel	General
James Holcombe	Program
Edward Brame	Exhibit
1992 - Philadelphia	
Karmie Galle	Governing Board Chair
Matthew Klee	General
Barry Lavine	Program
Edward Brame	Exhibit

PREVIOUS FACSS BOARD AND MEETING CHAIRS

1993 - Detroit

Robert Watters	Governing Board Chair
L. Felix Schneider and David Coleman	General
Julian Tyson	Program
Mildred Barber	Exhibit

1994 - St. Louis

Paul Bourassa	Governing Board Chair
Terry Hunter	General
John Koropchak	Program
Mildred Barber	Exhibit

1995 – Cincinnati

Jon W. Carnahan	Governing Board Chair
Joseph A. Caruso	General
Richard F. Browner and R. Kenneth Marcus	Program
Mildred Barber	Exhibit

1996 – Kansas City

Rachael Barbour	Governing Board Chair
O. Karmie Galle	General
William Fateley	Program
Scott McGeorge	Exhibit

1997 - Providence

Mildred Barber	Governing Board Chair
Chris Brown	General
John Olesik	Program
Scott McGeorge	Exhibit

1998 - Austin

John Graham	Governing Board Chair
David Laude	General
Isiah Warner and Linda McGown	Program
Scott McGeorge	Exhibit

1999 - Vancouver

Robert G. Michel	Governing Board Chair
Michael Blades	General
Ronald Williams	Program
Scott McGeorge	Exhibit

2000 - Nashville

John Koropchak	Governing Board Chair
Arlene Garrison	General
Michael Carrabba	Program
Scott McGeorge	Exhibit

2001 – Detroit

David A. Laude	Governing Board Chair
David Coleman and L. Felix Schneider	General Co-Chairs
David J. Butcher	Program
Scott McGeorge	Exhibit

2002 – Providence

Michael Carrabba	Governing Board Chair
Robert G. Michel	General Chair
Mark A. Hayes	Program Chair
Scott McGeorge	Exhibits

2003 – Fort Lauderdale

Ronald Williams	Governing Board Chair
Rina Dukor	General Chair
James Rydzak	Program Chair
Scott McGeorge	Exhibit

2004 – Portland

Michael Blades	Governing Board Chair
David Trimble	General Chair
George Agnes	Program Chair
Scott McGeorge	Exhibit

2005- Quebec City, Canada

Mark Hayes	Governing Board Chair
Denis Boudreau	General Chair
Paul Farnsworth	Program Chair
Scott McGeorge	Exhibit

2006 – Orlando

Diane Parry	Governing Board Chair
Christine Wehlburg	General Chair
S. Douglass Gilman	Program Chair
Mike Carrabba	Exhibit

2007 – Memphis

James Rydzak	Governing Board Chair
Paul Bourassa	General Chair
Ian R Lewis	Program Chair
Mike Carrabba	Exhibit

2008 – Reno

Gary Brewer	Governing Board Chair
John Hellgeth	General Chair
Greg Klunder	Program Chair
Mike Carrabba	Exhibit

2009 – Louisville

Becky Dittmar	Governing Board Chair
Jessica Jarman	General Chair
Curtis Marcott	Program Chair
Mike Carrabba	Exhibit

SOCIETY AND COMMITTEE MEETINGS AND EVENTS

FACSS

Sunday, October 17

8:30 am Long Range Planning Meeting, *Chancellor Room, Marriott*

6:00 pm Program Committee, *Chancellor Room, Marriott*

Wednesday, October 20

9:00 am 2011 Planning/Budget Committee, *University C, Marriott*

11:00 am 2012 Planning/Budget Committee, *University C, Marriott*

1:00 pm Budget and Finance Committee, *University C, Marriott*

Thursday, October 21

1:00 pm Executive Committee, *Chancellor Room, Marriott*

6:30 pm Governing Board Meeting, *Chancellor Room, Marriott*

ASTM

Monday, October 18

4:00 pm E13.10 Molecular Spectroscopic Optical Imaging, *Room 301B, convention center*

6:00 PM Raman Reception, *Room 402, convention center*

Tuesday, October 19

4:00 pm E13.08 Raman Spectroscopy, *Room 301B, convention center*

COBLENTZ

Monday, October 18

12:30 pm Board Meeting, *Room 202, convention center*

SOCIETY FOR APPLIED SPECTROSCOPY

Saturday, October 16

4:00 – 9:00 pm SAS Executive Committee Meeting, *Chancellor Room, Marriott*

Monday, October 18

7:00 – 9:00 am Editorial Board Meeting, *Chancellor Room, Marriott*

12:00 – 2:00 pm Journal Taskforce, *Chancellor Room, Marriott*

Tuesday, October 19

8:00 – 10:00 am Publication Committee, *Chancellor Room, Marriott*

12:00 – 2:00 pm Membership Committee, *Chancellor Room, Marriott*

4:30 – 6:30 pm SAS Governing Board Meeting, *Chancellor Room, Marriott*

7:00 –11:00 PM SAS Wine and Cheese Reception, *State Ballroom, Marriott (members only)*

Wednesday, October 20

12:00 – 2:30 pm SAS Award Winner Luncheon, *Chancellor Room, Marriott*

6:00 PM
WEDNESDAY EVENING EVENT
Ballroom A

Analytical Chemistry....It is All Fun and Games at FACSS.

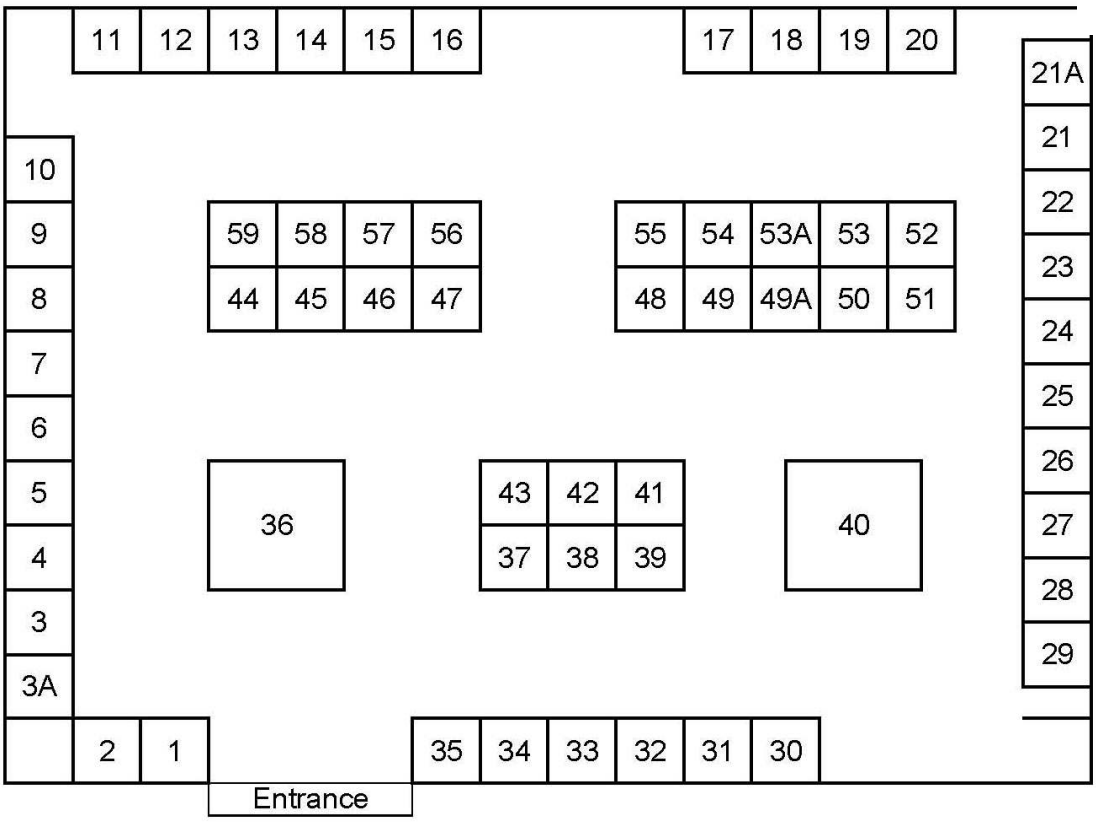
Join us for an evening of food, beverage, music, dancing, and games. Challenge a colleague to a game of Battleship or test your knowledge in Trivial Pursuit. Prove you are the Mastermind of FACSS. If you prefer to be more active, there is always FACSS Twister. Everyone will be entered into a game of chance with raffle prizes being awarded throughout the evening. A Wii Station will be available for you to play your favorite games with your friends.

There is no additional charge for the Wednesday Evening Event.

FACSS EXHIBITORS

ABB Analytical Measurement	Booth 58
Agilent Technologies	Booth 12
Anasys Instruments	Booth 34
Andor Technology	Booth 4
B&W Tek, Inc.	Booth 55
BioTools	Booth 57
Block Engineering	Booth 20
Brimrose Corporation of America	Booth 56
Bruker	Booth 38
CAMAG Scientific	Booth 9
CAMO Software	Booth 23
Centice	Booth 31
CeramOptec Industries	Booth 33
Cobalt Light Systems	Booth 59
Coblentz Society	Booth 17
Daylight Solutions	Booth 10
Dionex	Booth 52
Edinburgh Instruments	Booth 22
Eigenvector Research	Booth 48
Enwave Optronics	Booth 49A
Expo Technologies	Booth 53
FACSS	TBD
FiberTech Optica	Booth 51
Fiveash Data Management	Booth 53A
HORIBA Scientific	Booth 36
Ibsen Photonics	Booth 28
ICP Information Newsletter	Booth 21A
Innovative Photonic Solutions	Booths 44 & 45
JASCO	Booth 5
John Wiley & Sons	Table Top
Kaiser Optical Systems	Booths 1 &2
LEAP Technologies	Booth 29
Mettler Toledo	Booth 21
Nanonics Imaging	Booths 6 & 7
NSI Solutions	Booth 30
Ocean Optics	Booth 43
OI Analytical	Booth 50
Ondax	Booth 32
OPOTEK	Booth 37
Optigrate	Booth 8
Oriel Instrument	Booth 3
Oxxius	Booth 42
PAIR Technologies	Booth 49
PerkinElmer Life & Analytical Sciences	Booth 35
Princeton Instruments	Booth 39
Renishaw	Booths 46 & 47
RoMack	Booths 41
Royal Society of Chemistry	Booth 19
RPMC Lasers	Booth 54
Society for Applied Spectroscopy	Booths 14, 15, & 16
Spectroscopy Magazine / Advanstar	Booth 27
SPEX CertiPrep	Booth 3A
Taylor & Francis	Booth 13
Thermo Scientific	Booths 24, 25 & 26
Varian	Booth 11
Wasatch Photonics	Booth 18
WITec Instruments	Booth 40

EXHIBIT HALL LAYOUT



EXHIBITOR DESCRIPTIONS

ABB Analytical Measurement **Booth 58**
585 boul. Charest E., Ste 300
Quebec, PQ, G1K 9H4 CANADA
www.abb.com/analytical
Since 1973, ABB Analytical enables scientists around the world to perform through excellence in infrared spectroscopy. ABB is a market leader in Fourier Transform Infrared (FT-IR/FT-NIR) in terms of innovation, reliability and reproducibility. ABB designs, manufactures and markets high-performance spectrometers and offers turnkey analytical solutions for Academic, Petroleum, Chemical, Life Sciences, Semiconductor, Metallurgy, Remote Sensing and Aerospace markets. Our capabilities encompass one of the largest portfolios in the world for laboratory, at-line and process analyzers.

Agilent Technologies, Inc. **Booth 12**
2850 Centerville Rd.
Wilmington, DE 19808
www.agilent.com/chem
Agilent manufactures and distributes a complete line of instrumentation serving the clinical, analytical, biotech, environmental, pharmaceutical, forensic science, food and flavor, academia, and all other laboratory markets that have needs for the best in quality, performance, and serviceability in the instruments they purchase.

Anasys Instruments Corp. **Booth 34**
121 Gray Avenue, Ste 100
Santa Barbara, CA 93101
www.anasysinstruments.com
Anasys is a young but pioneering company in the field of nanoscale materials properties characterization. Our most recent technology breakthrough is the nanoIR platform for nanoscale IR Spectroscopy. Our previous technology breakthrough was the nanoTA platform for nanoscale thermal analysis

Andor Technology **Booth 4**
425 Sullivan Ave. #3
S. Windsor, CT 06074-1942
www.andor.com
Andor enables its customers to break new ground by performing light measurements previously considered impossible. Andor is a world leader in low light imaging with a portfolio spanning high-performance scientific digital cameras, spectrographs, and microscopy confocal and white light systems. Andor now counts over 250 employees in 17 offices worldwide, offering over 70 products to 10,000 scientific research and OEM customers, and acquired Bitplane in 2009 and Photonic Instruments in 2010. Andor Technology is quoted on the AIM market of the London Stock Exchange (LSE: AND).

B&W Tek, Inc. **Booth 55**
19 Shea Way, ste 301
Newark, DE 19713
www.bwtek.com
B&W Tek, Inc. is proud to announce our newest NIR spectrometer, the Sol2.2A, with a spectral range out to 2.2 microns. This spectrometer features a built-in AutoZero function resulting in exceptionally low noise and high dynamic range. It is designed with four settings of gain, producing great results for most applications in low light level NIR detection. The Sol2.2A is also equipped with a built-in three-stage cooler, with no external control box, allowing for easy integration just by providing +5VDC power.

BioTools, Inc. **Booth 57**
17546 Bee Line Highway
Jupiter, FL 33458
www.btools.com
BioTools, known worldwide for its expertise in characterization of molecular chirality and the structure of proteins, was the first company to introduce dedicated spectrometers for the measurement of VCD & ROA– the Chiral*IR-2X*™ and the Chiral*RAMAN-2X*™. VCD has recently evolved to become one of the most sought-after tools for the unambiguous determination of absolute configuration, as well as determination of enantiopurity and solution conformations. Our BioIR series of solutions for measurements and analysis of FT-IR spectra of proteins, viruses, sugars and nucleotides, lead by the best selling PROTA instrument, is the number one choice for scientists doing biopharmaceutical formulation. BioTools also offers spectroscopic accessories including extensive protein databases measured in solid and solution states using various techniques; unique sampling cells, holders, and accessories for temperature controlled studies. In addition to products, BioTools provides services and consulting for the characterization of chiral molecules and proteins with the aim of solving the full range of customer's particular needs.

Block Engineering **Booth 20**
377 Simarano Dr. #130
Marlborough, MA 01752
www.blockeng.com
Block Engineering has pioneered the evolving field of Mobile Instrumentation for identification and analysis of gases, liquids and solids. We developed a widely tunable Mid-IR Laser Absorption Spectroscopy System that can detect trace chemicals at a 2-3 ft standoff, analyze gases, inspect bulk materials and liquids, and can be incorporated into a microscope. Using our decades-long experience, we have developed legendary Standoff Fourier Transform Infrared (FTIR) spectrometers that can detect chemical clouds as far as three miles away. These field rugged instruments can be handheld or mounted in ground or air vehicles. We are now taking this unparalleled know-how one step further by developing Microelectromechanical Systems (MEMS) based, high resolution, pen-size FTIR that cost an order of magnitude less than similar laboratory instruments.

Brimrose Corporation of America **Booth 56**
19 Loveton Circle
Sparks Glencoe, MD 21152
www.brimrose.com
We will present rugged and miniaturized AOTF-NIR Analyzers as cost-effective solutions for various Real-time On-line applications such as non-destructive, non- contact process monitor and control of Drying, Blending and many more. We will be showing our brand new Luminar 7030, which is an all stainless steel unit in which it is IP55 rated. We will show advantages of using NIR for Real-time analysis on different types and configurations of Blenders and Fluid Bed Dryers. The 21CFR Part11 compliant data management system is used for on-line results monitoring, backing up data and controlling the processes

Bruker Optics, Inc. **Booth 38**
19 Fortune Drive
Manning Park
Billerica, MA 01821
www.brukeroptics.com

EXHIBITOR DESCRIPTIONS

CAMAG Scientific, Inc. **Booth 9**
515 Cornelius Harnett Dr.
Wilmington, NC 28401
www.camag.com
CAMAG Scientific, Inc. is the US subsidiary for CAMAG (headquartered in Switzerland). We opened our doors in 1980, and are located in Wilmington, NC. We take pride in our ability to deliver useful and cost effective instrumentation to the instrumental TLC market, and to then follow through with personalized service and support. Camag Scientific also operates a contract laboratory, providing specialized TLC/HPTLC services in a cGMP/cGLP environment. We offer a wide range of products that cover all aspects of both TLC and HPTLC. The exceptional performance and reliability of our instrumentation, as well as our experience and expertise, have ensured that we are the world leader in modern thin layer chromatography.

CAMO Software Inc **Booth 23**
One Woodbridge Center, Suite 319
Woodbridge, NJ 07095
www.camo.com
CAMO is a pioneer in providing solution, training and professional services around Multivariate Data Analysis. Its products offer the most definitive analytical modeling, prediction and optimization solutions. CAMO's flagship simulation and prediction software products is The Unscrambler®. The Unscrambler® X (a GenX suite of products) represents the single largest redesign of CAMO Software's flagship product The Unscrambler®. Featuring an enhanced user friendly interface, superior graphics and new state-of-the-art methods, The Unscrambler® X provides the most flexible and adaptable approach to Multivariate Data Analysis (MVA) and Design of Experiments (DoE) available. The Unscrambler® X reaches out to a wider audience of data analysts, and process control applications, while still maintaining a strong commitment to our spectroscopic & sensory data analysis users.

Centice Corporation **Booth 31**
215 Southport Dr., Ste 1000
Morrisville, NC 27560
www.centice.com
Centice Corporation leads Raman spectroscopic sensor innovation in solving real-world problems across a wide range of industries. Providing products and services for prescription drug verification and identification, Centice serves retail pharmacy and healthcare markets. The company's first product, PASS Rx is a compact and easy to use system that verifies dispensed medications through vials within seconds, thereby reducing human error and increasing patient safety. The second product, Rx Identifier, provides hospitals with the quick identification of unknown drugs in tablet or capsule form from a library of more than 2500 unique medications. Both systems incorporate a patented coded aperture for enhanced throughput without sacrificing spectral resolution.
www.centice.com

CeramOptec Industries Inc. **Booth 33**
515 Shaker Road
East Longmeadow, MA 06062
www.ceramoptec.com
CeramOptec® is optical fiber products and solutions. We offer a comprehensive range of superior fibers, bundles, assemblies, collimators and feedthroughs for virtually any application. Specialty silica fibers from 50 to 2000 microns with NA's from 0.06 to 0.66 for use from deep UV to NIR including non-circular core fibers. Silver halide fibers for MIR applications. Fibers for use at temperature ranges from -180C to 650C. Any of these fibers can be manufactured into assemblies, bundles, fused end bundles and vacuum products. Replacement fibers for most spectrometer systems and instruments. CeramOptec offers products for most markets with custom solutions for any application. From preform through finished product with unmatched quality and direct from the manufacturer pricing .

Cobalt Light Systems **Booth 59**
Fermi Avenue
Didcot
Oxon OX11 0QR, United Kingdom
www.cobaltlight.com
Cobalt develops and sells novel Raman instruments for spatially offset Raman spectroscopy (SORS) and transmission Raman (TRS). We offer targeted instruments and also bespoke research and production devices. The TRS100 is a high throughput pharmaceutical analysis tool for regulated testing environments. The Triton is for R&D into SORS, TRS and wide-area Raman spectroscopy. Our LiteThru platform is the basis of our bespoke devices for biological, chemical and other research.

Coblentz Society **Booth 17**
Dept of Chemistry and Biochem
Miami University
Oxford, OH 45056
www.coblentz.org
Professional organization that fosters the understanding and application of vibrational spectroscopic sciences: infrared, near infrared, Raman and chemometric methods used in these spectroscopies. Through the voluntary efforts of its members, the society sponsors scientific conferences, creates symposia for research presentations, provides social activities to stimulate informal discussion, and recognizes excellence in vibrational spectroscopy through three sponsored awards (the Coblentz, Williams-Wright, and Lippincott Awards). The society also administers the ABB Bomem-Michelson Award. The Coblentz website can be found at http://www.coblentz.org.

Daylight Solutions **Booth 10**
15378 Avenue of Science, Ste 200
San Diego, CA 92128
www.daylightsolutions.com
Daylight Solutions is a leading supplier of molecular imaging and sensing systems based on External Cavity Quantum Cascade Lasers. DLS also offers ECqcL laser sources for molecular spectroscopy, stand-off detection, microscopy, and imaging applications. Sources provide coverage from 3-12 microns in the mid-IR spectrum.

Dionex Corporation **Booth 52**
1228 Titan Way
PO Box 3603
Sunnyvale, CA 94088-3606
www.dionex.com
It's All About the Chemistry-Dionex delivers breakthrough chromatography solutions for better performance and higher productivity. The UltiMate® 3000 LCi series includes RSLC systems with UHPLC and HPLC capabilities, the new RSLCnano system for proteomics. The titanium solution with bioinert flow paths and ESA detectors expand capabilities for biomolecule detection. Our unique Reagent-Free™ IC systems feature Eluent Generation and Regeneration. We lead the industry with selective high-capacity IC and HPLC/RSLC columns and consumables and automated sample preparation and solid-phase extraction systems that significantly reduce labor and waste.

EXHIBITOR DESCRIPTIONS

Edinburgh Instruments Ltd **Booth 22**
Analytical Division
2 Bain Square, Kirkton Campus
Livingston, West Lothian EH54 7DQ UK
www.edinst.com
Edinburgh Instruments is recognized internationally as the leading supplier of advanced research grade modular fluorescence & laser flash photolysis spectrometers, TCSPC fluorescence lifetime instrumentation, and IR gas lasers. They also develop and manufacture high quality gas sensors, which ensure accurate and reliable measurement of target gases such as CO2, CO, CH4 and refrigerants. The company also represents femtosecond upconversion systems, pulsed TEA-CO2 lasers, DPSS lasers and THz oscillators, detectors & accessories from reputed manufacturers.

Eigenvector Research, Inc. **Booth 48**
3905 West Eaglerock Dr
Wenatchee, WA 98801
http://www.eigenvector.com
Eigenvector Research, Inc. (EVRI) is a full-service Chemometrics company, offering software, consulting and training. EVRI's mission is to provide advanced chemometrics support for a wide variety of industries and academia, including pharmaceutical, semiconductor, chemical process, consumer product manufacturers and analytical instrument developers. Our chemometric software products include our flagship MATLAB-based PLS_Toolbox and stand-alone Solo. We also offer add-ons for Multivariate Image Analysis, MIA_Toolbox and Solo+MIA, and applications for putting chemometric models on-line, such as Solo_Predictor. EVRI's services include chemometrics consulting and training. Our staff of six consultants has over 100 years of combined chemometric experience. Our chemometric short courses cover a wide range of topics and have been attended by over a thousand participants from industry and academia. Our goal is to be your complete source for state-of-the-art chemometric tools and know-how.

Enwave Optronics, Inc. **Booth 49A**
18200 W. McDurmott St., Ste B
Irvine, CA 92614
www.enwaveopt.com
Enwave Optronics provides a full line of routine Raman instrumentation solutions with outstanding performance and best value for measurements of solids, liquids and gases. The field portable EZRaman systems are ideal for product authentication, homeland security, incoming material inspection, and research/education laboratories. The high sensitivity ProRaman analyzers (CCD cooled to below –50 degree Celsius) are best for industrial process monitoring and laboratories applications requiring affordable solutions for high detection speed, sensitivity, and stability. We also offer Raman microscopes ranging from low cost to high-end confocal design, suitable for all levels of microscopic Raman analysis. Our instrument capability is specialized in saving Raman applications failed by other vendors.

Expo Technologies **Booth 53**
4041 Forest Park Ave
St. Louis, MO 63108
www.expoforpat.com
Expo Technologies expertise in spectroscopy, engineering and software come together to provide a complete range of PAT solutions based on NIR and UV/Vis and Fluorescence spectroscopy. The ePAT series is focussed on pharmaceutical unit operations such as reaction monitoring, blending, granulation and compaction whilst a complimentary range sample interface accessories and other engineering solutions provide for a robust lab to plant implementation. Expo's NovaPAC© software suite allows full migration of the PAT tool from the development laboratory through to manufacturing.

FACSS **Booth TBD**
2019 Galisteo St., Bldg I-1
Santa Fe, NM 87505
www.facss.org
The 2011 FACSS Conference will be held October 2-6th in Reno, NV. Combine a *world-class scientific conference* with a *beautiful vacation destination* and you have an event that is not to be missed. The annual FACSS meeting covers all aspects of analytical chemistry with an emphasis on emerging technology and brings together leading scientists across many disciplines for scientific exchange. This is accomplished through a strong technical program, exhibition, and numerous informal networking opportunities. The Reno/Tahoe location provides a beautiful setting and opportunities to see and experience the grandeur of the Sierra Nevada Mountains. The 2011 meeting will also introduce new awards for ***Scientific Innovation*** debuted at the FACSS meeting, see the website for details www.facss.org.

FiberTech Optica, Inc. **Booth 51**
330 Gage Avenue, Ste 11
Kitchener, ON, N2M 5C6 Canada
www.fibertech-optica.com
Designer and manufacturer of specialty fiber assemblies such as: Bundles, reflectance probes, spot-to-line converters, vacuum feedthrough, linear and spaced v-groove arrays, patchcords, pigtails and high power laser cables. Spectral bands coverage from deep UV to MIR. Distributor of specialty multimode all-silica, HCS, PCS, Hard Clad all-silica fibers**.** Core diameters from 10um to 2000um.

Fiveash Data Management, Inc. **Booth 53A**
211 Vista Rd.
Madison, WI 53726
www.fdmspectra.com
The new FDM Raman Minerals (11 libraries, 514, 532, 780 and 785 nm lasers, over 6,000 spectra) and the new FDM XRD Minerals will be demonstrated for the first time. The FDM Minerals spectral libraries are for the ID of minerals on laboratory and portable Raman and XRD spectrometers.\r\n\r\nThe new FDM Mixture Libraries and FDM Mixture Search will be available for demonstration. Both are for semiquantitative analysis of unknown mixtures. The FDM Mixture Libraries work with most FTIR search programs and require no additional software or training. The FDM Mixture Search is a new search algorithm built into the FDM Library Search and is used with Operant's Essential FTIR software. The FDM MultiSearch is a new feature of the FDM Library Search. Now, with the FDM MultiSearch, users can select multiple search algorithms and get a composite report. Users can effortlessly see which algorithms provide the best results

HORIBA Scientific **Booth 36**
Attn: Raman Spectroscopy
3880 Park Avenue
Edison, NJ 08820
www.molecularandmicroanalysis.com
Elemental or molecular spectroscopy, macro or micro, HORIBA Scientific has the right solution. We are the world leader in Raman and Fluorescence spectroscopy and imaging; offer the broadest line of spectroscopic components including sources, spectrometers, and detectors; plus EDXRF microscopes, Surface Plasmon Resonance (SPR) imaging, GDS and ICP OES. New innovations include the AccuRA transmission Raman system for solid dosage form and content uniformity analysis in the pharmaceutical industry. A new

affordable Raman inverted microscope the XploRA INV with DuoScan and microsecond imaging capability. ULF ultra low frequency accessory allowing filter based Raman spectroscopy down to less that 5cm-1. The XGT 7200 advanced XRF micoscope using SDD and small spot monocapillary technology. CLUE our new CL and Raman accessory and software for SEM and ESEM, and DynaMyc, a microspectrofluorometer featuring lifetime capabilities These join our complete family of Raman, elemental and fluorescence solutions, including XploRA - our powerful and low cost Raman microscope; SWIFT and DuoScan incredibly fast Raman hyperspectral chemical imaging solutions; steady-state and lifetime spectrofluorometers with TCSPC or phase and Photoluminor our complete line of PL spectrometers and solutions

Ibsen Photonics **Booth 28**
Ryttermarken 15-21
Farum, Other DK-3520 Denmark
www.ibsenphotonics.com
Ibsen Photonics is a global leader in holographic, fused silica, transmission gratings and spectrometer modules for a wide range of telecom, sensing and laser markets. Ibsen Photonics is a privately held company with headquarter in Farum, Denmark. For more information please visit www.ibsenphotonics.com.

ICP Information Newsletter, Inc. **Booth 21A**
PO Box 666
Hadley, MA 01035-0666
http:/icpinformation.org
ICP Information Newsletter, Inc. is a nonprofit corporation established in 1997 to foster science education, research, and study in spectroanalytical chemistry. The corporation includes three divisions: the ICP Information Newsletter, a monthly publication with international distribution that gathers all conference and published information related to plasma spectrochemistry; the Winter Conference on Plasma Spectrochemistry, a biennial meeting with international participation featuring state-of-the-art research developments in plasma spectrochemistry, and the University Research Institute for Analytical Chemistry, the research and development branch that provides provides specialty plasma spectrochemical analysis, method development, training, consulting, and applied research with ICP atomic emission and mass spectroscopy. The 2012 Winter Conferfence is scheduled for January 8-14, 2012. The ICP Information Newsletter now in its 36th year of publication is currently distributed to subscribers in computer-readable format on CD-ROM. Visit http://icpinformation.org for subscription details.

Innovative Photonic Solutions **Booth 44 & 45**
4260 U.S. Route 1, Suite 3
Monmouth Junction, NJ 08852
www.innovativephotonics.com
IPS is the leading manufacturer of Raman spectroscopy light sources for OEM and laboratory use. Our proprietary spectrum stabilized laser technology enables us to lock the laser to a specific wavelength and tailor the spectral linewidth without complex feedback mechanisms. The technology is applicable to both single and multi-mode lasers and enables the manufacture of both multi-watt multi-mode, and narrow linewidth (<100 KHz) single frequency lasers. IPS's lasers are frequently used for Raman spectroscopy, fiber laser seeding & pumping, THz Generation, remote sensing, interferometry and homeland security applications. Our products span the ~600 nm – 2400 nm wavelength range and are available in 14-Pin BF packages or in turn-key systems. Standard wavelengths include 730 nm, 740 nm, 785 nm, 808 nm, 830 nm, 976 nm, 1064 nm and 1550 nm. Other wavelengths are available upon request.

JASCO, Inc. **Booth 5**
28600 Mary's Court
Easton, MD 21601
http://www.jascoinc.com
JASCO specializes in analytical instruments for spectroscopy and chromatography applications, with over 50 years of experience. JASCO's worldwide presence, superior product quality and outstanding service and support make the company an industry leader. The full line of spectroscopy products includes FT-IR, Portable FT-IR, FT-IR microscopes, FT-Raman, UV-Vis/NIR, Fluorescence, Raman, portable Raman, NSOM, Polarimeters, Circular Dichroism and Dissolution testers. JASCO is also recognized for its robust and reliable chromatography instruments including SFC/SFE (analytical and preparative systems), HPLC and X-LC® (UHPLC).

Kaiser Optical Systems, Inc. **Booth 1 & 2**
371 Parkland Plaza
Ann Arbor, MI 48103
www.kosi.com
Kaiser Optical Systems, a Rockwell Collins Company, is recognized as a world leader in the design and production of Raman analyzers and components for spectroscopy. Our *RamanRxn Systems*™ suite of Raman analyzer includes ATEX certified process analyzers for classified installations, reaction analysis analyzers, bulk solids analyzers, gas-phase analyzers, Raman microscopes, and the *Raman WorkStation*™ featuring Kaiser's revolutionary fast, quantitative *P^{h}AT* technology and transmission Raman capability. Our components product lines include performance filters, high F/# spectrographs, and OEM systems. Raman analyzer installation locations include R&D, Pilot plant, manufacturing, and QA/QC. Pharmaceutical PAT applications include reaction monitoring, API production, polymorphic form quantitation, drug product unit operations (including blending, granulation, and tableting), and end product testing. Other Applications areas for *RamanRxn Systems* analyzers include biotech, semiconductors, nanotechnology, petrochemical, polymers, and specialty chemical. We invite you to visit our booth, learn about our products and discuss your applications needs.

LEAP Technologies **Booth 29**
PO Box 969
610 Jones Ferry Rd
Carrboro, NC 27510
http://www.leaptec.com/
LEAP offers an advanced H/D-X Sample Preparation workstation for scheduling and performing label experiments followed by direct injection to the LCMS. It is based on the CTC PAL robotic platform. H/D-X PAL™ is an easy-to-use system that provides an automated process for the scheduling and experimental execution of H/D-X experimental workflow. Reagent addition, timed reactions, quenching and injection are all handled using flexible control software. Temperature controlled zones allow reactions to take place at specified temperatures in the range of 1 – 40 Celsius. Columns and Injection valves are maintained at low temperature to minimize back-exchange. Experimental design is simplified by use of the advanced LEAP Shell scheduling software. Label experiments are synchronized with the MS and overlapped to provide optimal throughput. Reliable repeatable performance enhances the quality of experimental data. LEAP Technologies is the North American distributor for CTC Analytics, specializing in PAL sales, customization and support.

EXHIBITOR DESCRIPTIONS

Mettler Toledo **Booth 21**
7075 Samuel Morse Drive
Columbia, MD 21046
www.mt.com/autochem
METTLER TOLEDO is the world leader in *in situ* spectroscopy technology and provides scientists with tools to continuously monitor chemical reactions for a deeper understanding of reaction kinetics, mechanisms and pathways. ReactIR™ is used in the lab for organic synthesis, process development and as a PAT tool to enable scientists to gain a deeper insight into their chemistry. It is a valuable tool for measuring and understanding reaction progression, initiation, con¬version, intermediates and endpoint, determines if the desired reaction occurred and provides associated endpoint determination without offline analysis. ReactIR™ is used for scale-up studies, campaigns and production monitoring. It provides non-destructive, rapid, quantitative chemical analysis of key reaction components in the reactor. ReactIR™ for production supports the design of safe, robust processes that build quality into the commercial process. This allows for process consistency, batch repeatability and eliminates failures at the manufacturing scale.

Nanonics Imaging Ltd **Booth 6 & 7**
POB 48330
Manhat Technology Park
Jerusalem 91487, Israel
www.nanonics.co.il
Single & Multiprobe AFM/NSOM/SPM Systems hallmarked by transparent optical integration into all microscopes: upright, inverted and special dual microscope configurations. On-line AFM-Raman/TERS, confocal, SEMs, FIBs, 10°K operation. Ideal for multiprobe plasmonics, electrical characterization, photonics, thermal diffusivity, chemical writing and other unique SPM operations. Capable of all modes of AFM/NSOM with silicon or transparent AFM probes. Deep trench and side wall imaging capabilities, glass insulated electricalprobes, Nanoheater™ thermal conductivity, AFM-controlled gas/liquid nanochemical deposition, electrochemical probes.

NSI Solutions, Inc. **Booth 30**
7212 ACC Blvd
Raleigh, NC 27617
www.nsi-es.com
NSI Solutions is an ISO9001:2008 registered manufacturer of certified reference materials and laboratory reagent solutions for the environmental testing and pharmaceutical quality control laboratories. Products displayed include: A2LA Accredited PT Standards, Certified Quality Controls Standards for Environmental Analysis, Microbiological QC Standards, Ready to Use LCS/QC Check Standards in single use teflon tubes, ICP/ICP-MS standards, USP Dissolution Testing Media, USP Reagent Concentrates, Pharmaceutical HPLC OQ/IQ Standards, HPLC and IC Mobile Phase concentrates and custom formulation services.

Ocean Optics, Inc. **Booth 43**
830 Douglas Avenue
Dunedin, FL 34698
www.oceanoptics.com
Manufacturer of efficient, compact, and broadly tunable ultraviolet, visible, near and mid infrared solid-state laser systems based on OPOTEK patented Optical Parametric Oscillators (OPO). These products stand out in their efficiency, reliability and robustness as well as compact, all-in-one designs. Applications include mass spectrometry, photochemistry, photobiology, medical-diagnostics, spectral imaging and environmental monitoring. The systems are computer controlled via a single USB connection and require no expertise in laser operation to use. OPOTEK also manufacturers the HySPEC, a complete HyperSpectral Imaging system. The HySPEC combines spectroscopy and imaging to create a powerful tool for identifying and quantifying components in heterogeneous samples, e.g., pharmaceutical powders, raw materials, tablets, biological samples, food, lotions, textiles, consumer products, etc. The system collects complete spectra in seconds. The HySPEC will be available for demonstration at the exhibition.

OI Analytical **Booth 50**
151-A Graham Rd.
College Station, TX 77845
www.oico.com
OI Analytical will feature the iTOC-CRDS Isotopic Carbon Analyzer for measuring the total organic carbon (TOC) content of solid or liquid samples along with the δ 13C stable isotope ratio by cavity ring down spectroscopy. Also featured will be the IonCam™ 2020 transportable GC-MS with miniaturized double-focusing mass spectrometer and ion charge-coupled-device (IonCCD) array detector for real-time measurement of transient chemical species

Ondax, Inc. **Booth 32**
850 E. Duarte Rd.
Monrovia, CA 91016
www.ondax.com
Ondax Inc. is the leading manufacturer of ultra-narrow notch filters, ASE filters and Wavelength Stabilized Laser Diodes and Modules. The SureLock™ product line of single frequency and MM lasers are available in standard wavelengths of 405nm, 640nm, 658nm, 780.25nm, 785nm and 808nm. Custom laser wavelengths and powers are also available.The ultra-narrow (10cm-1) SureBlock™ laserline notch filters provide an O.D of 4 to 6 and the ASE NoiseBlock™ filters have a 0.1nm bandpass. These 2 product lines work with any of our standard or custom stabilized lasers to take your Raman system to the next level of performance. Ondax VHGs are manufactured using a very stable and robust proprietary glass to withstand harsh environments and do not degrade with time.More information can be obtained at www.ondax.com or by contacting sales@ondax.com.

OPOTEK, Inc. **Booth 37**
2233 Faraday Avenue
Suite E
Carlsbad, CA 92008
www.opotek.com
Manufacturer of efficient, compact, and broadly tunable ultraviolet, visible, near and mid infrared solid-state laser systems based on OPOTEK patented Optical Parametric Oscillators (OPO). These products stand out in their efficiency, reliability and robustness as well as compact, all-in-one designs. Applications include mass spectrometry, photochemistry, photobiology, medical-diagnostics, spectral imaging and environmental monitoring. The systems are computer controlled via a single USB connection and require no expertise in laser operation to use. OPOTEK also manufacturers the HySPEC, a complete HyperSpectral Imaging system. The HySPEC combines spectroscopy and imaging to create a powerful tool for identifying and quantifying components in heterogeneous samples, e.g., pharmaceutical powders, raw materials, tablets, biological samples, food, lotions, textiles, consumer products, etc. The system collects complete spectra in seconds. The HySPEC will be available for demonstration at the exhibition.

OptiGrate Corp **Booth 8**
3267 Progress Drive
Orlando, FL 32826
www.optigrate.com
OptiGrate Corp is a 10 year old company that manufactures ultra narrow band optical filters based on volume Bragg grating (VBG) Technologies in photosensitive glass. We supply holographic

optical elements to hundreds of customers on 5 continents. Our new product lines of ultra narrow band Notch and Bandpass filters, available at 488, 532, 633, 785, and 1064 nm wavelengths, will allow you to upgrade your Raman system to measure Stokes and Anti-Stokes frequencies down to 5 cm^{-1} with a single-stage monochromator. We can also provide filters at custom wavelengths in a range from 400 to 2000 nm.

Oriel Instrument, Inc. **Booth 3**
150 Long Beach Blvd.
Stratford, CT 06349
www.newport.com/oriel
Oriel® Instruments, a brand of Newport Corporation, has developmed recognition in the optical research field as a reliable source for well engineered, durable Light Sources and their dedicated Power Supplies, as well as Light Detection Systems and Spectroscopy Instrumentation. Oriel also manufactures dedicated broadband light sources, monochromatic light sources and detectors for light measurement & characterization in sophisticated dedicated instrumentation.

Oxxius **Booth 42**
4 rue Louis de Broglie
Lannion 22300, France
www.oxxius.com
New Product: SLIM DPSS at 553, 660 nm SLM lasers; LaserBoxx at 405, 488 and 640nm, circular beam Laser Diode Modules. Oxxius manufactures innovative Diode Pumped Solid-State Lasers and Laser diode Modules for industrial and scientific applications. Its compact laser modules, featuring wavelengths in the visible and near-UV spectrum, have outstanding performance with market-leading power levels. The SLIM line features a monolithic resonator technology that ensures true single-frequency emission along with excellent power, wavelength and pointing stability. Available wavelengths are 375, 405, 445, 473, 488, 532, 550, 561 and 660 nm.

PAIR Technologies **Booth 49**
1 Innovation Way, Ste 304
Newark, DE 19711
www.pairtech.com
PAIR Technologies, LLC is the world leader in planar array infrared spectroscopy. We are introducing our new double beam infrared spectroscopic instrument. Employing a true double beam optical path, the need for purging is eliminated and instrument drift is minimized. Rugged and stable, the spectrometer can measure extremely small absorbance differences between sample and references, making it useful for protein solutions, nanoparticle measurements, rapid reactions and irreversible processes. With measuring times in the milliseconds, molecular interactions can be observed and extremely thin films can be measured. The rugged construction allows use in laboratory and industrial environments with a degree of information gathering heretofore unavailable. PAIR Technologies brings unmatched signal to noise, stability and speed to IR spectroscopy.

PerkinElmer Life & Analytical Sciences **Booth 35**
710 Bridgeport Avenue
Shelton, CT 06484
www.perkinelmer.com
PerkinElmer is a global scientific company focused on improving health and safety of people and their environment, from earlier medical insights and more effective therapies to cleaner water, safer products and more secure homes. We provide laboratory services and analytical instrumentation in diverse markets including Thermal Analysis, FTIR, FT-NIR, UV/Vis/NIR & Raman, GC/MS, LC/MS, LC/UV, Thermal Desorption, Atomic Absorption, ICP-Emission and ICP-Mass Spectrometry technology. OneSource delivers the most comprehensive multi-vendor laboratory services portfolio, including repair, maintenance, lab relocation, asset management and regulatory compliance solutions.

Princeton Instruments, Inc. **Booth 39**
3660 Quakerbridge Rd.
Trenton, NJ 08619
www.princetoninstruments.com
Princeton Instruments (PI) is the proven choice for high-performance CCD camera, spectroscopy and optical coating solutions for demanding research and industrial applications, with over 30 years experience helping scientists solve challenging problems. PI's versatile TriVista Confocal Raman Microscope System combines the high resolution, low frequency and tuning capabilities of the renowned Acton Series triple spectrograph with our extensive range of high performance CCD detectors, Olympus microscopes and accessories, to provide systems with both macro and micro sampling capabilities. Many of our spectroscopy cameras, including the deeply cooled Spec-10 and popular PIXIS platforms, are now available with PI's exclusive eXcelon CCD technology, providing enhanced sensitivity, reduced etaloning and lower dark current. The LS-785 offers outstanding throughput and imaging performance in a NIR lens spectrograph. Applications and techniques include Raman, fluorescence, photoluminescence and biomedical spectroscopic imaging. New – ProEM EMCCD Camera for Spectroscopy – stop by Princeton Instruments at booth # 39 for more information.

Renishaw, Inc. **Booth 46 & 47**
5277 Trillium Blvd.
Hoffman Estates, IL 60192
www.renishaw.com

RoMack, Inc. **Booth 41**
PO Box 615
Lightfoot, VA 23090
www.romackfiberoptics.com
RoMack, Inc. manufacturers fiberoptic aassemblies, components and related products specifically tailored for spectroscopic, laser, pharma and medical applications. Products include probes, fiberoptics, connectors, adapters, patchcords, bundles, arrays, imagers, collimators, couplers, tapers and filter packages. RoMack routinely takes concept to product, creating solutions to the most difficult problems. For Sales please contact Lisa Young at contact@romackfiberoptics.com or by phone 1-757-258-4805.

Royal Society of Chemistry **Booth 19**
Thomas Graham House
Science Park, Milton Road
Cambridge, UK CB4 0WF
www.rsc.org
The Royal Society of Chemistry (RSC) is the largest organisation in Europe for advancing the chemical sciences, supported by 45,000 members worldwide and an internationally acclaimed publishing business. At FACSS 2010, meet RSC Publishing staff and visit our booth to pick up a free copy of Analyst, the journal for leading edge research in interdisciplinary analytical, bioanalytical and detection science, Journal of Analytical Atomic Spectrometry (JAAS), publishing fundamentals in elemental analysis and isotope ratio determinations, as well as our new journals Metallomics, providing insight into the role of trace elements in the life sciences, and Analytical Methods, highlighting new and improved methods for the practical application of analytical science.

EXHIBITOR DESCRIPTIONS

RPMC Lasers, Inc. **Booth 5**
203 Joseph Street
OFallon, MO 63366
www.rpmclasers.com
Since 1996, RPMC has been offering high quality solid state lasers and laser diode products from the world's leading manufactures. We offer a wide variety of CW and QCW diode pumped lasers, standard mil qualified diode pumped lasers, along with pulsed and cw fiber lasers. Our laser diode products include high power single emitters, bars, arrays, fiber coupled modules, and single mode diodes. Standard products range from 622 nm to 1.8 mm, power levels from 5mW to 120W CW. Custom diode packaging is also available. We can offer you a complete solution for your diodes requires including diode drivers, laser diode mounts and cooling systems.

Society for Applied Spectroscopy **Booth 14,15 &16**
201B Broadway Street
Frederick, MD 21701-6501
www.s-a-s.org
The Society for Applied Spectroscopy is a non-profit, membership organization dedicated to serving and educating scientists in the field of spectroscopy. Membership includes a subscription to the internationally recognized, peer-reviewed journal, Applied Spectroscopy.

Spectroscopy Magazine / Advanstar **Booth 27**
485 Route 1 South, Bldg F, 1st Fl
Iselin, NJ 08830
www.spectroscopyonline.com
Spectroscopy's mission is to enhance productivity, efficiency, and the overall value of spectroscopic instruments and methods as a practical analytical technology across a variety of fields. Scientists, technicians, and laboratory managers gain proficiency and competitive advantage for the real-world issues they face through unbiased peer-review technical articles, trusted troubleshooting advice, and best-practice application solutions. The Spectroscopy brand now encompasses a wide variety of vehicles, delivering industry-leading technical content to the largest audited circulation of spectroscopists in the U.S. using print, digital, e-newsletters, podcasts and webcasts, and our ever-expanding social networking groups on LinkedIn and Twitter

SPEX CertiPrep **Booth 3A**
203 Norcross Ave.
Metuchen, NJ 8840 USA
www.spexcsp.com
SPEX CertiPrep is the leading manufacturer of Organic and Inorganic Certified Reference Materials for Spectroscopy, Chromatography, and other analytical instrumentation. We are Certified by UL-DQS for ISO 9001 and accredited by A2LA for ISO/IEC 17025 and ISO Guide 34 with the most comprehensive scope in the industry. Providing scientists with certified reference materials and sample preparation equipment for a diverse range of analytical techniques.

Taylor & Francis **Booth 13**
325 Chestnut St., Ste 800
Philadelphia, PA 19106
www.taylorandfrancis.com
For two centuries Taylor & Francis has been fully committed to the publication of scholarly information of the highest quality, and today this remains our primary goal. Taylor & Francis has grown rapidly over the last two decades to become a leading international academic publisher. With offices throughout the world, the Taylor & Francis Group publishes more than 1,100 journals, including Analytical Letters, Applied Spectroscopy Reviews, and Spectroscopy Letters.

Thermo Scientific **Booth 24, 25 &26**
5225 Vernona Road
Madison, WI 53711
www.thermoscientific.com/scientificinstruments
www.thermoscientific.com/ahura
Stop by booth 24/25/26 to learn about Thermo Scientific high-end analytical instruments. Thermo Scientific instruments for elemental analysis range from AA and ICP to ICP-MS and high resolution inorganic mass spectrometry to Spark-OES and XRF. Our elemental analysis solutions combine reliability, superior performance, versatility and ease-of use to solve even complex problems. In addition, we offer high performance FT-IR, IR microanalysis and imaging, dispersive Raman and NIR technology that allow researchers the flexibility to build custom experiments and complete demanding analyses of molecular structures. The Thermo Scientific TruScan handheld instrument is a rugged, ultra-compact, field-enabled optical system for the immediate identification and verification of liquid and solid chemical substances. Designed to facilitate the inspection of incoming raw materials and identification of counterfeit pharmaceuticals, TruScan is now utilized by nine of the Top Ten global pharmaceutical manufacturers. This Raman based system allows users to realize significant time and cost savings in material sampling. TruScan supports 21 CFR part 11 compliance. Support this technology with our proven Laboratory Information Management Systems (LIMS) and Chromatography Data Systems (CDS) to help you lower costs, increase productivity and maximize uptime with our 24/7 worldwide support and service staff. For more information, visit www.thermoscisni.com.

Varian, Inc. – now Agilent Technologies **Booth 11**
2700 Mitchell Drive
Walnut Creek, CA 94598
800.926.3000
www.agilent.com
Agilent manufactures and distributes a complete line of instrumentation serving the clinical, analytical, biotech, environmental, pharmaceutical, forensic science, food and flavor, academia, and all other laboratory markets that have needs for the best in quality, performance, and serviceability in the instruments they purchase.

Wasatch Photonics **Booth 18**
Systems Division
4020 Stirrup Creeks Drive
Durham, NC 27703
www.wasatchphotonics.com
Wasatch Photonics specializes in high performance Volume Phase Holographic Gratings (VPHG) and Holographic Optical Elements. Our Systems Division has developed a variety of innovative designs to take advantage of our high performance VPHG in Raman Spectrometer and Optical Coherence Tomography (OCT) spectral engines and systems.

WITec Instruments **Booth 40**
200 East Broadway - Suite 30
Maryville, TN 37804
www.WITec-Instruments.com
WITec is a manufacturer of high resolution optical and scanning probe microscopy solutions for scientific and industrial applications. A modular product line allows the combination of different microscopy techniques such as Raman, NSOM or AFM in one single instrument for flexible analyses of optical, chemical and structural properties of a sample.

WORKSHOPS

Workshops are a valuable component of FACSS and are conducted by leading experts. There is an additional charge for workshops. See on-site registration form for costs.

ANALYTICAL RAMAN SPECTROSCOPY

Brian Marquardt, *University of Washington;* **Jeremy Shaver,** *Eigenvector Research, Inc.;* **Ian Lewis,** *Kaiser Optical*

Sunday, 8:00 am – Noon

The course will provide an overview of modern Raman spectroscopy beginning with an introduction to Raman scattering and the differences between IR and Raman spectra. It will include discussion of sampling, calibration, data analysis methods (pre-treatments and modeling approaches), and successful application developments. Modern instrument configurations and configuration choices will be covered. The course will include a thorough introduction to the major approaches to sample illumination and spectrum collection, emphasizing fiber optic probes and Raman microprobes. Raman imaging will be briefly discussed. The applicability of and successes with Raman will be surveyed with numerous applications examples. This ½ day course will be split 50 / 50 between a) Raman practical considerations and theory and b) applications.

INDUCTIVELY COUPLED PLASMA- MASS SPECTROMETRY (ICP-MS): INTRODUCTION

R. S. Houk, *Ames Laboratory USDOE, Iowa State University*

Sunday, 8:00 am – Noon

This course is meant for the beginner in ICP-MS.
Course Topics:

- The ICP as an Ion Source
- Ion Extraction and Beam Formation
- Operating Principles of Ion Lenses, Quadrupole Mass Analyzers, and Detectors
- Magnetic Sector Mass Analyzers with the ICP
- Causes of and Corrections for Spectral Interferences and Matrix Effects
- Survey of Methods to Remove Polyatomic Ions - Cool Plasma, Collision Cells, Solvent Removal Survey of Applications
- Designing a Sound Analytical Strategy Using ICP-MS

RAMAN CHEMICAL IMAGING AND TECHNOLOGIES AND METHODS

Ryan J. Priore and Matt Nelson, *ChemImage*

Sunday, 8:00 am – Noon, *Room 302B, CC*

Chemical Imaging is a rapidly maturing discipline that involves the integration of digital imaging with molecular spectroscopy, relying on Raman, fluorescence, visible, NIR and IR absorption/reflectance techniques. It has evolved as a powerful approach for non-invasive characterization of chemical heterogeneity. Among these techniques, Raman Chemical Imaging is particularly suited for microspectroscopy and imaging due to the inherent selectivity and sensitivity of Raman spectroscopy. Two different imaging approaches, spatial and wavelength scanning, are used to acquire a hyperspectral data cube containing wavelength, intensity, x, y and z- spatial information. This course emphasizes wavelength scanning approaches to Raman Chemical Imaging which enable rapid image acquisition with diffraction-limited spatial resolution. In widefield Raman Chemical Imaging, thousands of Raman spectra are simultaneously collected from a field of view. The spectral data is used to generate chemically-specific images. Intricate image and spectral processing techniques may be applied to the chemical imaging data to produce various qualitative and/or quantitative parameters associated with the often complex sample matrix. This short course is designed as a comprehensive overview of theoretical and practical aspects of Raman Chemical Imaging and associated instrumentation. The special emphasis is placed on Raman Chemical Imaging applications in pharmaceutical and biothreat detection fields. This course will be useful to analytical, forensic, biomedical and materials chemists.

CHEMOMETRICS WITHOUT EQUATIONS (or Hardly Any) - HANDS ON!

Jeremy Shaver and Neal Gallagher; *Eigenvector Research, Inc.*

Sunday and Monday, 9:00 am – 5:00 pm

Chemometrics without Equations concentrates on two areas of chemometrics: 1) exploratory data analysis and pattern recognition, and 2) regression. Participants will learn to safely apply techniques such as Principal Components Analysis (PCA), Principal Components Regression (PCR), and Partial Least Squares (PLS) Regression. Examples will include problems drawn from process monitoring and quality control, predicting product properties, and others. The target audience includes those who collect and/or manage large amounts of data that is multivariate in nature. This includes bench chemists, process engineers, and managers who would like to extract the most information from their measurements. The course will finish with a short section on how to apply these models for online predictions, Multivariate Statistical Process Control and inferential sensing. Students will work problems using MATLAB and PLS_Toolbox on computers provided (maximum of two students per computer).

WHAT MODERN NEAR-IR SPECTROSCOPY CAN DO FOR YOU

Donald Burns

Sunday, 9:00 am – 5:00 pm

Morning

Basic NIR Concepts:

Overview & history; principles & theory; advantages & disadvantages; basis of absorptions (overtones & combination bands).

Calibration & Wavelength Selection

Search strategies; teaching/learning & confirmation sets; MLR, PCR; hidden information (via derivatives & zero-crossing techniques).

Software & Chemometrics

Mahalanobis distances; discriminant analysis; spectral reconstruction; indicator variables; 3rd Party offerings; qualitative & quantitative analysis; error propagation.

Method Development

General rules; how many samples (and why?); feasibility studies; some basic statistics.

Afternoon

On-Line Analysis

Liquids, solids, gases; use of fiber optics; case study with plastics.

Applications

Tobacco & cigars; agricultural products; polymers; pharmaceuticals; medical uses; artwork; food & beverages; petrochemicals; textiles; lignin; actinides; detection of counterfeit items (drugs, ivory, animal feeds, turquoise, currency).

Instrumentation – who makes what?

Classification; principle of operation (filters, gratings, diode arrays, scanning, FT, AOTF); attachments (fiber optics, samplers); dedicated units.

The Future – How to Learn More

Meetings, courses, books & journals, audio seminars, groups to join, the Internet, a basic bibliography on disk.

WORKSHOPS

PROCESS ANALYTICAL CHEMISTRY: OUT OF THE LAB AND INTO THE PIPE

James Rydzak, *GlaxoSmithKline*

Tuesday, 9:00 am – 5:00 pm

Process Analyzers are becoming more important to the manufacturing industry by providing improved process quality, yields, uptimes and safety, while reducing hazards and environmental impact. This course will answer a question frequently posed by laboratory analytical chemists: "What is process analytical chemistry and how does it differ from more traditional laboratory-based analysis?" It will introduce basic relevant engineering concepts, and compare process analyzers with laboratory instrumentation. The course will primarily focus on on-line and in-line applications of optical and mass spectrometry, gas chromatography, and titrimetry as they are applied in the refining, chemicals, petrochemicals, food, personal care, pharmaceuticals, and life science industries.

ADVANCED CHEMOMETRICS WITHOUT EQUATIONS - HANDS ON!

Jeremy Shaver and **Neal Gallagher;** *Eigenvector Research, Inc.*

Tuesday, 9:00 am – 5:00 pm

The critical difference between inadequate and successful chemometric models is often data preprocessing, i.e. what is done to the data before using PCA, PLS etc. The goal of preprocessing is to remove variation not related to the problem of interest so that the variation of interest is more evident and can be more easily modeled. The variables selected, e.g. spectral regions, can also greatly affect the success of the application. This course focuses on advanced preprocessing methods, including Extended Multiplicative Scatter Correction (EMSC) and Generalized Least Squares (GLS), for improving models. Variable selection techniques, such as interval PLS (iPLS) are also considered. The effect of preprocessing and variable selection on robustness of the final models is also considered.

NEAR INFRARED CHEMICAL IMAGING

Linda Kidder and Kevin Dahl, *Malvern Instruments*

Tuesday, 1:00 – 5:00 pm

Near infrared chemical imaging (NIRCI) is widely used in the pharmaceutical and consumer products industries to measure the spatial distribution of chemical components; this structural information is often highly correlated with product performance. The technique's easy sample presentation and data acquisition have contributed to its popularity with laboratory personnel, while the information-rich data and automation of data processing into quantitative tabulated values make it extremely valuable for formulation and quality control managers. In this workshop addressed to beginners and intermediate level users, we will cover both the instrumentation and data analysis with relation to the broad range of applications typically tackled with this technique. For example, the elucidation of product structure, trouble-shooting of dissolution failure, formulation design and implications in QbD will be discussed. We invite attendees to contact the instructors ahead of the workshop if there are topics that you would like to discuss more in depth.

ANALYTICAL PROBLEM SOLVING FOR UNDERGRADUATES

Alan Ullman, *The Procter & Gamble Company*

Wednesday, 9:00 am – 5:00 pm No Charge

The objective of this workshop is to provide insight into the work of industrial analytical chemists. In a highly interactive forum, participants will explore the role of analytical chemist as problem solver using real problems encountered at Procter & Gamble. Participants have an opportunity to try their hand at solving real consumer product chemistry problems, and to get answers to some of their questions on industrial chemistry careers. The course is targeted at third-year undergraduates who have had some exposure to instrumental analysis; however, new undergraduate students, graduate students, and chemistry teachers have all reported that they found the short course highly beneficial. For additional information go to: www.pg.com/science/prof_chemists.jhtml.

PRACTICAL APPLICATIONS OF MASS SPECTROMETRY FOR SMALL MOLECULES

Michael Balogh, *Waters*

Wednesday, 9:00 am – 5:00 pm

Comprehensive focus on understanding the fundamentals of the most widely used mass spectrometers and sample inlet technologies: how and where are they employed to the practitioners' best advantage. The examples include how designing the separation and avoiding pitfalls of combining LC, GC and MS can increase analytical success. Discussion includes the latest technologies such as non-LC atmospheric ionization techniques DESI, DART, ASAP and MALDI imaging for tissues. The course is designed for anyone in pharma, industry or environmental practice employing MS for small molecule investigations including design of experiment considerations (column chemistries, solvents and ionization methods), quantitative considerations, qualitative considerations (spectral interpretation) and identification of target compounds in complex matrices. In addition to course materials, references for further study and glossaries explaining commonly used terms, the take-home value of attending for students is gaining an overall view of current mass spectrometry practice and the opportunity to address individual questions.

HANDS-ON CHEMOMETRIC ANALYSIS WITH THE UNSCRAMBLER®

Katherine Bakeev, ***CAMO Software***

Wednesday, 9:00 am – 5:00 pm

This workshop is aimed at giving an overview of the principles and use of multivariate methods with exercises and examples specific to spectroscopic data analysis. It provides a combination of theory and practice, with an emphasis on PCA and PLS model development and interpretation.

THIS YEAR ACS SPONSORED THREE WORKSHOPS AT FACSS 2010:

- Practical Approaches to Patents and Other Forms of Intellectual Property
- NMR Spectral Interpretation and Organic Spectroscopy: A Problem-Based Learning Approach
- Laboratory Safety

PROGRAM HIGHLIGHTS

SUNDAY	MONDAY	TUESDAY	WEDNESDAY	THURSDAY
	7:30 Wake up coffee Ballroom Lobby	7:30 Wake up coffee Ballroom Lobby	7:30 Wake up coffee Ballroom Lobby	7:30 Wake up coffee Ballroom Lobby
8:00 AM – 5:00 PM FACSS Workshops	*Ballroom A* Plenary Session 8:00 am Christy L. Haynes *University of Minnesota*	*Ballroom A* Plenary Sessions 8:00 am Charles Mann Award Richard McCreery *National Institute for Nanotechnology, Canada* 8:30 AM ANACHEM Award Marc D. Porter *Institute of Utah*	*Ballroom A* Plenary Sessions 8:00 am SAS Applied Spectroscopy William F. Meggers Award Paul Gemperline *East Carolina University* 8:30 AM Lester W. Strock Award Kay Niemax *BAM*	*Ballroom A* Plenary Sessions 8:00 am Coblentz Clara Craver Award Boris Mizaikoff *University of Ulm* 8:30 am Ellis R. Lippincott Award Martin Moskovits *Univ. of California, Santa Barbara*
	9:00 am – 5:00 pm Workshops	9:00 AM – 5:00 PM Workshops	9:00 – 5:00 pm Workshops	
		9:00 AM – 4:30 PM Exhibits Open *Ballroom B*	9:00 AM – 3:00 pm Exhibits Open *Ballroom B*	
	9:00 – 10:30 pm Poster Session and Break *Ballroom Lobby*	9:00 – 10:30 am Poster Session and Break *Ballroom B*	9:00 – 10:30 am Poster Session and Break *Ballroom B*	9:00 – 10:30 am Poster Session and Break *Ballroom Lobby*
	10:30 am – 12:30 pm Oral Symposia	10:30 am – 12:30 pm Oral Symposia	10:30 am – 12:30 pm Oral Symposia	10:30 am – 12:30 pm Oral Symposia
	12:30 pm Lunch on own	12:30 pm Lunch on own	12:30 pm Lunch on own	12:30 pm Lunch on own
	12:30 PM Lunch and Roundtable discussion for students. Sponsored by SABIC Innovative Plastics *Room 307*		12:30 pm FACSS Planning Meeting for conferees and exhibitors	
		1:30 pm Poster Viewing and Dessert Reception *Ballroom B*	1:30 pm Poster Viewing and Dessert Reception *Ballroom B*	1:30 pm Poster Viewing and Break *Ballroom Lobby*
4:30 – 7:00 pm What's Hot Vendor Presentations	2:00 – 4:00 pm Oral Symposia	2:00 – 4:00 pm Oral Symposia	2:30 – 4:30 Oral Symposia	2:00 – 4:00 pm Oral Symposia
	4:00 pm Poster Viewing and Break	4:00 pm Poster Viewing and Break *Ballroom B/C*		
	4:20 pm Plenary Session Uli Hacksell *ACADIA Pharmaceuticals*	4:20 pm Plenary Session Robert S. Houk *Iowa State University*	4:30 pm FACSS Award Presentations 4:40 pm Plenary Session Alex Scheeline *University of Illinois at Urbana-Champaign*	
		6:00 pm Raman Reception *Room 402, convention center*		
7:00 – 9:00 pm Welcome Mixer and SAS Sponsored Student Poster Session Coblentz Student Awards *Ballroom Lobby*	5:30 – 7:30 PM Exhibit Opening Reception *Ballroom B*	7:00 pm SAS Reception *(SAS members only)* *State Ballroom, Marriott*	6:00 pm FACSS All Inclusive Wednesday Evening Event *Ballroom A*	

PROGRAM OVERVIEW

SUNDAY

4:40 – 7:00 pm **What's Hot Vendor Presentations,** *Ballroom A*

7:00 – 9:00 pm **Welcome Mixer and SAS Sponsored Student Poster Session, Coblentz Student Awards,** *Ballroom Lobby*

MONDAY MORNING

8:00 am **PLENARY LECTURE,** *Ballroom A*
ACS Division of Analytical Chemistry Arthur F. Findeis Award for Achievement by a Young Analytical Scientist; **Christy L. Haynes**, page 45

9:00 am **POSTER SESSION,** page 45

10:30 am **SYMPOSIA,** page 47
Materials Characterization using Vibrational Spectroscopy and Mass Spectrometry, *Rm 301B*
Celebrating 30 Years of ICP-MS, *Rm 302A*
Laser Induced Breakdown Spectroscopy, *Rm 302B*
Novel But Important Data Analysis Techniques in Analytical Chemistry, *Rm 302C*
ACS Division of Analytical Chemistry Arthur F. Findeis Award, *Rm 304*
Portable NMR and Hyphenation of NMR to Chromatography and Electrophoresis, *Rm 305A*
Near and Mid-Infrared Imaging, *Rm 305B*
Special Memorial Seminar Session: How One Man Changed the World of FTIR and Infrared Spectroscopy: The Don Sting Legacy, *Rm 306A*
PAT in the Biopharmaceutical and Pharmaceutical Industries SAS Process Session, *Rm 306B*
Emerging Areas in Raman Spectroscopy, *Rm 306C*

MONDAY AFTERNOON

2:00 pm **SYMPOSIA,** page 49
Ionic Liquids and Spectroscopy: A Green and Happy Marriage, *Rm 301B*
Celebrating 30 Years of ICP-MS, *Rm 302A*
Laser Induced Breakdown Spectroscopy, *Rm 302B*
Chemometrics in the Pharmaceutical Industry, *Rm 302C*
ACS Division of Analytical Chemistry Arthur F. Findeis Award, *Rm 304*
Chromatography in the Pharmaceutical Industry, *Rm 305A*
Emerging and Non-Traditional Electrophoresis Techniques, *Rm 305B*
Quantum Cascade Laser Applications, *Rm 306A*
PAT: New Technology, *Rm 306B*
Biomedical Raman Spectroscopy, *Rm 306C*

4:00 pm **POSTER VIEWING**

4:20 pm **PLENARY LECTURE,** *Ballroom A*
The Role of Analytical Chemistry and Spectroscopy in Drug Discovery and Development: Challenges and Opportunities, **Uli Hacksell**, page 45

TUESDAY MORNING

8:00 AM **PLENARY LECTURES,** *Ballroom A*
Charles Mann Award, Richard McCreery, page 52
ANACHEM Award, Marc D. Porter, page 52

9:00 am **POSTER SESSION,** page 52

10:30 am **SYMPOSIA,** page 54
Headspace Analysis for Chemical Signatures, *Rm 301B*
Atomic Analyses in the Pharmaceutical and Neutraceutical Industry, *Rm 302A*
The JAAS Silver Anniversary Celebration: Highlighting Young Investigators in Atomic Spectroscopy, *Rm 302B*
Chemometrics in Forensics, *Rm 302C*
ANACHEM Award, *Rm 304*
Dielectrophoresis and Related Techniques, *Rm 305A*
Emerging Technologies for Standoff Detection for Homeland Security, *Rm 305B*
Special Session to Honor William G. Fately, *Rm 306A*
SAS Process Analytical Technology, *Rm 306B*
Raman Microscopy and Imaging, *Rm 306C*
Nanotechnology: Applications to Sensing and Energy, *Rm 307*

TUESDAY AFTERNOON

1:30 pm **POSTER VIEWING**

2:00 pm **SYMPOSIA,** page 57
MS Fundamentals and Gas-Phase Chemistry, *Rm 301B*
Chemistry in Art and Archaeology, *Rm 302A*
ICPMS – A Lot More Than Total Metals Analysis, *Rm 302B*
Chemometrics for Biological and Biomedical Spectroscopy, *Rm 302C*
Charles Mann Award, *Rm 304*
Practical Aspects of Chiral Analysis Using VCD and ROA, *Rm 305A*
Lasers in Analytical Chemistry: Celebrating the 50th Anniversary of the Laser, *Rm 305B*
Special Session to Honor William G. Fateley, *Rm 306A*
Next Generation Spectroscopic Methods for the Analysis of Pharmaceutical Systems, *Rm 306B*
Tip Enhanced Raman Spectroscopy – Pushing the Limits of Resolution, Rm *306C*
Nanotechnology: Applications to Sensing and Energy, *Rm 307*

4:00 pm **POSTER VIEWING**

4:20 pm **PLENARY LECTURE,** *Ballroom A*
Celebration of 30 Years of ICP-MS, **Robert S. Houk**, page 52

PROGRAM OVERVIEW

WEDNESDAY MORNING

8:00 am **PLENARY LECTURES,** *Ballroom A*
SAS Applied Spectroscopy William F. Meggers Award, Paul Gemperline, page 60
Lester W. Strock Award, Kay Niemax, page 60

9:00 am **POSTER SESSION,** page 60

10:30 am **SYMPOSIA,** page 62
Probing Interactions between Biomolecules and Nanomaterials, *Rm 301B*
Near-IR Spectroscopy: Applications and Calibrations, *Rm 302A*
Vibrational Spectroscopy at Work in the Pharmaceutical Industry, *Rm 302B*
Lester Strock Award – Atomic Spectroscopy Progress Reports, *Rm 304*
Bioanalytical Separation Science, *Rm 305A*
Optical Effects in Infrared Spectroscopic Imaging, *Rm 305B*
Two-Dimensional Correlation Spectroscopy, *Rm 306A*
Sustainability and PAT Applications, *Rm 306B*
New SERS Architectures, *Rm 306C*
Terahertz Spectroscopy, *Rm 307*

WEDNESDAY AFTERNOON

1:30 pm **POSTER VIEWING**

2:30 pm **SYMPOSIA,** page 64
Nanomaterials for Surface Plasmon Resonance, *Rm 301B*
Novel Sample Introduction Methods for ICP-MS and ICP-OES, *Rm 302A*
Fundamental Studies and Exciting New Applications of Glow Discharge Spectroscopy, *Rm 302B*
Advances in Mass Spectrometry Instrumentation, *Rm 302C*
Meggers Award – Advances in Hyperspectral Imaging, *Rm 304*
Student Awards, *Rm 305A*
Biomedical Applications of Spectroscopic Imaging, *Rm 305B*
Two-Dimensional Correlation Spectroscopy, *Rm 306A*
Monitoring Continuous Chemistry and Chemical Processes, *Rm 306B*
Applications of SERS, *Rm 306C*
Terahertz Spectroscopy, *Rm 307*

4:30 pm **FACSS Award Presentations,** *Rm 305A*

4:40 pm **PLENARY LECTURE,** *Rm 305A*
Distinguished Service: Becoming an Oxymoron?, **Alex Scheeline**, page 60

THURSDAY MORNING

8:00 AM **PLENARY LECTURES:** *Ballroom A*
Coblentz Clara Craver Award, Boris Mizaikoff, page 68
Ellis R. Lippincott Award, Martin Moskovits, page 68

9:00 am **POSTER SESSION,** page 68

10:30 am **SYMPOSIA,** page 70
Surface Plasmon Resonance: Instrumentation and Applications*, Rm 301B*
Plasmas for Atomic and Molecular Analyses, *Rm 302A*
Microreactors: Chemistry, Technology, and Success, *Rm 302B*
H/D Exchange Mass Spectrometry, *Rm 302C*
Lippincott Award, *Rm 304*
Contributed Chromatography, *Rm 305A*
From Forensics to Pharma – Applications of Chemical Imaging, *Rm 305B*
Raul Curbelo – The Hidden Innovator in FTIR, *Rm 306A*
Royal Society of Chemistry, Analytical Division: Application, Advances and Deduction by Vibrational Spectroscopy, *Rm 306B*
Raman Spectroscopy in the Pharmaceutical Industry, *Rm 306C*

THURSDAY AFTERNOON

1:30 pm **POSTER VIEWING**

2:00 pm **SYMPOSIA,** page 73
Surface Plasmon Resonance: Instrumentation and Applications, *Rm 301B*
Atomic Spectroscopy Techniques for Solid Surface Analysis in Biology, Geology, and Astronomy, *Rm 302A*
Drop Deposition and Dynamics, *Rm 302B*
Developments and Applications in Biological Mass Spectrometry, *Rm 302C*
Craver Award – Honoring Professor Boris Mizaikoff, *Rm 304*
Contributed Capillary Electrophoresis, *Rm 305A*
Biological and Biomedical Applications of Raman Spectroscopy, *Rm 305B*
Chemometrics Applied to Air, Oceanography, Marine Life and Coastal Waters, *Rm 306A*
Computer Directed/Supported Spectroscopy, *Rm 306B*
Pharmaceutical Raman, *Rm 306C*

TECHNICAL PROGRAM OVERVIEW BY TOPIC

AWARD SESSIONS

Monday AM
ACS Division of Analytical Chemistry Arthur F. Findeis Award, *Rm 304*
Monday PM
ACS Division of Analytical Chemistry Arthur F. Findeis Award, *Rm 304*
Tuesday AM
ANACHEM Award, *Rm 304*
Tuesday PM
Charles Mann Award, *Rm 304*
Wednesday AM
Lester Strock Award – Atomic Spectroscopy Progress Report, *Rm 304*
Wednesday PM
Meggers Award – Advances in Hyperspectral Imaging, *Rm 304*
FACSS / SAS Student Awards, *Rm 305A*
Thursday AM
Lippincott Award, *Rm 304*
Thursday PM
Craver Award Honoring Boris Mizaikoff, *Rm 304*

ANALYTICAL METHODS/INSTRUMENTATION

Monday AM
Materials Characterization Using Vibrational Spectroscopy and Mass Spectrometry, *Rm 301B*
Monday PM
Ionic Liquids and Spectroscopy: A Green and Happy Marriage, *Rm 301B*
Quantum Cascade Laser Applications, *Rm 306A*
Tuesday AM
Headspace Analysis for Chemical Signatures, *Rm 301B*
Tuesday PM
Lasers in Analytical Chemistry: Celebrating the 50th Anniversary of the Laser, *Rm 305B*
Thursday AM
RSC, Analytical Division: Applications, Advances and Deduction by Vibrational Spectroscopy, *Rm 306B*
Thursday PM
Drop Deposition and Dynamics, *Rm 302B*

ATOMIC SPECTROSCOPY

Monday AM
Celebrating 30 Years of ICP-MS, *Rm 302A*
Laser Induced Breakdown Spectroscopy, *Rm 302B*
Monday PM
Celebrating 30 Years of ICP-MS, *Rm 302A*
Laser Induced Breakdown Spectroscopy, *Rm 302B*
Tuesday AM
Atomic Analyses in the Pharmaceutical and Neutraceutical Industry, *Rm 302A*
The JAAS Silver Anniversary Celebration: Highlighting Young Investigators in Atomic Spectroscopy, *Rm 302B*
Tuesday PM
Chemistry in Art and Archaeology, *Rm 302A*
ICPMS – A Lot More Than Total Metals Analysis, *Room 302B*
Wednesday AM
Novel Sample Introduction Methods for ICP-MS and ICP-OES, *Rm 302A*
Fundamental Studies and Exciting New Applications of Glow Discharge Spectroscopy, *Rm 302B*
Thursday AM
Plasmas for Atomic and Molecular Analyses, *Rm 302A*
Thursday PM
Atomic Spectroscopy Techniques for Solid Surface Analysis in Biology, Geology, and Astronomy, *Rm 302A*

CHEMOMETRICS

Monday AM
Novel but Important Data Analysis Techniques in Analytical Chemistry, *Rm 302C*
Chemometrics in the Pharmaceutical Industry, *Rm 302C*
Tuesday AM
Chemometrics in Forensics, *Rm 302C*
Tuesday PM
Chemometrics for Biological and Biomedical Spectroscopy, *Rm 302C*
Thursday PM
Chemometrics Applied to Air, Oceanography, Marine Life and Coastal Waters, *Rm 306A*

CHROMATOGRAPHY

Monday AM
Portable NMR and Hyphenation of NMR to Chromatography and Electrophoresis, *Rm 305A*
Monday PM
Chromatography in the Pharmaceutical Industry, *Rm 305A*
Emerging and Non-Traditional Electrophoresis Techniques, *Rm 305*B
Tuesday AM
Dielectrophoresis and Related Techniques, *Rm 305A*
Wednesday AM
Bioanalytical Separation Science, *Rm 305A*
Thursday AM
Chromatography, *Rm 305A*
Thursday PM
Capillary Electrophoresis, *Rm 305A*

COMPUTATIONAL METHODS

Thursday PM
Computer Directed/Supported Spectroscopy, *Rm 306B*

HOMELAND SECURITY

Tuesday AM
Emerging Technologies for Standoff Detection for Homeland Security, *Rm 305B*

IMAGING

Monday AM
Near and Mid-Infrared Imaging, *Rm 305B*
Wednesday AM
Optical Effects in Infrared Spectroscopic Imaging, *Rm 305B*
Wednesday PM
Biomedical Applications of Spectroscopic Imaging, *Rm 305B*
Thursday AM
From Forensics to Pharma – Applications of Chemical Imaging, *Rm 305B*

MASS SPECTROMETRY

Tuesday PM
MS Fundamentals and Gas-Phase Ion Chemistry, *Rm 301B*
Wednesday PM
Advances in Mass Spectrometry Instrumentation, *Rm 302C*
Thursday AM
H/D Exchange Mass Spectrometry, *Rm 302C*
Thursday PM
Developments and Applications in Biological Mass Spectrometry, *Rm 302C*

TECHNICAL PROGRAM OVERVIEW BY TOPIC

MOLECULAR SPECTROSCOPY (IR and NIR)

Monday AM

Special Memorial Seminar Session: How One Man Changed the World of FTIR and Infrared Spectroscopy: The Don Sting Legacy*, Rm 306A*

Tuesday AM

Special Session to Honor William G. Fateley, *Rm 306A*

Tuesday PM

Practical Aspects of Chiral Analysis Using VCD and ROA, *Rm 305A*

Special Session to Honor William G. Fateley, *Rm 306A*

Next Generation Spectroscopic Methods for the Analysis of Pharmaceutical Systems*, Rm 306B*

Wednesday AM

Near-IR Spectroscopy: Application sand Calibrations, *Rm 302A*

Vibrational Spectroscopy at Work in the Pharmaceutical Industry, *Rm 302B*

Two-Dimensional Correlation Spectroscopy, *Rm 306A*

Wednesday PM

Two-Dimensional Correlation Spectroscopy*, Rm 306A*

Thursday AM

Raul Curbelo – The Hidden Innovator in FTIR, *Rm 306A*

NANOTECHNOLOGY

Tuesday AM

Nanotechnology: Applications to Sensing and Energy, *Rm 307*

Tuesday PM

Nanotechnology: Applications to Sensing and Energy, *Rm 307*

Wednesday AM

Probing Interactions between Biomolecules and Nanomaterials, *Rm 301B*

PROCESS, ANALYTICAL

Monday AM

PAT in the Biopharmaceutical and Pharmaceutical Industries, SAS Process Session, *Rm 306B*

PAT: New Technology, *Rm 306B*

Tuesday AM

SAS Process Analytical Technology, *306B*

Wednesday AM

Sustainability and PAT Applications, *Rm 306B*

Wednesday PM

Monitoring Continuous Chemistry and Chemical Processes, *Rm 306B*

RAMAN

Monday AM

Emerging Areas in Raman Spectroscopy, *Rm 306C*

Monday PM

Biomedical Raman Spectroscopy, *Rm 306C*

Tuesday AM

Raman Microscopy and Imaging, *Rm 306C*

Tuesday PM

Tip Enhanced Raman Spectroscopy – Pushing the Limits of Resolution, *Rm 306C*

Wednesday AM

New SERS Architectures, *Rm 306C*

Wednesday PM

Applications of SERS, *Rm 306C*

Thursday AM

Microreactors: Chemistry, Technology, and Success, *Rm 302B*

Raman Spectroscopy in the Pharmaceutical Industry, *Rm 306C*

Thursday PM

Biological & Biomedical Applications of Raman Spectroscopy, *Rm 305B*

Pharmaceutical Raman, *Rm 306C*

SURFACE PLASMON RESONANCE

Wednesday PM

Nanomaterials for Surface Plasmon Resonance, *Rm 301B*

Thursday AM

Surface Plasmon Resonance: Instrumentation and Applications, *Rm 301B*

Thursday PM

Surface Plasmon Resonance: Instrumentation and Applications, *Rm 301B*

TERHERTZ

Wednesday AM

Terahertz Spectroscopy, *Rm 307*

Wednesday PM

Terahertz Spectroscopy, *Rm 307*

TECHNICAL PROGRAM SUNDAY
Workshops – see page 36 for a list ◆ What's Hot Symposium

"What's Hot" Symposium, Presider: Brian Dable**,** *Ballroom A*

4:30 **Agilent**, "Today's Agilent – New Solutions in Atomic Spectroscopy"
4:40 **OPOTEK,** "The Advancement of Applications using Tunable OPO laser systems: From a Research Novelty to a Mature Scientific Device"
4:50 **Thermo Scientific**
5:00 **Princeton Instruments,** "Increasing the Resolution of Optical Spectrometers through the Development of Novel Aberration Correcting Optics"
5:10 **HORIBA Scientific,** "Rapid Raman and Fast Fluorescence Imaging on an Inverted Microscope"
5:20 **Daylight Solutions,** "Laser Access to Hydrocarbon Stretches at 3.25 μm: The new Über Tuner-3"
5:30 **Anasys Instruments,** "Nanoscale IR Spectroscopy: Breaking the Diffraction Limit"
5:40 **Eigenvector,** "PLS_Toolbox/Solo 6.0: Mastering Clutter for Better Models"
5:50 **JASCO,** "The Need for Speed - Instrumentation to Increase Raman Imaging Efficiency"
6:00 **Cobalt Light Systems, "**Raman Analysis: Scratching (Beyond) the Surface"
6:10 **Thermo Scientific**
6:20 **Nanonics,** "Tip Enhanced Raman Scattering"
6:30 **Innovative Photonics,** "Novel Single Frequency 532 and 785 nm Raman Excitation Sources"
6:40 **Kaiser,** "Advances in Process Raman Sampling"
6:50 **HORIBA, "**Extremely Low Frequency (<5cm-1) Raman Measurements with a Filter!"

SUNDAY 7:00 PM
WELCOME MIXER
SAS Sponsored Student Poster Session, Coblentz Student Awards
Ballroom Lobby

SAS
Student Poster Showcase and Awards

Please join us in celebrating the
future of spectroscopy as SAS students showcase their research and
compete for the annual SAS Student Poster Awards.

Sunday, October 17, 2010, 7-9 p.m. *(during the FACSS mixer)*

Sponsored by
The Society for Applied Spectroscopy and FACSS

SOCIETY SPONSORED / ORGANIZED SYMPOSIA

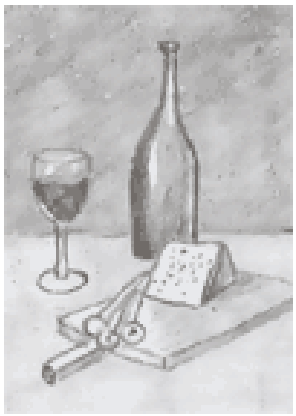
ACS
ACS Division of Analytical Chemistry Arthur F. Findeis Award

ASMS
MS Fundamentals and Gas-Phase Ion Chemistry
Advances in Mass Spectrometry Instrumentation
H/D Exchange Mass Spectrometry
Developments and Applications in Biological Mass Spectrometry

SAS
Lester Strock Award – Atomic Spectroscopy Progress Reports
Meggers Award – Advances in Hyperspectral Imaging
Lippincott Award
PAT Applications in the Pharmaceutical Industry
SAS Process Analytical Technology

COBLENTZ
Two-Dimensional Correlation Spectroscopy
Practical Aspects of Chiral Analysis Using VCD and ROA
NIR
Next Generation Spectroscopic Techniques for the Analysis of Pharmaceutical Systems
Quantum Cascade Laser Systems
Near and Mid Infrared Imaging
Ionic Liquids and Spectroscopy: A Green and Happy Marriage
Biomedical Applications of Spectroscopic Imaging
Vibrational Spectroscopy at Work in the Pharmaceutical Industry
Optical Effects in IR Imaging
Special Session to Honor William G. Fateley

RSC, Analytical Division
Applications Advances and Deduction by Vibrational Spectroscopy



The Society for Applied Spectroscopy
Cordially Invites
You to Join Us at Our Annual
Wine and Cheese Awards Reception
Tuesday, October 19, 2010 7:00 p.m.
In the State Ballroom of the
Marriott Raleigh City Center Hotel
This is a member's only event.

TECHNICAL PROGRAM – MONDAY

Plenary Lectures

Morning Presider: Charles Wilkins; Afternoon Presider: André Sommer

8:00 am Plenary
ACS Division of Analytical Chemistry Arthur F. Findeis Award, *Ballroom A*

(1) Electroanalytical Eavesdropping on Cellular Communication**; Christy Haynes;** University of Minnesota

4:20 pm Plenary, *Ballroom A*

The Role of Analytical Chemistry and Spectroscopy in Drug Discovery and Development: Challenges and Opportunities**; Uli Hacksell,** *ACADIA Pharmaceuticals*

MONDAY POSTER SESSION
9:00 – 10:30 am
Ballroom Lobby

All Monday posters should be put up between 7:30 – 8:00 am and removed between 5:00 – 6:00 pm. Odd numbered poster boards present between 9:00 – 9:45 am. Even numbered poster boards present between 9:45 – 10:30 am.

Bioanalytical

Board #

1 (3) **Dry Eye and Human Tear Lipid Compositional and Structural Relationships Using Spectroscopy.**; Douglas Borchman[1], Gary Foulks[1], Marta Yappert[1]; [1]University of Louisville

2 (4) **Development of a Peptidase-Resistant Reporter to Measure BCR-ABL Kinase Activity**; Angela Proctor[1], Qunzhao Wang[1], David Lawrence[1], Nancy Allbritton[1,2]; [1]University of North Carolina at Chapel Hill; [2]North Carolina State University

3 (5) **Enhanced Glycan ESI Response Using Neutral Hydrazide Reagents via Hydrophobic Tagging towards the Profiling of Cleaved N-Linked Glycans**; Hunter Walker[1], Brian Papas[1], Daniel Comins[1], David Muddiman[1]; [1]North Carolina State University

4 (6) **Detection of Mycoplasma Pneumoniae in a Clinical Background by Surface-Enhanced Raman Scattering Spectroscopy**; Suzanne Hennigan[1], Jeremy Driskell[1], Yiping Zhao[1], Richard A. Dluhy[1], Duncan C. Krause[1]; [1]University of Georgia

5 (7) **Application of Support Vector Regression in Non-Invasive Blood Glucose Detection Using Raman Spectroscopy**; Ishan Barman[1], Narahara Chari Dingari[1], Jeon Woong Kang[1], Chae-Ryon Kong[1], Ramachandra R. Dasari[1], Michael S. Feld[1]; [1]Massachusetts Institute of Technology

6 (8) **Cell Labeling with Plasmonic Particles**; George Chumanov[1], Kyle Dukes[1]; [1]Clemson University

7 (9) **Acid Cleavable Tagging for Biomolecule Analysis**; Dongmao Zhang[1], Karthikeswar Vangala[1], Michael Yanney[1], Shaoyong Li[1], Sygula Andrzej[1]; [1]Mississippi State University

8 (10) **Low-Shear Microfluidic Devices for Cell Culture and Analysis**; Dimitri Pappas[1]; [1]Texas Tech University

9 (11) **Application of GA-ANN for Prediction of the Selectivity Coefficients for the MIP Based Potentiometric Sensor**; Mahmood Reza Sohrabi[1], Pegah Nezakati[1], Meharn Javanbakht[2], Hoda Pasdar[1]; [1]Azad University; [2]Amirkabir University

Chemometrics

10 (12) **Facilitating the Teaching of Chemometrics through the Cyber-Enabled Virtual Chemometrics Lab**; Edward Duranty[1], Rebecca Horton[1], Morgan McConico[1], Frank Vogt[1]; [1]University of Tennessee

Board #

11 (13) **Comparing Variable Selection in Multivariate Model Development Using "Classical" and "Continuous" Haar Wavelet Transforms Coupled With Genetic Algorithm Optimization**; Lucy Botros[1], Kevin Judge[1]; [1]Molecular Biometrics Inc.

12 (14) **On-Line Chiral Analysis of Trace Biomarkers by UPLC-QToF Mass Spectrometry Using the Kinetic Method with Post-Column Derivatization**; Yong-Ill Lee[1], Hua Jin[1]; [1]Changwon National University

13 (15) **Optimized Spectral Data Fusion**; Heather Brooke[1], Kevin Johnson[1], Christian Minor[2]; [1]US Naval Research Laboratory; [2]Nova Research, Inc.

14 (16) **Adaptive Regression by Subspace Elimination**; Bryon Herbert[1], Karl Booksh[1]; [1]University of Delaware

15 (17) **Simultaneous Determination of Metformin Hydrochloride and Glibenclamide in Binary Mixtures Using Spectrophotometric Data and Wavelet Transform**; Naghmeh Kamali[1], Mahmoud Reza Sohrabi[1]; [1]Azad University, North Tehran Branch

16 (18) **Recognizing Patterns in Single Molecule Fluorescence Microscopy Using Multivariate Analysis**; Nathan Skinner[1], Gerhard Prinz[1], Derek Bailey[1], Jared Kindt[1], Madison Taylor[1], Michael Culbertson[1], Daniel Burden[1]; [1]Wheaton College

17 (19) **A Model Based Up on H-Point Technique for Simultaneous Spectrophotometric Determination of Dextromethorphan HBR and Psedoephedrin HCL in Farmacitical Formolation**; Mahmood Reza Sohrabi[1], Adeleh Ghobadi[2], Naser Goudarzi[3]; [1]Islamic Azad University,North Tehran Branch; [2]Azad University,North Tehran branch; [3]Industrial Shahrod University

General Analytical
(Education, Electrochemistry, Forensic, Instrumentation, Laser Ablation, Materials Characterization, Other, Pharmaceutical, Process/Control, Proteomics, Surface Characterization

18 (20) **ChemSpider – Building an Online Database of Open Spectra**; Antony Williams[1], Valery Tkachenko[1]; [1]Royal Society of Chemistry

19 (21) **Analysis of Anisotropic Local Surface Plasmon in a Thin Film of Gold Nano-Particles Studied by Visible Multiple-Angle Incidence Resolution Spectrometry**; Takeshi Hasegawa[1], Akiyoshi Kasuya[1], Yuki Itoh[1], Tetsuo Okada[1], Masatoshi Osawa[2]; [1]Tokyo Institute of Technology; [2]Hokkaido University

Board #

20 (22) **Food Authenticity Determination by Total Organic Carbon Isotope Analysis Using a Combine TOC-Cavity Ringdown Spectrometer (CRDS) Instrument**; Garrett Slaton[1], Jeff Lane[1], Richard Simon[1], Gary Engelhart[1], Trent Sprenkle[1]; [1]OI Analytical

21 (23) **Semi - Open Focussed Microwave Methodology for Fast Sequential Sample Preparation**; Bob Lockerman[1], David Barclay[1]; [1]CEM Corporation

22 (24) **Foreign Material Analysis in Pharmaceutical Forensics**; Cara Fowler[1]; [1]Eli Lilly and Company

23 (25) **Trace Drug Residue Analysis with Microscopy and Microextraction-Coupled to Nanospray Mass Spectrometry from Forensic Lifts**; William Hoffmann[1], Nicole Wallace[1], Dr. Guido Verbeck[1]; [1]University of North Texas

24 (26) **Characterization of Zinc Carbonate Basic Using Multiple Techniques in Support of a Toxicological Investigation**; Amal Essader[1], Scott Afton[1], Keith Levine[1], Todd Ennis[1], Brenda Fletcher[1], Kelly Amato[1], Andrea McWilliams[1], Glenn Ross[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS

Spectroscopy
(Absorption, Atomic Spectroscopy, Fluorescence, ICP, Infrared, Laser Spectroscopy, Mass Spectrometry, Molecular Spectroscopy, Near Infrared, NMR, Raman, Surface Enhanced Raman, Surface Plasmon Resonance)

25 (27) **Preparation and Characterization of ZnO/PVC Nanocomposites and Study the Physical Properties for their Complexes**; Islam Elashmawi[1], Nagwa Hakeem[1]; [1]National Research Centre

26 (28) **Searching for a Needle in a Haystack, Extracting Valuable Information from Chemical Images**; Paulette Guillory-Gardner; [1]Thermo Fisher Scientific

27 (29) **Near-Eye-Safe Trace Molecular Detection via SERS and SERRS at a Stand-Off Distance of 15 Meters**; Jonathan Scaffidi[1], Molly Gregas[1], Benoit Lauly[1], J. Chance Carter[3], S. Michael Angel[2], Tuan Vo-Dinh[1]; [1]Duke University, Biomedical Engineering and FIP; [2]Univ. of South Carolina, Dept. of Chem.; [3]Lawrence Livermore Nat'l Lab

28 (30) **Spectral DSC: Probing Phase Transformations with Coupled Raman and DSC**; Stephen Medlin[1], John Richmond[1]; [1]Bruker Optics

29 (31) **Use of an Inductively Coupled Plasma Atomic Emission Spectrometer as an Empirical Formula Detector for Gas Chromatography**; Carl Young[1], Meredith Lisle[1], Bradley Jones[1]; [1]Wake Forest University

30 (32) **Conformational Stability, r0 Structural Parameters, and Vibrational Assignment of 2,2-Difluoroethylamine**; Arindam Ganguly[1], James R Durig[1]; [1]University of Missouri-Kansas City

31 (33) **Survey of Pet Foods for Heavy Metal Content by ICP & ICP-MS.**; Ralph Obenauf[1], Vanaja Sivakumar[1], Patricia Atkins[1]; [1]SPEX CertiPrep, Inc.

32 (34) **Selenium Heavy Metal Antagonism in Soybeans**; Traci Hanley[1], Qilin Chan[1], Joseph Caruso[1]; [1]University of Cincinnati

33 (35) **Elemental Analysis of High Elevation Conifers to Investigate Effects of Acidic Deposition**; David J. Butcher[1], Matthew Rosenberg[1], Lucas Wilson[1]; [1]Western Carolina University

Board #

34 (36) **The Formation of Doubly Charged Ions In and Inductively Coupled Plasma**; Kyli McKay[1], Nicholas Taylor[1], Paul Farnsworth[1]; [1]Brigham Young University

35 (38) **The Spectral Game – Teaching NMR Spectroscopy Via a Web Browser**; Antony Williams[1], Jean-Claude Bradley[2], Robert Lancashire[3], Andrew Lang[4]; [1]Royal Society of Chemistry; [2]Drexel University; [3]The University of the West Indies; [4]Oral Roberts University

36 (39) **The Effect of Matrix Composition on Several Fundamental Parameters in an Emission ICP**; Nick Taylor[1], Paul Farnsworth[1]; [1]Brigham Young University

37 (40) **Evaluation of Ion Transmission and Shock Structure of Various Skimmer Cone Designs in an ICP-MS**; Alisa Smith[1], Nick Taylor[1], Ross Spencer[1], Paul Farnsworth[1]; [1]Brigham Young University

38 (41) **Spectroscopic Study of the Electronic States of Liquid Ketones and Ethers by Using Attenuated Total Reflection - Far Ultraviolet Spectroscopy**; Yusuke Morisawa[1], Kyoko Takaba[1], Akifumi Ikehata[2], Noboru Higashi[3], Yukihiro Ozaki[1]; [1]Kwansei Gakuin University; [2]National Food Research Institute; [3]KURABO

39 (42) **Effects of Hydrogen Bondings on an Electronic State of Acetone Studied by Using Attenuated Total Reflection - Far Ultraviolet Spectroscopy**; Yusuke Morisawa[1], Akifumi Ikehata[2], Noboru Higashi[3], Yukihiro Ozaki[1]; [1]Kwansei Gakuin University; [2]National Food Research Institute; [3]KURABO

40 (43) **Selective Oxidation and Vapour-Phase FT-IR Spectrometric Determination of Histidine, Threonine and Cysteine in Pharmaceutical Formulations**; Kazem Kargosha[1], S.Hamid Ahmadi[1], Mohsen Zeeb[1], Jila Azad[2]; [1]Chem. &Chemic. Engineering Research Center of Iran; [2]Alzahra University

41 (44) **Increasing Accessibility to Instrumentation: A "Cutting Edge Metal Detector" for Field Applications**; Summer N. Hanna[1], Bradley T. Jones[1]; [1]Wake Forest University

42 (45) **Large Area Nanopillars SERS Arrays**; Tiziana Bond[1], Elaine Behymer[1], Hoang Nguyen[1], Cindy Larson[1], James Chan[1], Robin Miles[1], Mihail Bora[1], Logan Liu[2], Zidar Xu[2], Manas Gartia[2]; [1]Lawrence Livermore National Laboratory; [2]University of Illinois, Urbana Champaign

43 (46) **Novel Observations of Absorption Bands of Liquid Alkanes and Polyethylene by Attenuated Total Reflectance-Far UV**; Tachibana Shin[1], Morisawa Yusuke[1], Ikehata Akifumi[2], Sato Harumi[1], Higashi Noboru[3], Ozaki Yukihiro[1]; [1]Kwansei Gakuen University; [2]National Food Research Institute; [3]KURABO

44 (47) **Effects of Intramolecular Hydrogen Bonding on OH Bands of Phenol and Halogenated Phenols in the Visible, Near-Infrared and Infrared Spectra**; Takayuki Gonjo[1], Yuusuke Morisawa[1], Toshiaki Suzuki[1], Yukihiro Ozaki[1]; [1]Kwansei Gakuin University

45 (48) **Robust SERS Substrates Generated by Coupling a Bottom-Up Approach and Atomic Layer Deposition**; Eric Formo[1]; [1]Center for Nanophase Materials Sciences, ORNL

46 (49) **Enhanced Plasmonic Light Beaming Induced By Near Field Resonance**; Pengyu Chen[1], Lin Zhu[1], Qiaoqiang Gan[2], Filbert Bartoli[2]; [1]Clemson University; [2]Lehigh University

Board #

47 (50) **Analytical Evaluation of a Helium-Argon Radiofrequency Glow Discharge Coupled to Time of Flight Mass Spectrometry**; Cristina González Gago[1], Nerea Bordel[1], Rosario Pereiro[1], Alfredo Sanz-Medel[1]; [1]University of Oviedo

48 (51) **Identification of Counterfeit Whisky Using Mid-Infrared Spectrometry with ATR Probes and Polycrystalline Silver Halide Optical Fibres**; Allyson McIntyre[1], Alison Nordon[1], David Littlejohn[1], Gary Colquhoun[2]; [1]University of Strathclyde, Glasgow UK; [2]Fibre Photonics, Livingston UK

49 (52) **Structural and Functional Study on High-Reflection Black Pigment by NIR Spectroscopy and XRD**; Nomura Satoshi[1], Morisawa Yusuke[1], Sanada Kazutoshi[2], Shinsuke Maruyama[2], Ozaki Yukihiro[1]; [1]Kwansei Gakuin University; [2]Toda Kogyo Corporation

50 (53) **The Dependence of Measurement Precision and Bubble Reproducibility on the Quality of Focusing Optics for Dual-Pulse LIBS Measurements in Water**; Christopher Gordon[1], S. Michael Angel[1]; [1]University of South Carolina

51 (54) **Numerical Simulation of the Effects of Helium Added to an Argon Inductively Coupled Plasma**; Helmut Lindner[1], Annemie Bogaerts[1]; [1]University of Antwerp

52 (55) **Characterization of Di-Isohexyl Phthalate (DIHxP) Using GC/MS and NMR in Support of Toxicological Studies**; Joseph Licause[1], Jason Burgess[1], James Blake[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

53 (56) **Surface-Enhanced Raman Scattering Substrates Based On Heat Shrinkable Polystyrene**; Joseph Mannion[1], George Chumanov[1]; [1]Clemson University

54 (57) **Detection of Cyanide Using Raman Spectroscopy on Metal Halide Films**; C.V. Gopal Reddy[1,2], Fei Yan[1,2], Yan Zhang[1,2], Tuan Vo-Dinh[1,2]; [1]Fitzpatrick Institute for Photonics; [2]Department of Biomedical Engineering

55 (58) **Hydrogen/Deuterium Exchange Mass Spectrometry System for Investigating Conformational Changes in Calmodulin Protein Upon Calcium Binding**; LeRoy Martin[1], Joomi Ahn[1], Martha Stapels[1], Michael Eggerston[1], Keith Fadgen[1], Rebecca Rose[2,3], Ying Qing Yu[1], Albert J. R. Heck[2,3]; [1]Waters Corporation, Milford, MA, USA; [2]Biomolecular Mass Spectrometry; [3]Netherlands Proteomics Centre, Padualaan; [4]

Monday Morning, *Room 301B*
MATERIALS CHARACTERIZATION USING VIBRATIONAL SPECTROSCOPY AND MASS SPECTROMETRY
Organizers and Presiders: David Hercules and Bruce Chase

10:30 (59) **Vibrational Spectroscopy of High Strength Polymeric Fibers**; Bruce Chase[1]; [1]Pair Technologies LLC

10:50 (60) **Interfacing Mass Spectrometry with Liquid and Gas Phase Separations for Synthetic Polymer Analysis**; Chrys Wesdemiotis[1], Xiapeng Li[1], Nilüfer Solak[1], George R. Newkome[1], Stephen Z.D. Cheng[1]; [1]The University of Akron

11:10 (61) **Molecular Structures of Polymer Surfaces and Buried Polymer Interfaces Studied by Sum Frequency Generation Vibrational Spectroscopy**; Zhan Chen[1]; [1]University of Michigan

11:30 (62) **Mass Spectrometry of Polyurethanes: MS/MS and IMS**; David Hercules[1], Anthony Gies[1]; [1]Vanderbilt University

11:50 (63) **Nanoscale Infrared Spectroscopy and Imaging of Polymer Microdomains**; Curtis Marcott[1], Michael Lo[2], Kevin Kjoller[2], Craig Prater[2], Gloria Story[3], Isao Noda[3]; [1]Light Light Solutions; [2]Anasys Instruments; [3]The Procter & Gamble Company

12:10 (64) **Using Mass Spectrometry to Study Polymer Processing**; Anthony Gies[1], David Hercules[1]; [1]Vanderbilt University

Monday Morning, *Room, 302A*
CELEBRATING 30 YEARS OF ICP-MS
Organizers and Presiders: Steven Ray and David Koppenaal

10:30 (65) **Building the First Commercial ICP-MS**; Don Douglas; [1]University of British Columbia

10:50 (66) **A Diamond Jubilee - 30 Years and Counting with ICPMS**; David W. Koppenaal[1]; [1]Pacific Northwest National Laboratory

11:10 (67) **ICP-MS Performance after 30 Years: Filling in the Blanks**; Paul Farnsworth[1], Nicholas Taylor[1], Ross Spencer[1]; [1]Brigham Young University

11:30 (68) **Has the Battle to Eliminate Spectral Overlaps in ICP-MS Been Won?**; John Olesik[1], Patrick Gray[1]; [1]Ohio State University

11:50 (69) **Magnetic Sector ICPMS: Enhancements to Sensitivity and Dynamic Range and the Analysis of Small Sample Amounts**; Charles Douthitt[1], Dan Wiederin[2]; [1]Thermo Fisher Scientific; [2]Elemental Scientific, Inc.

12:10 (70) **ICP-MS: 30 Years After – Nothing Left To Do?**; Norbert Jakubowski[1], Larissa Waentig[1], Peter Roos[2]; [1]BAM – Federal Institute for Materials Research and; [2]IfADo – Leibniz Research Centre for Work

Monday Morning, *Room 302B*
LASER INDUCED BREAKDOWN SPECTROSCOPY
Organizer and Presider: Mike Angel

10:30 (71) **Laser Induced Breakdown Spectroscopy: Application to Slurry Samples**; Jagdish P Singh[1], Krishna K. Ayyalasomayajula[1], Fang Yu Yueh[1], Laura T. Smith[1]; [1]Mississippi State University

11:10 (72) **Advances in Gunshot Residue Analysis by LIBS**; Christopher Dockery[1]; [1]Kennesaw State University

11:30 (73) **Combined Standoff LIBS and Raman System for Detection of Elemental Composition and Structure of Minerals**; Shiv Sharma[1], Anupam Misra[1], Paul Lucey[1], David Bates[1]; [1]Hawaii Inst. of Geophys. & Planetology

11:50 (74) **Planetary Geochemical Explorations by Remote Laser – Induced Breakdown Spectroscopy (LIBS)**; Samuel Clegg[1], Roger Wiens[1], Olivier Forni[2], Sylvestre Maurice[2], Shiv Sharma[3], Darby Dyar[4]; [1]Los Alamos National Laboratory; [2]Centre d'étude Spatiale des Rayonnement; [3]University of Hawaii; [4]Mt. Holyoke College

12:10 (75) **Test for Validity of the Boltzmann Plot Method: Implications from Plasma Modeling**; Igor Gornushkin[1], Sergei Shabanov[2], Sven Merk[1], Elisabetta Tognoni[3], Ulrich Panne[1]; [1]BAM, Berlin, Germany; [2]University of Florida, Gainesville, USA; [3]INO-CNR, Pisa, Italy

TECHNICAL PROGRAM – MONDAY
Orals 10:30 am – 12:30 pm

Monday Morning, *Room 302C*
NOVEL BUT IMPORTANT DATA ANALYSIS TECHNIQUES IN ANALYTICAL CHEMISTRY
Organizer and Presider: Barry Lavine

10:30 (76) **Covering My ARSE: Recent Advances in Adaptive Regression by Subspace Elimination**; Karl Booksh[1], Bryon Herbert[1], Seong-Soo Kim[2], Boris Mizaikoff[2], Chance Carter[3]; [1]University of Delaware; [2]Georgia Tech; [3]LLNL;

10:50 (77) **Genetic Algorithms for Identification of Cancer Markers from Mass Spectral Profiles**; Barry Lavine[1], Nikhil Mirjankar[1], Yehia Mechref[2], David Clemmer[3], Marie Hanigan[4], Matthew West[4]; [1]Oklahoma State University; [2]Texas Tech University; [3]Indiana University; [4]Oklahoma Health Sciences Center

11:30 (78) **Determining Microalgal Biodiversity as Novel Environmental Indicator - Combining Spectroscopic, Imaging and Prior Information through Bayesian Statistics**; Frank Vogt[1], Edward Duranty[1], Rebecca Horton[1], Morgan McConico[1]; [1]University of Tennessee, Department of Chemistry

11:50 (79) **Calibration and Classification Transfer Using Stacked Methods**; Steven Brown[1]; [1]Univ. Delaware

12:10 (80) **Practical and Theoretical Implications of PLS Constraints**; William Rayens[1], Aric Schadler[1]; [1]University of Kentucky

Monday Morning, *Room 304*
ACS DIVISION OF ANALYTICAL CHEMISTRY ARTHUR F. FINDEIS AWARD
Organizer and Presider Charles Wilkins

10:30 (81) **Neuropeptide Discovery: From New Characterization Approaches to Function**; Jonathan Sweedler[1]; [1]Univeristy of Illinois

11:10 (82) **Sensitivity of Carbon-Fiber Electrodes**; Mark Wightman[1]; [1]University of North Carolina at Chapel Hill

11:50 (83) **Design of Multifunctional Nanoparticle Probes for Molecular Imaging and Sensing in Single Living Organisms**; Dr. X. Nancy Xu[1], Prakash D. Nallathamby[1], Tao Huang[1], Kerry J. Lee[1], Lauren M. Browning[1]; [1]Old Dominion University

Monday Morning, *Room 305A*
PORTABLE NMR AND HYPHENATION OF NMR TO CHROMATOGRAPHY AND ELECTROPHORESIS
Organizer and Presider: Joana Diekmann

10:30 (84) **Smaller, Cheaper, and More User-Friendly: Are We Really Talking About NMR?**; Timothy Peck[1]; [1]Protasis Corporation

10:50 (85) **NMR with Small Magnets**; Bernhard Bluemich; [1]RWTH Aachen University

11:10 (86) **Bench-top and On-line High Resolution Permanent Magnet 60 MHz NMR for Reaction Monitoring and Process Control**; John Edwards[1], Paul Giammatteo[1]; [1]Process NMR Associates, LLC

11:30 (87) **Capillary Electrophoresis Hyphenated with Slotted Microstrip Nuclear Magnetic Resonance Detection**; Roland Hergenroeder[1], Joerg Lambert[1]; [1]ISAS

11:50 (88) **Mass Limited Sample Detection Using cITP Microcoil NMR**; Christopher Jones[1], Cynthia Larive[2]; [1]UC Riverside; [2]UC Riverside

12:10 (89) **Advanced LC/Microcoil NMR Methods for Structure Determination and Metabolite Profiling**; Daniel Raftery[1], Ravi KC[1], Emmanuel Appiah-Amponsah[1], Kwadwo Owusu-Sarfo[1], Tao Ye[1], G. A. Nagana Gowda[1]; [1]Purdue University

Monday Morning, *Room 305B*
NEAR AND MID-INFRARED IMAGING
Organizer and Presider: Chieu Tran

10:30 (90) **Recent Advances in IRIRI of Polymers**; Marek W. Urban, Biswajit Ghosh, Dhanya Ramachandran; [1]USM

10:50 (91) **Mid-Infrared Imaging of Tissue: Reduction of Confounding Scattering Effects**; Max Diem; [1]Northeastern University

11:10 (92) **Dynamic Chemical Imaging in the Near Infrared**; Patrick Treado[1], Matthew Nelson[1], Charles Gardner[1], Ryan Priore[1]; [1]ChemImage Corporation

11:30 (93) **From Formulating for Performance to Fingerprinting Counterfeits: A Review of the Roles of NIR Chemical Imaging in the Pharmaceutical Industry**; Janie Dubois[1], Linda H. Kidder[1], E. Neil Lewis[1]; [1]Malvern Instruments Inc.

11:50 (94) **Characterization and Monitoring Dynamic Processes of Nanomaterials by Near-infrared Spectroscopic Imaging Technique**; Chieu Tran[1]; [1]Marquette University

12:10 (95) **Enhanced Models for Fourier Transform Infrared (FT-IR) Spectroscopic Imaging of Human Tissue Specimens**; Rohith Reddy[1, 2], Brynmor Davis[1], Rohit Bhargava[1,2]; [1]University of Illinois at Urbana Champaign; [2]Beckman Institute for Advanced Science

Monday Morning, *Room 306A*
SPECIAL MEMORIAL SEMINAR SESSION: HOW ONE MAN CHANGED THE WORLD OF FTIR AND INFRARED SPECTROSCOPY: THE DON STING LEGACY
Organizer and Presider: John Coates

10:30 (96) **The Don Sting Legacy: Making FTIR Accessible to the World**; John Coates[1], James Rydzak[2]; [1]Caotes Consulting; [2]GlaxoSmithKline

10:50 (97) **Making Concepts into Products: From Barnes to A2…the History, the Companies and the Products**; Steven Donahue[1], Robert Messerschmidt[2]; [1]A2 Technologies; [2]Rare Light, Inc.

11:10 (98) **Don Sting the Engineer, the Innovator and the Inventor**; Scott Little[1], Gregg Ressler[2]; [1]Focal Point International LLC; [2]A2 Technologies, Inc.

11:30 (99) **FT-IR : Migration from the Lab to the Field**; David Schiering[1], John Seelenbinder[2]; [1]Smiths Detection; [2]A2 Technologies

11:50 (100) **Bringing FT-IR Spectroscopy and Microscopy Together – Another Don Sting Legacy**; John Reffner[1], Greg Ressler[2], Robert Messerschnidt; [1]John Jay College, CUNY; [2]A2 Technologies

12:10 (101) **Understanding the Chemistry and the Process: Tools for Reaction Monitoring**; Alan Rein[1], Henry Dubina[2]; [1]A2 Technologies; [2]Mettler Toledo Autochem

TECHNICAL PROGRAM – MONDAY
Orals 10:30 am – 12:30 pm ◆ Orals 2:00 – 4:00 pm

Monday Morning, *Room 306B*
PAT IN THE BIOPHARMACEUTICAL AND PHARMACEUTICAL INDUSTRIES, SAS Process Session
Organizers and Presiders: Brandye Smith-Goettler and Edita Botonjic-Sehic

10:30 (102) **Spectroscopic Solutions to Support the Design of an Attribute Based Manufacturing Process**; John Bobiak[1], Dongsheng Bu[1], Dimuthu Jayawickrama[1], Kevin Macias[1], Tim Stevens[1], Boyong Wan[1], Gary McGeorge[1]; [1]Bristol-Myers Squibb Co

10:50 (103) **Monitoring Stem Cell Cultivations with NIR Spectroscopy**; Francisca Folque[1], Tiago Fernandes[1], Margarida Diogo[1], Joaquim Cabral[1], José Cardoso de Menezes[1]; [1]IBB,Technical University of Lisbon (IST)

11:10 (104) **Development and Application of a General NIR Model for Monitoring the Moisture During Fluid-bed Drying**; Patrick Rameas[1], Antonio Peinado Amores[1]; [1]GlaxoSmithKline

11:30 (105) **The Choice of Validation Level as the Basis for Critical Limits in Raw Material Identification and Process Monitoring**; Frank Westad; [1]Camo Software

11:50 (106) **Application of Multivariate Data Analysis to Assess Scalability and Identify Scale-Dependent Variables of a Yeast Fermentation Process**; Louis Obando[1], Thomas Potgieter[1], Venkata Mangalampalli[1], Charles Miller[1]; [1]Merck

12:10 (107) **At-Line PAT Methods in Supporting Active Precision Coating End-point Determination and Coating Process Development**; Fan Zhang-Plasket[1], John Higgins[1], Ramasamy Manoharan[1], Charles Miller[1], Louis Obando[1], Bruce Thompson[1], Gert Thurau[1]; [1]Merck Sharp and Dohme Co

Monday Morning, *Room 306C*
EMERGING AREAS IN RAMAN SPECTROSCOPY
Organizers and Presiders: Ian Lewis and Pavel Matousek

10:30 (108) **Using Mid-Infrared Spectroscopic Imaging Basis Sets for Quantitative Raman Spectroscopy**; Matthew Schulmerich[1], Michael Walsh[1], Matthew Kole[1], Michael Asensio[1], Rohit Bhargava[1]; [1]Univ. of IL., Urbana-Champaign

10:50 (109) **Raman Spectroscopy and the Search for Life on Mars: a Biological and Geological Perspective**; Howell GM Edwards; [1]University of Bradford

11:10 (110) **Transmission Raman Spectroscopy as a Tool for Quantifying Polymorphic Content of Pharmaceutical Formulations**; Jonathan Burley[1], Michael Hargreaves[2], Adeyinka Aina[1], Pavel Matousek[3]; [1]University of Nottingham; [2]Cobalt Light Systems Ltd; [3]Central Laser Facility, STFC

11:30 (111) **Raman Mapping of Biological Tissue Using Clustering Analysis Based on the Pearson Correlation Coefficient**; Frederic Festy[1], Frances Downey[1], Richard Cook[1], Nic Cade[1], Cheryl Gillett[1], David Richards; [1]King's College London

11:50 (112) **Scanning Angle Total Internal Reflection Raman Microscopy**; Emily Smith[1,2], Kristopher McKee[1,2]; [1]US DOE, Ames Laboratory; [2]Iowa State University

12:10 (113) **Effect of Laser Angle of Interrogation on Raman Signal**; Phillip Wilcox[2], Jason Guicheteau[2], Ian Pardoe[1], Steven Christensen[2], Darren Emge[2]; [1]Excet, Inc.; [2]Edgewood Chemical Biological Center

SPECIAL INVITATION TO STUDENT ATTENDEES
12:30 pm, *Room 307*
Free Lunch and Employment Discussion for Students
Hosted by SABIC Innovative Plastics

Eat lunch and chat with professionals from a wide range of professional fields (academic, government, chemical industry, pharmaceuticals, goods and services, etc.) It's a unique opportunity to ask questions, get helpful tips, and discuss topics that relate to your specific career-seeking situation within the current job market. *Sign up at the FACSS registration desk.*

Monday Afternoon, *Room 301B*
IONIC LIQUIDS AND SPECTROSCOPY: A GREEN AND HAPPY MARRIAGE
Organizer and Presider: Chieu D. Tran

2:00 (114) **Spectroscopic Characterization of Natural Fiber Welding**; Hugh De Long[2], Luke Haverhals[1], Zane Fayos[1], Hadley Sulpizio[1], W. Matthew Reichert[1], Matthew Foley[1], Paul Trulove[1]; [1]United States Naval Academy; [2]Air Force Office of Scientific Research

2:20 (115) **Ionic Liquids in Separations and Mass Spectrometry, a New Frontier**; Daniel Armstrong[1]; [1]UT Arlington

2:40 (116) **NanoGUMBOS: A Novel Concept for the Design of Nanomaterials**; Isiah Warner[1], Bilal El-Zahab[1], Min Li[1], Susmita Das[1]; [1]Department of Chemistry,Louisiana State University

3:00 (117) **How Proteins Dance and Fold in Ionic Liquids**; Frank Bright[1], Nadine Kraut[1], Bharathwaj Sathamoorthy[1], Michael Dabney[1], Kiran Singarapu[1], David Parish[1], Gary Baker[2], Thomas Ssyperski[1], Taylor Page[1]; [1]UB, SUNY; [2]Oak Ridge National Laboratory

3:20 (118) **Photonic Ionic Liquids: Blurring the Line Between Solvent and Probe**; Gary A. Baker[1], Ka Yi Yung[2], Nadine D. Kraut[2], Frank V. Bright[2]; [1]Oak Ridge National Laboratory; [2]University at Buffalo

3:40 (119) **Ionic Liquids for and by Spectroscopy**; Chieu Tran[1]; [1]Marquette University

Monday Afternoon, *Room 302A*
CELEBRATING 30 YEARS OF ICP-MS
Organizers and Presiders: David Koppenaal and Steven J. Ray

2:00 (120) **Isotope Ratio Analysis of Fast Transient Samples with a Mattauch-Herzog Mass Spectrograph**; Jeremy Felton[1], Steven Ray[1], Roger Sperline[2], M. Bonner Denton[2], Charles Barinaga[3], David Koppenaal[3], Gary Hieftje[1]; [1]Indiana University; [2]University of Arizona; [3]Pacific Northwest National Laboratory

2:20 (121) **Real Time Monitoring and Determination of Trace Elements in Single Airborne Nanoparticles (ANPs) by Continuous Introduction into ICP-MS**; Naoki Furuta[1], Hikaru Sato[1], Shimpei Hikida, Yoshinari Suzuki[1]; [1]Chuo University

2:40 (122) **Agilent Technologies-Growth and Innovations in ICP-MS**; Amir Liba[1]; [1]Agilent Technologies

3:00 (123) **An Introduction to the Next Generation of Inductively Coupled Plasma Mass Spectrometers**; Kaveh Kahen[1], Hamid Badiei[1]; [1]Perkin Elmer Sciex

3:20 (124) **Fully Simultaneous ICP-MS**; Dirk Ardelt[1], Willi Barger[1], Ulrich Heynen[1]; [1]SPECTRO Analytical Instruments GmbH

3:40 (125) **Inductively-Coupled Plasma Time-of-Flight Mass Spectrometry: Quo Vadis?**; Steven J. Ray[1], Elise Dennis[1], Alexander Graham[1], Christie Enke[1], Gary M. Hieftje[1], Charles Barinaga[2], David Koppenaal[2]; [1]Dept. of Chemistry - Indiana University; [2]Pacific Northwest National Laboratory

Monday Afternoon, *Room 302B*
LASER INDUCED BREAKDOWN SPECTROSCOPY
Organizer and Presider: Mike Angel

2:00 (126) **Changing the Perception of LIBS: Fundamentals and Applications**; Richard E. Russo[1,2], Travis Owens[1], Jong Yoo[2], Jhanis Gonzalez[1,2], Alex Bolshakov[2]; [1]Lawrence Berkeley National Laboratory; [2]Applied Spectra Inc

2:40 (127) **Laser-Induced Breakdown Spectroscopy (LIBS) for the Rapid Identification and Classification of Pathogenic Bacteria**; Steven Rehse[1], Qassem Mohaidat[1], Sunil Palchaudhuri[2], Hossein Salimnia[3, 4]; [1]Wayne State Univ., Dept of Physics and Astronomy; [2]Wayne State Univ., Dept of Microbiology; [3]Wayne State Univ., Dept of Pathology; [4]Detroit Medical Center Univ. Laboratorie

3:00 (128) **Improving Standoff Sensor Detection Performance for Explosive Residues via Fusion of Shortwave Infrared, Raman and LIBS Imaging Data**; Matthew Nelson[1], Paul Mangold[1], Robert Schweitzer[1], Patrick Treado[1]; [1]ChemImage Corporation[4]

3:20 (129) **Application of LIBS to Aqueous Solutions**; Scott Goode, Amelia Taylor-Perry; [1]Univ of So Carolina

3:40 (130) **Archaeological Applications of LIBS: An Example from the Coso Volcanic Field, CA Using Advanced Statistical Signal Processing Analysis**; Russell S. Harmon[1], Jeremiah J. Remus[2], Jennifer L. Gottfried[3]; [1]ARL Army Research Office; [2]Clarkson University; [3]Army Research Office

Monday Afternoon, *Room 302C*
CHEMOMETRICS IN THE PHARMACEUTICAL INDUSTRY
Organizer and Presider: Barry Lavine

2:00 (131) **Classification Modeling for Pharmaceutical Applications**; Katherine Bakeev[1]; [1]CAMO Software Inc

2:20 (132) **Data Fusion for Analysis of Pharmaceutical Systems**; Steven Brown[1], Maureen Lanan[2], Mike Koenigbauer[3]; [1]Univ. Delaware; [2]Biogen; [3]Astra Zeneca

2:40 (133) **Wavelets and Genetic Algorithms for Multivariate Calibration of NIR Data of Low Content Drug Tablets**; Barry Lavine[1], Nikhil Mirjankar[1], Mehul Vora[2]; [1]Oklahoma State University; [2]Clarkson University

3:00 (134) **Network Analysis of Pharmaceutical Process Analytical Technology (P-PAT) Research Trends**; Karl Booksh, University of Delaware

3:20 (135) **Multiple Fluorescent Label Capillary Electrophoresis Detection via Supercontinuum Rapid Excitation-Emission Matrix (ScREEM)**; Timothy Corcoran[1], Christopher Dettmar[1], Jacob Balthazor[1], Phillip Allen[1], Jose Chavez[1], Ivonne de la Torre[1], Alisha Lewis[1], Neda Nouri Nassr[1], Hossein Ahmadzadeh[1]; [1]California State Polytechnic University, Pomona

3:40 (136) **Multivariate Analysis (MVA) Applied to Drug Substance Development**; Susan Barnes[1], Christian Airiau[1], Vern De Biasi[1]; [1]GlaxoSmithKline

Monday Afternoon, *Room 304*
ACS DIVISION OF ANALYTICAL CHEMISTRY ARTHUR F. FINDEIS AWARD
Organizer and Presider: Charles Wilkins

2:00 (137) **Determination of Encapsulation Efficiency by Single Vesicle Analysis**; Michael Heien[1]; [1]University of Arizona

2:40 (138) **Segmented Flow for High Throughput Analysis at the Nanoliter Scale**; Robert Kennedy; [1]University of Michigan

3:20 (139) **Electrochemical Evaluation of Dopamine and Serotonin Neurotransmission in the Fruit Fly Brain**; B. Jill Venton[1], Trisha Vickrey[1], Huai-fang Fang[1]; [1]University of Virginia

Monday Afternoon, *Room 305A*
CHROMATOGRAPHY IN THE PHARMACEUTICAL INDUSTRY
Organizer and Presider: Charles Goss

2:00 (140) **The Past, Current, and Future of Reverse-phase HPLC Method Development in Pharmaceutical Industry**; Shujun Chen[1], Gerald Terfloth[1], Alireza Kord[1]; [1]GlaxoSmithKline

2:20 (141) **Peak Tailing Suppression of Phosphoric Prodrug in RP Chromatographic Separation Using Acidic Mobile Phase**; Jin Zhng[1], Qinggang Wang[1], Lydia Breckenridge[1], Brent Kleintop[1]; [1]Bristol Myers Squibb

2:40 (142) **Application of Preparative Chromatography to Accelerate Drug Development**; Leo Hsu[1], Xiqin Yang[1]; [1]GlaxoSmithKline

3:00 (143) **The Use of SFC from Method Development to Purification of Hundreds Grams of Racemic Material**; Manon Villeneuve[1]; [1]GlaxoSmithKline

3:20 (144) **Lessons in Implementing Open-Access Chromatography Across the Globe**; Steve Cole[1], Helen Weston[1], Bill Young[1], James Roberts[1]; [1]GlaxoSmithKline, plc

3:40 (145) **Fast GC for Rapid Solvent Composition and Purity Analysis**; Charles Goss[1], Will Canoy[1], Hamid Shafiei[1], Francis DeMartin[1]; [1]GlaxoSmithKline

Monday Afternoon, *Room 305B*
EMERGING AND NON-TRADITIONAL ELECTROPHORESIS TECHNIQUES
Organizer and Presider: Jonathan Shackman

2:00 (146) **Microfluidic GEMBE of "Real-World" Samples and Nanofluidic Separation of DNA**; Elizabeth A. Strychalski[1], Alyssa C. Henry[2], Henry W. Lau[3], Lynden A. Archer[3], David Ross[1]; [1]National Institute of Standards and Technology; [2]Applied Research Associates, Inc.; [3]Cornell University

2:20 (147) **Electrofocusing in Gradient Monoliths**; Cornelius Ivory[1]; [1]Washington State University

2:40 (148) **Gradient and Discontinuous Electrokinetics**; Mark Hayes[1]; [1]Arizona State University

3:00 (149) **Evaluation of the Precision of Dual-Opposite-Injection Capillary Electrophoresis and Comparison with Conventional Capillary Electrophoresis**; Joe Foley[1], Donna Blackney[1]; [1]Drexel University

3:20 (150) **Using Buffer Additives to Improve Analyte Stream Stability in Micro Free Flow Electrophoresis**; Nicholas Frost[1], Michael Bowser[1]; [1]University of Minnesota

3:40 (151) **Determination of Inorganic Ions in Mineral Water by Gradient Elution Moving Boundary Electrophoresis**; Paul Flanigan IV[1], David Ross[2], Jonathan Shackman[1]; [1]Temple University; [2]NIST

Monday Afternoon, *Room 306A*
QUANTUM CASCADE LASER APPLICATION
Organizer and Presider: Michael W. George

2:00 (152) **Quantum Cascade Laser for Quantitative Analysis in Liquid Phase**; Bernhard Lendl[1], Markus Brandstetter[1], Andreas Genner[1], Wolfgang Ritter[2]; [1]Vienna University of Technology; [2]QuantaRed Technologies

2:20 (153) **Mid-Infrared Absorption Spectroscopy using Quantum Cascade Lasers**; Adam Erlich[1]; [1]Block Engineering

2:40 (154) **Challenges and Opportunities in Biomedical IR Imaging with QCLs**; Rohit Bhargava[1], Rohith Reddy[1], Matthew Schulmerich; [1]University of Illinois at Urbana-Champaign

3:00 (155) **Recent Results from Broadly Tunable External Cavity Quantum Cascade Lasers**; Michael Radunsky[1], David Caffey[1], Vince Cook[1], Tim Day[1], Martin Algots[2]; [1]Daylight Solutions; [2]Algots Design

3:20 (156) **Nanosecond Time-Resolved IR Spectroscopy in Conventional and Supercritical Fluids Using External Cavity Quantum Cascade Lasers**; Mike George[1], James Calladine[1]; [1]University of Nottingham

3:40 (157) **First Application of External-Cavity Quantum Cascade Lasers for Nanosecond Time-Resolved Infrared Detection of Intermediates Generated by Pulse Radiolysis**; David Grills[1], Andrew Cook[1], Etsuko Fujita[1], Michael George[2], Jack Preses[1], James Wishart[1]; [1]Brookhaven National Laboratory; [2]University of Nottingham

Monday Afternoon, *Room 306B*
PAT: NEW TECHNOLOGY
Organizer and Presider: Brian Marquardt

2:00 (158) **A Robust, Reproducible, and Inexpensive Optical Oxygen Sensor for Process Analysis**; Charles Branham[1], Tom Dearing[1], Lauren Hughs[1], Kent Mann[2], Brian Marquardt[1]; [1]University of Washington; [2]University of Minnesota

2:20 (159) **Laboratory and Process Based Analytical and Sample Conditioning Applications for Modular Sample Conditioning Systems**; Mike Cost; Parker Hannifin Corporation

2:40 (160) **Process Optimisation in Microreactors Based on Flow Rate Manipulation and Real-Time Non-Invasive Measurements by Raman Spectrometry**; David Littlejohn[1], Alison Nordon[1], Sergey Mozharov[1], Charlotte Wiles[2], Paul Watts[2], Paul Dallin[3], John Girkin[4]; [1]University of Strathclyde; [2]University of Hull; [3]Clairet Scientific; [4]University of Durham

3:00 (161) **Multi-Plasma Laser-Induced Breakdown Spectroscopy (Multi-Plasma LIBS): New Developments and Further Evaluations**; Galan Moore, Douglas Jennings, Michael Carson; [1]Corning Incorporated

3:20 (162) **LED Array Based Light Induced Fluorescent Sensor for Real-time Monitoring**; Jason Dickens[1], Mervin Taylor[1], Mike Vaughn[1], Mike Ponstingl[2]; [1]GlaxoSmithKline; [2]Custom Sensors and Technology

3:40 (163) **The Next Generation of Near-Infrared Spectral Sensing Systems**; John Coates[1], Nada O'Brien[2], Fred Van Milligen[2]; [1]Coates Consulting; [2]JDSU

Monday Afternoon, *Room 306C*
BIOMEDICAL RAMAN SPECTROSCOPY
Organizer and Presider: Francis W. L. Esmonde-White

2:00 (164) **Support Vector Regression and Wavelength Interval Selection in Biological Raman Spectroscopy**; Narahara Chari Dingari[1], Ishan Barman[1], Jeon Woong Kang[1], Chae-Ryon Kong[1], Ramachandra R. Dasari[1], Michael S. Feld[1]; [1]Massachusetts Institute of Technology

2:20 (165) **Confocal Raman Microscopy Reveals the Origin of Refractive Index Variation in a Living Cell**; Jeon Woong Kang[1], Niyom Lue[1], Ishan Barman[1], Narahara Chari Dingari[1], Chae-Ryon Kong[1], Ramachandra R. Dasari[1], Michael S. Feld[1]; [1]Massachusetts Institute of Technology

2:40 (166) **Advanced Single Cell Analysis by Means of Raman Spectroscopy**; Juergen Popp[1,2], Benjamin Dietzek[0], Michael Schmitt[1], Christoph Krafft[2], Robert Moeller[1,2], Petra Roesch[2]; [1]Friedrich-Schiller University Jena; [2]Institute of Photonic Technology Jena

3:00 (167) ***Ex vivo* Determination of Breast Tissue Margins Using Spatially Offset Raman Spectroscopy**; Anita Mahadevan-Jansen[1], Daniel Masters[1], Matthew Keller[3], Mark Kelley[2]; [1]Dept of Biomedical Engineering, Vanderbilt Univ; [2]Dept of Surgery, Vanderbilt Univ; [3]Lockheed Martin Aculight

3:20 (168) ***In vivo* Dental Caries Assessment with Polarized Raman Spectroscopy: A Pilot study**; Lin-P'ing Choo-Smith[1], Mark Hewko[1], Michael Sowa[1]; [1]NRC-Inst. for Biodiagnostics

3:40 (169) **Transcutaneous Raman Spectroscopy of *ex-vivo* Human Bone**; Francis Esmonde-White[1], Karen Esmonde-White[1], Blake Roessler[1], Michael Morris[1]; [1]University of Michigan

4:00 pm – Poster Viewing and Break, *Ballroom Lobby*

4:20 pm Plenary, *Ballroom A*

The Role of Analytical Chemistry and Spectroscopy in Drug Discovery and Development: Challenges and Opportunities**;**
Uli Hacksell, *ACADIA Pharmaceuticals*

TECHNICAL PROGRAM – TUESDAY

Plenary Lectures

Morning Presider: Pavel Matousek; Afternoon Presider: André Sommer

Charles Mann Award

8:00 am Plenary, *Ballroom A*

Richard McCreery

(171) **Raman Spectroscopy of Active Molecular Electronic Devices**; Richard McCreery[1,2], Andrew Bonifas[2,3], Lian Shoute[1,2]; [1]University of Alberta; [2]National Institute for Nanotechnology; [3]The Ohio State University

Refer to page 12 for biographical information

ANACHEM Award

8:30 am Plenary, *Ballroom A*

Marc Porter

(172) **Nanomaterial Strategies for Immunodetection**; Marc Porter; University of Utah

Refer to page 12 for biographical information

Plenary Lecture

4:20 pm, *Ballroom A*

Robert S. Houk

Celebration of 30 Years of ICP-MS, Robert S. Houk, Iowa State

TUESDAY POSTER SESSION

9:00 – 10:30 am

Exhibit Hall – Ballroom B

All Tuesday posters should be put up between 7:30 – 8:00 am and removed between 5:00 – 6:00 pm. Odd numbered poster boards present between 9:00 – 9:45 am. Even numbered poster boards present between 9:45 – 10:30 am.

Bioanalytical

Board #

1 (173) **A New Technology to Isolate Muscle Stem Cells for Muscular Dystrophy Research and Treatment**; Nicholas Dobes[1], Wei Xu[1], David Detwiler[1], Chris Sims[1], Joe Kornegay[1], Nancy Allbritton[1,2]; [1]University of North Carolina Chapel Hill; [2]North Carolina State University

2 (174) **Synthesis of Fluorescent Peptide Substrates for Capillary Electrophoresis-Based ErbB-2 Kinase Assay**; Abigail Turner[1], Ryan Phillips[1], Angela Proctor[1], David S. Lawrence[1], Nancy L. Allbritton[1,2]; [1]University of North Carolina at Chapel Hill; [2]North Carolina State University

3 (175) **Characterization of Bacterial Endospores Using Fluorescence Spectrometry**; Paul DeRose[1]; [1]NIST

4 (176) **Identification and Quantification of Human Growth Hormone by MALDI –TOF/TOF MS with On-Target Digestion**; Renee N. Easter[1, 2], Colin G. Barry[2]; [1]Department of Chemistry, University of Cincinnati,; [2]Forensic Chemistry Center, U.S. Food and Drug

5 (177) **Analytical Method Development for 3,3',4,4'-Tetrachloroazobenzene (TCAB) in Rodent Mammary Gland Tissue in Support of a Short-Term Toxicology Study**; Franz Thomas[1], James Blake[1], Stephen Cooper[1], Kelly Amato[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

6 (178) **A Highly Fluorescent Conjugated Polymer Nanoparticle for Measuring pH in Acidic Compartments of Living Cells**; Prakash Kandel[1], Lawrence Fernando[1], P. Christine Ackroyd[1], Kenneth A. Christensen[1]; [1]Clemson University

Board #

7 (179) **Analytical Method Development and Validation for Safrole in Gavage Dose Formulations for Rodent Toxicology Studies**; Gwendolyn McNeill[1], Jennifer Gilliam[1], James Blake[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International, RTP; [2]NIEHS,NTP

8 (180) **Apoptosis Detection by Fluorescence Correlation Spectroscopy in an Affinity Microdevice**; Michelle Martinez[1], Randall Reif[1], Dimitri Pappas[1]; [1]Texas Tech University

9 (181) **High Definition sFTIR Imaging of Plaques in Alzheimer Disease Mouse Model Tissue**; Marzena Kastyak[1], Michael Nasse[2], Carol Hirschmugl[2], Kathleen Gough[1]; [1]University of Manitoba, MB, Canada; [2]Synchrotron Radiation Center (SRC) University

10 (182) **Polymer Fiber-Based Platforms for Measuring Gene Expression**; Kenneth Christensen[1]; [1]Clemson University

11 (183) **Encapsulated Silver Nanoparticles as Optical Labels for Biomolecules**; Kyle Dukes; [1]Clemson University

Chromatography (Chromatography, Electrophoresis, Separations)

12 (184) **Increasing HPLC Throughput for Analysis of Luciferin, its Intermediates, and Derivatives**; Celine Maravick[1], Leonard Moothart[1], Laurent Bernad[1]; [1]Promega Biosciences

13 (185) **Development of Novel Chiral Polymerizable Surfactants: Comparison for Chiral Separations in MEKC and CEC**; Jun He[1], Congying Gu[1], Shahab Shamsi[1]; [1]Georgia State University, Department of Chemistry

Board #

14 (186) **Analytical Method Development and Validation for Di (2-Ethylhexyl) Phthalate (DEHP) in Rodent Feed for Developmental and Reproductive Toxicology Studies**; Charles Crafford[1], Jennifer Gilliam[1], Gwendolyn McNeill[1], Donna Browning[1], Shaun Norton[1], James Blake[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

15 (187) **Investigations on the Extensibility of Electrochemically Modulated Separations (EMS) Using Ion Selective Electrodes Coupled with ICP-MS Detection**; Katy Fordyce[1], Kate Ziegelgruber[1], Michael Green[1], Shane Peper[1], Douglas C. Duckworth[1]; [1]PNNL

16 (188) **Simlutaneous Estimation of Silylated Monosaccharides and Bio-Ethanol in Lignocellulose Hydrolysate by Gas Chromatography**; Anju Chopra[1], Dheer Singh[1], Ravi Sahai[1], Ravinder Kumar[1], M.B. Patel[1], A.S. Sarpal[1]; [1]Indian Oil Corporation, R&D Centre

17 (189) **Portable Microcoil NMR Detection Coupled to CE for the Analysis of Perfluoro Organic Acids**; Joana Diekmann[1], Kristl L. Adams[2], Gregory L. Klunder[2], Lee Evans[2], Carla Vogt[1], Julie L. Herberg[2]; [1]Leibniz University Hannover; [2]Lawrence Livermore National Laboratory

18 (190) **Superheated Water Extraction for the Determination of Aliphatic Hydrocarbons in Source Rock and its Application in Geochemical Exploration**; Akinsehinwa Akinlua[1], Roger M Smith[2]; [1]Obafemi Awolowo University, Ile-Ife, Nigeria; [2]Loughborough University, Loughborough,UK

19 (191) **The Importance of Protein Phosphorylation in Cerebral Spinal Fluid for the Development of Biomarkers Preventing Strokes**; Karolin K. Kroening[1,2], Renee N. Easter[1,2], Joseph F. Clark[3], Joseph A. Caruso[1,2]; [1]Agilent Technologies Metallomics Ctr of Americas; [2]Univ of Cincinnati, Chemistry Dept; [3]Univ of Cincinnati, Neurology Dept

General Analytical
(Education, Electrochemistry, Forensic, Instrumentation, Laser Ablation, Materials Characterization, Other, Pharmaceutical, Process/Control, Proteomics, Surface Characterization

20 (192) **Investigation of Black Particles in Melt Granulation**; Frances Liu[1], Charles Pan[1], Greg Argentieri[1], Don Drinkwater[1], Ferris Harmon[1], Rosario LoBrutto[1]; [1]Novartis Pharm.

21 (193) **Development and Validation of an X-Ray Fluorescence Method for Detection of Toxic Metals in Pharmaceutical Products**; Sergey Arzhantsev[1], Lucinda Buhse[1], Benjamin Westenberger[1], John Kauffman[1]; [1]US Food and Drug Administration

22 (194) **PAT for API Drying Process Understanding and Control**; Charles Goss[1], Susan Barnes[1], Dennis Crowe[1], Brian Crump[1], Erwin Irdam[1], Rahn McKeown[1], Ailette Tobien[1]; [1]GlaxoSmithKline

23 (195) **Utilizing Spectral Counting to Quantitatively Characterize the Effects of Abundant Protein Depletion and the ALiPHAT Method in Human Plasma Proteomics**; Christopher M. Shuford[1], Adam M. Hawkridge[1], John C. Burnett, Jr.[2], David C. Muddiman[1]; [1]North Carolina. State University; [2]Mayo Clinic College of Medicine

Board #

24 (196) **Characterization of Core-Shell Structured Nanofibers Prepared by Coaxial and Triaxial Electrospinning**; Wenwen Liu[1], Yilin Liu[1], Giriprasath Gururajan[1], D. Bruce Chase[1], John F. Rabolt[1]; [1]University of Delaware[4]

25 (197) **Glycomic Quantification Using Surface Enhanced Raman Tagging Method**; Dongmao Zhang[1], Karthikeshwar Vangala[1], Michael Yanney[1], Roneasa Garner[1], Andrzej Sygula[1]; [1]Mississippi State University

26 (198) **Sequence Specific Genotoxicity Sensing: An Electrochemical Approach**; Eli G. Hvastkovs, Jennifer E. Satterwhite, Amanda M. Pugh, Allison S. Danell; [1]East Carolina University

27 (200) **Measurements of Sample Heating by a Laser Induced Air Plasma in Orthogonal Dual-Pulse Laser Induced Breakdown Spectroscopy**; Janna Register[1], S. Michael Angel[1]; [1]University of South Carolina

28 (201) **Determination of Praziquantel and Fenbendazole by Second Derivative Spectrophotometry**; M. Ines Toral[1], Cesar Soto[2], Romina Otipka[1], Sandra Orellana[1]; [1]Faculty of Science. University of Chile; [2]Univesity of Concepcion

Spectroscopy
(Absorption, Atomic Spectroscopy, Fluorescence, ICP, Infrared, Laser Spectroscopy, Mass Spectrometry, Molecular Spectroscopy, Near Infrared, NMR, Raman, Surface Enhanced Raman, Surface Plasmon Resonance)

29 (202) **Raman Spectroscopy Using a Spatial Heterodyne Spectrometer**; Nathaniel Gomer[1], Christopher Gordon[1], S. Michael Angel[1]; [1]University of South Carolina

30 (203) **Optical Fiber-Based Tools for the Analysis of Zinc in Aqueous Environments**; Steven Kopitzke[1], Peter Geissinger[1]; [1]University of Wisconsin-Milwaukee

31 (204) **Polarized Fourier Transform Infrared Spectroscopic Study on Molecular Orientation in Electrospun Polymer Fibers**; Xiaoqian Ma[1], Bruce Chase[1], John Rabolt[1]; [1]Materials Sci. and Eng., Univ. of Delaware

32 (205) **Application of DRIFTS for Direct On-Filter Characterization of Extrathoracic Wood Dust Collected in Working Environments**; Madalina Chirila[1], Taekhee Lee[1], Michael Flemmer[1], James Slaven[1], Martin Harper[1]; [1]Nat. Inst. for Occupational Safety and Health

33 (206) **Globalization of Spectroscopic Data Access**; David Joyce[1], Steve Best[1]; [1]Thermo Fisher Scientific (Informatics)

34 (207) **Investigation of Excitation / Ionization Processes of High-Power Pulsed Microplasma for Aqueous Sample Analysis**; Yoichi Nagata[1], Yuichiro Takahashi[1], Yuta Negishi[1], Kenji Kodama[2], Hidekazu Miyahara[1], Kuniyuki Kitagawa[2], Akitoshi Okino[1]; [1]Tokyo Institute of Technology; [2]Nagoya University

35 (208) **Newly Observed Effects of Anions on the Raman Bending Vibration of Water**; Henk-Jan van Manen, Bojk Berghuis, Aurelie Arrouet, Rob Bloemenkamp, Oscar van den Brink; AkzoNobel RD&I

Board #

36 (209) **Evaluation of a Portable SERS Substrate Reader for Point of Use Analysis**; David Eustace[1], Alastair Mcinroy[1], Bryan Ray[2], Rick Cox[2]; [1]Renishaw Diagnostics; [2]DeltaNu-Intevac Photonics

37 (210) **Investigating the Relationship of Instrument Parameters and Sample Complexity Towards the Analysis of Label-Free Relative Quantification Using Spectral Counts**; Genna Andrews[1], Adam Hawkridge[1], David Muddiman[1]; [1]North Carolina State University

38 (211) **Surface-Enhanced Raman Scattering for the Detection of Lipid Mediators Secreted by Mast Cells During Allergic Response**; Audrey Guerard[1], Kyle Bantz[1], Christy Haynes[1]; [1]University of Minnesota

39 (212) **Detection of Fungal Exudates Using Synchrotron FTIR**; Kathleen Gough[1], Merrill Isenor[1], Susan Kaminskyj[2], Carol Hirschmugl[3], Michael Nasse[3], Rusty Rodriguez[4], Regina Redman[4]; [1]University of Manitoba; [2]University of Saskatchewan; [3]Synchrotron Radiation Center; [4]University of Washington

40 (213) **Determination of Melamine and Cyanuric Acid in Contaminated Pet Food and Milk Products Using Surface-Enhanced Raman Scattering**; Ngee-Sing Chong[1], Sunil Kumar Setti[1], Beng Guat Ooi[1]; [1]Middle Tennessee State University

41 (214) **Quantitative Measurements of Biomass-Derived and Other Oxygenate Additives in Gasoline and Diesel Fuels by Infrared Spectroscopy**; Beng Guat Ooi[1], Joe Boachie[1], Ngee-Sing Chong[1]; [1]Middle Tennessee State University

42 (215) **Confocal Raman Imaging – A New Method for Drug Characterization and Design**; Jiangyoung Yang[1], Ute Schmidt[1], Andrea Jauss[1], Thomas Dieing[1], Fernando Vargas[1], Olaf Hollricher[1]; [1]WITec GmbH

43 (216) **Surface Plasmon Resonance (SPR) Biosensing within Electrokinetic Channels**; Qiongjing Zou[1], Karl Booksh[1]; [1]University of Delaware

44 (217) **Conservation of Historical Pigments on Sultan Baybas Qur'an and a 14th century Mamluk Qur'an by Raman Spectroscopy**; Enrique Lozano Diz[1], Colin Baker[2], Paul Garside[2], David Jacobs[2], Barr Knight[2], Dean Brown[1]; [1]PerkinElmer; [2]The British Library

45 (218) **Utility of Raman Spectroscopy in a Materials Science Environment**; Shawn Mehrens[1], Slobodan Sasic[1], Linda Lohr[1]; [1]Pfizer Global R&D

46 (219) **Measurement of Trace Atmospheric Pollutants by Broadband Cavity-Enhanced Absorption Spectrometry with an FT-IR Spectrometer**; Ben Perston[2], Cathryn Langley[1], Gus Hancock[1], Wolfgang; [1]University of Oxford; [2]PerkinElmer; [3]Oxford Medical Diagnostics;

47 (220) **Direct Detection of Components and Nanoparticles in Smoke by Time-of-Flight Mass Spectrometry**; Yoko Nunome[1, 2], Kenji Kodama[2], Kozo Matsumoto[2];Hyunkook Park[3], Sang Chun Lee[4], Kuniyuki Kitagawa[2]; [1]Graduate School of Engineering, Nagoya Univ.; [2]EcoTopia Science Institute, Nagoya Univ.; [3]Korea Maritime Univ.; [4]Department of Chemistry, Kyungnam Univ.

Board #

48 (221) **Time Resolved Fourier Transform Infrared (TR-FTIR) Studies Employing Micro Fluidic Mixers**; Christoph Wagner[1], Martin Kraft[2], Michiel Vellekoop[1], Bernhard Lendl[1]; [1]Vienna University of Technology; [2]Carinthian Tech Research AG

49 (222) **Molecular Spectroscopy Diagnostic: Aluminum Monoxide**; Christian Parigger[1], James Hornkohl[1], Burl Donaldson[2], Thomas Sanchez[3]; [1]Univ. Tennessee Space Inst.; [2]New Mexico State Univ.; [3]Omicron Safety & Risk Technologies, Inc.

50 (223) **Analysis of Lanthanide Elements in Molten LiCl-KCl Eutectic Salt Using Laser-Induced Breakdown Spectroscopy**; Dong Hyoung Lee[1], Bong Young Kim[1], Tae Hyeong Kim[1], Jong-Il Yun[1]; [1]KAIST

51 (224) **Mid-IR Spectroscopy as a Quality Control Tool for Traditional and Herbal Medicines**; Ben Perston[1], Patrick Courtney[1], Chris Lynch[1]; [1]PerkinElmer

52 (225) **Fluorescence Guided Ingredient Specific Particle Sizing of Nasal Suspension Formulations**; Ryan Priore[1], Oksana Olkhovyk[1], Oksana Klueva[1], Michael Fuhrman[1]; [1]ChemImage

53 (226) **Development of a Fieldable Sensing System for Rapid Pesticide Exposure Analysis**; Kevin Spencer[1], Susan Clauson[1], Sarah Spencer[1], Jim Sylvia[1], Quirina Vallejos[2], Sara Quandt[2], Thomas Arcury[2]; [1]EIC Laboratories, Inc; [2]Wake Forest University School of Medicine

54 (227) **Biofuels: Properties and Contaminants by FT-IR Analysis**; Ben Perston[1], Aniruddha Pisal[1], Dean Brown[1]; [1]PerkinElmer

55 (228) **Explosives Detection in the Presence of Real-World Interferences using Surface-Enhance Raman Spectroscopy**; Kevin Spencer[1], Sarah Spencer[1], Susan Clauson[1], James Sylvia[1]; [1]EIC Laboratories, Inc

56 (229) **Realistic Resolution Targets for Chemical Imaging of Pharmaceuticals: PEG-Embedded Polydimethylsiloxane Devices**; Laura C. Mecker[1], John F. Kauffman[1]; [1]Food and Drug Administration

57 (230) **Quantitative Proteomic Analysis of Human Embryonic Stem Cells During Early Stage Differentiation Utilizing SILAC Labeling and High Resolution Mass Spectrometry**; Timothy Collier[1], Prasenjit Sarkar[1], Balaji Rao[1], David Muddiman[1]; [1]North Carolina State University

58 (231) **Advancements in Low-Frequency Raman Spectroscopy Using Ultra-High Performance Holographic Notch Filters**; Frank Havermeyer[1], Christophe Moser[1]; [1]Ondax, Inc

59 (232) **Reduction of Spectral Interferences in Inductively Coupled Plasma Mass Spectrometry Using a Universal Cell Technology**; Hamid Badiei[1], Kaveh Kahen[1]; [1]PerkinElmerSCIEX

Tuesday Morning, *Room 301B*
HEADSPACE ANALYSIS FOR CHEMICAL SIGNATURES
Organizer and Presider: Greg Klunder

10:30 (233) **Detection of Drugs and Explosives in Large Volume Headspace Using Planar Solid Phase Microextraction and Ion Mobility Spectrometry**; Jose Almirall[1], Wen Fan[1], Mimy Young[1]; [1]Florida International University

10:50 (234) **Development of Odor Mimics for Improved Detection of Forensic Specimens by Canines and Instruments**; Kenneth Furton[1], Katylynn Beltz[1], Norma Caraballo[1], Lauryn DeGreeff[1], DeEtta Mills[1]; [1]Florida International University

11:10 (235) **Characterization of Volatile Compound Evolution from Complex Synthetic and Natural Polymeric Materials: Methodologies, Statistical Data Analysis and Applications**; James Lewicki[1], Sarah Chinn[1], Christopher Harvey[1], Cynthia Alviso[1], John Liggat[2], Lorriane Gibson[2], Robert Maxwell[1]; [1]Lawrence Livermore Nat'l Laboratory; [2]University of Strathclyde, Glasgow

11:30 (236) **Metabolomic Profiling of Exhaled Breath to Differentiate between Asthma and COPD**; Michael Schivo[2], Abhinav Bhushan[1], Weixiang Zhao[1], Nicholas J. Kenyon[2], Cristina E. Davis[1]; [1]UC Davis, Dept Mech & Aeronaut Engineering; [2]UC Davis, Dept Int Med, Div Pulm Med

11:50 (237) **Clues Overhead: GC/MS profile of VOCs Produced by Algae as Markers of Contaminated Water**; Andrew Callender; Tennessee Technological University

12:10 (238) **Analysis of Microbial and Fungal Toxins in Airborne Grain Dust**; Mustafa Selim[1], S. L. Kinney[1], H. D. Patel[1]; [1]East Carolina University

Tuesday Morning, *Room 302A*
ATOMIC ANALYSES IN THE PHARMACEUTICAL AND NEUTRACEUTICAL INDUSTRY
Organizers and Presiders: Ken Marcus and Nancy Lewen

10:30 (241) TBD

10:50 (242) **A Survey of Metal Contamination in Pharmaceuticals and Dietary Supplements**; John Kauffman[1], James Guthrie[2], J. David Rovertson[2,3]; [1]FDA Division of Pharmaceutical Analysis; [2]University of Missouri Research Reactor; [3]University of Missouri Dept of Chemistry

11:10 (243) **Regulation of Elemental Impurities**; Mamata De[1]; [1]US Goverment, CDER, FDA

11:30 (244) **ICP-OES Analysis of Pharmaceutical Materials: USP Elemental Impurities**; Timothy Shelbourn[1]; [1]Eli Lilly and Company

11:50 (245) **Phosphorus and Sulfur as Internal Tags for Pharmaceutical Analysis by ICPMS**; Joseph Caruso[1], Brittany Catron[1], Renee Easter[1], Kirk Lokits[1], Pat Limbach[1]; [1]University of Cincinnati

12:10 (246) **HPLC-Particle Beam Mass Spectrometry for Metal Speciation and Actives Profiling in Botanical Products**; R. Kenneth Marcus[1]; [1]Clemson University

Tuesday Morning, *Room 302B*
THE JAAS SILVER ANNIVERSARY CELEBRATION: HIGHLIGHTING YOUNG INVESTIGATORS IN ATOMIC SPECTROSCOPY
Organizers: Norbert Jakubowski, Spiros Pergantis, and Steven Ray; Presider: Norbert Jakubowski

10:30 (247) **FACSS STUDENT AWARD - Investigations of Fundamental Processes and Ion Chemistry of the Flowing Atmospheric-Pressure Afterglow and Low-Temperature Plasma Probe Ambient Ionization Sources**; Jacob T. Shelley[1], Joshua S. Wiley[2], Carsten Engelhard[1], Ayanna U. Jackson[2], R. Graham Cooks[2], Gary M. Hieftje[1]; [1]Dept. of Chemistry - Indiana University; [2]Dept. of Chemistry - Purdue University

10:50 (248) **Elemental Bioimaging by Pulsed Radio-Frequency Glow Discharge – Monochromatic Imaging Spectrometry**; Carsten Engelhard[1,2], Steven J. Ray[1], Wolfgang Buscher[2], Maxim Voronov[3], Volker Hoffmann[3], Gary M. Hieftje[1]; [1]Indiana University, Dept. of Chemistry; [2]University of Münster, Institute of Inor; [3]Leibniz Institute for Solid State

11:10 (249) **Analytical Methods for the Diagnosis of Death-By-Drowning**; Maite Aramendia Marzo[1], Maria Rosario Flórez[2], Martín Resano[2], Michel Piette[1], Frank Vanhaecke[1]; [1]Ghent University; [2]Universidad de Zaragoza

11:30 (250) **Depth Profile Capabilities of Pulsed RF Glow Discharge TOFMS. How Far Are We from SIMS?**; Jorge Pisonero[1], Nerea Bordel[1], Alfredo Sanz-Medel[1], Antonino Licciardello[2]; [1]University of Oviedo, Spain; [2]University of Catania, Italy

11:50 (251) **New Possibilities for Quantitative Analysis in Environmental and Life Sciences Using (Hetero)Element Tags and ICP-MS Detection**; Daniel Proefrock[1], Andreas Prange[1]; [1]GKSS Research Centre

12:10 (252) **Application of Labelled Antibodies for ICP-MS Detection**; Charlotte Giesen[1,2], Michael G Weller[0], Norbert Jakubowski[1], Ulrich Panne[1,2]; [1]BAM; [2]Humboldt University Berlin

Tuesday Morning, *Room 302C*
CHEMOMETRICS IN FORENSICS
Organizer: Barry Lavine; Presider: Stephen Morgan

10:30 (253) **Multivariate Analysis of Variance for Forensic Trace Evidence Decision-Making**; Stephen L. Morgan[1]; [1]University of South Carolina

10:50 (254) **Improving Investigative Lead Information and Evidential Significance Assessment for Automotive Paint by Development of Pattern Recognition Based Library Searching Techniques**; Barry Lavine[1], Nikhil Mirjankar[1], Mark Sandercock[2]; [1]Oklahoma State University; [2]Royal Candadian Mounted Police

11:10 (255) **Application of Chemometric Methods and Advanced Pattern to Trace Evidence Analysis**; Nicholas Petraco[1]; [1]John Jay College of Criminal Justice; [2]The Graduate Center/CUNY

11:30 (256) **Application of Target Factor Analysis to the Classification of Ignitable Liquids from Fire Debris**; Michael Sigman[1], Mary Williams[1]; [1]University of Central Florida

11:50 (257) **The Design of an Infrared Imaging System for Blood Stains at Crime Scenes Using a Chemometrics Simulation-Driven Process**; MIchael L. Myrick[1], Heather Brooke[1], Megan R. Baranowski[1], Jessica N. McCutcheon[1], Stephen L. Morgan[1]; [1]University of South Carolina

12:10 (258) **Linear Discriminant Analysis-Parallel Factor Analysis for Classificaiton of Biological Particles by Fluorescence**; Karl Booksh, University of Delaware

Tuesday Morning, *Room 304*
ANACHEM AWARD SYMPOSIUM
Organizer and Presider: Greg M. Swain

10:30 (259) **Novel Nanorod Array Substrates as a Platform for SERS-Based Biosensing of Infectious Disease**; R.A. Dluhy[1], J.D. Driskell[1], Y.-P. Zhao[1], P. Rota[2], R.A. Tripp[1]; [1]University of Georgia; [2]Centers for Disease Control

10:50 (260) **Using Spectroscopy to Reveal Dynamics in Self-Assembled Systems**; Gary Blanchard[1], Heather Pillman[1], Monika Dominska[1], Benjamin Oberts[1]; [1]Michigan State University

11:10 (261) **Modified Electrodes in the Solid State: Molecules as Circuit Components**; Richard McCreery[1,2], Adam Bergren[1], Haijun Yan[1, 3], Andrew Bonifas[1, 3]; [1]National Institute for Nanotechnology; [2]University of Alberta; [3]Ohio State University

11:30 (262) **Holey Zeolites: Chemistry and Biology in Confined Spaces**; Prabir Dutta; [1]The Ohio State University

11:50 (263) **Optically Transparent Diamond Electrodes for Use in UV/Vis and IR Transmission Spectroelectrochemical Measurements**; Greg Swain[1], Chen Qiu[1], Denis Proshlyakov[1]; [1]Michigan State University

Tuesday Morning, *Room 305A*
DIELECTROPHORESIS AND RELATED TECHNIQUES
Organizers: Blanca Lapizco Encinas and Alexandra Ros; Presider Alexandra Ros

10:30 (265) **Manipulating Biomolecules by Dielectrophoresis**; Alexandra Ros[1], Asuka Nakano[1], Lin Gan[1], Tzu-Chiao Chao[1]; [1]Arizona State University

10:50 (266) **Insulator-Based Dielectrophoresis of Particles Employing Extremely Low Frequency Alternating Current Electric Fields**; Javier L. Baylon-Cardiel[1], Nadia M. Jesus-Perez[1], Ana V. Chavez-Santoscoy[1], Sergio O Martinez-Chapa[1], Blanca H. Lapizco-Encinas[2]; [1]Tecnológico de Monterrey, Mexico; [2]CINVESTAV-Monterrey, Mexico

11:10 (267) **Quantitative Analysis of Erythrocyte Rupturing in an Alternating Current Dielectrophoretic Field as Compared to Theoretical Observations**; Kaela Leonard[1], Adrienne Minerick[1]; [1]Michigan Technological University

11:30 (268) **Dielectrophoresis at Conductive Liquid Interfaces**; Zachary Gagnon[1]; [1]Johns Hopkins University

11:50 (269) **Ion excluded Volume Effects on Dielectrophoresis of a Colloidal Particle**; Hui Zhao; [1]University of Nevada Las Vegas

12:10 (270) **Dielectrophoretic Manipulation of Particles and Cells in Curved Microchannels**; Xiangchun Xuan[1]; [1]Clemson University

Tuesday Morning, *Room 305B*
EMERGING TECHNOLOGIES FOR STANDOFF DETECTION FOR HOMELAND SECURITY
Organizer and Presider: Michael Shepard

10:30 (271) **US Department of Homeland Security Counter-IED Detection Programs and Priorities**; Michael Shepard[1]; [1]US Department of Homeland Security

10:50 (272) **Vibrational Sum Frequency Spectroscopy for Stand-Off Detection of Chemicals on Surfaces**; William Asher[1], Ella Willard-Schmoe[1]; [1]University of Washington

11:10 (273) **Sensitive Standoff Detection of Chemical Agents By Nonlinear Multi-Photon Laser Wave-Mixing Spectroscopy**; William Tong[1], Marc Gregerson[1], Tiffany Neary[1], Marcel Hetu[1], Manna Iwabuchi[1], Jorge Jimenez[1], Ashley Warren[1]; [1]San Diego State University

11:30 (274) **Photoacoustic Standoff Detection Using Atomic Vapor Filters**; Dimitri Pappas[1]; [1]Texas Tech University

11:50 (275) **Single Ultrafast Pulse Excitation for Remote Coherent Anti-Stokes Raman Spectroscopy (SUPER-CARS) for Standoff Detection**; Marcos Dantus; [1]Michigan State University

12:10 (276) **Differential Laser-Induced Perturbation Spectroscopy (DLIPS) for Standoff Detection**; David Hahn[1], Sarah Smith[1], Jonathan Merten[1], Nicolo Omenetto[1]; [1]University of Florida

Tuesday Morning, *Room 306A*
SPECIAL SESSION TO HONOR WILLIAM G. FATELEY
Organizers: Bruce Chase and Jeff White; Presider: Jeff White

10:30 (277) **Raman Scattering as a Probe of Structure in Polymeric Fibers**; Bruce Chase[1]; [1]Pair Technologies LLC

11:10 (278) **Effect of Hydrogen Bond Strength on the Vibrational Relaxation of Interfacial Water**; Ali Eftekhari-Bafrooei[1]; [1]Temple University

11:30 (279) **Applications of Nanoscale Imaging and Spectroscopy Using Tip Enhanced Raman Spectroscopic Techniques**; Ira Levin[1], Zachary Schultz[1,2], Taner Ozel[1], Tsoching Chen; [1]National Institutes of Health; [2]University of Notre Dame

12:10 (280) **FT-IR Gas Analysis: What We've Learned in 25 Years**; Martin Spartz[1]; [1]Prism Analytical Technologies, Inc.

Tuesday Morning, *Room 306B*
SAS PROCESS ANALYTICAL TECHNOLOGY
Organizers and Presiders:: Edita Botonjic-Sehic and Brandye Smith-Goettler

10:30 (281) **Design Considerations and Best Practices, for the Implementation of a Fluorescence Spectrophotometer in Laboratory and Plant Pharma PAT;** Susan Bragg, Paul Davies, Expo Technologies

10:50 (282) **The Application of PAT to Continuous Processes**; Martin Warman[1], Justin Pritchard[1], Gregory Connelly[1], Aude Legos[2], Trevor Page[2]; [1]Vertex Pharmaceuticals Inc; [2]GEA Pharma Systems

11:10 (283) **Fundamental Studies in Powder Drying Using *in situ* Spectroscopic and Off-Line Particle Size Analysis Techniques**; Peter Hamilton[1], Elana Duff[1], David Littlejohn[1], Alison Nordon[1], Jan Sefcik[1], Paul Slavin[2], Paul Dallin[3], John Andrews[3]; [1]University of Strathclyde; [2]GlaxoSmithKline; [3]Clairet Scientific

11:30 (284) **On-line Mass Spectroscopy in the Pharmaceutical Industry**; Charles Goss[1], James Rydzak[1], Gregory Gervasio[1]; [1]GlaxoSmithKline

11:50 (285) **NIR Monitoring and Control of a Constant Volume Distillation**; Bob Cooley[1], Russ Fitzgerald[1], Delphi Burton[1], Ming Li Lim[2], Jeremy Yeo[2]; [1]GlaxoSmithKline - RTP; [2]GlaxoSmithKline - Jurong

12:10 (286) **Combination of PAT and Data Fusion for the Characterization of a Chemical Process**; Thomas Dearing[1], Brian Marquardt[1]; [1]Applied Physics Laboratory

TECHNICAL PROGRAM – TUESDAY
Orals 10:30 am – 12:30 pm ◆ Poster Viewing 1:30 pm ◆ Orals 2:00 – 4:00 pm

Tuesday Morning, *Room 306C*
RAMAN MICROSCOPY AND IMAGING
Organizers and Presiders: Andrew Whitley and Eunah Lee; Presider: Andrew Whitley

10:30 (287) **Utilizing High-Resolution Raman Spectroscopy to Examine Molecular Interactions Within Lithium Battery Electrolyes**; Wesley Henderson[1], Daniel Seo[1], Qian Zhou[1]; [1]NC State University

10:50 (288) **Design and Self-Assembly of Surface-Enhanced Raman Scattering (SERS) Platforms: Building an Optical Biosensor**; Betty C. Galarreta[1], Peter R. Norton[1], Francois Lagugne-Labarthet[1]; [1]The University of Western Ontario

11:10 (289) **Applications of Raman Spectroscopy for Cancer Cells Detection**; Alexandru R. Biris[1, 3], Meena Mahmood[1], Yang Xu[1], Alokita Karmakar[1], Anindya Gosh[2], Ashley Fejleh[1]; [1]University of Arkansas, Applied Science; [2]University of Arkansas, Chemistry Dept; [3]National Institute for R&D, Romania

11:30 (290) **Raman Spectroscopy of Algae Biopetroleum Hydrocarbons from Botryococcus Braunii**; Taylor L. Weiss[1], Hye Jin Chun[2], Jaan Laane[2], Shigeru Okada[3], Tim P. Devarenne[1]; [1]Texas A&M University, Dept. of Biochem./Biophys.; [2]Texas A&M University, Dept. of Chemistry; [3]University of Tokyo, Agr. & Life Sci.

11:50 (291) **Coupling Raman and Fluorescence for Confocal Imaging of Biological and Pharmaceutical Samples**; I. Chourpa[1], E. Munnier[1], A. Paillard[1], E. Allard[1], C. Linassier[1], S. Cohen-Jonathan[1], R. Lewandowska[3], E. Lancelot[3], E. Garcion[2], P. Dubois[1], M. L. Saboungi[4]; [1]Universite Francois Rabelais de Tours; [2]INSERM; [3]Horiba Jobin Yvon; [4]UMR

12:10 (292) **Lean Raman Imaging for Rapid Assessment of Homogeneity in Pharmaceutical Formulations**; Stephanie Brown[1], Mike Claybourn[1], Chris Ashman[1]; [1]AstraZeneca

Tuesday Morning, *Room 307*
NANOTECHNOLOGY: APPLICATIONS TO SENSING AND ENERGY
Organizers: Francis Zamborini, Jayne Garno, and Shouzhong Zou; Presider: Francis Zamborini

10:30 (293) **Hydrogen Sensing with a Single Palladium Nanowire**; Reginald Penner[1], Fan Yang[1]; [1]Univ. of California, Irvine

10:50 (294) **Electrochemically-Fabricated Metal/Organic/Metal Junctions for Electronic Switching and Sensing Applications**; Francis Zamborini[1], Radhika Dasari[1]; [1]University of Louisville

11:10 (295) **Nitric Oxide-Releasing *in vivo* Glucose Biosensors**; Mark Schoenfisch[1], Ahyeon Koh[1], Dan Riccio[1], Bin Sun[1], Yuan Lu[1]; [1]University of North Carolina

11:30 (296) **Ultrasensitive Electrochemical Detection for DNA Arrays Based on Silver Nanoparticles**; Danke Xu[1], Hui Li[1], Ziyin Sun[1], Hong-Yuan Sun[1]; [1]Nanjing University

11:50 (297) **Nanocatalysts for Electrochemical Energy Conversion: The Challenges for Synthesis and Characterization**; Keith Stevenson[1], Anthony Dylla[1], Sankaran Murugesan[1], Sebatien Verret[1], Salome Nagita[1], Corrinne Atkinson[1]; [1]University of Texas at Austin

12:10 (298) **Vertically Oriented Nanogap Substrates for Surface-Enhanced Raman Scattering**; Kyle Bantz[1], Hyungsoon Im[1], Nathan Lindquist[1], Sang-Hyun Oh[1], Christy Haynes[1]; [1]University of Minnesota

1:30 pm – Poster Viewing and Dessert Break, *Exhibit Hall*

Tuesday Afternoon, *Room 301B*
MS FUNDAMENTALS AND GAS-PHASE ION CHEMISTRY
Organizer and Presider: Allison S. Danell

2:00 (299) **Protein Structure in the ESI Transition Regime: Where Does Solution End and the Gas Phase Begin?**; Ryan Julian[1]; [1]University of California Riverside

2:20 (300) **The Analysis of Protein Carbonyl Modifications from *in vitro* and *in vivo* Oxidative Stress**; Scott Gronert[1], David Simpson[1], Zafer Ugur[1]; [1]Virginia Commonwealth University

2:40 (301) **When CID ≠ IRMPD and ECD ≠ ETD: Contradictions to Conventional Wisdom**; Gary Glish[1], Alessandra Ferzoco[1], Natalie Thompson[1], Daniel Thomas[1], Takashi Baba[1]; [1]University of North Carolina

3:00 (302) **Structures and Energetics of Transition Metal Cation N-Donor Ligand Complexes from Collision-Induced Dissociation and Theoretical Studies**; Mary T. Rodgers[1], Nalaka S. Rannulu[2], Holliness Nose[1]; [1]Wayne State University; [2]University of New Orleans

3:20 (303) **Characterization of Peptide-Metal Complexes: To ESI or Not to ESI**; Allison Danell; [1]East Carolina University

3:40 (304) **MS Approaches for the Investigation of Tertiary and Quaternary Structure of Nucleic Acids**; Daniele Fabris; [1]University at Albany

Tuesday Afternoon, *Room 302A*
CHEMISTRY IN ART AND ARCHAEOLOGY
Organizer and Presider: Mary Kate Donais

2:00 (305) **Archaeometry: Combining Analytical Technique with Archaeological Interpretation to Find a Meaningful Relationship**; Michael D. Glascock[1]; [1]University of Missouri

2:40 (306) **XRF Analysis of Elementally Non Uniform Materials**; Bruce Kaiser; [1]Bruker AXS

3:00 (307) **Spectroscopic Investigations of Archaeological Sample from the Coriglia, Castel Viscardo Excavation Site, Italy**; Mary Kate Donais[1], Anna Daigle[1], David George[2]; [1]Saint Anselm College Chemistry Department; [2]Saint Anselm College Classics Department

3:20 (308) **Handheld XRF Analysis of the 6000 Year Old Nahal Mishmar Hoard of Copper Alloyed Artifacts**; Aaron Shugar[1]; [1]Buffalo State College

3:40 (309) **LA-ICP-MS Microsampling of Human Bones: The Dynamic Interaction between Sample, Introduction Method and Target Data for Organic Minerals**; Ian Scharlotta[1]; [1]Baikal Archaeology Project, University of Alberta

TECHNICAL PROGRAM – TUESDAY
Orals 2:00 – 4:00 pm

Tuesday Afternoon, *Room 302B*
ICPMS – A LOT MORE THAN TOTAL METALS ANALYSIS
Organizer and Presider: Joe Caruso

2:00 (310) **From Phosphorylation to Metalloproteins as Biomarkers for Hemorrhagic Stroke**; Joseph Caruso[1], Karolin Kroening[1], Yaofang Zhang[1], Renee Easter[1]; [1]University of Cincinnati

2:20 (311) **Metallomics for Non-Covalently Attached Metalloproteins: What Are the Limitations?**; James Holcombe[1], Isaac Arnquist[1], Haley Finley-Jones[1]; [1]Univ. of Texas at Austin

2:40 (312) **Examination of Trace Arsenic Species in Fruit Juices by LC-ICPMS**; Kevin Kubachka[1], Traci Hanley[1], Nohora Shockey[1]; [1]US Food and Drug Administration

3:00 (313) **Metallomics Approach in Diabetes Research: Relation between Metal/Metalloid Status and Some Clinical Parameters, Typically Evaluated in Diabetic Patients**; Katarzyna Wrobel[1,2], Kazimierz Wrobel[1,2]; [1]University of Guanajuato, Department of Chemistry; [2]Metallomics Center of America

3:20 (314) **Application of Elemental and Molecular Mass Spectrometry and qNMR for the Determination of Arsenobetaine**; Zoltan Mester[1], Anthony Windust[1]; [1]National Research Council

3:40 (315) **Measurements of Metals in Microbes - New Metallomics Techniques**; David W. Koppenaal[1], M Liz Alexander[1], Himadri Pakrasi[2], Jana Stockel[2], Charles J Barinaga[1]; [1]Pacific Northwest National Laboratory; [2]Washington University, St Louis

Tuesday Afternoon, *Room 302C*
CHEMOMETRICS FOR BIOLOGICAL AND BIOMEDICAL SPECTROSCOPY
Organizer and Presider: Barry Lavine

2:00 (316) **What Chemometrics Tells Us About the Design of Optical Instruments**; Karl Booksh[1]; [1]University of Delaware

2:20 (317) **Traps and Pitfalls when Applying Chemometrics to Biomedical Problems**; Jerome Workman[1]; [1]Technology Business Associates

2:40 (318) **Nocturnal Hpoglycemic Alarm Based on Noninvasive Near-Infrared Spectroscopy**; Gary Small[1]; [1]University of Iowa

3:00 (319) **Spectrochemical Monitoring of the Chemical Composition of Microalgae in Response to Changing Environmental Conditions**; Frank Vogt[1], Edward Duranty[1], Rebecca Horton[1], morgan McConico[1]; [1]University of Tennessee, Department of Chemistry

3:20 (320) **Classification of Individual Phytoplankton Cells via Imaging Multivariate Optical Computing**; Laura Hill[1], Tammi L. Richardson[1], Timothy Shaw[1], Michael L. Myrick[1]; [1]University of South Carolina

3:40 (321) **Detection of Mastitis in Cows during Milking Using DRIFTS and the Wavelet Packet Tree to Mine NIR Data**; Barry Lavine[1], Nikhil Mirjankar[1], Roumiana Tsenkova[2]; [1]Oklahoma State University; [2]Kobe University

Tuesday Afternoon, *Room 304*
CHARLES MANN AWARD SYMPOSIUM
Organizers: Richard McCreery and Pavel Matousek;
Presider: Pavel Matousek

2:00 (322) **Raman Scattering as a Probe of Structure Development in Electro-Spun Fibers**; Bruce Chase[1], John Rabolt[2], Giri Gururajan[3]; [1]Pair Technologies LLC; [2]University of Delaware; [3]Connoco Phillips

2:40 (323) **Colorimetric-Solid Phase Extraction (C-SPE): Water Quality Monitoring for Crew Health on the International Space Station**; Marc Porter[1]; [1]Nano Institute, University of Utah

3:20 (324) **Evolution Trend in Raman Spectroscopy Instrumentation**; Jun Zhao, Juergen Sawatzki; [1]Bruker Optics

3:40 (325) **Characterizing Mechanisms and Dynamics of Emulsion Free Radical Polymerization by *in-situ* Raman Spectroscopy**; R. Thomas Cambron[1], Ed Grundner[1], Tom Desmarais[1]; [1]Procter and Gamble

Tuesday Afternoon, *Room 305A*
PRACTICAL ASPECTS OF CHIRAL ANALYSIS USING VCD AND ROA
Organizer and Presider: Douglas James Minick

2:00 (326) **VCD in Pharmaceutical Discovery**; Don Pivonka[1], Steve Wesolowski[1]; [1]AstraZeneca

2:20 (327) **Advances in Pharmaceutical Applications of Vibrational Circular Dichroism**; Laurence Nafie; [1]Syracuse University

2:40 (328) **Practical Considerations for Rapid And Accurate Stereochemical Assignments Using VCD**; Feng Qiu, Yingru Zhang, Michael Reily, David Wang-Iverson, Adrienne Tymiak; [1]Bristol-Myers Squibb Co

3:00 (329) **A Theoretical and Experimental Study of Solvent Effects on the VCD Spectrum of 1-(2-Methylbenzoyl)-2-Pyrrolidinemethanol**; James Cheeseman[1], Douglas Minick[2]; [1]Gaussian, Inc.; [2]GlaxoSmithKline

3:20 (330) **Calculating Confidence Limits for Ab Initio VCD Structural Assignments: A Comparison of Two Approaches**; Dean Phelps[1], Douglas Minick[1]; [1]GlaxoSmithKline, Molecular Discovery Research

3:40 (331) **Applications of Raman Spectroscopy in the Identification of Extraneous Particulats and the Analysis of ProteinTherapeutics**; Xiaolin Cao[1]; [1]Amgen Inc

Tuesday Afternoon, *Room 305B*
LASERS IN ANALYTICAL CHEMISTRY: CELEBRATING THE 50TH ANNIVERSARY OF THE LASER
Organizer and Presider: James R. Gord

2:00 (332) **Automated Pulse Shaping and Compression Based on Multiphoton Intrapulse Interference Phase Scan (MIIPS) Enables Development of Novel Biomedical Analytical Methods**; Marcos Dantus[1]; [1]Department of Chemistry, Michigan State University

2:20 (333) **New Frontiers for Old Nonlinear Optical Effects**; Garth Simpson[1]; [1]Purdue University

2:40 (334) **Plasmon-Controlled Fluorescence: A New Paradigm in Fluorescence Spectroscopy**; Krishanu Ray[1], Joseph R. Lakowicz[1]; [1]Univ of Maryland School of Medicine

TECHNICAL PROGRAM – TUESDAY
Orals 2:00 – 4:00 pm ◆ Poster Viewing 4:00 pm ◆ Plenary Lecture 4:20 pm

3:00 (335) **Reacting Flow Diagnosis Using Ultrafast Lasers**; Sukesh Roy[1], James R. Gord[2]; [1]Spectral Energies, LLC; [2]Air Force Research Laboratory

3:20 (336) **Mass Spectrometric Imaging at the Cellular and Subcellular Level by Laser Desorption/Ionization**; Edward Yeung[1], DC Perdian[1], Sangwon Cha[1], Sangwon Cha[1], Young-Jin Lee[1]; [1]Ames Laboratory, Iowa State University

3:40 (337) **Nanomaterial Design for Quantitative Surface Enhanced Raman Scattering (SERS) Detection**; Amanda Haes[1], Marie C. Pierre[1], Sudip Nath[1], Maryuri Roca[1], Binaya Shrestha[1]; [1]University of Iowa

Tuesday Afternoon, *Room 306A*
SPECIAL SESSION TO HONOR WILLIAM G. FATELEY
Organizers: Bruce Chase and Jeffrey White; Presider: Bruce Chase

2:00 (338) **The Fateley/KSU Years**; Robert M. Hammaker[1]; [1]Kansas State University

2:40 (339) **Analytical Spectroscopy: Dow-Then and Dow-Now**; J.D. Tate, Paul Chauvel, Anne Leugers, Marianne McKelvy; [1]The Dow Chemical Company

3:00 (340) **Fabrication of Substrates for SERS and TERS by Electroless Deposition**; Peter Griffiths[1], Przemyslaw Brejna[1]; [1]University of Idaho

3:40 (341) **Hadamard to Terahertz**; Jeffrey White[1], William Fateley[2]; [1]Picometrix LLC; [2]Kansas State University

Tuesday Afternoon, *Room 306B*
NEXT GENERATION SPECTROSCOPIC METHODS FOR THE ANALYSIS OF PHARMACEUTICAL SYSTEMS
Organizer and Presider: Lynne Taylor

2:00 (342) **Pharmaceutical Applications of Nanoscale Infrared Spectroscopy and Imaging**; Curtis Marcott[1], Michael Lo[2], Kevin Kjoller[2], Craig Prater[3], Bernard Van Eerdenbrugh[3], Lynne Taylor[3]; [1]Light Light Solutions; [2]Anasys Instruments; [3]Purdue University

2:20 (343) **Fast Raman Imaging Using Compressive Sampling Detection**; Dor Ben-Amotz[1], Brandon Davis[1], Amanda Hemphill[1], Derya Cebeci[1], Bradley Lucier[2]; [1]Purdue University, Dept. of Chemistry; [2]Purdue University, Dept. of Mathematics

3:00 (344) **Nonlinear Optical Imaging of Organic Crystal Nucleogenesis**; Garth Simpson, Duangporn Wanapun, Umesh Kestur, Lynne Taylor; [1]Purdue University

3:40 (345) **Applications of Two Dimensional Correlation Spectroscopy to Pharmaceutical Drug Interactions**; David Heaps[1], Alfred Rumondor[1]; [1]AstraZeneca

Tuesday Afternoon, *Room 306C*
TIP ENHANCED RAMAN SPECTROSCOPY – PUSHING THE LIMITS OF RESOLUTION
Organizer and Presider: Volker Deckert

2:00 (346) **Tips for TERS: Improving Their Efficiency and Stability**; Alexei Sokolov[1]; [1]University of Tennessee, Knoxville

2:20 (347) **Near-Field Raman Spectroscopy for Nano-Scale of Chemical Analysis of Structured Carbons**; Yuika Saito[1], Mitsuhiro Honda[1], Yoshikiyo Moriguchi[1], Kyoko Masui[1], Prabhat Verma[1], Satoshi Kawata[1]; [1]Department of applied physics, Osaka University

2:40 (348) **Optical Nano-Crystallography by Tip-Enhanced Raman Spectroscopy**; Markus Raschke[1]; [1]University of Washington

3:00 (349) **Spectroscopy of Single Semiconductor Nanowires: From Confocal Raman Microscopy to TERS**; François Lagugné-Labarthet[1]; [1]University of Western Ontario

3:20 (350) **TERS as a Diagnostic Tool in Bioanalytics**; Dana Cialla[1], René Boehme[1], Tanja Deckert-Gaudig[2], Volker Deckert[1,2], Robert Moelle[2], Juergen Popp[1,2]; [1]Friedrich-Schiller University Jena, [2]Institute of Photonic Technology

3:40 (351) **Tip-Enhanced Raman Scattering on DNA/RNA Strands**; V. Deckert[1,2];R. Treffer[1], X. Lin[1], [1]Institute of Photonic Technology, [2]Institute of Physical Chemistry

Tuesday Afternoon, *Room 307*
NANOTECHNOLOGY: APPLICATIONS TO SENSING & ENERGY
Organizers: Francis Zamborini, Jayne Garno, and Shouzhong Zou; Presider: Jayne Garno

2:00 (352) **Aerogels as Reaction Platforms – Progress and Pitfalls**; R. Lloyd Carroll[1]; [1]West Virginia University

2:20 (353) **Bottom-Up Nanofabrication of Organosilane Films for Organic Photovoltaic Applications Prepared by Particle Lithography**; Jayne C Garno, Evgueni Nesterov; [1]Louisiana State University

2:40 (354) **Vibrational Spectroscopy as Probe of Nanoscopic Environments in Polymer Electrolyte Membrane**; Carol Korzeniewski, Chang Kyu Byun; [1]Texas Tech University

3:00 (355) **Highly Efficient Nanoparticle Catalyst for Fuel Cell Applications**; Shouheng Sun[1]; [1]Brown University

3:20 (356) **Electrocatalysis on Facet-Controlled Pt-Alloy Nanocrystals**; Shouzhong Zou[1], Hongzhou Yang[1]; [1]Miami University

3:40 (357) **Depolarized Scattering from Silver Nanoparticles for Analytical Applications**; John Heckel[1], George Chumanov[1]; [1]Clemson University

4:00 pm – Poster Viewing and Break, *Exhibit Hall*

4:20 pm Plenary, *Ballroom A*
Celebration of 30 Years of ICP-MS, **Robert S. Houk**, *Iowa State*

TECHNICAL PROGRAM – WEDNESDAY

Plenary Lectures

Morning Presider: Pavel Matousek; Afternoon Presider: André Sommer

Applied Spectroscopy William F. Meggers Award

8:00 am Plenary, *Ballroom A*

Paul Gemperline

(359) **Kinetic Modeling of Multivariate Spectroscopic Images**; Paul Gemperline[1], Patrick Cutler[2], David Haaland[3], Erik Andries[2]; [1]East Carolina University; [2]University of New Mexico; [3]Sandia National Laboratories

Refer to page 15 for biographical information

Applied Spectroscopy Lester W. Strock Award

8:30 am Plenary, *Ballroom A*

Kay Niemax

(360) **Chemical Characterization of Micro- and Nanoparticles by ICP Spectrometry**; Kay Niemax[1], Sebastian Groh[2], Ayrat Murtazin[1], Carmen C. Garcia[3]; [1]BAM Berlin; [2]ISAS Dortmund; [3]Institute for Transuranium Elements

Refer to page 14 for biographical information

FACSS Award Presentations and Plenary Lecture

4:30 pm, *Room 305A*

Alexander Scheeline

(532) **Distinguished Service: Becoming an Oxymoron?**; Alexander Scheeline[1]; [1]University of Illinois at CU

Refer to page 10 for biographical information

WEDNESDAY POSTER SESSION

9:00 – 10:30 am

Exhibit Hall – Ballroom B

All Wednesday posters should be put up between 7:30 – 8:00 am and removed by 5:00 pm. Odd numbered poster boards present between 9:00 – 9:45 am. Even numbered poster boards present between 9:45 – 10:30 am.

Bioanalytical

Board #

1 (361) **Transparent Magnetic Photoresists for MEMS and BioMEMS Applications**; Philip Gach[1], Christopher Sims[1], Nancy Allbritton[1,2]; [1]University of North Carolina at Chapel Hill; [2]North Carolina State University

2 (362) **Isolation and Identification of Potential Chemical Attractants from Rudbeckia Inflorescences**; Patricia L. Lang[1], Ashley N. Simpson[1], Gary N. Dodson[1]; [1]Ball State University

3 (363) **The Exploration of Selenopeptides in Cerebral Spinal Fluid Using Complementary SEC-ICPMS and LC-MALDI for Elemental and Molecular Identification**; Renee N. Easter[1], Karolin K. Kroening[1], Gail Pyne-Gaithman[2], Joseph A. Caruso[1]; [1]University of Cincinnati/Agilent Technologies Meta; [2]Department of Neurology, University of Cincinnati

4 (364) **Investigation of Capacitance Effects on Liposomes Containing pH Gradients.**; Josemar Castillo[1]; [1]Arizona State University

5 (365) **ATR Imaging of Engineered Cartilage Constructs**; Somaieh Moghadam[1], David Reiter[2], Onyinyechi Irrechukwu[2], Ping-Chang Lin[2], Richard Spencer[2], Nancy Pleshko[1]; [1]Temple University, College if Engineering; [2]National Institues of Health

6 (366) **Peroxidase Activated Nanoprobe for SERS Imaging**; Hsiangkuo Yuan[1], Benoit Lauly[1], Christopher Khoury[1], Jonathan Scaffidi[1], Hsin-neng Wang[1], Catherine Ibarra[1], Tuan Vodihn[1]; [1]Duke University

7 (367) **Development of Spectral Markers Using Infrared Imaging to Access Cardiac Remodeling**; Naomi D\'souza[1], Nancy Pleshko[1]; [1]Temple University

8 (368) **Formulation and Confirmation of Diisobutylphthalate in Rodent Feed Mixes in Support of Developmental and Reproductive Toxicology Studies.**; Shaun Norton[1], Jennifer Gilliam[1], Gwendolyn McNeill[1], Donna Browning[1], James Blake[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

9 (369) **A Lab-on-a-Chip FRET Biosensor for Pharmaceutical Applications**; Annadele Herman[1], Hamed Shadpour[1], Jon Zawistowski[2], Klaus Hahn[2], Nancy Allbritton[1,2,3]; [1]Dept. of Chemistry, UNC, Chapel Hill; [2]Dept. of Pharmacology, UNC, Chapel Hill; [3]Dept. of Biomedical Eng., UNC-NCSU

Environmental

10 (370) **Development of a FRET-Peptide Sensor for Trace and Ultratrace Metal Detection**; Shelly Casciato[1], James Holcombe[1]; [1]The University of Texas at Austin

11 (371) **Phytoremedation of Metals in Soils by ICP-OES**; Joseph Sneddon[1], Carey Hardaway[1], Venkate Salla[1]; [1]McNeese State University

12 (372) **Application of Dendrimers for Water Purification**; Priyanka Bhattacharya[1], Pengyu Chen[1], Seung Ha Kim[2], Monica H. Lamm[2], Pu Chun Ke[1]; [1]Clemson University; [2]Iowa State University

13 (373) **Environmental Studies of Metals by ICP-OES in Southwest Louisiana**; Caray Hardaway[1], Joseph Sneddon[1], Shilpa Vootla[1], Venkatesula Salla[1]; [1]McNeese State University

14 (374) **A Comparison of Methods for Analysis of Dispersed Oil in Water and Soil by FT-IR Spectrometry**; Ben Perston[1], Aniruddha Pisal[1], Dean Brown[1]; [1]PerkinElmer

TECHNICAL PROGRAM – WEDNESDAY
Posters 9:00 – 10:30 am

Board #

15 (375) **Evaluation of Laboratory Productivity for Environmental Applications**; Laura Thompson[1], Paul Krampitz[1], Stan Smith[1], Praveen Sarojam[1], Zoe Grosser[1]; [1]PerkinElmer, Inc.

16 (376) **Parallel Extraction of Oxytetracycline and Flumequine from Fish Food and Its Determination by Derivative Spectrophotometry**; M. Ines Toral[1], Jairo Zuniga[1], Sandra Orellana[1], Cesar Soto[2]; [1]Faculty of Science. University of Chile; [2]University of Concepcion

17 (377) **Levels of Sulphur Dioxid and the Correlation to the Total Suspended Particulate Matter in Polluted Air Samples**; EL Mukhtar Belgasem, Ramadan Damja; [1]al-Fateh University

18 (378) **Analysis of Suspended Particulates for Their Trace Element Contentes**; Ramadan Damja, EL Mukhtar Belgasem; [1]Al-Fateh University

General Analytical
(Education, Electrochemistry, Forensic, Instrumentation, Laser Ablation, Materials Characterization, Other, Pharmaceutical, Process/Control, Proteomics, Surface Characterization

19 (379) **Photon Trapping Spectroscopy: Prototype Optimization and Application to Air Monitoring**; John Frost[1], Joseph Aldstadt[1], Peter Geissinger[1], Jorg Woehl[1]; [1]University of Wisconsin Milwaukee

20 (380) **Detection of Diethylene Glycol Impurity in Propylene Glycol by Near-Infrared Spectroscopy: Method Development and Validation**; Xiang Li[1,2], Sergey Arzhantsev[2], John Kauffman[2], Benjamin Westenberger[2], Lucinda Buhse[2], John Spencer[2]; [1]Penn State University; [2]FDA-Division of Pharmaceutical Analysis

21 (381) **Forensics Applications of Vibrational Spectroscopy and X-Ray Fluorescence to Nail Polishes and Their Signatures**; Dale L Perry[2], Shinobu T Heier[1]; [1]ThermoFisher Scientific; [2]Lawrence Berkeley National Laboratory

22 (382) **Signatures of Inorganic Materials by Use of Multiple Spectroscopic Approaches**; Dale L Perry[1]; [1]Lawrence Berkeley National Laboratory

23 (383) **Tablet Identification Using an FT-Near-IR Integrating Sphere with Principal Component Analysis**; Frank Weston[1]; [1]Varian, Inc

24 (384) **Module for the Measurement of Photoluminescence in NIR and MIR Spectral Ranges**; Sergey Shilov[1], Michael Joerger[1]; [1]Bruker Optics

25 (385) **Determination of Derivatized Compounds in a Supersonic Jet**; Steven Goates[1], Lindsey Mills[1], Amy Felsted[1], Andrew Orton[1]; [1]Brigham Young University

26 (386) **Analysis of Composite Interphase Degradation**; Christopher N. Young[1], Clive R. Clayton[1], Richard D. Granata[2]; [1]Stony Brook Univ, Dept of Materials Science; [2]Florida Atlantic Univ, Dept Ocean Eng.

Microfluidics/Nanotechnology

27 (387) **Vesicle-Based Tools for Single Cell Analysis**; Michelle L. Kovarik[1], K. Scott Phillips[1], Hsuan-Hong Lai[1], Nancy L. Allbritton[1]; [1]University of North Carolina - Chapel Hill

28 (388) **Trapping of Proteins by Insulator-Based Dielectrophoresis**; Asuka Nakano[1], Sanchari Bhattacharya[1], Tzu-Chiao Chao[1], Alexandra Ros[1]; [1]Arizona State University

Board #

29 (389) **Dielectrophoretic Single Cell Trapping in a Microfluidic Lab-on-Chip Device**; Sanchari Bhattacharya[1], Tzu-Chiao Chao[1], Prof. Alexandra Ros[1]; [1]Arizona State University

30 (390) **Internally-Etched Silica Encapsulated Gold Coated Silver Nanostructures for Improved SERS Detection**; Sudip Nath[1], Binaya Shrestha[1], Amanda Haes[1]; [1]The University of Iowa

31 (391) **Use of Sonication Power to Control Length Distributions of SWNTs in Aqueous Suspensions Used for Network Deposition**; Meagan A. Cauble[1], Pornnipa Vichchulada[1], Jihye Shim[2]; [1]University of Georgia; [2]Kyung Hee University, Korea

32 (392) **Quantitative Study of Ligand Adsorption onto Metal Nanoparticle Using Surface Enhanced Raman Internal Reference Method**; Siyam Ansar[1], Dongmao Zhang[1]; [1]Mississippi State University

33 (393) **Whispering Gallery Resonating Nanoparticles**; Zachary Koontz[1], George Chumanov[1]; [1]Clemson University

34 (394) **Time-Resolved Spectroscopy of Plasmon-Enhanced Luminescence from Rare-Earth Ions**; Jaetae Seo[1], Maria Veronica Rigo[1]; [1]Hampton University

35 (395) **Ambient Measurements of Charge Transport with Designed Surface Structures of cobaltacarborane Porphyrins Using Conductive Probe Atomic Force Microscopy**; Venetia D. Lyles, Wilson K. Serem, Erhong Hao, M. Graca H. Vicente, Jayne C. Garno; [1]Louisiana State University

36 (396) **Controlling the Surface Density of Organosilane Nanostructures: Particle Lithography Strategies for Preparing Nanostructures with Well-Defined Periodicity and Geometries**; ChaMarra K. Saner, Kathie L. Lusker, Zorabel M. LeJeune, Jayne C. Garno; [1]Louisiana State University

37 (397) **Plasmonic Nanometals and Semiconductor Nanocrystals for the Fabrication of Hybrid Optical Material Structure**; Maria Veronica Rigo[1], Jaetae Seo[1]; [1]Hampton University

38 (398) **Detection of SMC1/SMC3 Protein Binding to DNA via Quartz Crystal Microbalance**; Laura Steller[1], Rolf Jessberger[2], Hagen Schmidt[1], Magdalena Laugsch[2], Electra Gizeli[3]; [1]IFW, Dresden, Germany; [2]Inst. of Physiol. Chem., Dresden; [3]Biosensors Lab, Heraklion, Greece

39 (398a**) Synthesis and Application of Multishell Silver Core Nanoparticles**; Whitney Snyder[1], George Chumanov[1]; [1]Clemson University

Spectroscopy
(Absorption, Atomic Spectroscopy, Fluorescence, ICP, Infrared, Laser Spectroscopy, Mass Spectrometry, Molecular Spectroscopy, Near Infrared, NMR, Raman, Surface Enhanced Raman, Surface Plasmon Resonance)

40 (399) **Advances in Structural Characterization and High Spatial Resolution Imaging of Lipid Species with Ion Mobility Mass Spectrometry**; Michal Kliman[1], Jay G. Forsythe[2], John A. McLean[3]; [1]Department of Chemistry, Vanderbilt University

Board #

41 (400) **Quantitative Measure of Soybean Protein and Lipid Content Using Transmission Raman Spectroscopy**; Matthew Schulmerich[1], Michael Asensio[1]; [1]The Beckman Institute; [2]The National Soybean Research Laboratory; [3]Illinois Crop Improvement Association

42 (401) **X-Ray and UV Excited Luminescence of Doped Y2O3 Nanocrystals**; Yan Zhang[1], CV Gopal Reddy[1], Tuan Vo-Dinh[1]; [1]Duke University

43 (402) **Modification and Characterization of a Commercial FT-IR Accessory for Surface Plasmon Resonance Spectroscopy**; Nicola Menegazzo[1], Laurel Kegel[1], Karl Booksh[1]; [1]University of Delaware

44 (403) **Standoff Resonance Enhanced Multiphoton Ionization (REMPI) for Detection of Hazardous Materials.**; Maria Damian[1], Janna Register[1], S. Michael Angel[1]; [1]University of South Carolina

45 (404) **Use of a Handheld NIR Spectrometer to Assess Agricultural Materials**; David Himmelsbach; [1]Light Light Solutions, LLC

46 (405) **Spectral Imaging of Coupled Localized and Propagating Surface Plasmon Resonance in Nanohole Arrays**; Laurel Kegel[1], Karl Booksh[1]; [1]University of Delaware

47 (406) **Physicochemical Determination of the Role of Methylene Chloride and Phenol in Paint Strippers**; Christopher Young[1], Kelly Watson[2], James Yesinowski[3], Clive Clayton[1], James Wynne[3], Young Han[4]; [1]Stony Brook University; [2]Science Applications International Corp.; [3]Naval Research Laboratory; [4]Naval Air Systems Command

48 (407) **Raman Spectroscopy of Supported Lipid Bilayers**; Zhorro Nickolov[1], Selver Ahmed[2], Stephanie Wunder[2]; [1]Centralized Research Facility, Drexel University; [2]Dept of Chemistry, Temple University

49 (408) **Lipid Analysis Using Nanostructure-Initiator Mass Spectrometry**; Jay Forsythe[1], Michal Kliman[1], Joshua Broussard[1], Donna Webb[1], John McLean[1]; [1]Vanderbilt University

50 (409) **Infrared Spectral Imaging Analysis of Cartilage Repair Tissue Over Time**; Madhuri Penmatsa[1], Paul West[2], Xu Yang[2], Nancy Pleshko[1]; [1]Temple University, PA; [2]Hospital for Special Surgery, NY

51 (410) **Coating Effects on Fabrid Infrared Reflectance Spectra**; Megan Baranowski[1], Heather Brooke[1], Jessica McCutcheon[1], Stephen Morgan[1], Michael Myrick[1]; [1]Univeristy of South Carolina

52 (411) **Mass Spectrometry-Based Analysis of the Lung Proteome of Mice Infected with Aspergillus fumigatus**; Chengsi Huang[1], Jason McCarthy[2], Marta Feldmesser[2], Vicki H. Wysocki[1]; [1]University of Arizona; [2]Albert Einstein College of Medicine

53 (412) **Frequencies and Absorption Intensities of Fundamentals and Overtones of NH Stretching Vibrations of Pyrrole and Pyrrole**; Yukihiro Ozaki[1], Yoshisuke Futami[1], Yasushi Ozaki[2], Yoshiaki Hamada[3], Marek Wojcik[4]; [1]Kwansei-Gakuin University; [2]Josai University; [3]The Open University of Japan; [4]Jagiellonian University

Board #

54 (413) **Microsecond Time-Resolved Desorption Electrospray Ionization Mass Spectrometry**; Zhixin Miao[1,2,3], Hao Chen[1,2,3]; [1]Department of Chemistry and Biochemistry; [2]Center for Intelligent Chemical Instrume; [3]Ohio University

55 (414) **Advancing Calibration Strategies For Plasma Spectrometry: Different Operation Modes Of A Dual Drop-On-Demand Aerosol Generator For Micro-Volume Sample Introduction**; J. Niklas Schaper[1], Jan Massmann[1], Jan H. Petersen[1], Nicolas H. Bings[1]; [1]University of Mainz, Analytical Chemistry

56 (415) **FAIMS as a Filter for Biomarker Detection**; Charles Harrison[1], Alessandra Ferzoco[2], Mark Ridgeway[2], Desmond Kaplan[1], Kevin Dixon[1], Melvin Park[1], Gary Glish[2]; [1]Bruker Daltonics; [2]The University of North Carolina

57 (416) **Improving the Accuracy of Calculated VCD Spectra Using Multilayer Computational Analysis**; Douglas J. Minick[1], Dean P. Phelps[1], Randy D. Rutkowske[1], Luke A. D. Miller[1]; [1]GlaxoSmithKline

58 (417) **Shock Behavior and Analyte Transport in the ICP-MS via the Direct Simulation Monte Carlo Algorithm**; Ross Spencer[1], Steven Schmidt[1], Paul Farnsworth[1]; [1]Brigham Young University

59 (418) **The Potential of Autofluorescence Spectroscopy to Detect Human Urinary Tract Infection**; Unnikrishnan Kuzhiumparambil[1], Sandeep Menon Perinchery[1], Subramanyam Vemulpad[1], Ewa M. Goldys[1]; [1]Macquarie University

Wednesday Morning, *Room 301B*
PROBING INTERACTIONS BETWEEN BIOMOLECULES AND NANOMATERIALS
Organizer and Presider: Wenwan Zhong

10:30 (419) **Engineered Nanoparticle - Biological Interactions and Their Role**; Kevin Dreher[1]; [1]EPA

11:10 (420) **DNA-Carbon Nanotube Interaction: Fundamentals and Applications**; Ming Zheng; [1]National Institute of Standards and Technology

11:30 (421) **Immunological Properties of Engineered Nanoparticles**; Marina Dobrovolskaia; [1]SAIC-Federick Inc

11:50 (422) **Measuring Affinity between Proteins and Nanoparticles Using Capillary Electrophoresis**; Wenwan Zhong[1], Ni Li[1], Shang Zeng[1]; [1]University of California, Riverside

12:10 (423) **Characterization and Detection of Non-Covalent Binding of Single-Stranded Oligonucleotides to Single-Walled Carbon Nanotubes**; Meagan A. Cauble[1]; [1]University of Georgia

Wednesday Morning, *Room 302A*
NEAR-IR SPECTROSCOPY: APPLICATIONS AND CALIBRATIONS
Organizer and Presider: J. Clay Harris

10:30 (424) **Near-IR Spectroscopy of Clay Minerals and Amorphous Silicates: Understanding Aqueous Alteration Environments on Mars**; Elizabeth Rampe[1], Nina Lanza[2], Thomas Sharp[1]; [1]Arizona State University; [2]University of New Mexico

10:50 (425) **The Role of Near-Infrared in Pharmaceutical Counterfeit Prevention**; Frederick Haibach[1]; [1]Polychromix, Inc

11:10 (426) **Near- Versus Mid-Infrared Diffuse Reflectance Spectroscopic Analysis of Compostable Utensils and Biochars: Results Can Be Surprising**; James B. Reeves III[1], Walter W. Mulbry III[1], Heekwon Ahn[1]; [1]EMBUL, ARS, USDA

11:30 (427) **NIR Instrument Calibration and Quality Control Testing Via a Center of Gravity Algorithm**; Mark Henson[1], Kevin Judge[1]; [1]Molecular Biometrics, Inc.

11:50 (428) **Development of a Robust Calibration Model for Monitoring Alcoholic Fermentation Process by Using Near-Infrared and Infrared Dual-Wavelength System**; Takuma Genkawa[1], Masahiro Watari[2], Mitsue Satou[2], Mikiko Konta[1], Yukihiro Ozaki[1]; [1]Kwansei Gakuin University; [2]Yokogawa Electric Corporation

12:10 (429) **What Color is Your Yellow Cake? Vis/NIR Spectroscopic Analysis of Uranium Ore Concentrate Samples**; Greg Klunder[1], Paul Spackman[1], Pat Grant[1], Martin Robel[1], Lars Borg[1], Rachel Lindvall[1], Ian Hutcheon[1]; [1]Lawrence Livermore National Laboratory

Wednesday Morning, *Room 302B*
VIBRATIONAL SPECTROSCOPY AT WORK IN THE PHARMACEUTICAL INDUSTRY
Organizer and Presider: Linda Kidder

10:30 (430) **Raman, NIR and FT-IR Spectroscopic Quality Control by Hand-Held Instrumentation**; Heinz W. Siesler[1]; [1]University of Duisburg-Essen

10:50 (431) **Exploring the Limitations of Spectroscopic Techniques for Tablet Analysis**; Kevin Macias[1], John Bobiak[1], Dongsheng Bu[1], Gary McGeorge[1]; [1]Bristol-Myers Squibb Co

11:10 (432) **The Spectroscopic Options for Real Time Tablet Content Uniformity: A Comparison Study of NIR Reflectance, NIR Transmission and Raman Transmission**; Yang Liu[1], Stephanie Dolph[1]; [1]Pfizer Global Research and Development

11:30 (433) **Multivariate Image Analysis of NIR Chemical Images of Polymeric Gel Strips**; Rodolfo J Romanach[1], Jackeline Jerez Rozo[1], Jose M Prats Montalban[2], Alberto Ferrer[2]; [1]Univ.Puerto Rico _Mayaguez Campus; [2]Polytechnic University of Valencia

11:50 (434) **A Comprehensive Exploration of Imaging Technologies for Pharmaceutical Drug Development**; Pascal Chalus[1]; [1]F-Hoffman-La Roche AG., PTDFA, Basle, Switzerland

12:10 (435) **NIR Spectral and Imaging Analysis for Support of a Pharmaceutical Manufacturing Process**; Boyong Wan[1], Kevin Macias[1], Gary McGeorge[1], Douglas Both[1]; [1]Analytical R&D, Bristol-Myers Squibb Co

Wednesday Morning, *Room 304*
LESTER STROCK AWARD SYMPOSIUM – ATOMIC SPECTROSCOPY PROGRESS REPORTS
Organizer: Kay Niemax; Presider: David J. Butcher

10:30 (436) **On the Usefulness of the Spectral Fluctuation Approach in Laser Induced Plasma Spectroscopy**; Nicolo Omenetto, Heh-Young Moon, Daniel Shelby, Jonathan Merten, Benjamin Smith; [1]University of Florida

10:50 (437) **Imaging the Ion Beam in the Second Vacuum Stage of an Inductively Coupled Plasma Mass Spectrometer**; Paul Farnsworth[1], Nicholas Taylor[1]; [1]Brigham Young University

11:10 (438) **Laser Ablation Based Chemical Analysis: Macroscale, Nanoscale, Fundamentals and Commercialization**; Richard E. Russo[1,2], Vassilia Zorba[1], Xianglei Mao[1], Jhanis Gonzalez[1,2], Jong Yoo[2]; [1]Lawrence Berkeley National Laboratory; [2]Applied Spectra Inc

11:30 (439) **LA-ICP-MS: A Status Report**; Joachim Koch[1]; [1]Laboratory of Inorganic Chemistry, ETH Zurich

11:50 (440) **Time-Resolved Measurements of Ions Produced from Individual Droplets and Particles in Plasmas**; John Olesik[1], Patrick Gray[1], Josh Dettman[1]; [1]Ohio State University

12:10 (441) **Field-Flow Fractionation Inductively Coupled Plasma Spectrometry: Status Report 2010**; Ramon Barnes[1], Atitaya Siripinyanond[2], Supharart Sangsawong[2], Juwadee Shiowatana[2]; [1]ICP Information Newsletter Inc; [2]Mahidol University

Wednesday Morning, *Room 305A*
BIOANALYTICAL SEPARATION SCIENCE
Organizer and Presider: Neil D. Danielson

10:30 (442) **Liquid Chromatography of Biomolecules using an Alkylammonium Formate Ionic Liquid Mobile Phase**; Neil Danielson[1], Matthew Collins[1], Shau Grossman[1]; [1]Miami University

10:50 (443) **CE-LIF Studies to Facilitate Bioprobe Design and Microbe Detection**; Christa Colyer, Xiuli Lin, Tara Massie, Stephanie Rockett, Jennifer Kneezel; [1]Wake Forest University

11:10 (444) **Micellar Electrokinetic Chromatography Coupled to Atmospheric Pressure Photoionization Mass Spectrometry for Analysis of Chiral Benzoin Derivatives Using Mixed Molecular Micelles**; Shahab Shamsi[1], Jun He[1]; [1]Georgia State University

11:30 (445) **Bioseparations Using Self-Assembled Phospholipids**; Lisa Holland[1], Stephanie Archer-Hartmann[1], Ted Langan[1]; [1]West Virginia University

11:50 (446) **Incorporation of Guanosine Compounds into Sieving Gels for DNA Separations in Capillary Gel Electrophoresis**; Linda McGown[1], Yingying Dong[1]; [1]Rensselaer Polytechnic Institute

12:10 (447) **Monitoring Anti-Cancer Drug Metabolism with Capillary Electrophoresis**; Amanda Jones[1], Varuni Subramaniam[1], Amanda Haes[1]; [1]The University of Iowa

Wednesday Morning, *Room 305B*
OPTICAL EFFECTS IN INFRARED SPECTROSCOPIC IMAGING
Organizer and Presider: Rohit Bhargava

10:30 (448) **Comparison of Infrared and Raman Spectral Imaging to Study Stem Cell Differentiation**; Max Diem[1]; [1]Northeastern University

10:50 (449) **FTIR Can Detect DNA Conformational Changes in Cells and Tissue in Response to Hydration: Implications for Disease Diagnosis**; Bayden Wood[1], Donna Whelan[1], Keith Bambery[1], Don McNaughton; [1]Monash University

11:10 (450) **Infrared Microscopy of Cells and Tissue: Disentangling Scattering from Absorption**; Peter Gardner[1], Paul Bassan[1], Achim Kohler[2]; [1]University of Manchester; [2]Nofima Mat

11:30 (451) **IR Reflectance Microspectroscopy of Particles on a Mirrored Surface: Tools for Estimating Absorbance and Optical Properties**; Michael Myrick[1], Heather Brooke[2], Burt Bronk[3]; [1]University of South Carolina; [2]Naval Research Laboratory; [3]Army Research Laboratory

11:50 (452) **An Empirical Study to Understand Optical Anomalies in Infrared Microspectroscopy: A Step Forward in Disease Detection**; Heather Gulley-Stahl[1], Andre Sommer[2]; [1]Lexmark International; [2]Molecular Microspectroscopy Laboratory

12:10 (453) **Modeling Distortions in Infrared Spectroscopic Imaging**; Rohit Bhargava[1], Paul Carney[1], Rohith Reddy[1], Brynmor Davis[1], Anil Kodali[1]; [1]University of Illinois at Urbana-Champaign

Wednesday Morning, *Room 306A*
TWO-DIMENSIONAL CORRELATION SPECTROSCOPY
Organizer and Presider: Isao Noda

10:30 (454) **Projection Two-Dimensional Correlation Spectroscopy**; Isao Noda[1]; [1]The Procter and Gamble Co

10:50 (455) **Two-Dimensional Correlation Spectroscopy of Poly(3-hydroxyalkanoate)s in Terahertz Frequency Region**; Hiromichi Hoshina[1], Yusuke Morisawa[2], Harumi Sato[2], Isao Noda[3], Yukihiro Ozaki[2], Chikoi Otani[1]; [1]RIKEN Advanced Science Institute; [2]Kwansei Gakuin University; [3]The Procter & Gamble Company

11:10 (456) **2D Correlation Raman Spectroscopy for Kinetic Studies of Polypeptide Folding and Aggregation**; Igor Lednev[1], Vitali Sikirzhytski[1], Natalya Topilina[1], Seiichiro Higashiya[1], John Welch[1]; [1]University at Albany, SUNY

11:30 (457) **2D-COS Applications of Vibrational Optical Activity in Proteins**; Laurence A Nafie[1, 3], Soo Ryeon Ryu[2], Young Mee Jung[2], Rina K Dukor[3]; [1]Syracuse University; [2]Kangwon University; [3]BioTools, Inc

11:50 (458) **Self-Modeling Mixture Analysis in Combination with Correlation Spectroscopy**; Willem Windig; [1]Eigenvector Research, Inc

12:10 (459) **Two-Dimensional Correlation Spectroscopic Analysis of Concentration-Dependent Solvent-Solvent Interactions**; Heinz W. Siesler[1]; [1]University of Duisburg-Essen

Wednesday Morning, *Room 306B*
SUSTAINABILITY AND PAT APPLICATIONS
Organizer and Presider: J. D. Tate

10:30 (460) **Advanced Combustion Diagnostics for Large Scale Fired-Equipment**; J.D. Tate[1], Linh Le[1], Trevor Knittel[2], Jie Zhu[2], Alan Cowie[2]; [1]The Dow Chemical Company; [2]Yokogawa Corporation of America

10:50 (461) **Combustion Diagnostics Using *in-situ* Tunable Diode Laser Spectroscopy**; Alan Cowie[1], Jie Zhu[1]; [1]Yokogawa Corporation of America

11:10 (462) **Ambient Air Monitoring Applications for Fluorocarbon Production Units**; Troy Francisco[1]; [1]DuPont

11:30 (463) **PAT an Enabler of Sustainability**; Darryl Ertl, Sean Sisk, Bob Cooley, Charlie Goss, Susan Barnes, Greg Gervasio; [1]GlaxoSmithKline

11:50 (464) **Green PAT in Pfizer Manufacturing Plants**; Hiwot Isaac[1], Bronwyn Grout[1]; [1]Pfizer Inc

12:10 (465) **Temporary On-line Spectroscopic Analysis for Process Troubleshooting**; Serena Stephenson[1], Wendy Flory[1], Lamar Dewald[1], Roger Gagnon[1]; [1]Dow Chemical Company

Wednesday Morning, *Room 306C*
NEW SERS ARCHITECTURE
Organizers: Duncan Graham, Karen Faulds, Michael Natan;
Presider: Duncan Graham

10:30 (466) **Nanoparticle Arrays Tunable in Size and Gap Distance for SERS Detection of Explosives**; Jean-Francois Masson[1]; [1]Université de Montréal

10:50 (467) **Controlled Formation of SERS Hot Spots in Gold Nanoclusters.**; Rene Lopez[1], Kristen Alexander[1]; [1]University of North Carlinat at Chapel Hill

11:10 (468) **Rapid, Sensitive TNT Detection Using SERS Nanotags**; Michael Natan[1], Brad Brown[1], Becky Golightly[1]; [1]Oxonica Materials Inc

11:30 (469) **SERS Multilayer Substrate Optimization Using SAM Dielectric Spacers**; Charles Klutse[1], Brian Cullum[1]; [1]University of Maryland, Baltimore County

11:50 (470) **Optimization of Strain Promoted Azide-Alkyne Cycloadition for the Development of Glycan Microarray Technology via Surface Enhanced Raman Spectroscopy**; Sharon Martin[1], Richard Dluhy[1], Jun Guo[2], Gert Jan Boons[2], Yiping Zhao[3]; [1]UGA Department of Chemistry; [2]UGA Complex Carbohydrate Research Center; [3]UGA Department of Physics and Astronomy

12:10 (471) **Nanostructure Junctions for High Sensitivity Raman Detection and Imaging**; Zachary Schultz, University of Notre Dame

Wednesday Morning, *Room 307*
TERAHERTZ SPECTROSCOPY
Organizer: Gilbert Pacey; Presider: James Gord

10:30 (472) **Terahertz Measurements of Nonwoven Products**; Jeffrey White[1], John Riccardi[1], Irl Duling[1], David Zimdars[1], Greg Fichter[1]; [1]Picometrix LLC

11:10 (473) **Nondestructive Evaluation of Sol-Gels Using Terahertz Time-Domain Reflectance Spectroscopy to Sol-Gel Aging**; Gilbert Pacey[1], Anita Taulbee-Combs[2], James Cox[1]; [1]Miami University; [2]University of Dayton Research Institute

11:50 (474) **High-Speed Terahertz Imaging**; Jeffrey White[1], Irl Duling[1], David Zimdars[1], Greg Fichter[1], John Duquette[1], Chris Megdanoff[1]; [1]Picometrix LLC

12:30 pm – FACSS Planning Meeting for Conferees and Exhibitors, *location TBD*

1:30 pm – Poster Viewing and Dessert Break, *Exhibit Hall*

Wednesday Afternoon, *Room 301B*
NANOMATERIALS FOR SURFACE PLASMON RESONANCE
Organizer: Jean-Francois Masson; Presider: Karl Booksh

2:30 (475) **Nonplasmonic Nanoarrays in Laser Assisted Desorption and Ionization Mass Spectrometric Imaging**; Lin He[1]; [1]North Carolina State University

2:50 (476) **Flow-Through Plasmonic Nanosensors Using Embedded Annular Nanoband Electrodes**; Paul Bohn[1], Sean Branagan[1]; [1]University of Notre Dame

3:10 (477) **Nanoholes on Metals and Their Application in Bioanalytical Chemistry**; Alexandre Broloa[1]; [1]University of Victoria

3:30 (478) **Sensing Properties of Au, Ag, and Au on Ag Nanohole Arrays**; Jean-Francois Masson[1], Marie-Pier Murray-Méthot[1], Mathieu Ratel[1]; [1]Université de Montréal

3:50 (479) **Plasmonics for SERS Sensing: Platform Design, Fabrication and Applications**; Tuan Vo-Dinh[1]; [1]Duke University

4:10 (480) **Detecting Vitamin D with Localized Surface Plasmon Resonance Sensors**; Amanda Haes[1]; [1]University of Iowa

Wednesday Afternoon, *Room 302A*
NOVEL SAMPLE INTRODUCTION METHODS FOR ICP-MS AND ICP-IES
Organizer and Presider: Doug Duckworth

2:30 (481) **Low Flow Small Sample MC-ICP-MS Analysis of U and Pu Using Electrochemically Modulated Separations.**; M. Liezers[1], D.C Duckworth[1], G.C. Eiden[1], G. Gill[1], M.S. Good[1], G.J. Posakony[1], B.E. Watson[1]; [1]Pacific Northwest National Laboratory

2:50 (482) **A New System for Automated Analysis of Micro Volume Liquid Samples by ICPMS**; Daniel Wiederin[1], Kyle Uhlmeyer[1], Cory Gross[1], Patrick Sullivan[1], Nathan Saetveit[1]; [1]Elemental Scientific

3:10 (483) **Heat-Assisted Argon Electrospray Interface for Low-Flow Rate Liquid Sample Introduction in Plasma Spectrometry**; Ryan Brennan[1], Savelas Rabb[1], Michael Winchester[1], Gregory Turk[1]; [1]National Institute of Standards and Technology

3:30 (484) **Potenital of a Novel Low-Flow Drop-On-Demand Aerosol Generator for Plasma Spectrochemical and Speciation Analysis**; J. Niklas Schaper[1], Jan Maßmann[1], Jan H. Petersen[1], Nicolas H. Bings[1]; [1]University of Mainz, Analytical Chemistry

3:50 (485) **Development of a New Torch/Fastening Design for Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Low Argon Consumption**; Wolfgang Buscher[1,2], Thorben Pfeifer[1], Michael Sperling[1,2], Rasmus Janzen[1]; [1]Analytical Chemistry, University of Muenster; [2]EVISA; VI for Speciation Analysis

4:10 (486) **Interfacing Field Flow Fractionation Techniques to an ICP–MS for the Characterization of Naturally Occurring Particles and Engineered Nano-Particles**; Trevor Havard[1], Soheyl Tajiki[1], Thorsten Klein[1,2]; [1]Postnova Analytics USA; [2]Postnova Analytics Germany

Wednesday Afternoon, *Room 302B*
FUNDAMENTAL STUDIES AND EXCITING NEW APPLICATIONS OF GLOW DISCHARGE SPECTROSCOPY
Organizer and Presider: Jorge Pisonero Castro

2:30 (487) **Fundamental Properties of Non Equilibrium Radiofrequency Plasmas Used for Material Analysis**; Philippe Belenguer[1], Philippe Guillot[2], Thomas Nelis[2], Abdelattif Zahri[1], Laurent Therese[2]; [1]LAPLACE, CNRS, Toulouse; [2]DPHE, CUFR

2:50 (488) **Spatial Distribution and Temporal Dependence of the Optical Emission in a Pulsed Radiofrequency Glow Discharge**; Nerea Bordel[1], Rebeca Valledor[1], Jorge Pisonero[1], Thomas Nelis[2], Alfredo Sanz-Medel[1]; [1]University of Oviedo, Spain; [2]CFUR J. F. Champollion, Albi. France

3:10 (489) **Characterisation of Nanostructured Materials by Plasma Profiling Ion Mass Spectrometry**; Agnès Tempez[1], Lara Lobo[2], Abdelhak Bensaoula[3], Nunzio Tuccitto[4], Jorge Pisonero[2], Antonino Licciardello[4], Chris Boney[3], Nacer Badi[3], Nerea Bordel[2], Patrick Chapon[1]; [1]Horiba Jobin Yvon; [2]University of Oviedo; [3]University of Houston; [4]University of Catania

3:30 (490) **Making Lemonade from Lemons: Using Metal Oxide Ions in GDMS for Materials Speciation**; Fred King[1], Jennifer Robertson-Honecker[1], Na Zhang[1], Megan DeJesus[1], Guodong Gu[1]; [1]West Virginia University

3:50 (491) **Distance-of-Flight Mass Spectrometry: A New Instrumental Concept for Elemental Mass Spectrometry**; Alexander W.G. Graham[1], Steven J. Ray[1], Christie G. Enke[2], Charles J. Barinaga[3], David W. Koppenaal[3], Gary M. Hieftje[1]; [1]Department of Chemistry, Indiana University, Bl; [2]Department of Chemistry, New Mexico U; [3]Pacific Northwest National Laboratory

4:10 (492) **Particle Beam/Hollow Cathode Optical Emission Spectroscopy (PB/HC-OES) as a Tool for the Study of Metal Binding of Proteins**; R. Kenneth Marcus[1], C. Derrick Quarles, Jr.[1]; [1]Clemson University

Wednesday Afternoon, *Room 302C*
ADVANCES IN MASS SPECTROMETRY INSTRUMENTATION
Organizers: John A. McLean and Jody C. May;
Presider: Jody C. May

2:30 (493) **The Analytical Capabilities of FAIMS: Back to the Basics and More**; Gary Glish[1], Mark Ridgeway[1], Alessandra Ferzoco[1], Alice Pilo[1], Desmond Kaplan[2], Melvin Park[2]; [1]University of North Carolina; [2]Bruker Daltonics

2:50 (494) **Applying Digital Waveforms to Mass Spectrometry**; Peter Reilly[1], Maxwell Marino[2], Hideya Koizumi[3], William Whitten[4]; [1]Washington State University; [2]Colorado College; [3]Arkansas State University; [4]Oak Ridge National Laboratory

3:10 (495) **Metastable Atom-Activated Dissociation of Glycopeptides, Nitrosylated Peptides and Non-Peptidic Analytes**; Glen P Jackson[1], Shannon L Cook[1]; [1]Ohio University

3:30 (496) **Incorporating a Surface-Induced Dissociation Device into an Ion Mobility-Tandem Mass Spectrometer for Structural Analysis of Protein Complexes**; Chengsi Huang[1], Mowei Zhou[1], Anne E. Blackwell[1], Eric Dodds[1], Ünige Laskay[1], Vick H. Wysocki[1]; [1]University of Arizona

3:50 (497) **A New Segmented Rectilinear Ion Trap with Modified Smalley Nozzle for Creation of New Catalysts and Dielectric Materials**; Guido Verbeck[1]; [1]University of North Texas

4:10 (498) **New Concepts in Ion Mobility-Mass Spectrometry Instrumentation**; Jody C. May[1, 2, 3], Sevugarajan Sundarapandian[1,2,3], John A. McLean[1,2,3]; [1]Department of Chemistry, Vanderbilt University; [2]Vanderbilt Institute of Chemical Biology; [3]VIIBRE

TECHNICAL PROGRAM – WEDNESDAY
Orals 2:30 – 4:30 pm

Wednesday Afternoon, *Room 304*
MEGGERS AWARD SYMPOSIUM – ADVANCES IN HYPERSPECTRAL IMAGING
Organizer: Paul Gemperline; Presider: David Haaland

2:30 (499) **Single Particle Tracking Analyses of Hyperspectral Fluorescent Images**; Patrick Cutler[1], Michael Malik[2], Fang Huang[2], Diane Lidke[1], Keith Lidke[2]; [1]Department of Pathology, University of New Mexico; [2]Department of Physics and Astronomy, UNM

2:50 (500) **Integrating Physics with Chemometrics for Enhanced Vibrational Spectroscopic Imaging**; Rohit Bhargava[1], Anil Kodali[1], Matthew Schulmerich[3], Xavier Llora[1], Rohith Reddy[1]; [1]University of Illinois at Urbana-Champaign

3:10 (501) **Autonomous Hyperspectral Imaging in Real-Time**; Patrick Treado[1], Robert Schweitzer[1], Arjun Bangalore[1]; [1]ChemImage Corporation

3:30 (502) **Hyperspectral Imaging of Microalgae Using Two-photon Excitation**; Howland Jones[1], Michael Sinclair[1], Ting Luk[1], Bryce Ricken[1], Thomas Reichardt[1], Omar Garcia[1], Jerilyn Timlin[1]; [1]Sandia National Labororatories

3:50 (503) **Micoralgal Biodiversity as Novel Indicator for Marine Ecosystems - Combining Spectroscopy, Imaging and Prior Information through Bayesian Statistics**; Frank Vogt[1], Morgan McConico[1]; [1]University of Tennessee, Department of Chemistry

4:10 (504) **Passive Standoff Detection of Solid Explosive Residues on Soil via Infrared Hyperspectral Imaging**; Neal Gallagher[1], J. F. Kelly[2], T. A. Blake[2]; [1]Eigenvector Research, Inc.; [2]Pacific Northwest National Laboratory

Wednesday Afternoon, *Room 305A*
FACSS / SAS STUDENT AWARDS
Organizer: Pavel Matousek; Presider: Ramachandra Dasari

Tomas Hirschfeld Scholar

2:30 (505) **Rational Design of Nanostructured Probes for Surface Enhanced Raman Spectroscopy**; Anil Kodali[1], Matthew Schulmerich[1], Xavier Llora[1], Rohit Bhargava[1]; [1]Univ of Illinois at Urbana Champaign

Tomas Hirschfeld Scholar

2:50 (506) **Correction for Physiological Dynamics Significantly Improves Spectroscopy Based Transcutaneous Blood Glucose Predictions**; Ishan Barman[1], Chae-Ryon Kong[1], Narahara C. Dingari[1], Jeon-Woong Kang[1], Ramachandra R. Dasari[1], Michael S. Feld[1]; [1]Massachusetts Institute of Technology

3:10 SAS Poster Awardee

3:30 SAS Poster Awardee

3:50 SAS Poster Awardee

4:10 SAS Poster Awardee

Wednesday Afternoon, *Room 305B*
BIOMEDICAL APPLICATIONS OF SPECTROSCOPIC IMAGING
Organizer: Sergei Kazarian;
Presider: Sergei Kazarian and Nick Stone

2:30 (507) **Vibrational Spectroscopy and Microspectroscopic Imaging: Applications to Skin Pharmacology and Wound Healing**; Richard Mendelsohn, Carol Flach; [1]Rutgers University; [2]Rutgers University

2:50 (508) **Detection of Viral Infection by Spectral Cytopathology**; Max Diem; [1]Northeastern University[4]

3:10 (509) **Applications of ATR-FTIR Spectroscopic Imaging to Biomedical Samples**; Sergei Kazarian[1], Andrew Chan[1]; [1]Imperial College London

3:30 (510) **Turning ATR Imaging Upside Down: A New Microscope Specifically Designed for Pathological Investigations**; Andre Sommer[1], Craig Damin[1]; [1]Miami University

3:50 (511) **Exploring the Potential of Vibrational Spectroscopy for Determination of Malignant Lymph Nodes**; Nick Stone[1], Jonathan Horsnell[1], Linda Orr[1], Martin Isabelle[1], Jenny Smith[1], Keith McCarthy[1], Jonathan Christie-Brown[1], Neil Shepherd[1], Charlie Chan[1], Hugh Barr[1]; [1]Gloucestershire Hospitals NHS

4:10 (512) **Evaluation of FTIR Imaging Spectroscopy-Derived Parameters on ACI Treated Human Repair Cartilage**; Arash Hanifi[1], Sally Roberts[2], James Richardson[2], Nancy Pleshko[1]; [1]Temple University; [2]Keele University

Wednesday Afternoon, *Room 306A*
TWO-DIMENSIONAL CORRELATION SPECTROSCOPY
Organizer and Presider: Isao Noda

2:30 (513) **2D Correlation Spectra Based on Higher Order Moments for Correlation Analysis with Constituent Concentrations**; Jun Uozumi[1]; [1]Hokkai-Gakuen University, Faculty of Engineering

2:50 (514) **Use of Infrared Correlation Spectroscopy to Characterize Polymer Materials**; Georgia Arbuckle-Keil[1], Frank Weston[2], Isao Noda[3]; [1]Rutgers University; [2]Varian, Inc; [3]The Procter & Gamble Company

3:10 (515) **A Depth Profiling and Heterogeneity Investigation of Polymers, including Multi-Laminate and Conductive**; Frank Weston[1], Georgia Arbuckle[2], Isao Noda[3]; [1]Varian, Inc.; [2]Rutgers University; [3]Procter & Gamble

3:30 (516) **Multiple-Perturbation Two-Dimensional Correlation Analysis of Cellulose by Attenuated Total Reflectance Infrared (ATR IR) Spectroscopy**; Yukihiro Ozaki[1], Hideyuki Shinzawa[2]; [1]Kwansei Gakuin University; [2]Advanced Industrial Science and Technolo

3:50 (517) **Recent Trends in Multiple-Perturbation 2D Correlation**; Hideyuki Shinzawa[1], Yizhuang Xu[2], Yuqing Wu[3], Isao Noda[4]; [1]Advanced Industrial Science and Technology (AIST); [2]Peking University; [3]Jilin University; [4]The Procter & Gamble Company

4:10 (518) **2D Correlation Spectroscopy As A Tool for Spectral Interpretation**; Franklin Barton[1], James de Haseth[1]; [1]Light Light Solutions, LLC

Wednesday Afternoon, *Room 306B*
MONITORING CONTINUOUS CHEMISTRY AND CHEMICAL PROCESSES
Organizer and Presider: James Rydzak

2:30 (519) **Real-Time *in situ* FTIR Analytics as a PAT Tool for Batch and Continuous Processes**; Wes Walker[1]; [1]METTLER TOLEDO

2:50 (520) **From Batch to Continuous – The GSK Continuous Hydrogenation Workflow**; Robert Yule[1], Gary Kelly[1]; [1]GSK

3:10 (521) **NIR Monitoring of Hot Melt Extrusion Processes**; Brandye Smith-Goettler[1], Robert Meyer[1], Neil MacPhail[1], Colleen Gendron[1]; [1]Merck Sharp and Dohme Corp

3:30 (522) **Determining the Efficacy of Rapid Spectroscopic Techniques for *in-situ* Characterisation of Polymorph Contamination**; Michelle C Hennigan[1], Yun Hu[1], Alan G Ryder[1]; [1]School of Chemistry, NUI Galway, Ireland

3:50 (523) **Controlling the Crystalline State of Electrospun Nylon 6 by Varying the Solvent Evaporation Kinetics**; Carl Giller[1], Bruce Chase[1], John Rabolt[1], Christopher Snively[2]; [1]University of Delaware, Dept. Mat. Sci./Eng.; [2]SCHOTT North America, Inc.

4:10 (524) **Accelerating Drug Development Using Flow/Continuous Processes**; Sandeep Kedia, Thomas Lovelace; Chemical Development, GlaxoSmithKline

Wednesday Afternoon, *Room 306C*
APPLICATIONS OF SERS
Organizers: Duncan Graham, Karen Faulds, Michael Natan;
Presider: Karen Faulds

2:30 (525) **Engineering SERS Tags for Cytometry**; John Nolan[1]; [1]La Jolla Bioengineering Institute

3:10 (526) **Plasmonics Nanoprobes and Nanochips for Environmental Sensing and Medical Diagnostics**; Tuan Vo-Dinh ; [1]Duke University

3:30 (527) **Integrated SERS Sensors Based on Modified Particles Stabilised in Polymer Gels**; Steven Bell, David Jones[1], Colin McCoy[1], Maighread McCourt[1], Alan Stewart[1], Steven Bell, Steven Bell, Steven Bell, Steven Bell, Steven Bell; [1]Queen's University of Belfast

4:10 (528) **Sensitive SERRS Detection of DNA by Novel Signal Amplification Based Approach**; Jennifer A. Dougan[1], Kristy McKeating[1], Duncan Graham[1], Karen Faulds[1]; [1]University of Strathclyde

Wednesday Afternoon, *Room 307*
TERAHERTZ SPECTROSCOPY
Organizer and Presider: Gilbert Pacey

2:30 (529) **Continuing Development of Standoff Chemical Sensing and Concealed-Threat Detection with Millimeter-Wave and THz Radiation**; Michael Gord[1], Anita Taulbee-Combs[2], David Hufnagle[2], Gilbert Pacey[2], Carla Benton[3], Douglas Petkie[3], Satya Ganti[3], Jason Deibel[3], Michael Moulton[4], James Gord[4]; [1]Dayton Christian High School; [2]Miami University; [3]Wright State University; [4]Air Force Research Laboratory

3:10 (530) **Using Terahertz Pulses in the Process World**; Philip Taday[1], Mike Evans[1], Axel Zeitler[3], Yaochun Shen[2], Dipankar Dey[4]; [1]TeraView; [2]Liverpool; [3]Cambridge; [4]Oystar-Manesty

3:50 (531) **Terahertz Computed Tomography Measurements**; Jeffrey White[1], David Zimdars[1], Greg Fichter[1], Chris Megdanoff[1], John Duquette[1], Irl Duling[1]; [1]Picometrix LLC

4:30 pm FACSS AWARD PRESENTATIONS, *Room 305A*

FACSS Student Award
Jacob Shelley, *Indiana University*

Tomas Hirschfeld Scholars
Ishan Barman, *Massachusetts Institute of Technology*
Anil Kumar Kodali, *University of Illinois at Urbana*

FACSS Distinguished Service Awards
Scott McGeorge, *Transition Technologies*
Alexander Scheeline, *University of Illinois at CU*

4:40 pm Plenary, *Room 305A*
(532) Distinguished Service: Becoming an Oxymoron?; **Alexander Scheeline**; University of Illinois at CU

TECHNICAL PROGRAM – THURSDAY

Plenary Lectures

Presider: Pavel Matousek

Coblentz Clara Craver Award

8:00 AM Plenary Lecture, *Ballroom A*

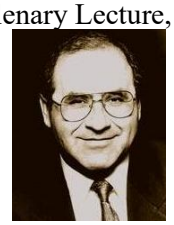
Boris Mizaikoff

(533) **Advanced Sensor Technologies for Enhanced Mid-Infrared Diagnistics**; Boris Mizaikoff; University of Ulm

Refer to page 22 for biographical information

Ellis R. Lippincott Award

8:30 AM Plenary Lecture, *Ballroom A*



Martin Moskovits

(534) **Transforming SERS into a Reliable, Ultrasensitive Analytical Tool**; Martin Moskovits; University of California Santa Barbara

Refer to page 14 for biographical information

THURSDAY POSTER SESSION

9:00 – 10:30 am

Ballroom Lobby

All Thursday posters should be put up between 7:30 – 8:00 am and removed by 5:00 pm. Odd numbered poster boards present between 9:00 – 9:45 am. Even numbered poster boards present between 9:45 – 10:30 am.

Bioanalytical

Board #

1 (535) **Development and Application of an UPLC/UV Method for Determination of Resveratrol in Rat Urine at Parts-Per-Billion Levels**; Teruyo Uenoyma[1], Stephen Cooper[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

2 (536) **Development and Validation of a New LC/MS/MS Bio-Analytical Method for the Determination of Curcumin in Human Plasma Samples**; Sunny Chopra[1], Saurabh Arora[1], Kanchan Kohli[3]; [1]Faculty of Pharmacy,Jamia Hamdard,New Delhi,India

3 (537) **Microbeam Radiation Therapy of Cells on Microchips**; Jocelyn Wang[1], Hamed Shadpour[1], Jared Snider[2], Jian Zhang[2, 3], Guang Yang[2,3], Adrienne Cox[2], Sha Chang[2, 3], Nancy Allbritton[1, 4]; [1]Dept. of Chemistry, UNC, Chapel Hill; [2]Dept. of Radiation Oncology, UNC; [3]Dept. of Physics and Materials Sci., UNC; [4]Dept. of Biomedical Eng., UNC-NCSU

4 (538) ***In vitro* Monitoring Vitamin C in Human Urine by Flow-Injection Chemiluminescence with Luminol-Dissolved Oxygen System**; Donghua Chen[1], Zhenghua Song[1]; [1]Northwest University, China

5 (539) **Development of a Quartz Crystal Microbalance (Qcm) Immuno Sensor for Sesame Protein Detection in Complex Food Matrices**; Fatima Tazeen Husain[1], Margit Cichna-Markl[1], Romana Schirhagl[1], Franz Ludwig Dickert[1]; [1]University of Vienna

6 (540) **Tip Enhanced Raman Spectroscopy and Imaging of Lipid Membranes**; Tsoching Chen[1], Ira Levin[1]; [1]National Institutes of Health

7 (541) **Infrared Imaging Analysis of Skin Mineralization in the Genetic Disease Pseudoxanthoma Elasticum**; Nadire Beril Kavukcuoglu[1], Qiaoli Li[2], Jouni Uitto[2], Nancy Pleshko[1]; [1]Temple University, Mechanical Engineering; [2]Jefferson Medical Collage, Dermatology

Board #

8 (542) **Infrared Spectroscopy to Quantify Collagen in Infarcted Myocardium after Targeted VEGF Treatment**; Rabee Cheheltani[1], Jenna M. Rosano[1], Bin Wang[1], Nancy Pleshko[1], Mohammad Kiani[1]; [1]Temple University

9 (543) **Using Contact Printing Method to Modify Surface of Arrayed Microstructures**; Wei Xu[1], Yuli Wang[1], Jonathan Clark[1], Christopher E. Sims[1], Nancy L. Allbritton[1]; [1]University of North Carolina

10 (544) **Development of "Protectides" to Prolong the Lifetime of Peptide Reporters for Intracellular Abl Kinase Activity**; shan yang[1], Lauren L. Cline[1], Marcey L. Waters[1], Nancy L. Allbritton[1]; [1]Dept. of Chemistry, University of North Carolina

General Analytical
(Education, Electrochemistry, Forensic, Instrumentation, Laser Ablation, Materials Characterization, Other, Pharmaceutical, Process/Control, Proteomics, Surface Characterization

11 (545) **Spectroelectrochemical Characterization of Polymerized Hemoglobins**; Scott Dorman[1], Serena Murphy[1]; [1]Birmingham-Southern College

12 (546) **Polymer Blend Characterizations Utilizing FRET and Multivariate Fluorescence Correlation Spectroscopy**; Carol Roach[1], Sharon Neal[1]; [1]University of Delaware

13 (547) **Development of a Spectroelectrochemical Assay for Serum Bilirubin**; Paul Flowers[1], Megan Alexander[1]; [1]Univ North Carolina Pembroke

14 (548) **Fibre Spectroscopy with Clean in Place Probes for PAT**; Viacheslav Artyushenko[1], Joachim Mannhardt[1], Gary Colquhoun[1]; [1]Fibre Photonics Ltd

15 (549) **Proteomic Analysis of The Rice Blast Fungus Magnaporthe Oryzae**; Emine Gokce[1], Timothy Collier[1], Yeon Yee Oh[2], William Franck[2], Ralph Dean[2], David Muddiman[1]; [1]W.M. Keck FT-ICR Mass Spectrometry Laboratory; [2]Center for Integrated Fungal Research

16 (550) **Chemical and Surface Structure Correlations Using Tip Enhanced Raman Spectroscopy**; James Marr[1], Zachary Schultz[1]; [1]University of Notre Dame

Board #

17 (551) **Quantitative Determination of Polymorphic Purity of Crystalline API in Tablets Using Raman Spectroscopy and Multivariate Analysis**; Yong Xie[1], Rick Chiu[1], Nina Cauchon[1]; [1]Analytical Research and Development, Amgen Inc

18 (552) **Design Considerations and Best Practices, for the Implementation of a Fluorescence Spectrophotometer in Laboratory and Plant Pharma PAT.**; Susan Bragg[1]; [1]Expo Technologies

Imaging / Microscopy and Spectral Analysis

19 (553) **Spectroscopic Analyses for Quantification of Key Biological Components in Microalgae as Indicators of Environmental Change**; Rebecca Horton[1], Edward Duranty[1], Morgan McConico[1], Frank; [1]University of Tennessee, Department of Chemistry

20 (554) **Bayesian Classification of Microalgae FTIR Spectra as Innovative Method to Detect Chemical Changes in Ecosystems**; Morgan McConico[1], Edward Duranty[1], Rebecca Horton[1], Frank Vogt[1]; [1]University of Tennessee, Department of Chemistry[4]

21 (555) **Fast Methods for Simultaneous Wavelength Selection and Grouping**; Erik Andries[2], John Kalivas[1]; [1]Central New Mexico Community College; [2]Center for Advanced Research Computing; [3]Idaho State University

22 (556) **Calmagite Assay for Quickly Screening Potential Magnesium Chelators**; Joshua Kimball[1], Leonard Moothart[1], Laurent Bernad[1], Thomas Kirkland[1]; [1]Promega Biosciences LLC

23 (557) **Spatial and Time Resolved Measurements by a New Acousto-Optical Imaging Spectrometer in Combination with Glow Discharge Sources**; Maxim Voronov[1], Volker Hoffmann[1], Till Wallendorf[2], Swen Marke[2], Gary Hieftje[3], Steven Ray[3], Carsten Engelhard[3], Wolfgang Buscher[4]; [1]IFW Dresden, Institute for Complex Materials; [2]IfU Diagnostic Systems GmbH; [3]Indiana University; [4]University of Münster

24 (558) **Single Modified Starch Granules Rapidly Imaged with Focal Plane Array FT-IR Microspectrometer Illuminated by Multiple Combined Synchrotron Beams**; David Wetzel[1,2], Michael Nasse[3,4]; [1]Microbeam Molecular Spectroscopy Laboratory; [2]Kansas State University; [3]Synchrotron Research Center; [4]University of Wisconsin-Milwaukee

25 (559) **Remote Hyperspectral Imaging of Human Skin**; Kerri Moloughney[1], Kiersten Schiliro[2], Diane Williams[3]; [1]Oak Ridge Institute of Science and Education; [2]FBI, Operational Technology Division; [3]FBI, Laboratory Division

26 (560) **Cellular Crystallography: Generation and Detection of 2D Membrane Protein Crystals in Living Cells.**; Ellen Gualtieri[1], Fei Guo[1], David Kissick[1], Joyce Jose[1], Richard Kuhn[1], Wen Jiang[1], Garth Simpson[1]; [1]Purdue University

27 (561) **Characterization and Differentiation of Phthalates Using FTIR Fingerprinting, NMR Spectroscopy, and GC/MS.**; Martin Best[1], Melanie Silinski[1], Joseph Licause[1], James Blake[1], Stephen Cooper[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

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28 (562) **Highly Sensitive Imaging of Protein Crystals at Cryogenic Temperatures**; David Kissick[1], Ellen Gualtieri[1], Kevin Battaile[3], Michael Becker[3], Robert Fischetti[3], Steve Ginell[3], Lisa Keefe[3], Anne Mulichak[3], Vadim Cherezov[2], Garth Simpson[1]; [1]Purdue University; [2]Scripps Research Institute; [3]Advanced Photon Source

29 (563) **Second Harmonic Generation in NaCl**; Scott Toth[1], Garth Simpson[1]; [1]Purdue University

30 (564) **NIR Assessment of Water Correlates to Mechanical Properties in Articular Cartilage**; Alireza Hosseini[1], Somaieh Moghadam[1], Roza Mahmoodian[2], Sorin Siegler[2], Nancy Pleshko[1]; [1]Temple University; [2]Drexel University

Spectroscopy (Absorption, Atomic Spectroscopy, Fluorescence, ICP, Infrared, Laser Spectroscopy, Mass Spectrometry, Molecular Spectroscopy, Near Infrared, NMR, Raman, Surface Enhanced Raman, Surface Plasmon Resonance)

31 (565) **A Novel Matrix Modifier for the Analysis of Arsenic and Antimony in High-Sulfate Acid Mine Drainage**; Ronald Smith[1]; [1]Indiana Geological Survey

32 (566) **Development and Validation of an Analytical Method for the Determination of Zinc Carbonate Basic in Zinc-Deficient Rodent Feed.**; Randy Price[1], Glenn Ross[1], Jason Perlmutter[1], Keith Levine[1], Donna Browning[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

33 (567) **Co(II) Determination by Photoacoustic Spectroscopy with 3-(2-Pyridyl)-5,6-bis(4-sulfophenyl)-1,2,4-triazine as Ligand**; M. Ines Toral[1], César Soto[2], Jorge Yañez[2], Renato Saavedra[2], Ivo Fustos[2], Cristian Candia[2]; [1]Faculty of Science. University of Chile; [2]Univesity of Concepcion

34 (568) **Optical Optimization of Phytoplankton Classification instrument**; Joe Swanstrom[1], Laura Hill[1], Tammi Richardson[1], Timothy Shaw[1], Michael Myrick[1]; [1]University of South Carolina

35 (569) **The Dual-Sample CapNMR Probe: Application Diversity and Performance Enhancement through Miniaturization and Automation**; Timothy Peck[1], James Norcross[1], Craig Milling[1], Robert Albrecht[1], Dean Olson[1], Steve Xu[1]; [1]Protasis Corporation

36 (570) **Microscopic FTIR/DSC System Used to Investigate the Thermal-induced Intramolecular Cyclization of Eudragit E Film or PVA Copolymer Film**; Shan-Yang Lin[1], Wen-Ting Cheng[1], Yen-Shan Wei[1], Yu-Ting Huang[1]; [1]Yuanpei University

37 (571) **Formation and Characterization of Nanosized Organic Molecular Crystals on Engineered Surfaces**; Andrea Centrone[1], Kitae Kim[2], T. Alan Hatton[1], Allan S. Myerson[2]; [1]Massachusetts Institute of Technology; [2]Illinois Institute of Technology

38 (572) **NIST SRM for the Relative Intensity Correction of Raman Spectrometers Utilizing 830 nm Excitation**; Aaron Urbas[1], Steven Choquette[1]; [1]National Institute of Standards and Technology

39 (573) **Imaging Nonlinear Optical Stokes Ellipsometry (iNOSE)**; Garth Simpson; [1]Purdue University[4]

40 (574) **Re-Investigation of Excited State Proton Transfer Reaction in 2-naphthol in the Presence of Sodium Acetate**; K Singh[1], G.C. Joshi[1]; [1]HNBGarhwal University,Srinagar

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41 (575) **New Directions in AFM-Raman BioImaging in Liquids**; David Lewis[1], Anatoly Komisar[1], Andrey Ignatov[1], Rimma Dekhter[1], Hesham Taha[1], Aaron Lewis[2]; [1]Nanonics Imaging Ltd.; [2]Hebrew University of Jerusalem

42 (576) **Proposal of s New Calibration Strategy gor LA-ICP-MS Based on Dried Residues of Individual Picoliter Droplets.**; Jan H. Petersen[1], Jan Massmann[1], Niklas Schaper[1], Nicolas H. Bings[1]; [1]University of Mainz, Analytical Chemistry

43 (577) **Multiphoton-Excited Intrinsic Fluorescence of Protein Crystals**; Jeremy Madden[1], Ellen Gualtieri[1], David Kissick[1], Garth Simpson[1]; [1]Purdue University

44 (578) **ETD and CID Characterization for the Automated Top-Down Analysis of Intact Large Proteins Via High Resolution FTMS**; Aaron Behr[1], Jeremy Wolff[2], Christopher Thompson[2]; [1]The Rivers School; [2]Bruker Daltonics, Inc.

45 (579) **Surface-Enhanced Raman Scattering from Au and Ag-Coated Magnetic Microspheres**; Gulay Ertas[1], Haci Osman Guvenc[1]; [1]Bilkent University, Chemistry Department

46 (580) **Toward High-Speed, Near-Field Raman Acquisition Utilizing Ag Nano Junctions**; Steven Asiala[1], Zachary Schultz[1]; [1]University of Notre Dame

47 (581) **Trace Element Analysis of Seminal Fluid by ICP-MS**; Todor Todorov[1], Gustavo Guandalini[2], Dennis Hoover[3], Larry Anderson[3], Jose Centeno[2], Katherine Squibb[4], Melissa McDiarmid[4]; [1]US Geological Survey; [2]Armed Forces Institute of Pathology; [3]University of Maryland; [4]Veterans Administration - Baltimore

48 (582) **Development of a High-Speed and High-Sensitivity Near-Infrared Spectrometer and Short - Time Transmission Measurement of Tablets by Using It**; Koudai Murayama[1], Makoto Komiyama[1], Takuma Genkawa[2], Mikiko Konta[2], Yukihiro Ozaki[2]; [1]Yokogawa Electric Corp.; [2]Kwansei Gakuin Univ

49 (583) **Identification of Fish Species by Protein Profiling Using MALDI-TOF Mass Spectrometry**; Alexander Post[1], Sergei Dikler[1]; [1]Bruker Daltonics Inc.

50 (584) **Chromatographic Detection Using a Micro-Fluidic Attenuated Total Internal Reflection (ATR) Cell Coupled to a Planar Array Infrared Spectrograph**; Willie Tran[1,2], Andre Sommer[1,2]; [1]Molecular Microspectroscopy Laboratory; [2]Miami University

51 (585) **Comparison of Atomic Absorption and Molecular Spectrophotometry for the Indirect Determination of Phosphate Compounds**; Neil Danielson[1], Matthew Collins[1]; [1]Miami University

52 (586) **Identification of Unknown Pharmaceuticals in Hospitals with a Small Coded Aperture Raman Spectrometer**; Prasant Potuluri[1], Brett Guenther[1], Yuting Qi[1]; [1]Centice Corporation

53 (587) **Development of Halide Sensor Based on 4-(2-Pyridylazo)resorcinol (PAR)**; Michal Sidlo[1], Premysl Lubal[1], Pavel Anzenbacher Jr.[2]; [1]Masaryk University Brno, Czech Republic; [2]Bowling Green State University, Ohio

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54 (588) **Improvement of Analytical Method for Methylmercury in Seafood by High Performance Liquid Chromatography-Inductively Coupled Plasma Mass Spectrometry**; Kyung-Yoal Yoo[1], Kyeong-Nyeo Bahn[1], Yang-Sun Kim[1], Eun-Jung Kim[1], Seong-Chul Shin[1], Ji-Hyeon Seok[1], Hye-Young Park[1], Mi-Sun Lee[1], Yeo-Won Sohn[1], Hae-Seong Yoon[1]; [1]Gyeongin Regional KFDA

55 (589) **Validation of Low-E Slides for FT-IRIS Polarization Measurements**; Arash Hanifi[1], Nancy Pleshko[1]; [1]Temple University

56 (590) **Calibration of NIR Water Assessment in Articular Cartilage Using a Model System of Gelatin and Chondroitin Sulfate**; Mugdha Padalkar[1], Karl Lewis[1], Nancy Pleshko[1], Richard Spencer[2]; [1]Temple University Philadelphia; [2]National Institute of Aging Baltimore

57 (591) **Enhanced Ion Mobility Shift Reagents for Peptide Labeling**; Thomas J. Kerr[1], Randi L. Gant-Branum[1], John A. McLean[1]; [1]Vanderbilt University

58 (592) **Elucidation of the Binding of Alkanes To Transition Metals Using Quantum Cascade Lasers and Time-Resolved Infrared and NMR Spectroscopies**; James A. Calladine[1], Olga Torres[2], Khuong Q. Vuong[1], Steven L. Matthews[2], Simon B. Duckett[2], Robin N. Perutz[2], Michael W. George[1]; [1]The University of Nottingham; [2]The University of York

Thursday Morning, *Room 301B*
SURFACE PLASMON RESONANCE: INSTRUMENTATION AND APPLICATIONS
Organizer: Jean-François Mason; Presider: Amanda Haes

10:30 (593) **SPR Signal Amplification with Nanoparticles and *in situ* Polymer Growth**; Q Cheng[1], Ying Liu[1]; [1]Univ of California Riverside

10:50 (594) **Localized Surface Plasmon Resonance Response of Surface-Attached Gold Nanoplates to Protein Binding: Effect of Binding Location and Distance**; Francis Zamborini[1], Srinivas Beeram[1]; [1]University of Louisville

11:10 (595) **Surface Optical Sensing: SPR, Interferometry and Grating Reflection**; Roger Terrill; [1]San Jose State University

11:30 (596) **Development of a Surface Plasmon Resonance Sensor for Monitoring Cytochrome P450 Activity**; Brent Cameron[1], Rui Zheng[1]; [1]University of Toledo

11:50 (597) **Direct Detection of Biomarkers in Whole Biological Matrixes Using Ultralow Fouling Peptide SAM and SPR**; Olivier R. Bolduc[1], Joelle N. Pelletier[1,4], Jean-François Masson[1,2,3]; [1]Département de Chimie, Université de Montréal; [2]Center for Self-Assembled Chem. Struct.; [3]Centre for Biorecognition and Biosensors; [4]PROTEO Network for Protein Structure

12:10 (598) **New Tools for Surface Science: Robust Multifunctional Chemically Patterned Amorphous Carbon Substrates**; Stephen Weibel; [1]GWC Technologies, Inc

TECHNICAL PROGRAM – THURSDAY
Orals 10:30 am – 12:30 pm

Thursday Morning, *Room 302A*
PLASMAS FOR ATOMIC AND MOLECULAR ANALYSES
Organizers: Carsten Engelhard and Jacob T. Shelley;
Presider: Carsten Engelhard

10:30 (599) **Indicator for Flagging Matrix Effects in Axial-Viewing Mode Inductively Coupled Plasma–Atomic Emission Spectrometry**; George Chan[1], Gary Hieftje[1]; [1]Indiana University

10:50 (600) **Tungsten Coil Atomic Emission Spectrometry**; Brad Jones[1]; [1]Wake Forest University

11:10 (601) **ICP Signals: Model and Experimental Results**; Josh Dettman[1], John Olesik[1]; [1]The Ohio State University

11:30 (602) **Optical and Mass Spectrometric Studies of a Helium Dielectric Barrier Discharge Used as an Ambient Ionization Source**; Matthew Heywood[1], Jonathan Wright[1], Paul Farnsworth[1]; [1]Brigham Young University

11:50 (603) **Atmospheric Desorption and Detection of Organic Compounds by Atmospheric Pressure Glow Discharge Mass Spectrometry (APGD-MS)**; Tim M. Brewer[1], Marcela Najarro[1], Jennifer Verkouteren[1], Greg Gillen[1]; [1]NIST

12:10 (604) **Examinations and Improvements in Low-Temperature Plasma (LTP) Ambient Desorption/Ionization Mass Spectrometry**; Joshua Wiley[1], Jacob Shelley[2], Ayanna Jackson[1], Gary Hieftje[2], R. Graham Cooks[1]; [1]Dept. of Chemistry - Purdue University; [2]Dept. of Chemistry - Indiana University

Thursday Morning, *Room 302B*
MICROREACTORS: CHEMISTRY, TECHNOLOGY, AND SUCCESS
Organizers and Presiders: Ian R. Lewis and Brian J. Marquardt

10:30 (605) **Enhanced Chemical Synthesis and Scale-Up in Micro Reactors**; Paul Watts; [1]University of Hull

10:50 (606) **Designing Sustainable Chemical Systems in a Process Intensified Environment**; Michael Gonzalez[1]; [1]United States Environmental Protection Agency

11:10 (607) **Benefits of On-line Sensors for Advanced Flow Reactor Analysis, Optimization and Control**; Brian Marquardt[1], Wesley Thompson[1], Thomas Dearing[1]; [1]University of Washington

11:30 (608) **Ultrasonically Levitated Microreactors: Continuously Stirred or Shaken?**; Rachel Behrens[1], Alexander Scheeline[1] ; [1]University of Illinois at CU

11:50 (609) **Microreactors for the Large Scale Manufacture of Life Science Compounds**; David Ager[1]; [1]DSM

12:10 (610) **Synthesis in Microreactors: From Materials to Small Molecules**; D. Tyler McQuade[1]; [1]Florida State University

Thursday Morning, *Room 302C*
H/D EXCHANGE MASS SPECTROMETRY
Organizer and Presider: Michael Chalmers

10:30 (611) **Mapping Contact Surfaces in Protein Complexes by Solution-Phase H/D Exchange Monitored by Ultrahigh Resolution FT-ICR Mass Spectrometry**; Alan Marshall[1], Greg Blakney[2], George Bou-Assaf[1], Mark Emmett[2], Christopher Hendrickson[2], Santosh Valeja[1], Hui-Min Zhang[2], Qian Zhang[1], Alan Marshall, Alan Marshall; [1]Florida State University; [2]Nat\'l High Magnetic Field Laboratory

10:50 (612) **Mass-Spectrometry-Based Hydrogen/Deuterium Exchange, PLIMSTEX, and Protein Digestion for Elucidating Metal Binding in Proteins**; Michael L Gross[1], Richard Huang[1]; [1]Washington Univ in St Louis;

11:10 (613) **SUPREX: An H/D Exchange and Mass Spectrometry-Based Protein-Ligand Binding Assay with a High Throughput Capability**; Michael C. Fitzgerald[1]; [1]Duke University

11:30 (614) **Mass Shift Perturbation Methods for Structural Proteomics**; David Schriemer[1], Andrew Percy[1]; [1]University of Calgary

11:50 (615) **The Utility of H/D-Exchange for Epitope Screening to Assist with Biopharmaceutical Candidate mAb Selection**; Jennifer F. Nemeth[1], Seng-Jiun Wu[1], Steve Tuske[2], Yoshi Hamuro[2]; [1]Centocor Research and Development; [2]ExSAR Corporation

12:10 (616) **Ligand Screening with Hydrogen Deuterium Exchange Mass Spectrometry**; Michael Chalmers[1], Jun Zhang[2], Rachelle Landgraf[1], Graham West[1], Janelle Lauer[1], Scott Novick[1], Scooter Willis[1], Bruce Pascal[1], Scott Busby[1], Patrick Griffin[1]; [1]TSRI

Thursday Morning, *Room 304*
LIPPINCOTT AWARD SYMPOSIUM
Organizer: Martin Moskovits; Presider: Ricardo Aroca

10:30 (617) **Expanding Versatility of SERS with Construction of Various Nanostructures**; Zhong-Qun Tian[1], Jiang-Feng Li[1], Yi-Fan Huang[1], Zheng Liu[1], Zhi-Lin Yang[2], Bin Ren[1]; [1]Chemistry Department, Xiamen University; [2]Physics Department, Xiamen University

10:50 (618) **Pathogen Detection with Plasmonic and Superparamagnetic Nanoparticles**; Li-Lin Tay[1], Peilin Chen[2], John Hulse[1], Jamshid Tanha[1]; [1]National Research Council Canada; [2]Academia Sinica, Taipei, Taiwan[4]

11:10 (619) **Biomolecule Sensing with Adaptive Plasmonic Nanostructures**; Vladimir Shalaev[1], Vladimir Drachev[1]; [1]Birck Nanotechnology Center, Purdue University

11:30 (620) **Surface-Enhanced Resonance Raman Scattering Imaging with Langmuir-Blodgett Monolayers**; Ricardo Aroca[1], Golam Moula[1], Nicholas Pieczonka[1]; [1]University of Windsor

11:50 (621) **Surface-Enhanced Spectroscopies of Biomolecules**; Naomi Halas; [1]Rice University

12:10 (622) **New Panel for SERS-Based Screening of Influenza Viral Nucleoproteins Using Anti-Influenza Aptamer**; Pierre Negri[1], Richard A.; [1]University of Georgia - Department of Chemistry

Thursday Morning, *Room 305A*
CONTRIBUTED CHROMATOGRAPHY
Organizer: Neil Danielson; Presider: Sarah Rutan

10:30 (623) ***In situ* Investigation of the Structure of the RPLC Stationary Phase with Sum Frequency Spectroscopy**; Arthur Quast[1], Alexander Curtis[1], Anthony Peterson[1], Steven Goates[1], James Patterson[1]; [1]Brigham Young University

10:50 (624) **Effect of Mobile Phase Modifiers on Chromatography and Negative Ion Electrospray Response: A Case Study with Flavonoids and Phenolic Acids**; Christine Hughey[1], Bruce Wilcox[1], Carina Minardi[2], Crisand Anderson[2]; [1]James Madison University; [2]Chapman University

11:10 (625) **The Development of an Interpolation-Based Approach to the Alignment of Fast LCxLC-DAD Chromatograms**; Robert Allen[1], Sarah Rutan[1]; [1]Virginia Commonwealth University

11:30 (626) **Biotransformation of Aflatoxin B1 in Soil**; Mustafa Selim[1]; [1]East Carolina University

11:50 (627) **The Metabolism, Excretion, and Pharmacokinetics of Resveratrol in Pregnant and Lactating Rats**; Brenda Fletcher[1], Franz Thomas[1], Teruyo Uenoyama[1], Norman Gaudette[1], Timothy Fennell[1], Stephen Cooper[1], Melanie Silinski[1], James Blake[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

Thursday Morning, *Room 305B*
FROM FORENSICS TO PHARMA – APPLICATIONS OF CHEMICAL IMAGING
Organizer and Presider: Linda Kidder

10:30 (629) **Hyperspectral Imaging of Post-Blast Explosives Residues**; Daniel Mabel[1, 3], Kerri Moloughney[1], Diane Williams[2]; [1]Oak Ridge Institute of Science and Education; [2]FBI, Laboratory Division; [3]Virginia Commonwealth University

10:50 (630) **Experimental Considerations for Quantitation of Solid Mixtures for Focal Plane Array Near-IR Imaging Data**; Mark Boatwright[1, 2], David Wetzel[1,2]; [1]Microbeam Molecular Spectroscopy Laboratory; [2]Kansas State University

11:10 (631) **Effect of Scale of Scrutiny in the NIR Spectroscopic Evaluation of Blend Homogeneity in Continuous and Batch Pharmaceutical Blending Processes**; Rodolfo Romañach[1], Yleana Colon[1], Jackeline Jerez-Rozo[1], Luis Obregon[2], Rafael Mendez[2], Carlos Velázquez-Figueroa[2]; [1]Dept of Chemistry, Univ. Puerto Rico-Mayaguez; [2]Chemical Eng., Univ.Puerto Rico-Mayaguez

11:30 (632) **Combining Physical Morphology and Spectroscopic Classification on Multi-Component Samples**; Justin Pritchard[1], Martin Warman[1]; [1]Vertex Pharmaceuticals

11:50 (633) **The Development of Infrared Spectroscopic Imaging-Based Histochemical Methods for the Prediction of Kidney Ischemia**; Scott Huffman[1], Caitlin Williams[1], Nicole Crane[2], Ira Levin[3], Eric Elster[2]; [1]Western Carolina University; [2]Naval Medical Research Center; [3]National Institutes of Health

12:10 (634) **Mid-IR Imaging for Identification of Cells and Mucin Subtype in the Gastrointestinal Tract**; Michael Walsh[1], Jason Ip[1], Caroline Caroline Cvetkovic[1], Rohit Bhargava[1]; [1]University of Illinois at Urbana-Champaign

Thursday Morning, *Room 306A*
RAUL CURBELO – THE HIDDEN INNOVATOR IN FTIR
Organizer and Presider: Ellen Miseo

10:30 **Rapid Time Resolved FT-IR Spectroscopy Before Step Scanning**; James deHaseth, LightLight Solutions

10:50 (636) **From Handheld to Huge to Handheld: Raul Curbelo and the Continuing Evolution of Commercial FT-IR Spectrometers**; Richard Crocombe[1]; [1]Thermo Fisher Scientific

11:10 (637) **Spectroscopy through the Engineering Lens: Lessons from Raul Curbelo**; Norman Wright[1]; [1]Applied Instrument Tech, Hamilton Sundstrand

11:30 (638) **Development of Digital Signal Processing (DSP) Software for Step-Scan Modulation Measurements**; Dave Drapcho[1]; [1]Thermo Fisher Scientific

11:50 (639) **On the Shoulders of Giants: an FT-IR Legacy**; Andrew Hind[1,2]; [1]Agilent Technologies; [2]Varian Inc.

Thursday Morning, *Room 306B*
ROYAL SOCIETY OF CHEMISTRY, ANALYTICAL DIVISION: APPLICATIONS, ADVANCES AND DEDUCTION BY VIBRATIONAL SPECTROSCOPY
Organizer and Presider: John M. Chalmers

10:30 (641) **Building a Community Resource of Open Spectral Data**; Antony Williams[1]; [1]Royal Society of Chemistry

10:50 (642) **Establishing Raman Spectroscopy as a First Choice Method in Forensic Casework**; Steven Bell[1], Samantha Stewart[1], W. James Armstrong[2], George Kee[2], S. James Speers[2]; [1]Queen's University; [2]Forensic Science (N.I.)

11:10 (643) **Raman Spectroscopy in Forensic Geoscience and Contraband Materials**; Howell GM Edwards; [1]University of Bradford

11:30 (644) **Raman and Near-Infrared Spectroscopy for the Forensic Analysis of Intact Tablets**; Tony Moffat[1], Sulaf Assi[1], Robert Watt[1]; [1]The School of Pharmacy, University of London

11:50 (645) **Raman Mapping of Chocolate and Cells**; Duncan Graham[1], Karen Faulds[1], Iain Larmour[1], Ross Stevenson[1], Joanna Loose[1]; [1]University of Strathclyde

12:10 (646) **Sampling for Success: Quantitative *in situ* Raman Spectroscopy**; Ian Lewis[1], Kevin Davis[1], Sean Gilliam[1], Maryann Cuellar[1], David Strachan[1], Carsten Uerpmann[2], Herve Lucas[2], Pat Wiegand[1], Joe Slater[1], ; [1]Kaiser Optical Systems, Inc.; [2]Kaiser Optical Systems, SARL

Thursday Morning, *Room 306C*
RAMAN SPECTROSCOPY IN THE PHARMACEUTICAL INDUSTRY
Organizer and Presider: Don Pivonka

10:30 (647) **Transmission Raman Spectroscopy for Quantitative Analysis**; Hanna Matic[1], Magnus Fransson[1], Jonas Johansson[1], Anders Sparén[1], Olof Svensson[1]; [1]AstraZeneca PAR&D, Mölndal, Sweden

10:50 (648) **Signal Intensity Dependence on Depth in Transmission Raman Spectroscopy of Pharmaceutical Tablets**; Pavel Matousek[1,2], Neil Everall[3], Alison Nordon[4], David Littlejohn[4], Matthew Bloomfield[2]; [1]Rutherford Appleton Laboratory; [2]Cobalt Light Systems; [3]Intertek MSG; [4]University of Strathclyde

11:10 (649) **Raman Reaction Monitoring in Opaque Vessels When No Ports are Available for a Raman Probe**; Michael Pelletier[1]; [1]Pfizer Global Research and Development

11:30 (650) **Application of *in-situ* Raman Spectroscopy in Pharmaceutical Chemical Development**; Susan Barnes[1], Gregory Gervasio[1], James Rydzak[1]; [1]GSK

11:50 (651) **Application of Raman Spectroscopy in Pharmaceutical Process Development**; Ming Huang[1], Robert Wethman[1], John Wasylyk[1]; [1]Bristol-Myers Squibb Co.

12:10 (652) **Evaluation of Granules Made by High Shear Granulation using Raman Mapping and Imaging**; Tatsuo Koide[1], Toru Kawanishi[1], Yukio Hiyama[1]; [1]National Institute of Health Sciences

1:30 pm – Poster Viewing and Break
Ballroom Lobby

Thursday Afternoon, *Room 301B*
SURFACE PLASMON RESONANCE: INSTRUMENTATION AND APPLICATIONS
Organizer and Presider: Jean-François Masson

2:00 (653) **Multiparametric Surface Plasmon Resonance Imaging Systems and Nano- Micro- Milli- Structured Biochip Substrates**; Michael Canva[1, 2], Anuj Dhawan[2], Aurélien Duval[1], Mohamed Nakkach[1], Buntha Ea-Kim[1], Alain Bellemain[1], Julien Moreau[1], Tuan Vo-Dinh[2]; [1]Institut d\'Optique; [2]Duke University

2:20 (654) **Characterization of Diazonium-Salt Monolayers as a Linker for Surface Plasmon Resonance Spectroscopy Analyses**; Karl Booksh[1], Nicola Menegazzo[1], Qiongjing Zou[1], Laurel Kegel[1]; [1]University of Delaware

2:40 (655) **Grating-Assisted Optical Fiber SPR Sensor with Self-Referencing Capability**; Jacques Albert[1]; [1]Carleton University

3:00 (656) **Extending SPR to Novel Substrates**; Josh Guske[1], Stefan Franzen[1]; [1]NCSU

3:20 (657) **Detection of Influenza Virus Using Evanescent Fields of Waveguide Modes**; Makoto Fujimaki[1], Subash Gopinath[1], Koichi Awazu[1]; [1]AIST

3:40 (659) **Surface Plasmon Resonance Imaging for Applications of Binding Kinetics in Multiplex Throughput**; Karen Gall[1], Philippe Kerouredan[1]; [1]Horiba Scientific

Thursday Afternoon, *Room 302A*
ATOMIC SPECTROSCOPY TECHNIQUES FOR SOLID SURFACE ANALYSIS IN BIOLOGY, GEOLOGY, AND ASTRONOMY
Organizer and Presider: Alan Koenig

2:00 (659) **Laser Ablation ICP-MS: A Progress Report of Problems, Pitfalls and Potential for Geological and Biological Applications**; Alan Koenig[1]; [1]US Geological Survey

2:20 (660) **From Sub-Micron Crater Size to High Repetition Rate Femtosecond Laser Ablation Inductively Coupled Plasma Time-of-Flight Mass Spectrometry.**; Jhanis Gonzalez[1], Vassilia Zorba[1], Dayana Oropeza[1], Travis Owens[1], Xianglei Mao[1], Richard Russo[1]; [1]L. Berkeley National Laboratory

2:40 (661) **Combined LA-ICP-MS and Raman Microspectroscopy for Pharmaceutical and Nutraceutical Imaging Analysis**; Todor Todorov[1], Alan Koenig[1]; [1]US Geological Survey

3:00 (662) **Liquid Nitrogen Cooled Stage for Laser Ablation Inductively Coupled Plasma Mass Spectrometry**; William Hoffmann[1], Aaron Hart[1], Dr. Guido Verbeck[1]; [1]University of North Texas

3:20 (663) **Characterization of Genesis Solar Wind Sample Surface Contamination by Total Reflection X-ray Fluorescence Spectrometry**; Martina Schmeling[1], Munir Humayun[2], Donald Burnett[3]; [1]Loyola University Chicago; [2]Florida State University; [3]California Institute of Technology

3:40 (664) **Multi-Element RIMS Analysis of Genesis Solar Wind Collectors**; Igor Veryovkin[1], Emil Tripa[1], Alexander Zinovev[1], Bruce King[1,2], Michael Pellin[1], Donald Burnett[3]; [1]Argonne National Laboratory; [2]University of Newcastle, Australia; [3]California Institute of Technology

Thursday Afternoon, *Room 302B*
DROP DEPOSITION AND DYNAMICS
Organizer and Presider: Karen Esmonde-White

2:00 (665) **Blood, Sweat and Tears: What Can DCDRS Tell Us About Bodily Fluids?**; Nicholas Stone[1, 2], Catherine Kendall[1,2], Jacob Filik[1]; [1]Gloucestershir Hospitals NHS Foundation Trust, UK; [2]Cranfield University, UK

2:20 (666) **Drops on Superhydrophobic Surfaces Provide New Analytical Tools**; Noah Weiss[1], Antonio Garcia[2], Mark Hayes[1]; [1]Arizona State University, Chemistry; [2]Arizona State University, Bioengineering

2:40 (667) **Morphological and Chemical Analysis of Dried Biofluid Drops**; Karen Esmonde-White[1], Francis Esmonde-White[2], Blake Roessler[1], Michael Morris[2]; [1]University of Michigan Medical School; [2]University of Michigan

3:00 (668) **Towards the Use of Levitated Drops as Microreactors to Study Enzyme Kinetics**; Alexander Scheeline[1], Zakiah Pierre[1], Oluwafemi Masha[1], Edward Chainani[1]; [1]University of Illinois at CU

3:20 (669) **Quantitative Protein Characterization Using Drop Coating Deposition Raman Spectroscopy**; Dongmao Zhang[1]; [1]Mississippi State University

3:40 (670) **Analysis of Rapidly Solidified TiO2 Droplets by Micro-Raman Spectroscopy**; Christopher Young[1], Jose Colmenares-Angulo[1], Giovanni Bolelli[2], Valeria Cannillo[2], Luca Lusvarghi[2], Clive Clayton[1]; [1]Stony Brook University; [2]University of Modena and Reggio Emilia

Thursday Afternoon, *Room 302C*
DEVELOPMENTS AND APPLICATIONS IN BIOLOGICAL MASS SPECTROMETRY
Organizer and Presider: Adam M. Hawkridge

2:00 (671) **Mass Spectrometry for Analyzing the Proteome of a Small Number of Cells**; Liang Li[1]; [1]University of Alberta

2:20 (672) **Microchip Separations with Integrated Electrospray Ionization**; J. Scott Mellors[1], Andrew Chambers[1], J. Michael Ramsey[1]; [1]University of North Carolina at Chapel Hill

2:40 (673) **A Proteomic Perspective of the Dynamic Interplay between Viruses and Hosts**; Ileana Cristea; [1]Princeton University

3:00 (674) **Isotopic Labeling Strategies for Quantitative Glycomics**; Ron Orlando[1]; [1]CCRC, University of Georgia; [2]BioInquire; [3]Walter Reed Army Med Cntr; [4]Windber Research Institute

3:20 (675) **Mass Spectrometry-Based Proteogenomic Approaches Reveal Insight into the Activities and Functions of Microbial Isolates and Communities**; Robert Hettich, Alison Russell[1], Brian Erickson[1], Chongle Pan[1], Nathan VerBerkmoes[1], Jillian Banfield[2]; [1]Oak Ridge National Lab; [2]University of California - Berkeley

3:40 (676) **Label-Free Mass Spectrometry-Based Proteomics Study of Ovarian Adenocarcinoma in the Chicken**; Adam Hawkridge[1], Rebecca Wysocky[1], James Petitte[1], Kenneth Anderson[1], Paul Mozdziak[1], Oscar Fletcher[1], Jonathan Horowitz[1], David Muddiman[1]; [1]NC State University

Thursday Afternoon, *Room 304*
2010 CRAVER AWARD SYMPOSIUM HONORING PROFESSOR BORIS MIZAIKOFF
Organizer: Boris Mizaikoff; Presider: Scott Little

2:00 (677) **Standoff Detection Using Raman Spectroscopy: Current Status and New Directions**; S. Michael Angel[1], J. Chance Carter[1]; [1]University of South Carolina

2:20 (678) **Integrated Optical Sensing: Microresonators, Metamaterials, and Chip Scale Integrated Optical Systems**; Nan Jokerst[1]; [1]Duke University

2:40 (679) **Planar Waveguide ATR Spectroscopy in the Time- and Frequency-Domains: Development and Application to Photochemical and Electrochemical Reactions in Molecular Films**; S. Scott Saavedra[1], Zeynep Ozkan Araci[1], Anne Simon[1], Anne Runge[1], Walter Doherty[1], Clayton R. Shallcross[1], Neal R. Armstrong[1]; [1]University of Arizona

3:00 (680) **RIfS: A Versatile Biosensor Technology for Label-Free Detection**; Guenther Proll[1]; [1]University of Tuebingen

3:20 (681) **Raman Spectroscopy for Environmental Analysis**; Karl Booksh[1]; [1]University of Delaware

3:40 (682) **Recent Advances in Surface-Enhanced Infrared Spectroscopy**; Peter Griffiths[1], Ayuba Fasasi[1]; [1]University of Idaho

Thursday Afternoon, *Room 305A*
CONTRIBUTED CAPILLARY ELECTROPHORESIS
Organizer and Presider: Neil Danielson

2:00 (683) **Integrated Probing of Protein Purity and Conformation by Capillary Electrophoresis with Wavelength-Resolved Fluorescence Detection**; Bregje J. de Kort[1], Gerhardus J. de Jong[1], Govert W. Somsen[1]; [1]Utrecht University, the Netherlands

2:20 (684) **Characterization of an Automated Capillary Electrophoresis System for Single Cell Analysis**; Alexandra J. Dickinson[1], Dechen Jiang[1], Christopher E. Sims[1], Nancy L. Allbritton[1, 2]; [1]University of North Carolina; [2]North Carolina State University

2:40 (685) **Investigation of Electroosmotic Flow Dynamics in Response to Biological Sample Introduction for Capillary Electrophoresis**; Funda Kizilkaya[1], S. Douglass Gilman[1]; [1]Louisiana State University Department of Chemistry

3:00 (686) **Single Cell Analysis of EGFR Activity by Capillary Electrophoresis**; Ryan Phillips[1], Nancy Allbritton[1,2,3], David Lawrence[1,2]; [1]Department of Pharmacology, UNC Chapel Hill; [2]Department of Chemistry, UNC Chapel Hill; [3]Joint BME Program, UNC Chapel Hill/ NCSU

3:20 (687) **Particle Isolation by Insulating Gradient Dielectrophoresis (iDC-GDEP)**; Sarah Staton[1], Kang Ping Chen[2], Tom Taylor[3], Jose Rafael Pacheco[2, 4], Mark Hayes[1]; [1]Arizona State University Dept. of Chemistry; [2]Mechanical and Aerospace Engineering; [3]Mathematics and Statistical Sciences; [4]Center for Environmental Fluid Dynamics

3:40 (688) **Magnetic Bead Microreactors for Studies of On-Column Two-Enzyme Reactions**; Rachel Henken[1], S. Douglass Gilman[1]; [1]Louisiana State University

Thursday Afternoon, *Room 305B*
BIOLOGICAL AND BIOMEDICAL APPLICATIONS OF RAMAN SPECTROSCOPY
Organizer Ian Lewis: Presider: Francis Esmonde-White

2:00 (689) **Developing a New Toolbox for Analysis of Warrior Wound Biopsies: Vibrational Spectroscopy**; Nicole Crane[1], Eric Elster[1,2,3]; [1]Naval Medical Research Center; [2]National Naval Medical Center; [3]USUHS

2:20 (690) **Raman Spectra of Scars**; Adrian Eugenio VIllanueva Luna[1], Jorge Castro Ramos[1], Sergio Vazquez-Montiel[1], Jose Alberto Delgado Atencio[1]; [1]Instituto Nacional de Astro., Op y Elec.(INAOE)

2:40 (691) **Raman Spectroscopy of Neonatal Mouse Skull Soft Spot Reveals Cyclic Mineral Deposition Dynamics**; John-David McElderry[1], Guisheng Zhao[2], Renny Franceschi[2], Michael Morris[1]; [1]Department of Chemistry, U of Michigan; [2]School of Dentistry, U of Michigan

3:00 (692) **Non Invasive Pathology the 'Raman Style': Detecting Breast Cancer by Probing Microcalcifications**; Marleen Kerssens[1,2], Keith Rogers[2], Pavel Matousek[3], Nick Stone[1,2]; [1]Gloucestershire Hospitals NHS Foundation Trust; [2]Cranfield University; [3]Central Laser Facility, RAL

3:20 (693) **Assessing Bone Fragility in Chemically-Aged Bone with Raman Spectroscopy**; Jessica Lopez[1], Gurjit S. Mandair[1], Michael D. Morris[1]; [1]University of Michigan, Department of Chemistry

3:40 (694) **Deep Ultraviolet Resonance Raman Spectroscopy of Formulated Insulin**; Sergey Arzhantsev[1], Connie Gryniewicz-Ruzicka[1], John Kauffman[1]; [1]US Food and Drug Administration

Thursday Afternoon, *Room 306A*
CHEMOMETRICS APPLIED TO AIR, OCEANOGRAPHY, MARINE LIFE AND COASTAL WATERS
Organizer: Barry Lavine; Presider: Gregory Hall

2:00 (695) **Application of Chemometric Techniques to Open-Path FT-IR Spectra Measured under Conditions of Severe Interference**; Peter Griffiths[1], Limin Shao[2], April Leytem[3]; [1]University of Idaho; [2]University of Science and Technology of; [3]U. S. Department of Agriculture

2:40 (696) **Airborne Monitoring of Volatile Organic Compounds by Passive Infrared Spectroscopy**; Gary Small[1]; [1]University of Iowa

3:00 (697) **Predicting Wastewater Treatment Efficacy**; Lisa Morkowchuk[1], Michael Krein[1], Alison Kennicutt[2], Curt Breneman[1], James Kilduff[2]; [1]Rensselaer Polytechnic Inst, Chemistry & Chem Bio; [2]Rensselaer Polytechnic Inst. Civil & Env

3:20 (698) **Multiway Analysis of EEMs to Analyze Microbial Carbon Cycling in Estuarine Waters**; Gregory Hall[1], Jennifer Edmonds[2]; [1]U.S. Coast Guard Academy; [2]University of Alabama

3:40 (699) **Using Lignin-Derived Phenols to Measure Terrestrial Organic Matter Inputs to Western Long Island Sound**; Eric Miller[1], Annelie Skoog[1], Greg Hall[2]; [1]University of Connecticut, Dept. of Mar. Science; [2]U.S. Coast Guard Academy, Science Dept

Thursday Afternoon, *Room 306B*
COMPUTER DIRECTED/SUPPORTED SPECTROSCOPY
Organizer and Presider: Don Pivonka

2:00 (700) **Computational Aspect of Two-Dimensional Correlation Spectroscopy**; Isao Noda; [1]The Procter & Gamble Company

2:20 (701) **Methodologies for Extracting Useful Insights from Spectral Images**; Curtis Marcott[1]; [1]Light Light Solutions

2:40 (702) **Transfer and Implementation of Chemometric Models**; John Wasylyk[1], Ming Huang[1], Robert Wethman[1]; [1]Bristol-Myers Squibb Co.

3:00 (703) **Calculation of Vibrational Spectra for the Assignment of Chemical Structure**; Don Pivonka[1], Steven Wesolowski[1]; [1]AstraZeneca

3:20 (704) **Analysis of Data Arrays from Hyphenated Instrumentation - Multi-Way Methods Such as Parallel Factor Analysis (PARAFAC)**; Karl Booksh[1]; [1]University of Delaware

3:40 (705) **Mid-Infrared Imaging as a Label-Free Alternative to Immunohistochemistry for Breast Cancer Pathology**; Michael Walsh[1], Andre Kajdacsy-Balla[2], Rohit Bhargava[1]; [1]University of Illinois at Urbana-Champaign; [2]University of Illinois at Chicago

Thursday Afternoon, *Room 306C*
PHARMACEUTICAL RAMAN
Organizer and Presider: Ian Lewis

2:00 (706) **Standardization of Raman Spectral Library Methods for Rapid Screening of Pharmaceutical Raw Materials**; Jason Rodriguez[1], Lucinda Buhse[1], Benjamin Westenberger[1], John Kauffman[1]; [1]FDA Div. of Pharmaceutical Analysis, St. Louis, MO

2:20 (707) **Raman Analysis of Pharmaceutical Powders and Tablets**; David Littlejohn[1], Pamela Allan[1], Luke Bellamy[1], Alison Nordon[1], Nichola Townshend[1], John Andrews[2], Paul Dallin[2]; [1]University of Strathclyde; [2]Clairet Scientific

2:40 (708) **Using PCA to Understand Raman-DSC Data**; Richard Spragg, Kevin Menard; [1]PerkinElmer LAS, Shelton, CT

3:00 (709) **Assessing the Sample Heterogeneity and Determining the Minimum Sampling Ratio for the Healthy Statistical Representation using Raman Spectroscopy**; Eunah Lee[1], Sergey Mamedov[1], Fran Adar[1], Andrew Whitley[1]; [1]HORIBA Scientific

3:20 (710) **Accuracy of Quantification of Pharmaceutical Formulations Using Transmission Raman Spectroscopy**; Matthew Bloomfield[1], Darren Andrews[1], Craig Tombling[1], Paul Loeffen[1], Pavel Matousek[1,2]; [1]Cobalt Light Systems; [2]Central Laser Facility, RAL, England

3:40 (711) **Calibration Transfer Across Multiple Portable Raman Spectrometric Instruments Used for Pharmaceutical Surveillance**; Connie Gryniewicz-Ruzicka[1], Sergey Arzhantsev[1], Benjamin Westenberger[1], Lucinda Buhse[1], John Kauffman[1]; [1]US Food and Drug Administration

(1) **Electroanalytical Eavesdropping on Cellular Communication**

Christy Haynes[1]; [1]University of Minnesota

Carbon-fiber microelectrochemistry methods afford unique chemical and biophysical insights into cellular secretion of chemical messenger molecules. Using single cell cyclic voltammetry, it is possible to identify the chemical species secreted while amperometry reveals details about chemical messenger concentration, association with other species, cell membrane characteristics, and release kinetics. Herein, these two techniques are exploited for both fundamental and applied studies in cellular communication. Investigation of the fundamental properties of serotonin storage and release from blood platelets, the first real-time measurements of secretion from individual platelets, has revealed granular serotonin concentration, storage mechanism, and secretion driving forces as well as the role of membrane cholesterol in cell function. Additionally, carbon-fiber microelectrochemistry has been employed as a quantitative and direct measure of cell behavior following nanoparticle exposure. Measurements of this type have become critical as nanoparticle exposure increases drastically without sufficient toxicological data. In this work, the chemical messenger secretion behavior of control primary culture mast and chromaffin cells has been compared to cells exposed to noble metal nanoparticles. Detailed analysis of amperometric data facilitates not only an assessment of nanoparticle safety but also reveals the nanoparticle-cell interactions for future nanoparticle design considerations.

(3) **Dry Eye and Human Tear Lipid Compositional and Structural Relationships Using Spectroscopy**

Douglas Borchman[1], Gary Foulks[1], Marta Yappert[1]; [1]University of Louisville

Knowledge of tear film lipid compositional, structural, conformational and functional relationships could facilitate the development of therapies to alleviate symptoms related to meibomian gland dysfunction (MGD) and dry eye symptoms and to diagnose the disease. Toward this goal, we evaluated the wealth of information available from IR, NMR, Raman and MALDI-TOF mass spectroscopes to define tear lipid composition and structure relationships with age, sex and meibomian gland dysfunction. Spectroscopic methods are advantageous over chemical assays because the same sample can be used in multiple assays. Spectra of meibum from 41 patients diagnosed with MGD (Md) and 27 normal donors (Mn) were measured. We first measured lipid composition, and conformation using infrared and Raman spectroscopes. We then used 1H-NMR spectroscopy to quantify the major classes of lipid. MALDI-TOF mass spectroscopy was used as a final assay to quantify specific species of lipid. Principal component analysis was used to quantify the variance between the spectra. 1H-NMR confirmed our IR data showing wax esters were the predominant lipid in human meibum (57 %) and cholesterol esters were found to decrease by 21% with MGD. Using FTIR and Raman spectroscopes we found that changes in lipid composition induced a decrease in the strength of lipid-lipid interactions with age. At physiological temperature, lipid order (stiffness) decreased with increasing age. A training set of spectra were used to discriminate between Mn and Md with an accuracy of 93%. This shows that eigenvectors contain compositional and structural information about the changes that occur with MGD. Compared to normal age matched subjects, lipid order and phase transition temperature were significantly higher and similar to levels of meibum from younger normal subjects. By manipulating the cation and matrix composition added to tear lipid samples for MALDI-TOF MS analysis, we were able to quantify and identify lipid components such as cholesterol, phospholipids, hydrocarbons and wax esters with a sensitivity of 9 pmoles. This work highlights the power of spectroscopy to characterize tear film lipid composition-structure relationships with age and dry eye symptoms.

(4) **Development of a Peptidase-Resistant Reporter to Measure BCR-ABL Kinase Activity**

Angela Proctor[1], Qunzhao Wang[1], David Lawrence[1], Nancy Allbritton[1,2]; [1]University of North Carolina at Chapel Hill; [2]North Carolina State University

Patients with chronic myelogenous leukemia (CML) often have a chromosomal aberration caused by a reciprocal translocation between chromosomes 9 and 22. This results in formation of a fusion protein known as BCR-ABL, which increases the tyrosine kinase activity of the ABL protein, allowing for increased survival and proliferation of tumorigenic cells. Imatinib mesylate (Gleevec) and other second and third generation relatives have been developed that specifically inhibit the BCR-ABL protein and are effective treatments for CML. Current technology for determining the presence of BCR-ABL focuses on identifying the mutant DNA, for example, by karyotyping, fluorescence *in situ* hybridization (FISH) or PCR techniques. However, these methods can be time-consuming and do not measure the activity of the fusion protein itself. The activity of BCR-ABL in cells shows considerable variability between patients and patients can also develop resistance to Gleevec or its relatives. Thus, a rapid tool to measure the activity of BCR-ABL in single cells from a patient would be of high value for both diagnostics as well as individualized therapy. This work focuses on the development of a reporter to measure BCR-ABL kinase activity in single patient cells. We have developed a peptide substrate that is resistant to degradation by intracellular peptidases and is efficiently phosphorylated by ABL kinase. Capillary electrophoresis of synthesized peptide fragments was used to determine the location of initial peptidase cleavage sites following incubation of the peptide in a cell lysate. The peptide reporter was then stabilized against peptidase activity using an iterative process to substitute non-native amino acids into the sequence at the residues adjacent to the cleavage site. The stabilized reporter's half-life in a cell lysate with unrestricted protease activity was 19.07 +/- 3.35 minutes, while a control peptide in the same lysate possessed a half-life of 1.31 +/- 0.36 minutes. Capillary electrophoresis was also used to quantify the efficiency of reporter phosphorylation by ABL kinase. Future studies include single-cell level characterization of the reporter in model cell lines with varying BCR-ABL activity and in primary patient cells.

(5) **Enhanced Glycan ESI Response Using Neutral Hydrazide Reagents via Hydrophobic Tagging towards the Profiling of Cleaved N-linked Glycans**

Hunter Walker[1], Brian Papas[1], Daniel Comins[1], David Muddiman[1]; [1]North Carolina State University

This study has shown the development, optimization, and application of novel hydrazide reagents toward the hydrophobic tagging and increased electrospray response of proteoglycans in nanoLC-FTICR mass spectrometry. The hydrophobicity of tagging reagents has been exploited often in proteomics (ALiPHAT), and this study demonstrates the application to glycans. Also, it has been assumed that a permanent charge on the reagent molecule will facilitate gas phase ionization, yet here, it is shown that with all other properties held constant, the permanent charge is detrimental to glycan analysis. Two different pairs of charged and neutral reagents (one pair synthesized at NC State and the other commercially available) clearly indicate that a permanent charge decreases the abundance of the tagged glycan relative to its neutral counterpart, and, as hypothesized, an increase in the hydrophobicity of the tagging reagent results in increased electrospray response of the derivatized glycan. In this study, the hydrophobicity of the

reagents has been estimated using non-polar surface area calculations, and each pair of molecules has been calculated to have the same NPSA (regardless of charged or neutral), and thus hydrophobicity. This validates the fact that the permanent charge is causing the decrease in signal. The coupling reaction (via hydrazone formation) has been optimized, and the reaction efficiency of complex glycans is greater than 95%, requiring no clean-up stage before introduction onto the nanoLC column. It is shown that though a large excess of reagent must be used, the tagged glycans out-compete the free reagent in the electrospray droplet, and the measurement is not affected. In addition, using the most effective tag, standard glycoproteins were separated using SDS-PAGE and the glycans were cleaved from the proteins "in-gel," cleaned up using SPE, and tagged. All glycans detected were tagged, confirming complete reaction efficiency. This provides a viable avenue to measure the glycome of a specific sample and directly relate the detected glycans back to a specific protein, thus delving deeper into the relation between glycosylation and protein abundance.

(6) **Detection of Mycoplasma pneumoniae in a Clinical Background by Surface-Enhanced Raman Scattering Spectroscopy**

Suzanne Hennigan[1], Jeremy Driskell[1], Yiping Zhao[1], Richard A. Dluhy[1], Duncan C. Krause[1]; [1]University of Georgia

Mycoplasma pneumoniae(MPN) is an important human pathogen, causing 20% of the community-acquired pneumonias in adults; furthermore, it is the primary cause of atypical pneumonia and tracheobronchitis in children aged 5 - 9. Common symptoms include fever, a non-productive cough, malaise, and headache. Current detection strategies for MPN include enzyme-linked immunoassays (ELISAs) and immunofluorescence antibody assays; however, these methods suffer from low sensitivity and poor detection limits. We have evaluated surface enhanced Raman spectroscopy as a clinical method for detection and identification of MPN with species and strain specificity. Our laboratories have developed an oblique angle vapor deposition protocol to prepare aligned high aspect ratio Ag nanorod array substrates that yield SERS enhancement factors of >10E8. This nanorod array (NA)-SERS platform, when used in conjunction with chemometric analysis, has been shown to exhibit outstanding sensitivity and specificity for detection and differentiation of viral pathogens. In this work we demonstrate the capacity of NA-SERS to detect and distinguish closely related MPN strains in pure culture and in mock-infected throat swab samples.

(7) **Application of Support Vector Regression in Non-Invasive Blood Glucose Detection Using Raman Spectroscopy**

Ishan Barman[1], Narahara Chari Dingari[1], Jeon Woong Kang[1], Chae-Ryon Kong[1], Ramachandra R. Dasari[1], Michael S. Feld[1]; [1]Massachusetts Institute of Technology, MA

Failure to adequately regulate blood glucose may lead to both acute (ketoacidosis) and chronic (diabetic retinopathy) health complications making frequent monitoring of glucose levels critical. Near-infrared Raman spectroscopy, which provides the dual advantage of substantial penetration depth and excellent chemical specificity, provides an important tool to accomplish this in a painless and non-invasive manner. However, despite encouraging results in serum and whole blood samples, the development of a clinically accurate and robust algorithm capable of prospective prediction in a human population has proven to be difficult. Multiple factors can be attributed to the current difficulty in achieving successful calibration transfer including variations in tissue absorption and scattering (turbidity), ambient temperature, autofluorescence levels and associated quenching and differences in glucose concentrations in the blood and interstitial fluid compartments, in addition to chance spurious correlations. To develop robustness in the calibration models, we propose the application of kernel-based support vector regression. This shift from the conventional linear calibration schemes is based on the understanding that the linearity assumption between the spectral and concentration datasets may fail under the influence of fluctuations in the aforementioned process and system variables. In this talk, we will present our first results showing that support vector regression provides a significant improvement over the PLS models with an increase in correlation between the actual and predicted concentrations by nearly 40% and a concomitant decrease in the prediction error values by a factor of two or more, when multiple human subjects are included in the analysis. We also observe that PLS implementation on the human subject dataset clearly shows the presence of curved effects in the spectra-concentration relationship, validating our understanding and providing the reason for lack of robustness in linear models. Furthermore, we show for the first time that application of support vector regression enables Raman spectroscopy to provide clinically accurate predictions in the hypoglycemic range in human subjects.

(8) **Cell Labeling with Plasmonic Particles**

George Chumanov[1], Kyle Dukes[1]; [1]Clemson University

Silver nanoparticles (NPs) exhibit large scattering cross section in the visible spectral range due to the excitation of plasmon resonances. In fact, the excitation of plasmon resonances in Ag NPs represents the most efficient mechanism for the interaction of light with matter. For these reasons, these particles are nearly ideal optical labels for bioanalytical applications including cell sorting. Here, biocompatible optical labels based on Ag NPs encapsulated into a silica shell with a hydrophobic barrier and further modified with neutravidin are reported. The silica shell and the hydrophobic barrier afford long term stability of the particles in physiological solutions. Neutravidin provides a universal linker to any bitinalated proteins including antibody. Other advantages of these labels include the photochemical stability, the lack of photoblinking, and the possibility for single particle detection. These labels were applied in flow cytometry applications and demonstrated two orders of magnitude improvement of scattering detection as compared to the nonlabeled cells.

(9) **Acid Cleavable Tagging for Biomolecule Analysis**

Dongmao Zhang[1], Karthikeswar Vangala[1], Michael Yanney[1], Shaoyong Li[1], Sygula Andrzej[1]; [1]Mississippi State University

Protein and dye conjugation is probably the most widely used strategy in biomedical and biological research. Various fluorescence-based bioanalytical and biophysical techniques have been developed for studying protein folding, antibody-antigen binding and comparative proteomic analysis, etc. Because of their extended *f*à conjugation electrons, dye molecules usually have large normal Raman and surface enhanced Raman spectroscopic (SERS) activities and thus might serve as Raman tags for sensitive Raman and SERS detection of proteins and protein modifications. However, despite the wide availability of dye-conjugated proteins and tremendous interests in developing ultrasensitive bioanalytical method, there are very few reports claiming successful SERS detection of protein-dye conjugates, which is in stark contrast to the ultrahigh sensitive SERS detection (single molecule in some cases) commonly reported with free dyes. Our previous research demonstrated that, because protein compete with dye for direct absorption onto SERS substrate, the presence of protein drastically reduces even entirely eliminates the SERS signal intensity of the dye molecules that is either covalently conjugated to protein or mixed with a protein solution. To explore ways for effective translation of the dye SERS sensitivity for protein-dye conjugate,

we recently developed a novel acid cleavable SERS tagging (ACST) strategy for SERS detection of protein-dye conjugates. Because of the dye molecule can be cleaved and separated from protein before its SERS spectral acquisition, the overall SERS detection sensitivity of the protein-dye conjugation is drastically improved. In addition to the ACST tag design, preparation and its SERS spectral features, we will also discuss the potential applications of this new biomolecule tagging method in a wide range of bioanalytical problems.

(10) **Low-Shear Microfluidic Devices for Cell Culture and Analysis**

Dimitri Pappas[1]; [1]Texas Tech University

Microfluidic methods have proven to be robust and powerful technologies for cell analysis. Affinity separations, cell lysis, cell culture, and analysis of cellular products have all been realized using novel lab-on-a-chip approaches. Our lab has incorporated cell separations, chemical separations, and laser spectroscopy to interrogate cultured cells. A new form of cell separations, Differential Mobility Cytometry (DMC), has been developed for cell analysis. DMC uses a novel separation approach for cell capture, coupled to sensitive fluorescence measurements. DMC will be demonstrated for surface adhesion, cell detachment, apoptotic cell capture, and other applications. Fluorescence monitoring of intracellular processes of DMC-captured cells will elucidate the temporal mechanics of several biological processes.

(11) **Application of GA-ANN for Prediction of the Selectivity Coefficients for the MIP Based Potentiometric Sensor**

Mahmood Reza Sohrabi[1], Pegah Nezakati[1], Meharn Javanbakht[2], Hoda Pasdar[1]; [1]Azad University, North Branch, Tehran, Iran; [2]Department of Chemistry, Amirkabir University

A MIP based potentiometric sensor has the ability of discerning between hydroxyzine and the other metabolites with similar structure, being present in tablets and biological fluids (e.g.cetrizine). A quantitative structure-property relationship (QSPR) study was applied to provide a model to predict the matched potential method (MPM) selectivity coefficients for the hydroxyzine ion-selective sensor by relating the structures of compounds to their . The QSPR model provides significant additional insight into the relationship between the molecular structure and fundamental processes and phenomena in chemistry. In order to calculate the theoretical descriptors, molecular structures were drawn with Hyperchem version 6.0. Theoretical molecular descriptors were calculated by Dragon 2.1 software. A genetic algorithm was written in MATLAB (version 7.5, MathWorks Inc.) environment was used to select the best subset of descriptors. 15 descriptors were employed as input for generated 15-5-1 artificial neural network (ANN). A model was built up by using artificial neural network. Artificial neural network as a non-linear technique is a quite popular technique which has surprising ability to model properties of interest. A commonly used architecture is a three-layer feed-forward network which is comprised of (i) input layer, (ii) hidden layer, and (iii) output layer. In this study, the correlation between experimental and predicted values of selectivity coefficients was equal to 0.98, indicating an excellent correlation between the predicted and experimental values of the .In the end, for validation of the constructed model by ANN, cross validation and external validation procedures were utilized. The proposed method, due to low values of root mean square error of cross validation (RMSECV) and relative error of prediction set (REP) has a high predictive ability and could be useful aid to the costly and time-consuming experiments for determining the selectivity coefficients for the MIP based potentiometric sensors.

(12) **Facilitating the Teaching of Chemometrics through the Cyber-Enabled Virtual Chemometrics Lab**

Edward Duranty[1], Rebecca Horton[1], Morgan McConico[1], Frank Vogt[1]; [1]University of Tennessee, Department of Chemistry

Adequate preparation of the next generation of analytical chemists mandates the coverage of multivariate data analyses on both upper-division undergraduate and 1st year graduate level. However, three practical issues hinder a broad introduction of chemometrics into the analytical curriculum. (1) There is a lack of introductory teaching material, i.e. sufficiently detailed chapters in analytical standard textbooks as opposed to texts solely dedicated to chemometrics. Furthermore, exposing students to real-world situations/applications strengthens their career readiness. However, obtaining large, experimental, multi-component data sets requires a more laborious effort than generating simulated data. (2) Presenting all mathematical details of chemometric algorithms to a student audience requires a time-intensive math review. Commercial software packages are designed to alleviate this but typically offer more options than necessary and are thus distracting for introductory purposes. (3) Licensing costs for commercial chemometric or programming packages can be prohibitive for classroom settings. Thus, mathematical details of chemometric algorithms and complex software packages should be postponed for the introductory level and straightforward computation tools along with experimental data sets must be made available to students. In order to overcome these restrictions, the innovative 'Virtual Chemometrics Lab' has been developed which is a remotely-accessible, multi-user tool and shields the algorithmic details from the students via a graphical user interface. Furthermore, it is supported by a chemometrics primer which provides sufficient technical details to understand basic algorithms like Classical Least-Squares (extended Beer's Law), Principal Component Regression and Partial Least-Squares along with application-dependent data pre-processing methods. To be real-world relevant, this Virtual Chemometrics Lab has been coupled to a pre-compiled database containing ~1500 FTIR spectra acquired from 30+ analytes for teaching, homework and experimental purposes. Utilizing this software package will facilitate teachers to efficiently introduce chemometrics and will allow students to independently investigate the power of multi-component analyses which preserve selectivity even in the case of cross-sensitive signatures of multiple analytes. These tools enhance student training through applications which are more relevant and powerful than univariate Beer's Law. Instructors can easily design their own hands-on experiments based on the material covered in class without investing unreasonable amounts of time and cost.

(13) **Comparing Variable Selection in Multivariate Model Development Using "Classical" and "Continuous" Haar Wavelet Transforms Coupled with Genetic Algorithm Optimization**

Lucy Botros[1], Kevin Judge[1]; [1]Molecular Biometrics Inc.

Previously published methods on parsimonious multivariate model development of NIR spectral data have incorporated a son Haar wavelet transform and genetic algorithm (GA) search method to objectively optimize variable selection. While this method has been shown to be effective, the 'classical' son Haar wavelet transform is limited as it does not incorporate continuous spectral information. A 'continuous' Haar wavelet transform creates a greater number of overlapping wavelet variables of identical window size that contain more chemically relevant information, while discarding many irrelevant 'classical' wavelets. Although the 'continuous' method increases the computational time when coupled with the GA, it increases the probability of developing a more analytically accurate model. When applied to modeling efforts using NIR spectral data

collected from differing embryo culture media, validation of models that used 'continuous' wavelet transforms resulted in greater predictive power than models developed using the 'classical' method.

(14) **On-line Chiral Analysis of Trace Biomarkers by UPLC-QToF Mass Spectrometry Using the Kinetic Method with Post-Column Derivatization**

Yong-Ill Lee[1], Hua Jin[1]; [1]Changwon National University

A novel method was developed for chiral analysis utilizing on-line ultra performance liquid chromatography in combination with quadrupole time-of-flight mass spectrometry (UPLC-QToF-MS). This newly developed method was successfully applied to the discrimination and accurate quantification of chiral biomarkers, D/L-phenylmercapturic acid (PMA) and D/L-benzylmercapturic acid (BMA). The approach was established on the Cooks' single ratio kinetic method, which is in turn based on the kinetics of competitive unimolecular dissociation of transition metal complexes. Initially, UPLC was used for separation of the mixture of the two biomarker compounds and then the enantiomeric differentiation was successfully performed on-line by mass spectrometry. After the molecular separation of the two species is achieved, a mixture of appropriate metal ion and reference ligand were added on-line to the eluents for post-column derivatization to produce trimeric M(II)-bound complex ion, [MII(A)(ref*)2-H]+ (MII, divalent transition metal ion; A, analyte; ref*, reference ligand) for the biomarkers. Collision-induced dissocation of this trimeric complex ion resulted in two dimeric complex ions, [MII(A)(ref*)-H]+ and [MII(ref*)2-H]+. The ratio of the abundances for the two product ions, that is, the branching ratio was established for one enantiomer and it was expressed relative to that for the other enantiomer, which effectchiral discrimination. Cu(II)-L-Pro and Mn(II)-Gly-Phe are selected for the optimum distinction of chiral PMA and BMA. Rapid quantitative chiral analyses of PMA and BMA were achieved by constructing calibration curves derived from the kinetic method, related to the ratio of the branching ratios against the enantiomeric composition of their mixture.

(15) **Optimized Spectral Data Fusion**

Heather Brooke[1], Kevin Johnson[1], Christian Minor[2]; [1]US Naval Research Laboratory; [2]Nova Research, Inc.

Currently there is no systematic framework for characterizing fused, multisensory systems, and therefore the comparison of multiple independent systems is difficult without extensive field-testing. Development of a framework would allow for theoretical comparisons and enable more rapid prototyping of fused sensor systems, guidance for design from existing sensor components, and more effective engineering of new sensors optimized for use in fused sensor systems. Recent research at NRL has focused on characterizing Fourier transform infrared spectroscopy (FTIR) and mass spectrometry data for fused, multisensor applications to enhance chemical detection and discrimination in the presence of complex interfering backgrounds. An information theoretic approach has been used to elucidate the information content available from spectral data, quantify the ability of these sensing techniques to distinguish chemicals, and determine their susceptibility to noise and resolution limitations. The approach has also been applied to feature extraction and data fusion techniques on these data. Results characterizing the effectiveness of a fused multisensor system combining FTIR and mass spectrometry are presented.

(16) **Adaptive Regression by Subspace Elimination**

Bryon Herbert[1], Karl Booksh[1]; [1]University of Delaware

Adaptive Regression by Subspace Elimination enables the reliable estimation of analyte concentration with vibrational spectroscopy when uncalibrated spectral interferents are present within future samples. Utilizing mid-infrared spectroscopy as a tool to analyze complex systems, chemically relevant spectral features are attributed to molecular vibrational modes of reagents, intermediates and products. Mathematical modeling of these features assigns quantitative information through the formulation and implementation of a calibration set. A prediction generated from a calibration model is only as accurate as the chemical variations attributed within the calibration spectral set. A regression model is subject to errors when an uncalibrated chemical species is found within a prediction set. A method was developed to adaptively isolate and omit the inclusion of uncalibrated chemical features found within each spectrum of the prediction set. By transforming a mid-infrared spectrum into the wavelet domain, a localized multi-resolution representation of the vibrational features is presented in a more isolated manner. Wavelet coefficients are selectively evaluated for inclusion or omission based on their presence and performance within the Partial Least Squares calibration model space. The model adapts and updates the sample space by reducing the outlying spectral features in the spectrum and a prediction is made on the remaining coefficients. Adaptive regression by subspace elimination has been applied to mid-infrared spectra hydrocarbon mixtures and demonstrated a 15% improvement in the precision of the calibration model when no spectral interferents are present and a 90% reduction in the impact of spectral interferents on the quantitative performance of the calibration model.

(17) **Simultaneous Determination of Metformin Hydrochloride and Glibenclamide in Binary Mixtures Using Spectrophotometric Data and Wavelet Transform**

Naghmeh Kamali[1], Mahmoud Reza Sohrabi[1]; [1]Azad University, North Tehran Branch

In this work, a combined discrete and continuous wavelet transform analysis was developed for simultaneous spectrophotometric determination of metformin hydrochloride and glibenclamide, two antidiabetic drugs, in binary mixtures and pharmaceutical dosage form without any chemical pre-treatment. Absorption spectra of reference standard and test solutions were recorded over the wavelength range 200-400 nm. All spectra were subjected to the one-dimensional discrete wavelet transform (DWT) (3-level Daubechies 4 family) for signal de-noising. Various continuous wavelet transform (CWT) families were applied on these de-noised signals for choosing optimum wavelet processing conditions. Two CWT families, rbio 3.1 with scaling factor a=80 and gausian 2 with scaling factor a=60 were selected. Finally, a zero-crossing technique was used for construction of calibration curves for both drugs. The proposed method were validated by analyzing twelve synthetic mixtures of the investigated drugs with various concentrations in linear calibration region,between 1 and 20 mg/L. The amount of metformin hydrochloride and glibenclamide were determined by using CWT amplitudes in zero-crossing points. The results showed that this method can predict the concentrations of both drugs precisely and accurately. The model was used for determination of drugs in pharmaceutical dosage form. The results of accuracy and precision tests showed a good accordance between real and calculated concentrations of both drugs. The obtained results showed that the developed method is a simple, rapid and precise procedure for simultaneous determination of metformin hydrochloride and glibenclamide in binary mixtures and pharmaceutical dosage forms.

(18) **Recognizing Patterns in Single Molecule Fluorescence Microscopy Using Multivariate Analysis**

Nathan Skinner[1], Gerhard Prinz[1], Derek Bailey[1], Jared Kindt[1], Madison Taylor[1], Michael Culbertson[1], Daniel Burden[1]; [1]Wheaton College

In single-molecule tracking experiments, photophysical and kinetic differences between species are often small. This obscures species identification and prohibits analyses in complex media, such as lipid and cell membranes. We are developing an algorithm to analyze single-molecule fluorescent microscopy videos for up to fifteen observable parameters related to particle kinetics, spot shape, and intensity. We use the output from the algorithm in conjunction with multivariate analysis techniques such as PCA and SIMCA to classify single molecules diffusing in 2D. This presentation demonstrates how pattern recognition can be used to identify individual molecular components, extending the repertoire of single-molecule analysis tools beyond traditional histograms. Theoretical testing of simulated fluorescent microscopy videos confirms that this technique can be used to identify closely related species. We also show results from TIRF and wide-field fluorescence microscopy where the approach is applied to two fluorescent species diffusing in a lipid membrane.

(19) **A Model Based upon H-point Technique for Simultaneous Spectrophotometric Determination of Dextromethorphan HBR and Psedoephedrin HCL in Farmacitical Formolation**

Mahmood Reza Sohrabi[1], Adeleh Ghobadi[2], Naser Goudarzi[3]; [1]Islamic Azad University,North Tehran Branch; [2]Azad University,North Tehran Branch; [3]Industrial Shahrod University

Dextromerthorphan-P Syrup is composed of two components : dextromethorphan HBR and psedoephedrin HCL. Conventional method for simultaneous determination of dextromethorphan HBR and psedoephedrin HCL is high liquid chromathography (HPLC) that is complicated and expensive moreover solvents in HPLC cause pollution in inviroment. Spectrometric analysis of this mixture is useless because of overlapping of their absorbance spectra. Chemometrics is the best method inorder to remove these limitation. One of this method that attention to is H-point standard addition method (HPSAM). (HPSAM) was applied to the simultaneous determination of dextromethorphan HBr (DXT) and psedoephedrin HCL (PSU) in dextromethorphan-P after optimizing working condition with appropriate selection of wavelength 203 and 210 (nm) , the proposed method was applied to the simultaneous determination of real sample and results were satisfactory. DXT and PSU can be determined simultaneously in the rang of 0.50-5.00 ƒÝgmL-1 and 0.4-8.00 ƒÝgmL-1 respectively with satisfactory accuracy and precision. A limit of detection (LOD) of DXT and PSU was obtained 0.33 And 0.72 ƒÝgmL-1 respectively for synthetic samples. The standard error (Er) of DXT and PSU in real sample was Obtained 2.2% and 4% respectively. calibration graph were linear with the correlation coefficients of 0.989 for both analytes. This procedure for simultaneous determination of DXT and PSU is sensitive and easy to perform. Key words: Dextromethorphan, pseudoephedrine , simultaneous determination , HPSAM . spectrophotometry.

(20) **ChemSpider – Building an Online Database of Open Spectra**

Antony Williams[1], Valery Tkachenko[1]; [1]Royal Society of Chemistry

ChemSpider is an online database of almost 25 million chemical compounds sourced from almost 400 different sources including government laboratories, chemical vendors, public resources and publications. Developed with the intention of building community for chemists ChemSpider allows its users to deposit data including structures, properties, links to external resources and various forms of spectral data. Over the past three years ChemSpider has aggregated almost 3000 high quality spectra and continues to expand as the community deposits additional data. The majority of spectral data is licensed as Open Data allowing it to be downloaded and reused in presentations, lesson plans and for teaching purposes.

(21) **Analysis of Anisotropic Local Surface Plasmon in a Thin Film of Gold Nano-Particles Studied by Visible Multiple-Angle Incidence Resolution Spectrometry**

Takeshi Hasegawa[1], Akiyoshi Kasuya[1], Yuki Itoh[1], Tetsuo Okada[1], Masatoshi Osawa[2]; [1]Tokyo Institute of Technology; [2]Hokkaido University

A new combination technique of visible multiple-angle incidence resolution spectrometry (Vis-MAIRS) and hetero two-dimensional correlation spectroscopy (hetero-2DCOS) is proposed for analysis of characters of light absorption by local surface plasmon (LSP) in a metal thin film prepared by vacuum deposition. Plasmon absorption by metal-particle aggregates has long been studied by comparing with idealized theoretical models considering single/twin particles or well-defined particle arrays. The idealized models are far from the structure of the evaporated thin films, in which metal particles with a different size and shape are randomly dispersed or connected. To explore the LSP in the film, anisotropic absorptions by plasmon parallel (IP) and perpendicular (OP) to the film should be studied, for which Vis-MAIRS is one of the most suitable measurement techniques. In the present study, the spectral variations of a gold thin film in an aging process appeared in both MAIRS-IP and -OP spectra have been found largely different from each other. Since both spectra are derived from an identical sample, they were preferably applied to the hetero-2DCOS analysis, which has unveiled a deep insight involved in the complicated spectral changes. As result, the absorption bands near 520 nm in both IP and OP spectra were attributed to the quadrupole interaction of plasmon, whereas the band 676 nm available only in the IP spectrum was assigned to the dipole interaction.

(22) **Food Authenticity Determination by Total Organic Carbon Isotope Analysis Using a Combine TOC-Cavity Ringdown Spectrometer (CRDS) Instrument**

Garrett Slaton[1], Jeff Lane[1], Richard Simon[1], Gary Engelhart[1], Trent Sprenkle[1]; [1]OI Analytical

As food sources become more global, the importance of authenticating and tracing the origins of foodstuffs increases. One method for food authentication is fingerprinting by stable-isotope measurements using isotope ratio mass spectrometry (IRMS). These measurements have been used to determine both the authenticity of and the geographic origin of foodstuffs such as fruit juices, honey, butter, and wines. This poster will present a new approach to foodstuff authentication using a hybrid instrument combing a total organic carbon (TOC) analyzer and in-board computer-controlled gas dilution apparatus with a cavity ringdown spectrometer (CRDS) tuned to observe the 13CO2/12CO2 ratio in sampled gases derived from both solid and liquid samples. This hybrid instrument provides a level of isotope ratio measurement precision comparable to that of an IRMS instrument, with significantly less cost of ownership and greater ease of use. Representative data will be presented demonstrating the precision and accuracy of the measurements obtained from this technique on olive oil, honey, and maple syrup, which are desirable targets for adulteration, along with comparisons to traditional IRMS data.

(23) **Semi - Open Focussed Microwave Methodology for Fast Sequential Sample Preparation**

Bob Lockerman[1], David Barclay[1]; [1]CEM Corporation

Pressurized microwave digestion and solvent extraction technology has become widely accepted as a rapid and convenient method of sample preparation. Elevation of the reagent temperature above its' atmospheric boiling point results in rapid and complete digestion or extraction of the analytes of interest. However, traditional methodology for this type of instrumentation has been based on batch systems of closed pressurized vessels or single station open sequential instruments. Advantages for the analyst can be found in a combination of the instrumentation. This paper describes the development of a microwave heating platform and associated hardware specifically for analytical sample preparation which utilises the advantages of a focussed system – high watt density, individual control for each sample, in process focussed cooling – with the advantages of higher pressure vessels more commonly associated with the batch systems. Automation of the process will be demonstrated with an inbuilt control network allowing fast sequential processing of multiple samples requiring multiple reagents and methodologies. This automated control system allows a sample weight to volume ratio far in excess of any previous technology reducing reagent use and maximising analyte concentration. Results will be presented demonstrating the use of this methodology for real world samples as well as total digestions of reference materials. Significant reduction in the time of sample preparation will be shown with an improvement in ease of use and automation for the analyst.

(24) **Foreign Material Analysis in Pharmaceutical Forensics**

Cara Fowler[1]; [1]Eli Lilly and Company

Foreign material analysis can be challenging due to small sample size and complex matrices. However, identification of foreign material found in drug product, raw materials, and manufacturing equipment is critical to making quality decisions. Some of the techniques used in foreign material analysis include image analysis, optical microscopy, SEM/EDS, FTIR, and GC-MS. This presentation will cover the Forensics testing strategy at Eli Lilly and Company. Case studies involving some of the techniques listed will be presented.

(25) **Trace Drug Residue Analysis with Microscopy and Microextraction-Coupled to Nanospray Mass Spectrometry from Forensic Lifts**

William Hoffmann[1], Nicole Wallace[1], Dr. Guido Verbeck[1]; [1]University of North Texas

Forensic lifts, such as finger prints, electrostatic prints, and crime scene fibers are often analyzed for the print image only, and not chemical trace embedded or associated with evidence. The extraction of trace drug residues from various surfaces is often an arduous or unfeasible task due to the microscopic size of particles within the extracted matrix. Locating the drug particles for extraction may be difficult due to the type of surface material the particles may be imbedded in. Initial image probing with microscopy coupled with nanomanipulation-nanospray ionization mass spectrometry provides a great method to achieve trace particle extraction from challenging surfaces. Utilizing microscopy, the surface topography can be magnified sufficiently to locate drug particles trapped within so that extraction can take place. Differentiating between the drug particles and other trace debris within surface material can also be done with UV fluorescence or Raman imaging to allow the desired particle extractions to be made. Imaging of particles electrostatically lifted on metallic film or finger print lifts can be viewed and extracted with ease directly from the surface. Drug particles within fibers can be located and extracted skillfully without destruction of the material. The sensitivity of nanospray ionization-mass spectrometry is very prominent as to allow ample signal for single crystal extraction. Image probing-coupled with nanomanipulation-nanospray ionization-mass spectrometry provides a more effective method of drug particle extraction while still achieving high sensitivity with ultra trace particles.

(26) **Characterization of Zinc Carbonate Basic Using Multiple Techniques in Support of a Toxicological Investigation**

Amal Essader[1], Scott Afton[1], Keith Levine[1], Todd Ennis[1], Brenda Fletcher[1], Kelly Amato[1], Andrea McWilliams[1], Glenn Ross[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS

A critical component of any toxicological investigation is complete characterization of the test chemical. Inorganic test chemicals have unique properties and often require the use of several analytical techniques to achieve a successful characterization. Zinc carbonate basic, commonly used in water-based drilling fluids to scavenge hydrogen sulfide, is the test chemical in an ongoing investigation. In support of this study, the identity of the zinc carbonate basic bulk test chemical was unequivocally established, major impurities were identified, and stability at ambient and freezer temperatures was tested. To achieve these characterization objectives, multiple analytical techniques were used, including Karl Fischer titration for the determination of water content, combustion for carbon and hydrogen content, inductively coupled plasma optical emission spectrometry (ICP-OES) for quantitative metal analysis, x-ray diffraction (XRD) for the structural information, Fourier transform infrared (FTIR) spectroscopy for identification of functional groups, x-ray fluorescence (XRF) spectrometry for qualitative elemental determination, and thermogravimetric analysis (TGA) for evaluating purity as related to thermal decomposition behavior. Data collected from these analytical techniques signify that zinc carbonate basic is a stable compound and that no degradation is observed when comparing the ambient temperature (bulk) and frozen reference samples. In addition, results indicated that zinc oxide is the primary impurity with calcium and magnesium as minor impurities.

(27) **Preparation and Characterization of ZnO/PVC Nanocomposites and Study the Physical Properties for Their Complexes**

Islam Elashmawi[1], Nagwa Hakeem[1]; [1]National Research Centre

ZnO/PVC nanocomposites films have been prepared by solvent casting method and investigated by various techniques. All results show a good dispersion of ZnO nanoparticles in the polymeric matrix. XRD revealed that pure PVC films is partially crystalline with hallow peak but pure ZnO nanoparticles has Wurtzite structure and the nanocomposites films were almost the same as that of ZnO with decrease of the degree of crystallization and causes increase in the amorphous region. FT-IR presented the same spectra for nanocomposites in the wavenumber range of 700–3100 cmtext-1, weak band located at 500–700 cmtext-1 which attributed to stretching of Zn–O bond and an increase of the band of C=O at 1631 cmtext-1. The surface of the films was analyzed by SEM which becomes rough with some small aggregates compared with pure PVC with well distribution in the entire surface region with bright spots. TEM revealed a regular crystalline lattice superimposed on an amorphous background due to the carbon support and the PVC matrix and the structure of these particles is hexagonal. In addition, the nanocomposites films have higher glass transition temperature, specific heat and thermal stability related pure PVC because of strong interaction among ZnO nanoparticles and PVC.

(28) **Searching for a Needle in a Haystack, Extracting Valuable Information from Chemical Images**

Paulette Guillory-Gardner; [1]Thermo Fisher Scientific

Vibrational spectroscopy is a powerful tool for precise identification of materials. Coupled with microscopy the resulting technique is a powerful method of surface chemical analysis. FT-IR and Raman microspectroscopy are easily combined with post processing analytical methods resulting in specific identification and quantification of molecular species. Microspectroscopy coupled with a high precision stage allows for the creation of large chemical images that display the chemical properties of the video or mosaic image through user control of pixel representation. These pixels typically indicate a single spectrum and taken as a whole will create the chemical image. Chemical images contain many spectra, possibly up to several thousands of individual spectra. Most samples analyzed by vibrational techniques involve classification and identification and searching through thousands of individual spectra. Thermo Scientific offers a comprehensive mapping and imaging software program that comes with various post processing options to include principal components analysis, multivariate curve resolution, and simple area and peak mathematics. These can be combined with image analyses to extract valuable quantitative information from the map data. Using these techniques information such as specific identification of heterogeneous microscopic regions can be extracted from the data.

(29) **Near-Eye-Safe Trace Molecular Detection via SERS and SERRS at a Stand-Off Distance of 15 Meters**

Jonathan Scaffidi[1], Molly Gregas[1], Benoit Lauly[1], J. Chance Carter[3], S. Michael Angel[2], Tuan Vo-Dinh[1]; [1]Duke University, Biomedical Engineering and FIP; [2]Univ. of South Carolina, Dept. of Chem.; [3]Lawrence Livermore Nat'l Lab, M Division

We report the first demonstration of surface-enhanced Raman spectroscopy (SERS) detection of para-mercapto benzoic acid (pMBA) and surface-enhanced resonance Raman spectroscopy (SERRS) detection of brilliant cresyl blue (BCB) and cresyl violet perchlorate (CVP) with continuous-wave excitation from a stand-off distance of 15 meters. We further report the first stand-off SERRS detection of BCB and CVP at that same distance in the presence of ambient fluorescent and incandescent / blackbody background light. These preliminary results suggest that it is possible to detect sub-nanomole amounts of material at reasonable distances with eye-safe laser powers using stand-off SERRS, and serve as proof-of-concept highlighting the potential extension of stand-off Raman spectroscopy to include SERS and SERRS for remote, eye-safe chemical detection, analysis and imaging in the presence of ambient background light.

(30) **Spectral DSC: Probing Phase Transformations with Coupled Raman and DSC**

Stephen Medlin[1], John Richmond[1]; [1]Bruker Optics

Understanding chemical and physical properties of solids as they undergo thermally-induced transitions, such as polymorphic changes or solvate changes—are an important analytical need. Polymorphism is the ability of a chemical to exist as two or more crystalline phases with different molecular conformations. Solvates are crystalline solids that contain stoichiometric or nonstochiometric amounts of solvents that are incorporated into the lattice. Polymorph or solvate changes can result in dramatic differences in chemical and physical properties, such as melting point, chemical reactivity, optical properties, solubility, and so forth. In the pharmaceutical world, these changes can result in changes in bioavailability, toxicity, and so forth. Polymorphs in food applications, for example, can change melting, processability, and mouth taste. Differential Scanning Calorimetry (DSC) is typically used to measure heat flows associated with polymorphic and solvate changes. However, DSC may not fully characterize the transitions. Similarly, Raman or Near Infrared spectroscopy can be used to probe these changes, but these techniques do not provide information regarding the thermal or temporal dependence of these phase changes. Bruker Optics has coupled DSC with either Raman or NIR to form Spectral DSC. A Bruker Sentinel Raman spectrometer was coupled to a Q2000 DSC (TA Instruments) via a fiber-optically coupled probe. The Q2000 sample compartment was modified to accept the probe which was located over the sample. Various temperature profiles were applied and the resulting DSC data was collected simultaneously with Raman spectra. Spectral changes can be assigned to the various polymorph changes indicated by the DSC. The result is an analytical tool that can provide comprehensive information on these solid-state changes. In this talk, we present results of the Raman Spectral DSC for several applications. Spectral DSC will provide critical information in fields of pharmaceuticals, food, and polymers.

(31) **Use of an Inductively Coupled Plasma Atomic Emission Spectrometer as an Empirical Formula Detector for Gas Chromatography**

Carl Young[1], Meredith Lisle[1], Bradley Jones[1]; [1]Wake Forest University

An inductively coupled plasma atomic emission spectrometer (ICP-AES) will be used to detect crude extracts from natural product samples subsequent to separation by gas chromatography (GC). Natural products are compounds derived from plants and they account for more than 40% of the newly registered hits in the drug discovery field [1]. The majority of natural products contain only carbon, hydrogen, nitrogen, oxygen, and sulfur. The argon plasma of an ICP reaches temperatures of 6,000 to 10,000 K, which is hot enough to reduce any natural products to their elemental components. Inside the plasma, these elements will be excited to a higher energy level, and emit radiation at a unique wavelength in the ultraviolet (UV) or near infrared (NIR), which will be monitored by a charge couple device (CCD) detector. The high temperature and inert argon environment of the plasma facilitate mass determination of each element for empirical formula determination of any natural product, regardless of structure. 1. Exarchou et al. Magn. Reson. Chem. 2005; 43: 681–687

(32) **Conformational Stability, r0 Structural Parameters, and Vibrational Assignment of 2,2-Difluoroethylamine**

Arindam Ganguly[1], James R Durig[1]; [1]University of Missouri-Kansas City

The infrared spectra (3500 to 50 cm^{-1}) of the gaseous $HCF_2CH_2NH_2$, have been recorded. Additionally the infrared spectra (3500 to 400 cm^{-1}) of liquid xenon solutions have been recorded at variable temperatures (-55°C to -100°C) from which the enthalpy difference between the three conformers of the 2,2-difluoroethylamine has been determined. *Ab initio* calculations utilizing various basis sets up to 6-311G(2d,2p) with and without diffuse functions have been used to predict the conformational stabilities. A complete vibrational assignment is proposed for all three conformers based on the infrared band contours, infrared band intensities, and Raman activities, which was supported by normal coordinate calculations with scaled force constants from MP2(full)/6-31G(d) calculations. Utilizing previously reported microwave rotational constants for three isotopomers along with the *ab initio* predicted structural parameters from the MP2(full)/6-311+G(d,p) calculation, the r_0 parameters have been obtained for all three of the conformers. The determined heavy atom structural parameters for the Gg1{Gg2}[Gt] conformers are: the distances C_1-C_2 = 1.507{1.512}[1.514](3), C_2-N_3 = 1.458{1.453}[1.452](3), C_1-

F_4 = 1.370{1.369}[1.368](3), C_1-F_6 = 1.365{1.366}[1.368](3), and the angles in degrees ∠NC_2C_1 = 108.8{114.8}[115.0](5), ∠$F_4C_1C_2$ = 109.3{109.7}[110.3](5), ∠$F_6C_1C_2$ = 110.0{109.9}[110.3](5), ∠$F_4C_1F_6$ =107.0{107.1}[106.5](5), ∠$H_{10}N_3H_9$ =107.5{107.9}[107.3](5), and τF_4CCN = 64.5{61.2}[58.6](5), τF_6CCN = -178.1{178.8}[-58.6](5), τH_9NCC = -72.1{-56.6}[-59.2](5), τ$H_{10}NCC$ = 69.0{63.5}[59.2](5). The results are discussed and compared to the corresponding properties of some similar molecules.

(33) **Survey of Pet Foods for Heavy Metal Content by ICP & ICP-MS**

Ralph Obenauf[1], Vanaja Sivakumar[1], Patricia Atkins[1]; [1]SPEX CertiPrep, Inc.

The melamine pet food scare of 2007 rippled through the $45 billion pet supply industry and affected millions of pets and people. Not only is the illegal supplementation of protein sources with melamine and cyanuric acid an issue, but there are additional potentially controversial ingredients and additives that could be contained in pet food products such as selenium, BPA and phthalates. Further, other potential harmful components such as heavy metals, pentobarbital and ethoxyquin make it difficult for consumers to choose healthy pet foods. The selection of a healthy pet food now goes beyond the choice of a name brand food, or good ingredients on a label. The purpose of this study was to survey a variety of brands of commercial dog and cat foods (both wet and dry food) for detectable levels of heavy metals (Hg, Pb, As, Cr, Co). In addition, the levels of sodium selenite were measured to determine if the concentration of selenite exceeded current guidelines. Samples of pet foods were obtained from personal donations, pet supply stores, discount stores and super markets. The quality and price of these samples ranged from budget foods to premium pet foods. Dry samples were uniformly ground using the SPEX SamplePrep Geno Grinder. Wet samples were homogenized in a blender. Samples were then digested via microwave and analyzed by ICP and ICPMS. A number of samples showed high concentrations of potentially dangerous toxic heavy metals.

(34) **Selenium Heavy Metal Antagonism in Soybeans**

Traci Hanley[1], Qilin Chan[1], Joseph Caruso[1]; [1]University of Cincinnati

Gailer and co-workersa make the remarkable statement that, “Among the most startling observations in mammalian toxicology is that a lethal dose of selenium can be overcome by an otherwise lethal dose of arsenic….” And this is so with mercury as well. There have been many animal studies regarding these phenomena, but far fewer studies in plants. Yet, plants are often the route to heavy metal toxicity in the animal kingdom. Earlier studies in this laboratory have led to reports involving Se/Hg antagonism in green onion and soybean plantsb. These studies involve the powerful tools of ICPMS and ESIMS, along with chromatographic sample introduction. Further as a follow-up, X-ray fluorescence studies were undertaken. Taken together, these studies showed clearly that Hg and Se were sequestered in the roots, while only selenium was translocated to the aerial portions of the plant. Further, the XRF studies clearly showed that some Se was transported through the epidermis of the root, and that Hg remained on the outer portion of the roots, plus some selenium. In this presentation, the continuing investigation of the Se/Hg antagonism in soybeans will be presented. As yet the identity of the high molecular weight species has not been identified although there is speculation involving some type of Se/Hg complex with possible protein interactions. Additionally, the precise location of this or other sequestered species by the root is not yet known. The presentation will involve the latest studies on both the identity (via mass spectrometry) and the location of these species (XRF studies).

(35) **Elemental Analysis of High Elevation Conifers to Investigate Effects of Acidic Deposition**

David J. Butcher[1], Matthew Rosenberg[1], Lucas Wilson[1]; [1]Western Carolina University

The spruce-fir forest in the Southern Appalachian Mountains of North America is a unique ecosystem that consists of red spruce (Picea rubens) and Fraser fir (Abies fraseri). These forests are found at elevations exceeding 1400 m and are boreal reminants of the last ice age. These forests have undergone significant decline in recent years due to a combination of factors that may include exotic insect predation and acidic deposition induced by fossil fuel combustion. As the natural buffering capacity of the soil is exceeded, nutrients such as calcium and magnesium are leached from the soil and toxic aluminium becomes more available to the trees. It is proposed to determine these tree elements in the foliage and surrounding soil of these trees to characterize the effects of acidic deposition upon forest health. In this presentation, the results of a long-term study of health of these high elevation conifers is discussed. Following an overview of this environmental problem, sample locations and sampling procedures, the analytical conditions used for inductively coupled plasma optical emission spectrometry will be described. Statistical analysis will be employed to evaluate the effects of elevation, proximity to fossil fuel power plants, life stage of the trees, along with comparison to previous studies in the literature.

(36) **The Formation of Doubly Charged Ions in and Inductively Coupled Plasma**

Kyli McKay[1], Nicholas Taylor[1], Paul Farnsworth[1]; [1]Brigham Young University

The inductively coupled plasmas used as ion sources for elemental mass spectrometry efficiently ionize most of the metals in the periodic table. That ionization efficiency leads to very low detection limits and has made inductively coupled plasma mass spectrometry (ICP-MS) the method of choice for most trace elemental analysis. For analytical purposes, it is desirable to have the plasma convert as much of a target analyte as possible into a single ionic form for presentation to the mass spectrometer. For ICP-MS, that form is the atomic ion with a single positive charge. Some elements with low second ionization potentials form doubly-charged ions in significant quantities. Barium is a prime example of such an element, and it is used routinely in diagnostic routines for ICP-MS to detect and protect against over production of doubly-charged ions. Recent studies of the effect of the sampling interface on the plasmas used in ICP-MS have suggested that barium is atypical, and that doubly-charged barium ions may be present in the plasma at much higher concentrations than are indicated in the mass spectra derived from the ICP source. We have hypothesized that Ba2+ is produced in a single step by collision with a ground-state argon ion. This production pathway is not available to two other alkaline earth metals, Ca and Sr. The doubly-charged alkaline earth ions cannot be detected directly by conventional optical spectroscopy, so we are using indirect evidence for their presence in the form of the temporal response of emission from highly excited singly-charged ions to pulsed interruption of the power delivered to the ICP. We will present the results of our time-resolved emission measurements, and discuss their implications for the production of doubly-charged ions in the ICP.

(38) **The Spectral Game – Teaching NMR Spectroscopy via a Web Browser**

Antony Williams[1], Jean-Claude Bradley[2], Robert Lancashire[3], Andrew Lang[4]; [1]Royal Society of Chemistry; [2]Drexel University; [3]The University of the West Indies; [4]Oral Roberts University

We report on the implementation of the Spectral Game, a web-based game where players try to match molecules to various forms of interactive spectra including 1D/2D NMR. Each correct selection earns the player one point and play continues until the player supplies an incorrect answer. The game is played using a web browser interface and use spectra from the ChemSpider database (www.chemspider.com) for the problem sets together with structures extracted from the website. The spectra are displayed using JSpecView, an Open Source spectrum viewing applet which affords zooming and integration of JCAMP spectra. Players of the game provide both active and passive feedback regarding the quality of the spectral data resulting in crowd sourced curation and validation of the data.

(39) **The Effect of Matrix Composition on Several Fundamental Parameters in an Emission ICP**

Nick Taylor[1], Paul Farnsworth[1]; [1]Brigham Young University

The inductively coupled plasma (ICP) is widely used as an extremely efficient atmospheric ionization source for elemental analysis. Many mechanisms have been proposed for the ionization of these atomic species in the plasma including charge transfer, electron impact, and Penning ionization. Since the introduction of the ICP as an analytical tool, Penning ionization has been proposed as a potentially significant mechanism from which the analyte ions are generated. To better understand the impact Penning and Penning "like" processes have on the excitation/ionization of analyte species in the plasma, we have mapped argon metastable populations with various matrix compositions using an absorption pump-and-probe technique. Additionally, we have measured the repopulation rate of the argon metastable state following the pulsed dye laser depletion. We have further characterized the analyte environment by investigating fluorescence quantum efficiencies in the plasma. Previous studies have shown atomic fluorescence quantum efficiencies to be very high relative to other high temperature and pressure atom reservoirs. A comparison between atomic, ionic, and ion excited state quantum efficiencies for various matrices was developed by obtaining the saturation parameter for each system. We have concluded our study with the evaluation of spatially resolved argon metastable absorption line profiles under the influence of various matrices. The matrix elements used throughout this study include Mg, Ca, Sr, Ba, Cu, Y, La, Nd, Sm, and Gd. In this presentation, we will compare the argon metastable populations, repopulation rates, quantum efficiencies, and absorption line profiles in the presence of the various matricies to those obtained in the absence of a matrix. The impact the Penning mechanism has on the ionization of elements used in this work will be discussed.

(40) **Evaluation of Ion Transmission and Shock Structure of Various Skimmer Cone Designs in an ICP-MS**

Alisa Smith[1], Nick Taylor[1], Ross Spencer[1], Paul Farnsworth[1]; [1]Brigham Young University

Inductively coupled plasma-mass spectrometers have matured into the dominate trace elemental analysis technique used in typical analytical labs. Despite the success, higher sensitivities and lower detection limits are still in high demand in several areas of science. One area of significant signal loss is the extraction of ions from the plasma. Since the plasma is an atmospheric ionization source multiple vacuum stages are required to extract the ions into an acceptable pressure environment. The amount of ions that transmit through these stages is heavily dependent on the design of the interface through which they pass. A comprehensive evaluation of five commercially available skimmer cones will be discussed. The first study involves the measurement of ion transmission through the various skimmer cones. The percent ion transmission is determined by comparing the populations upstream and downstream from the skimmer cone tip by spatially resolved laser-induced fluorescence. It will be shown that the transmission of barium and calcium ions through the various skimmer cones is significantly lower than what would be expected under ideal skimming conditions. In order to evaluate the effects of space charge on the transmission of the selected ions barium neutral transmission was also evaluated. The barium neutral demonstrated approximately 5% increase in transmission compared to the barium ion. It can be concluded that space charge has little impact on the ion transmission; therefore, another mechanism must be effecting the transmission of ions through the skimmer cones. Additional space charge studies will be discussed. The second study involves the measurement of argon metastable line profiles upstream and downstream from the tip of the five various skimmer cones. The profiles from each of the five cones will be evaluated for the presence of a shock formation that is created due to the skimmer cone design. It will be shown that the presence of a shock structure adversely affects the transmission of ions through the skimmer cone. In addition, experimental profiles will be compared to calculated profiles obtained from a fluid dynamic model using Direct Simulation Monte Carlo (DSMC). An overall evaluation of the various skimmer cones will be thoroughly discussed.

(41) **Spectroscopic Study of the Electronic States of Liquid Ketones and Ethers by Using Attenuated Total Reflection - Far Ultraviolet Spectroscopy**

Yusuke Morisawa[1], Kyoko Takaba[1], Akifumi Ikehata[2], Noboru Higashi[3], Yukihiro Ozaki[1]; [1]Kwansei Gakuin University; [2]National Food Research Institute; [3]KURABO

Spectra in the far ultraviolet (FUV) region have been studied for molecules in the gas phase for a long time. These researches revealed that many kinds of molecules have strong absorptions due to electronic transitions to low-lying Rydberg states in the region until the vertical ionized energy of these. Recently we have uniquely developed the attenuated total reflection technique in the FUV region (ATR-FUV). By use of ATR-FUV, spectra in the region from 8.55 to 4.13 eV (145 to 300 nm) can be measured for many kinds of liquid, such as of aqueous solutions, neat water and neat alcohols without spectral saturation . Such the strong absorption will use in novel analytical method. The knowledge about Rydberg transition in the liquid phase, however, has not been collected well. In this paper, the new results about FUV spectra of several kinds of liquid ketones and ethers are reported. The differences in the electronic states of molecules between gas and liquid phases still have not been understood well. For example, the Ã-X absorption of water appears at 7.4, 8.3 and 8.6 eV (167, 149 and 144 nm) for vapor, liquid, and solid phases, respectively. These large blue shifts upon going from the gas phase to the condensed phases should come from changes in the ground and excited states caused by molecular interactions and exchanging repulsive interaction between excited electron and ground state electrons of neighbor molecules. Chergui and Schwentner discussed the Rydbergization of valence type anti-bonding orbital. In the rare gas matrixes of Ar, Kr and Xe, the shifts of transition energy from the gas phase are continuously increased by lattice constant in the solid. In this presentation the FUV absorption spectra of liquid ketone and ether are reported in the region of 8.55 to 6.20 eV (145 to 200 nm). The two or three distinguished absorption bands were observed in these spectra. The assignments for the absorptions are attempted by comparison with theoretical method.

(42) **Effects of Hydrogen Bondings on an Electronic State of Acetone Studied by Using Attenuated Total Reflection - Far Ultraviolet Spectroscopy**

Yusuke Morisawa[1], Akifumi Ikehata[2], Noboru Higashi[3], Yukihiro Ozaki[1]; [1]Kwansei Gakuin University; [2]National Food Research Institute; [3]KURABO

Molecules with oxygen atom have a transition of lone pair electron (n orbital) in the ultraviolet (UV) and far ultraviolet (FUV) region. Because an electron in lone pair orbital plays an important role in a hydrogen bonding, it is interesting to know how the transition behaves in the change of a hydrogen bonding state. The aqueous solution of acetone is a benchmark molecule to test the solvatochromism of nπ* electronic transition. The n π* transition, however, is symmetrically forbidden, and thus it is difficult to know the exact vertical excitation energy. The allowed electronic transition, n-3s Rydberg transition, appears in FUV region. Because of the difficulty of the experiment, the solvatochromism of this transition has still been unknown yet. We originally have developed attenuated total reflection (ATR) - FUV spectroscopy. This method can be used to observe the very strong absorption in the FUV region without peak saturation. This feature allowed easily measuring the FUV spectrum of aqueous solutions. In the previous FACSS meeting we reported the FUV spectra of H2O, CH3OH (alcohol), C2H5OC2H5 (ether) and CH3COCH3 (ketone) with ATR-FUV in the region from 8.55 to 6.20 eV. In this presentation we will report the electronic spectrum of acetone neat liquid, its aqueous and ethanol solutions in the range from 8.55 - 6.20 eV. From this result we found that the absorption which is observed at 6.75 eV in the neat liquid moves to 6.88 in the ethanol and 7.05 eV in the aqueous solution. We will discuss the relation between electronic spectrum and hydrogen bonding.

(43) **Selective Oxidation and Vapour-Phase FT-IR Spectrometric Determination of Histidine, Threonine and Cysteine in Pharmaceutical Formulations**

Kazem Kargosha[1], S.Hamid Ahmadi[1], Mohsen Zeeb[1], Jila Azad[2]; [1]Chem.&Chemic.Engineering Research Center of Iran; [2]Alzahra University

A novel method for selective oxidation and determination of Histidine (His.), Threonine (Thr.) and Cysteine (Cys.) based on Vapour- generation and infrared spectrometry is reported. In this producer, Thr., His., and Cys., were selectively oxidized into different infrared absorbing gaseous species by adjusting PH at fitted values and using proper oxidants. A glass Vial of 20 ml volume containing 10 ml of sample with adjusted PH is placed in a water-bath at 65,,aC, N2 flow is passed through the vial, oxidant solution with definite concentration is injected and the vapour generated in vial is transported to a lab made gas cell of the FT-IR spectrometer using nitrogen carrier flow of 4.0 ML Min-1. The vapour spectra is continuously recorded as a function of time between 2500 and 2100 cm-1. Threonine was oxidized into CO2 using sodium metaperiodate at PH of 2.5 . The maximum absorbance of this gas was measured at 2360 cm-1. The calibration graph in Thr. Measurement was linear in the range of 50-450 ppm and RSD was 2.10% (n=5). Histidine was also oxidized into CO2 by using sodium metaperiodate but at PH of 6.10. Calibration graph in His. Determination was linear from 10 up to 250 ppm. RSD was 1.2% (n=5). Cysteine at PH of 3 was oxidized into CO by using potassium iodate. The maximum absorbance of CO at 2170 cm-1 was selected as a measurement criterion. The calibration graph in this producer was linear over the range 6-300 ppm and RS (n=5) was close to 1.7%. This producer could be also used for individual determination of L-cysteine and L-Cystine. After measuring total concentration of L-Cysteine and L-Cystine, the former was masked with P-benzoquinone and the latter was determined. The amount of L-Cysteine was obtained by difference. The proposed method has been used for determination of His., Thr. and Cys. in pharmaceutical formulations.

(44) **Increasing Accessibility to Instrumentation: A "Cutting Edge Metal Detector" for Field Applications**

Summer N. Hanna[1], Bradley T. Jones[1]; [1]Wake Forest University

Accessibility to instrumentation has been a challenge for scientists in developing countries for some time. Across the globe, countries are seeing federal funds for science and technology regularly slashed, while the demand for scientific growth continues. This leaves scientists with little options beyond creating novel instrumentation from equipment that is readily available and within a modest operating budget. We have attempted to address these challenges through the development of a transportable flame atomic emission spectrometry device. It utilizes a tungsten coil as an electrothermal vaporization source, which allows for the use of small sample volumes, and is coupled to an oxygen-acetylene flame. The tungsten coil is simple in design and commercially available as a 150 W, 15 V microscope light bulb, while the flame source is constructed from a welder's metal-cutting torch. The marriage of these commercial devices has given rise to the "Cutting Edge Metal Detector (CEMD)," which has been capable of characterizing over twenty elements with LODs as low as 0.9 ng L-1 for Ca and 8.0 ng L-1 for In. Thirteen of these elements were determined using certified reference materials and results did not differ significantly beyond a 95% confidence level. Detection using a handheld CCD spectrometer, powered by a laptop computer, creates a truly portable instrument capable of field analysis with a minimal level of required user training. Considering the extremely small sample volumes required for analysis, the CEMD will rival modern complex instrumentation such as ICP-OES while operating within a modest budget, ensuring easy access to laboratories throughout the developing world.

(45) **Large Area Nanopillars SERS Arrays**

Tiziana Bond[1], Elaine Behymer[1], Hoang Nguyen[1], Cindy Larson[1], James Chan[1], Robin Miles[1], Mihail Bora[1], Logan Liu[2], Zidar Xu[2], Manas Gartia[2]; [1]Lawrence Livermore National Laboratory; [2]University of Illinois, Urbana Champaign

Large Area Nanopillars SERS Arrays James Chan, Elaine Behymer, Hoang Nguyen, Cindy Larson, Robin Miles, Mihail Bora, Allan Chang, and Tiziana Bond Lawrence Livermore National Laboratory Manas Gartia, Xu Zidar and Gang Logan Liu, University of Illinois, Urbana Champaign We introduce a novel and flexible fabrication approach for high density, large area, and high uniformity Surface Enhanced Raman Spectroscopy (SERS) platforms. We demonstrate this by presenting two kinds of arrays: vertically coupled and individually tapered nanopillars. The basic 3D templates are fabricated by a top-down process approach, relying on laser interference lithography and reactive ion etching of SiO2 vertical nanopillars on the entire surface of a 4" wafer. By proceeding with atomic layer deposition of Al2O3 followed by sputtering of a thin film of silver or gold, the gaps between the pillars are narrowed enough to create coupled plasmonic cavities that acts as 3D hotspots, with a density of 3.85•108 cavities/cm2. Confinement factors over 103 for each vertical cavity are possible due to plasmon focusing in the inter-wire space. Alternatively, by controlling the lithography exposure doses and dry etching parameters we can generate tapered nanopillars that when sidecoated with metal by angled e-beam depositions, translate into array of vertical nanoantennas. In this case, the tip of the nanopillar is the hotspot, coupling the light along the metal coated side to the holey nano pattern surface of the substrate. In both structures the hot spots are enough far apart to dismiss any coupling effect, since the size of the nanopillars is an order of magnitude larger than the penetration depth of the electric field in the metal. In addition, the

plasmon resonances can be adjusted for maximum overlap with the absorbance of the active material and the Raman resonances of the molecules. Far field absorbance measurements of the featured nanoarrays show plasmonic resonances in the convenient vis-NIR range and uniform Raman signals across the sample with enhancement EF=(I_SERS N_NR)/(I_NR N_SERS) 1/m 1/n^2 ~ 106 – 5x107 for 1,2bis-(4-pyridyl)-ethylene (BPE) or benzenethiol (BT) (m = area reduction factor to account for additional surface area; n = refractive index of bulk solution). Raman spectra were acquired with a confocal inverted microscope stage setup equipped at 532nm, 660nm or 785nm. In conclusion, laser interference lithography approach offers great potentials for batch nanofabrication of sensitive and tunable active SERS substrates that can be scaled to any size.

(46) **Novel Observations of Absorption Bands Of Liquid Alkanes and Polyethylene by Attenuated Total Reflectance-Far UV**

Tachibana Shin[1], Morisawa Yusuke[1], Ikehata Akifumi[2], Sato Harumi[1], Higashi Noboru[3], Ozaki Yukihiro[1]; [1]Kwansei Gakuen University; [2]National Food Research Institute; [3]KURABO

The spectroscopy in the far ultraviolet (FUV) region has been used for exploring the electronic states, and discussing Rydberg transition in the gas phase. However, in the region of shorter than 190 nm, it is difficult to measure the spectra in the condensed phase because of too strong absorption. In order to resolve the situation for the condensed phase we developed the attenuated total reflection technique (ATR) in the FUV. The spectra of alkanes in the FUV region have been published only for the gas phase. The present research gives spectra of liquid alkanes (normal and branched) and solid polyethylene (PE) in the region from 145 - 200 nm. According to the analysis of the liquid n-alkanes observed, the absorption band shifted to low energy side by increasing carbon numbers. The degree of the shift is proportional to the densities of alkanes. The ionized energy is proportional to the shift, too. As to liquid branched-alkanes, the peak of absorption was observed in lower energy compared to n-alkanes which have the same number of carbon atoms. The different shape of absorption was observed for different branced styles. These features were same as gas phase. On the other hand, the spectra of several kinds of PE, such as high density PE (HDPE), linear low density PE (LLDPE), low density PE (LDPE), were different from each other. These differences should be caused by carbon chain length and branch style. The spectra of a commercial wrap made by PE were similar to that of LDPE. In conclusion, the ATR-FUV technique will promote the research for the electronic states of alkanes and develop the discrimination of PEs.

(47) **Effects of Intramolecular Hydrogen Bonding on OH Bands of Phenol and Halogenated Phenols in the Visible, Near-Infrared and Infrared Spectra**

Takayuki Gonjo[1], Yuusuke Morisawa[1], Toshiaki Suzuki[1], Yukihiro Ozaki[1]; [1]Kwansei Gakuin University

Visible, near-infrared and infrared spectra were measured for phenol and halogenated phenol in hexane and CCl4. We observed OH stretching vibration bands from the fundamental to the third overtone. The three parameters of solvent shift, bandwidth and intensity could be derived from the observed spectra. Comparing these parameters, we have investigated differences in the intramolecular hydrogen bonding between the phenol and halogenated phenols. In the present research, we investigated particularly solvent shift and intensity. The solvent shift means the difference in wavenumber of absorption peak between gas and the solutions, it indicates the strength of intermolecular interaction. From the comparison of solvent shift, we found a result that the observed molecules which have stronger intramolecular hydrogen bonding got weaker intermolecular interaction from solvents. As for intensity, it is generally known that the intensity increase for the fundamental of OH stretching depends on formation of hydrogen bond, while, it has been pointed out that no such intensity increase occurs for overtone. Simple explanations have been given for this difference between the fundamental and first overtone. However, there is no corresponding study on OH stretching vibration bands through the fundamental to the third overtone. By observing not only the fundamental and first overtone but also second and third overtone, we will discuss about effects of intramolecular hydrogen bonding. In the presentation, we will compare the measured datas with theoretical calculations. In order to investigate the effect of intra and intermolecular interaction for the intensity and solvent shift of observed spectra, we carried out the quantum chemical calculations based on the one-dimensional wave equation.

(48) **Robust SERS Substrates Generated by Coupling a Bottom-Up Approach and Atomic Layer Deposition**

Eric Formo[1]; [1]Center for Nanophase Materials Sciences, ORNL

We will report on the development of a novel thermally stable Surface Enhanced Raman Scattering (SERS) substrate. Specifically, these substrates can withstand high temperatures in air for an extended period of time without the loss of their enhancement capabilities. To accomplish this we utilized a bottom-up approach, in which the polyol reduction process was used to synthesize silver nanowires (NW) that were roughly 90 nm wide to act as the SERS active moiety. Subsequently, the NW were deposited onto a glass substrate and then coated with a thin protective layer of Al2O3 via Atomic Layer Deposition (ALD). After heating these SERS substrates at 400oC for 24h in air it was found that the coated samples maintained a significant enhancement of the Raman signal, with further heating resulting in effectively no change in the SERS spectrum. The stability imbued by the ALD coating stems from limiting surface oxidation along with impeding Ostwald ripening that occurs at the higher temperature which would otherwise lead to the destruction of the nanomorphology and complete loss of the SERS capabilities. These highly stable SERS substrates highlight the potential application of SERS in investigation of high-temperature chemical reactions and catalytic processes.

(49) **Enhanced Plasmonic Light Beaming Induced By Near Field Resonance**

Pengyu Chen[1], Lin Zhu[1], Qiaoqiang Gan[2], Filbert Bartoli[2]; [1]Clemson University; [2]Lehigh University

The focus of this research is to understand the enhancement of plasmonic light beaming efficiency by the near field resonance in a subwavelength metallic slit-groove beaming structure. Here, we illustrate the plasmonic light beaming effects by use of COMSOL Multiphysics. We provide a detailed analysis of the role of near field resonance in plasmonic light beaming. We show that, by varying the film thickness and the separation distance of the beaming grating, the intensity of the optical near field can be greatly enhanced by resonance, which leads to an increase in beaming efficiency. Moreover, we obtain a nanocavity above the nanoslit by integrating a metal nanostrip with the beaming structure. The resonance of this nanocavity can further enhance near field intensity and improve beaming efficiency. In summary, we have studied the role of the near field resonance in light beaming for different metallic slit-groove beaming structures. The understanding of controlling collimated light in plasmonic structures is of great importance and can provide a wide range of applications in various integrated optical and nanodevices.

(50) **Analytical Evaluation of a Helium-Argon Radiofrequency Glow Discharge Coupled to Time of Flight Mass Spectrometry**

Cristina González Gago[1], Nerea Bordel[1], Rosario Pereiro[1], Alfredo Sanz-Medel[1]; [1]University of Oviedo

Pure argon has been the most common plasma gas used in analytical glow discharges (GDs). However, alternative gases (He, Ne, Kr) or gas mixtures (Ar with He, N2, O2, H2) have been investigated aiming at improving the performance (e.g. sensitivity or depth resolution) of the glow discharge. It would be supposed that helium gas would give rise to higher emission or ionic signals than argon because of its high excitation and ionization potential (first ionization potential 24.5 eV), but argon is a much more efficient sputtering agent than helium due to its greater mass. Therefore, the investigation of helium-argon mixtures results of great interest because they could combine the high ionization potential of He with the sputtering ability of Ar. The addition of small concentrations of He to the discharge could be specially useful for the analysis of elements with a first ionization potential close or higher than that for argon, which are ionized with small efficiency in argon plasmas. In this work, an in-house GD ion source coupled to an orthogonal time of flight mass spectrometer (TOFMS) was used to investigate the effect of He addition to the argon plasma in terms of signal intensities, ion yields, sputtering rates and depth resolution. Total gas flow as well as He concentration (varied in the 0-15% range) were optimized to improve sensitivities for different conductor and non-metallic matrices including samples containing fluorine (first ionization potential 17.42 eV). Moreover, and considering that the shape of sputtered craters is an important aspect of the quality of depth profiling analysis, the shape of the craters in the samples were obtained at different He concentrations. After such experiments, the possibility of achieving improved qualitative in-depth profiles by resorting to He-Ar mixtures was evaluated.

(51) **Identification of Counterfeit Whisky Using Mid-Infrared Spectrometry with ATR Probes and Polycrystalline Silver Halide Optical Fibres**

Allyson McIntyre[1], Alison Nordon[1], David Littlejohn[1], Gary Colquhoun[2]; [1]University of Strathclyde, Glasgow UK; [2]Fibre Photonics, Livingston UK

Counterfeit operations cost the Scotch Whisky industry a large amount of money each year with equally important threats to the reputation of the product. Various analytical techniques can be used to identify counterfeit whisky and indicate the way in which the fake product has been produced, which can range from modifying real Scotch to the use of locally produced spirit passed off as Scotch. However, there is a growing need for measurements that can be made out of the laboratory and molecular spectroscopy techniques offer opportunities for "in the field" analysis to check authenticity. Developments in probe technology and optical fibres in particular have improved the accessibility of *in situ* process measurements by MIR spectrometry. Two approaches have been devised to investigate the authenticity of whisky samples using attenuated total reflectance (ATR) probes connected to a MIR spectrometer using novel polycrystalline silver halide fibres. The methods allow rapid measurement of alcohol content and assessment of the method used to colour the whisky. Principal component analysis readily identified the samples in the test set that were counterfeit material and those that were genuine product. The approaches used will be described and the result obtained will be presented to illustrate the efficacy of the methodology.

(52) **Structural and Functional Study on High-Reflection Black Pigment by NIR Spectroscopy and XRD**

Nomura Satoshi[1], Morisawa Yusuke[1], Sanada Kazutoshi[2], Shinsuke Maruyama[2], Ozaki Yukihiro[1]; [1]Kwansei Gakuin University; [2]Toda Kogyo Corporation

High-reflection black pigment which has the function of reflecting the sunlight in the NIR region has been developed recently. Because of this function, this pigment is an environmental function material for suppressing the urban heat island. This pigment reflects sunlight from 700 to 1100 nm wavelength well, while in the region from 1100 to 2000 nm the absorption appears. In order to reveal the origin of this absorption, we have investigated about the relation between the NIR absorption and structure of the pigment.The measured samples of the pigment and related materials were prepared on PET films. For the NIR measurements, the samples were sandwiched by a window of the integrating sphere and a gold coated reflector.This pigment was made by CoCO3, Fe2O3, Al(OH)3 and Mg(OH)2 burned at 1100 ºC. In the present research, we observed NIR spectra of the films of the pigment and its components and oxidized components. We found absorptions originated from CoO and Fe2O3 in the NIR region. However, these signals are different from that of pigment. In order to explain the differences in the spectra, we measured XRD and NIR spectra of 8 pigments which were burned at 500 to 1200 ºC. As the result, some XRD peaks and the NIR absorption at 1670 nm behave synchronously. In the NIR spectra, absorbance gradually falls in the spectrum of the sample heated up to 900 ºC and slightly rises in that of the sample heated above 1000 ºC. In the XRD dates, there are corresponding peaks. From these features, we concluded that the structure of pigment changes near 1000 ºC.

(53) **The Dependence of Measurement Precision and Bubble Reproducibility on the Quality of Focusing Optics for Dual-Pulse LIBS Measurements in Water**

Christopher Gordon[1], S. Michael Angel[1]; [1]University of South Carolina

The performance of focusing optics of varying qualities in the analysis of bulk aqueous solutions using dual-pulse laser-induced breakdown spectroscopy (DP-LIBS) is evaluated. Shadowgraphic imaging of vapor bubbles formed by a laser-induced plasma (LIP) in water using bi-convex, plano-convex and achromatic doublet lenses demonstrates that imaging quality focusing optics produce more well defined focal volumes which results in more frequent production of well-shaped, spherical vapor bubbles under comparable experimental conditions. This in turn leads to higher precision in formation of a second laser-induced plasma within the vapor bubble. Bubbles that are formed using an achromatic lens are larger, more spherical, more reproducible and longer-lived in solution. As a result, the range of inter-pulse delays (the time separating the first and second laser pulse) in which a DP-LIBS signal can be observed increases with the use of optics that correct for spherical aberrations, which is promising for the analysis of high-pressure bulk solutions where the interpulse delay is a critical parameter in determining the emission strength for dissolved species in high pressure bulk aqueous solutions. In addition to an increase in the production of strong oxygen emission from each vapor bubble, there is an increase in the fraction of well-formed vapor bubbles, from 26% to 100% for plano-convex and achromatic lenses, respectively. These results have implications to the use of LIBS for expeditionary oceanographic research.

(54) **Numerical Simulation of the Effects of Helium Added to an Argon Inductively Coupled Plasma**

Helmut Lindner[1], Annemie Bogaerts[1]; [1]University of Antwerp

Inductively coupled plasmas (ICP) are widely used excitation sources for elemental analysis. The samples are injected into the

plasma as droplets or dry particles. The aerosol is evaporated in the hot plasma and the atoms are being excited and ionised. The analysis is done by detecting the optical emission of the atoms/ions or by mass spectrometry. One important way of producing dry aerosols is laser ablation of solid samples. Here, helium is often used as carrier gas since it improves the laser ablation conditions. Numerical simulations provide deeper insight into effects taking place in the plasma. In the present work, we studied the effect of helium added to the gas streams of an argon ICP flowing into an ambient nitrogen atmosphere. The plasma is simulated as a 2d axial symmetric geometry. Local thermodynamic equilibrium (LTE) was assumed to determine the degree of ionisation. The external current was adapted automatically in order to achieve a fixed coupled power into the plasma. Different helium to argon ratios were applied for the injector gas flow as well as different flow rates for the injector gas. The plasma conditions, such as temperature distributions or power coupling, are analyzed for the different conditions. Emphasise is laid on the effects inside the central channel of the ICP as the analyte is flowing there. The addition of helium to the injector gas flow results in a strong increase of temperature on the axis of the plasma.

(55) **Characterization of Di-Isohexyl Phthalate (DIHxP) Using GC/MS and NMR in Support of Toxicological Studies**

Joseph Licause[1], Jason Burgess[1], James Blake[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

Phthalates, or phthalate esters, are esters of phthalic acid and are mainly used as plasticizers (substances added to plastics to increase their flexibility, transparency, durability, and longevity) to soften polyvinyl chloride. Phthalates are being phased out of many products in the United States and European Union over health concerns. Di-isohexyl phthalate (DIHxP) is a low production phthalate acid ester suspected as a developmental and reproductive toxin as well as a possible carcinogen in rodents. A critical component of any toxicological study is unequivocal identity and complete characterization of the test chemical. The process used to produce these long chain branched-phthalate esters such as DIHxP can generate a variety of other phthalates as impurities or result in isomeric mixtures. This can have unintended consequences or produce results that are different from the specified test chemical. The present study involved an analysis of a sample of DIHxP specified by the manufacturer as a pure chemical with purity >99%, but our analysis found it to be a mixture. A sample of DIHxP was analyzed using capillary Gas Chromatography/Mass Spectrometry (GC/MS) to effectively resolve the constituents and confirm the identity of the primary components. The material was comprised of two major components accounting for 99.9% of the total area. The two components were suggested as diastereomers of bis(4-methyl-2-pentyl) phthalate based on their resulting spectra and parallel analysis of a comparison sample of bis(4-methyl-2-pentyl)phthalate (CAS No. 146-50-9). Nuclear Magnetic Resonance (NMR) spectroscopy was then conducted to provide additional confirmation of the material. Proton-NMR results confirmed the material as bis(4-methyl-2-pentyl)phthalate and matched the spectrum supplied in the vendor's COA. However the original analysis did not have adequate resolution (300 MHz instrument) to discern the components of the material. The follow up 1H and 13C-NMR experiments using a high resolution instrument (500 MHz) later confirmed the material to be a mixture of diastereomers.

(56) **Surface-Enhanced Raman Scattering Substrates Based on Heat Shrinkable Polystyrene**

Joseph Mannion[1], George Chumanov[1]; [1]Clemson University

A novel surface-enhanced Raman scattering (SERS) substrate was developed based on a bi-axially oriented polystyrene sheet. Silver was deposited (75 nm) by vacuum thermal evaporation onto the polystyrene sheet, followed by the adsorption of a monolayer of poly(diallydimethylammonium) chloride and 100 nm silver nanoparticles (NPs). The substrate was then heated in an oven, causing the polystyrene sheet to shrink, the overlaying silver film to wrinkle, and the Ag NPs to move closer to one another. Various biomolecules were adsorbed to this substrate and SERS spectra were recorded. The SERS spectra were highly reproducible across the substrate and molecules such as adenine were detected at 1x10-9 M. The substrates are robust and can be made to any practical size or shape using readily available methods.

(57) **Detection of Cyanide Using Raman Spectroscopy on Metal Halide Films**

C.V. Gopal Reddy[1,2], Fei Yan[1,2], Yan Zhang[1,2], Tuan Vo-Dinh[1,2]; [1]Fitzpatrick Institute for Photonics; [2]Department of Biomedical Engineering

Cyanide is ubiquitous. It can come from sources such as industry pollutants, automobile exhaust and cigarette smoke. An HCN concentration of 300 ppm in air will kill a human within a few minutes. Raman spectroscopy is well known for its specificity in chemical and biological analysis, and offers some distinct advantages over other spectroscopic methods such as fluorescence spectroscopy. We developed a toxic cyanide detection system based on the principles of Raman spectroscopy that operated both in aqueous solutions and air, and provided fast detection and high sensitivity/selectivity. It was found that reaction of cyanide molecules from aqueous solution formed stable metal cyanide as a stable complex. This metal halide was vacuum sublimed on silicon wafers and upon exposure to solutions of NaCN, MCN was formed producing a single sharp band at 2171 cm-1. The intensity of this band varies linearly with NaCN concentration and it was shown that a detection limit below 100 ppb was achievable. Key words: Chemical warfare agents, cyanide, sensors, Raman spectroscopy

(58) **Hydrogen/Deuterium Exchange Mass Spectrometry System for Investigating Conformational Changes in Calmodulin Protein upon Calcium Binding**

LeRoy Martin[1], Joomi Ahn[1], Martha Stapels[1], Michael Eggerston[1], Keith Fadgen[1], Rebecca Rose[2,3], Ying Qing Yu[1], Albert J. R. Heck[2,3]; [1]Waters Corporation; [2]Biomolecular Mass Spectrometry; [3]Netherlands Proteomics Centre, Padualaan

Hydrogen/deuterium exchange mass spectrometry (HX MS) has proven to be a useful analytical method for the study of protein dynamics and changes to protein conformation. The applications in HX MS require a system that can perform rapid chromatographic separations at 0 ° C and accurate mass measurements of labeled proteins and peptides with small quantities of material. Recent improvements in LC-MS have made HX MS an indispensible tool for discovery and development of protein drugs. In this study, a nanoACQUITY UPLC system with HDX technology combined with a XEVO Q-Tof MS was used as a robust HX MS platform for protein conformational analysis. In this system, online pepsin digestion was coupled to highly reproducible UPLC performed at low temperature. MSE analyses show high confidence peptide identification, up to 94 % sequence coverage. To illustrate how these data are useful for HX MS, we describe a recent study of conformational changes in important intracellular protein, calmodulin upon calcium binding.

(59) **Vibrational Spectroscopy of High Strength Polymeric Fibers**

Bruce Chase[1]; [1]Pair Technologies LLC

High strength fibers derive their mechanical properties from molecular level effects including both structure and orientation. Structural effects can include conformational state populations as

well as short range order interactions and crystallinity. Chain orientation has a direct impact on modulus. Vibrational spectroscopy offers a probe that is sensitive to both effects. Raman scattering results on high strength fibers ranging from gel spun polyethylene to aramid fibers will be reviewed illustrating the information content available through vibrational spectroscopy.

(60) **Interfacing Mass Spectrometry with Liquid and Gas Phase Separations for Synthetic Polymer Analysis**

Chrys Wesdemiotis[1], Xiapeng Li[1], Nilüfer Solak[1], George R. Newkome[1], Stephen Z.D. Cheng[1]; [1]The University of Akron

Matrix-assisted laser desorption ionization (MALDI) and electrospray ionization (ESI) have enabled mass spectrometry (MS) analyses for a wide variety of synthetic polymers. MS experiments provide the mass-to-charge ratios (m/z) of the constituent n-mers of a polymeric analyte, from which compositional heterogeneity, molecular weight, and functionality distributions can be deduced. Considerable challenges still exist, however. Polymerizations often create complex mixtures that are difficult or impossible to characterize by single-stage MS because the ionization and detection of certain components is obstructed by discrimination effects. Furthermore, a polymer may contain isobaric components and/or a mixture of isomeric architectures that cannot be identified by m/z measurement alone. These problems can be resolved by interfacing mass spectrometry with liquid chromatography (LC) or ion mobility (IM) separation, i.e. by using two-dimensional LC-MS or IM-MS approaches. Interactive LC (i.e. adsorption-mode LC) is found ideally suitable for the separation of oligomer mixtures with constituents of different polarities. This capability will be demonstrated with poly(ethylene oxide) / poly(propylene oxide) copolymers as well as sorbitan- or glycoside-based nonionic surfactants. In LC-MS, separation takes place before ionization. IM-MS may be viewed as a chromatographic method that disperses post-ionization according to mass, charge, and shape. Since it does not involve interactions with a solid or liquid stationary phase, it is particularly useful for labile and weakly bonded species which may decompose or react if sent through an LC-column. Our group has applied IM-MS to characterize supramolecular polymers, self-assembled from designed building blocks via coordinative (i.e. metal-ligand) or π-π interactions. Self assembly generally creates many different isomers and conformers (architectures); these have identical m/z ratios but unique shapes and, thus, can be separated and identified by IM-MS, as will be shown for metallomacrocycles and π-π bonded nanoparticles. Further insight about the binding interactions in these materials is gained by their dissociation energetics, assessed through three-dimensional IM-MS2 experiments.

(61) **Molecular Structures of Polymer Surfaces and Buried Polymer Interfaces Studied by Sum Frequency Generation Vibrational Spectroscopy**

Zhan Chen[1]; [1]University of Michigan

Sum frequency generation (SFG) vibrational spectroscopy has been applied to investigate various polymer surfaces and buried interfaces involving polymers *in situ*. The molecular structures of silane molecules, which are widely used as adhesion promoters, have been investigated using SFG at buried polymer/silane and polymer/polymer interfaces, providing molecular-level understanding of polymer adhesion promotion. SFG was also used to study model compounds for epoxy resins used as underfills in flip-chip devices. Surfaces structures of epoxy materials before cure, after cure, and after exposure to moisture have been examined. Buried interfaces between polymers and cured epoxy have been investigated *in situ* using SFG, and structural information obtains by SFG was correlated to the adhesion testing result. SFG has also been developed into a powerful tool to elucidate molecular structures at buried polymer/metal interfaces.

(62) **Mass Spectrometry of Polyurethanes: MS/MS and IMS**

David Hercules[1], Anthony Gies[1]; [1]Vanderbilt University

Polyurethanes (PURs) can be very complex mixtures of different species, depending on the synthesis method used. Important questions relate to the nature of the hard- and soft-segments and end groups. The current study looks at polyester-based PURs formed from diol- terminated poly(butylene adipate) as the soft block and methylene diphenyl diisocyanate (MDI) as the hard block. Butane diol is used as the extender. Collision-induced fragmentation (MS/MS) is used to determine the low energy pathways for PURs. Polyesters show 1,5-hydrogen shifts as the major pathway, but the main fragmentation occurs around the urethane group. Both 1,5- and 1,3 H-shifts are observed for PURs, the latter appearing to be more extensive. Of particular interest is the difference between cyclic polyesters and PURs. The major ring-opening reaction in the former involves a 1,5-H shift, but for PURs, a 1,3 H-shift is dominant. MS/MS is also helpful in determining the segment sequence in PURs containing multiple polyester and MDI groups.

(63) **Nanoscale Infrared Spectroscopy and Imaging of Polymer Microdomains**

Curtis Marcott[1], Michael Lo[2], Kevin Kjoller[2], Craig Prater[2], Gloria Story[3], Isao Noda[3]; [1]Light Light Solutions; [2]Anasys Instruments; [3]The Procter & Gamble Company

Atomic Force Microscopy (AFM) and infrared (IR) spectroscopy have been combined in a single instrument capable of producing sub-micron spatial resolution IR spectra and images. This new capability enables the spectroscopic characterization of microdomain-forming polymers at levels not previously possible. Specifically, films of poly(3-hydroxybutyrate-co-3-hydroxyheanoate) were solution cast on ZnSe prisms. The AFM images suggest spherulites containing both crystalline and amorphous domains are formed on the prism. A tunable IR laser generating pulses of the order of 10 ns was used for excitation of the sample films. Short duration thermal waves, due to infrared absorption, were studied by monitoring the resulting excitation of the contact resonance modes of the AFM cantilever. The spectral resolution is better than 16 cm-1 across the entire spectral range (1200-3600 cm-1), which is good enough for the system to differentiate crystalline and amorphous microdomains at a spatial resolution of less than one micron. The results demonstrate that AFM-IR is a promising tool to complement standard techniques for the high resolution characterization of polymer microdomains.

(64) **Using Mass Spectrometry to Study Polymer Processing**

Anthony Gies[1], David Hercules[1]; [1]Vanderbilt University

This presentation will focus on the identification of the structural distributions generated during the synthesis and processing of poly(p-phenylene sulfide) (PPS) (Ryton®), polyphenylsulfidesulfone (PPSS), and their copolymers. The limits of MALDI-TOF MS, using the evaporation-grinding method (E-G method), for intractable polymer analysis will first be discussed. Initial PPS studies focus on the relationships between the following experiments: (1) effects of MALDI laser fluence and area of irradiation on model PPS phenyl-capped trimers and cyclics; (2) modification of PPS synthesis conditions; (3) thermal curing of PPS under various atmospheric (N2 and air) and thermal (100 – 300 ˚C) conditions; and (4) heated N-methyl-2-pyrrolidone (NMP) fractionation of high MW PPS (25, 50, 75, and 100 °C). These studies yielded a wealth of information on the mass, structure, and end-groups of species generated in the synthesis, post-synthesis modification, and thermal curing of PPS. Further, surface-modified

MALDI sample preparation techniques were shown to selectively isolate cyclic species from high molecular weight PPS. However, the degree of PPS cyclization, sulfoxide formation, and dibenzothiophene end group modification was difficult to predict and more definitive information was necessary to confirm the existence of these structures, and to rule out the occurrence of isobaric PPS structures. Following the above study, we examine the combination of the evaporation-grinding MALDI sample preparation method with ion mobility (IM) separation and TOF/TOF CID fragmentation to overcome some of the problems associated with mass spectral analysis of complex polymeric mixtures. In these examples, MALDI-IM/MS will be used to provide clear separation of linear and cyclic species, and distinction between the PPS trendline and the PPSS trendline. The MALDI-TOF/TOF CID fragmentation studies identified a weak link at the phenyl-sulfone bond, which was used to further refine our previously reported polyarylsulfone fragmentation mechanisms and obtain "fingerprint" identification of "suspect" species. This work presents definitive evidence for the existence of: (1) arylthio metathesis reactions, (2) NMP side reactions leading to PPS and PPSS end group modification, and (3) low kinetic energy "venting" of SO2 from PPSS.

(65) **Building the First Commercial ICP-MS**

Don Douglas; [1]University of British Columbia

The early development of inductively coupled plasma mass spectrometry at Sciex will be described. In 1980 and 1981, work at Sciex with a microwave induced plasma (MIP) had demonstrated many of the features that were to make ICP-MS such a success: simple spectra, relative freedom from spectral interferences, low detection limits and isotope ratio information. With the ICP as an ion source, three major technical problems had to be overcome. The very small ion sampling orifice (ca. 50 microns) used in the first ICP-MS work of Houk et al. (Anal. Chem., 52, 2283, 1980) clogged with solutions of even moderate salt content. When larger orifices were used, the ICP arced to orifice, rapidly eroding it. The ICP also produced a much higher continuum background than the the MIP. The use of substantially larger sampling orifices was made possible by using molecular beam sampling methods with a first stage pressure of about 1 Torr. Arcing of the ICP to the orifice was eliminated by modifying the ICP tank circuit to lower the ICP electrical potential. The high continuum background was eliminated by using a "Bessel box" with an on axis stop to prevent photons and energetic neutrals from reaching the detector. These developments, which are still in use on many ICP-MS systems, allowed Sciex to announce the first commercial ICP-MS system, the Elan 250, at the 1983 Pittsburgh Conference.

(66) **A Diamond Jubilee - 30 Years and Counting with ICPMS**

David W. Koppenaal[1]; [1]Pacific Northwest National Laboratory

Inductively coupled plasma mass spectrometry (ICPMS) has enjoyed 30 years of unparalleled success as an analytical technique. It remains the technique of choice for trace and ultra-trace elemental and isotopic analyses, and continues to evolve and improve to maintain its position as the premier metals analysis technique. My experience with ICPMS has been long, varied, and interesting, and this paper will recount these experiences with an eye towards illustrating the power and versatility of the technique. As we celebrate the diamond anniversary of ICPMS, however, we should also look forward, and this paper will also provide a projection and prediction of where ICPMS research will lead us in the future.

(67) **ICP-MS Performance after 30 Years: Filling in the Blanks**

Paul Farnsworth[1], Nicholas Taylor[1], Ross Spencer[1]; [1]Brigham Young University

In its thirty year existence ICP-MS has been studied and used so extensively that one could reasonably assume that all aspects of its performance are well understood. To the contrary, some features of ICP-MS performance have proven to be remarkably resistant to quantitative, fundamental characterization. These include: matrix-induced changes in the plasma ion source, ion losses in the vacuum interface, and matrix-induced changes in ion transport efficiency between the plasma and the mass analyzer. I will discuss our group's recent progress in understanding each of these features of ICP-MS performance. In particular, I will describe measurements of the effects of matrix on argon excited species populations in the plasma ion source, characterization of the effects of skimmer size and geometry on shock formation in the ICP-MS vacuum interface, and direct optical measurements of the size and intensity of the ion beam at the entrance to the mass analyzer of a working ICP-MS. I will conclude the presentation with a summary of what I perceive to be remaining gaps in our understanding of ICP-MS behavior.

(68) **Has the Battle to Eliminate Spectral Overlaps in ICP-MS Been Won?**

John Olesik[1], Patrick Gray[1]; [1]Ohio State University

Spectral overlaps have been recognized as a problem in ICP-MS since its infancy. All elements except In have at least one isotope that is free from isobaric overlaps with other elemental ions. Unlike ICP-OES, the continuum background in ICP-MS is extremely low. Ions at the same nominal m/z as the analyte ion isotope result in background that can degrade detection limits or result in erroneous concentration results. Three main approaches are available on some commerical ICP-MS instruments to reduce, minimize or eliminate spectral overlaps: mass spectral resolution up to 10,000 provided by sector field mass spectrometers, kinetic energy discrimination and ion-neutral gas reactions. Each of these three approaches can reduce spectral overlaps and has significantly improved ICP-MS. However, none provides a universal solution to eliminate all spectral overlaps. Double focusing, sector field mass spectrometers provide the ability to assess potential overlaps and make trade-offs between sensitivity and selectivity. However, isobaric overlaps due to elemental ions cannot be overcome and some molecular ions cannot be completely resolved from elemental ions especially if the ratio of elemental to molecular ion signals is very small. Ion-neutral reactions, such as charge transfer reactions, are highly specific, and sometimes so efficient that spectral overlap ion signals can be trully eliminated. However, this approach is not universal. In some cases collisionally induced dissociation provides sufficient reduction of spectral overlap ion signals. Kinetic energy discrimination can reduce molecular ion signals and in many, but not all, cases provide sufficient improvement to obtain accurate results for many, but not all, applications. The three different, post ion sampling approaches, to deal with potential spectral overlaps will be assessed with examples from a number of different applications.

(69) **Magnetic Sector ICPMS: Enhancements to Sensitivity and Dynamic Range and the Analysis of Small Sample Amounts**

Charles Douthitt[1], Dan Wiederin[2]; [1]Thermo Fisher Scientific; [2]Elemental Scientific, Inc.

The capabilities of both HR and MC-ICPMS instruments are undergoing significant enhancement, even as the platforms mature. Enhancements of sensitivity and abundance sensitivity, combined with the ability to analyze uL samples, are keeping magnetic sector ICPMS at the cutting edge of ICP-MS. 1. Improvements in sensitivity have led champion data approaching 4% ion yields for both MC-ICPMS and HR ICPMS. These improvements in

sensitivity arise from combinations of a new interface pump, new designs for sampler and skimmer cones, and new inlet systems in the ESI Apex and FAST product lines. These improvements in sensitivity have been confirmed for ICPMS systems using desolvating nebulizers, but full evaluation of the consequences for laser ablation and chromatographic inlet systems remains to be done. 2. The dynamic range of both HR and MC–ICPMS systems is close to 13 orders of magnitude, achieved by incorporating both electron multipliers and Faraday cups into the detection system. Making full use of such a large dynamic range requires the addition of retardation lenses; the Element XR has one retardation lens and the Neptune Plus has two retardation lenses as options. 3. High-precision isotopic analysis of very small sample amounts is required by both geo- nuclear chemists. While the increase in sensitivity for both HR and MC-ICPMS and technical development allowing ion counter arrays have allowed high precision analysis at hitherto-unattainable concentrations, they did not solve the sample handling issue for uL samples. The recent development of the micro-FAST inlet system and the use of increasingly low flow rates allows sustained analysis of samples in the tens of uL size range.

(70) **ICP-MS: 30 Years After – Nothing Left To Do?**

Norbert Jakubowski[1], Larissa Waentig[1], Peter Roos[2]; [1]BAM – Federal Institute for Materials Research and; [2]IfADo – Leibniz Research Centre for Work

ICP-MS has become a mature technique during the last 30 years and is found nowadays in numerous routine applications in elemental and isotopic analysis, nevertheless in some of our own applications we observe still an increasing demand for new instrumental development and improvements. An examples from our work will be presented where we reach presently already the limitations of up-to date instrumentation. This example is related to element-tagging of antibodies for an indirect detection of specific proteins. We have developed tagging methods for detection and quantification of element-tagged antibodies in Western blot assays. The tagging chemistry is based on a metal chelating compound such as DOTA which can be bound to a protein via a SCN-linker molecule. The labelled antibody binds to its antigen after sodium dodecyl-sulphate polyacrylamide gel electrophoresis (SDS-PAGE) separation and electroblotting onto a nitrocellulose membrane (Western blotting). The metal labelled antibody is ablated by a laser directly on the membrane material and detection of the metal label is performed by ICP-SFMS. We now have applied 5 different antibodies labelled with different lanthanide elements in a single Western blot and even want to extent the number of labels per assay, but we are limited already by the acquisition speed of our ICP-MS instrumentation. Thus new instrumental concepts in ICP-MS will be discussed, which allow simultaneous detection of all elements with a sufficiently high time resolution.

(71) **Laser Induced Breakdown Spectroscopy: Application to Slurry Samples**

Jagdish P Singh[1], Krishna K. Ayyalasomayajula[1], Fang Yu Yueh[1], Laura T. Smith[1]; [1]Mississippi State University

This invited talk examines the experimental conditions associated with the laser induced breakdown spectroscopy (LIBS) analysis of slurries in order to achieve better measurement precision. Various experimental configurations and sampling methods were tested. We found that using a pick up lens to direct couple the signal to the optical fiber aligned 45° with laser beam can improve LIBS signal about 5-10 times as compared to the standard backward detection method. Sample preparation procedures that can produce same thickness of samples for analysis have been developed based on a spin coating method. For this method, a drop of slurry was placed on the glass substrate. The slurry is then coated on the substrate via a spin coater machine. The thickness of the sample layered on the substrates is dependent upon the weight, original water content in the sample and the type of substrates. Different substrates for the sample preparation method have been evaluated. It was determined the double sided tape attached to a glass slide gave reproducible thickness for the samples and LIBS results without contribution/ interference from the glass substrate. Four calibration samples and an unknown were prepared by adjusting the base simulant composition for SRS Tank 8F sludge simulant. LIBS data of the calibration sample were taken to develop a calibration curve for specific slurry constituents such as Fe, Al, Ni, Ca, and Si. Various data processing techniques have been evaluated to develop these calibration curves. The concentration of the various elements from the unknown are measured and compared with inductively coupled plasma (ICP) data to evaluate the quantitative measurement capability of the LIBS techniques.

(72) **Advances in Gunshot Residue Analysis by LIBS**

Christopher Dockery[1]; [1]Kennesaw State University

Laser-induced breakdown spectroscopy (LIBS) has been used to determine the period of time that a shooter will test positive for gunshot residue (GSR) after firing a revolver. Multiple rounds of primer were fired and samples collected at multiple hour intervals using an adhesive tape pressed against the skin. Samples were analyzed directly using a commercially available laser-induced breakdown spectrometer where barium emission (originating from barium nitrate in the primer) was observed. Population statistics were used to compare suspected GSR to a library of blank samples from which a threshold value was established. Statistically significant results, positive for GSR, are obtained 5.27 days after a firearm discharge using these techniques. Additionally, we have developed an experiment for the undergraduate analytical or forensic chemistry laboratory in which GSR produced from toy cap guns are analyzed by LIBS. This project allows students to investigate the development of a forensic method while addressing proper sampling techniques used in forensic investigations. Students are able to develop a library of blank samples, establish signal detection limits to address legal considerations for determination of false positive and negative error rates, and optimize an emission spectrometer.

(73) **Combined Standoff LIBS and Raman System for Detection of Elemental Composition and Structure of Minerals**

Shiv Sharma[1], Anupam Misra[1], Paul Lucey[1], David Bates[1]; [1]Hawaii Inst. of Geophys. & Planetology

The University of Hawaii has developed a combined laser-induced breakdown spectroscopy (LIBS) and Raman system for standoff detection of minerals on planetary surfaces. The combined standoff LIBS and Raman system uses an 8-inch telescope as collection optics. A double-pulse frequency-doubled Nd:YAG pulsed laser source (532 nm, 100 mJ/pulse, 15 Hz, pulse width 8 ns) is used in a coaxial geometry to excite the target located at standoff distances. We have developed a custom transmission spectrograph with three volume phase gratings and is equipped with a gated thermo-electrically cooled ICCD detector. The spectrograph has spectral range from 457 to 850 nm, which covers the visible to near-infrared (NIR) range for LIBS and entire Raman spectral region for both Stokes and anti-Stokes Raman spectroscopy. The system records the Raman spectra of minerals when the laser is operated in low power density modes; either by reducing the laser power electronically or by increasing laser spot size on the target. Raman spectra of various minerals were obtained up to a target distance of 125 m with short integration time of 1 to 10 seconds. The standoff LIBS spectra of minerals and other materials of interest were obtained by focusing the beam at distance target at 9 m and recording the LIBS spectra in the visible-NIR range by exciting the sample with either a single laser pulse or a double pulse. We will

present Raman and LIBS data of various minerals from standoff distances. The LIBS data recorded on minerals under similar conditions suggest that the performance of the combined LIBS and Raman system is at least 10,000 times better than the commercially available LIBS spectrographs. The advantages of the combined LIBS and Raman system and its possible applications for analyzing minerals and other materials will be discussed.

(74) **Planetary Geochemical Explorations by Remote Laser – Induced Breakdown Spectroscopy (LIBS)**

Samuel Clegg[1], Roger Wiens[1], Olivier Forni[2], Sylvestre Maurice[2], Shiv Sharma[3], Darby Dyar[4]; [1]Los Alamos National Laboratory; [2]Centre d'Étude Spatiale des Rayonnement; [3]University of Hawaii; [4]Mt. Holyoke College

Remote Laser – Induced Breakdown Spectroscopy is an exciting new analytical approach for the challenges of planetary geochemical explorations. LIBS is especially well suited for planetary exploration because it can be used in either an *in situ* (< 10 cm standoff) or remote (> 10 cm standoff) configuration, it can detect all elements above the detection limit regardless of the elemental mass, sample interrogation is rapid, and does not require sample preparation. The ChemCam instrument selected for the Mars Science Laboratory Rover "Curiosity" includes an integrated remote LIBS instrument and a remote micro-imager (RMI). The ChemCam instrument will probe samples up to seven meters from the rover mast for the nominal three year mission. Recently, the Venus Surface and Atmosphere Geochemical Explorer (SAGE) mission was one of three finalists for a NASA New Frontiers Mission that includes a remote Raman – LIBS instrument. A remote Raman – LIBS instrument has also been developed for future Mars rover explorations and a remote LIBS instrument for lunar geochemical explorations is also under development. Novel multivariate analysis techniques have been developed along with the instrument development. Partial Least Squares (PLS) and other multivariate tools can be used to extract quantitative elemental chemistries of rocks and minerals. Principal Components Analysis (PCA), Independent Components Analysis (ICA), and Soft Independent Modeling by Class Analogy (SIMCA) are used to determine the sample rock type. In this presentation, the planetary instrumentation, the resulting LIBS spectra, and data analysis techniques used to process the data will be described.

(75) **Test for Validity of the Boltzmann Plot Method: Implications from Plasma Modeling**

Igor Gornushkin[1], Sergei Shabanov[2], Sven Merk[1], Elisabetta Tognoni[3], Ulrich Panne[1]; [1]BAM; [2]University of Florida; [3]INO-CNR

The validity of the popular Boltzmann plot method is theoretically tested for the case of a non-homogenious non-isothermal laser-induced plasma. A collisional-dominated plasma model is employed to generate synthetic spectra by solving the radiative transfer equation. The spectra are processed with home-made software that calculates values for the plasma temperature and concentrations using the Boltzmann plot approach. Both static and dynamic plasma are investigated at various temperature and density gradients. The plasma parameters obtained from the Boltzmann plots are subsequently compared with the exact parameters of the model. The results are shown to have a direct implication for calibration-free laser-induced breakdown spectroscopy (CF-LIBS). For many tested situations, the Boltzmann plot method was capable of only semi-quantitative analysis providing the accurate determination of concentrations for main plasma components and failing to accurately predict the concentrations of minor components and trace elements.

(76) **Covering My ARSE: Recent Advances in Adaptive Regression by Subspace Elimination**

Karl Booksh[1], Bryon Herbert[1], Seong-Soo Kim[2], Boris Mizaikoff[2], Chance Carter[3]; [1]University of Delaware; [2]Georgia Tech; [3]LLNL

Adaptive Regression by Subspace Elimination (ARSE) has been shown to significantly decrease the quantitative bias from uncalibrated interferents when applied to multivariate data. In some cases the impact of uncalibrated interferents was decreased by 90% or more with only a small increase in variance. This presentation will detail the theory and implementation of ARSE including stability with respect to the complexity of both the calibration model and interferent spectra. The performance of ARSE will be demonstrated on MIR spectra of hydrocarbons collected with a capillary waveguide sample cell.

(77) **Genetic Algorithms for Identification of Cancer Markers from Mass Spectral Profiles**

Barry Lavine[1], Nikhil Mirjankar[1], Yehia Mechref[2], David Clemmer[3], Marie Hanigan[4], Matthew West[4]; [1]Oklahoma State University; [2]Texas Tech University; [3]Indiana University; [4]Oklahoma Health Sciences Center

The development of a genetic algorithm (GA) that employs both supervised and transverse learning to mine proteomic data is reported. The pattern recognition GA selects features that increase clustering of the samples in the principal component plot of the data while simultaneously optimizing the separation of the classes in a plot of the two or three largest principal components of the data. Two specific studies illustrating the efficacy and efficiency of the pattern recognition GA will be described. (1) Time tags that could serve as potential biomarkers for liver cancer were identified from ion mobility mass spectral data. (2) Features from profiles of N-glycans derived from 10fÝl serum samples that could serve as potential biomarkers for pancreatic cancer were identified by mass spectrometry using the pattern recognition GA.

(78) **Determining Microalgal Biodiversity as Novel Environmental Indicator - Combining Spectroscopic, Imaging and Prior Information through Bayesian Statistics**

Frank Vogt[1], Edward Duranty[1], Rebecca Horton[1], Morgan McConico[1]; [1]University of Tennessee, Department of Chemistry

Environmental analytical chemistry often involves investigations of numerous, complex and interrelated effects on ecosystems. To study these impacts, novel chemical sensing strategies are required which yield a more comprehensive picture of chemical/biological trends compared to the measurement of a single chemical parameter. One innovative approach is to use biological entities as novel *in-situ* probes themselves, for instance microalgae cells. Microalgal biodiversity has been reported to respond quickly and sensitively to changes in marine ecosystems while adapting via homeostasis. For experimental studies, algae samples were cultured under different growing parameters focused on concentrations of different nutrient sources, specifically carbon (via CO3-) and nitrogen (via NO3- and NH4+). Then algae samples were dried, mixed with KBr powder and pressed into pellets of known algae weight percentages. Biomaterials contain many characteristic, infrared-active components including amides, amino acids, carbohydrates, and lipids. Thus, FTIR spectroscopy is a powerful technique to acquire experimental data for microalgae studies. Utilizing microalgae samples grown under well-maintained environmental conditions, it is hypothesized that one can establish a relation between the cells' chemical signatures and a marine ecosystem's chemical state. With these calibration procedures in place, chemometric methods for identification of species and

growing conditions could be developed. The biggest challenge for these classification methods is the large number of microalgae species which must be discriminated. This number of possible species is multiplied by different environmental conditions which modify the cells' chemical signature. In order to establish high species classification rates, algorithms were developed based on Bayesian statistics. This method tests the hypothesis whether an algae sample belongs to a certain class, i.e. a species in conjunction with a set of environmental conditions. Bayes' classification gains from incorporating (here) physical, chemical and geometric background information about the samples in addition to their spectroscopic signatures. These Bayesian classification methods along with an experimental calibration database have been incorporated into the remotely accessible, custom-made Virtual Chemometrics Lab for straightforward utilization of these tools. The expected significance of this research is to utilize microalgal biodiversity as a novel probe for assessing chemical changes in marine ecosystems.

(79) **Calibration and Classification Transfer Using Stacked Methods**

Steven Brown[1]; [1]Univ. Delaware

Classification of multi-response data such as spectra has become increasingly common. As in calibration, using a classifier developed on one set of data to classify samples from a second set of data may benefit from correcting for the instrumental and environmental differences – a transfer of the classification model. I report the use of stacking – the weighted combination of portions of the multivariate data - as a way to reduce the effects of changes in the instrumentation on models developed for calibration and classification. The benefits of stacking on calibration and classification are demonstrated on some example data sets.

(80) **Practical and Theoretical Implications of PLS Constraints**

William Rayens[1], Aric Schadler[1]; [1]University of Kentucky

The issue of constraints, both inherent and imposed, in PLS modeling has rarely been discussed in the literature. Certainly, any such discussion is confounded by the many constructs of interest (e.g. scores, directions, loadings) as well as by the different approaches to the actual way in which PLS is implemented (e.g. NIPALS or an optimization problem approach). In this talk we address some of these issues on a very fundamental level. As a result, we are able to point to similarities, as well as expose some irreconcilable differences, between the use of the NIPALS algorithm and eigenstructure methods.

(81) **Neuropeptide Discovery: From New Characterization Approaches to Function**

Jonathan Sweedler[1]; [1]Univeristy of Illinois

Neuropeptides are critical molecules that modulate the physiological activity of every neuronal circuit in the brain. Surprisingly, though, more and more brain peptides are being discovered. Even the rate of brain peptide discovery is increasing. What do these novel peptides do? Two major areas are addressed here, one technical and one biological. The first area highlights mass spectrometry-based technologies to characterize the brain peptides from samples ranging from brain regions to single cells. Using these cutting-edge mass spectrometry-based approaches, we generate lists of known and unique peptides from specific brain regions, with these lists reaching to hundreds of peptides. Even for small brain areas, we still detect hundreds of peptides, making follow-up studies daunting. The second area addresses the question of which peptides are worth extensive follow-up studies. Using the suprachiasmatic nucleus (SCN) as an example, we describe functional studies such as measuring the peptides released from this region in an activity dependent manner and at specific times of the day. Knowing that a novel peptide is only detectable at a specific time of the day or under specific electrical stimulation protocols yields critical information on potential peptide function. For example, one SCN peptide "little SAAS" exhibits robust stimulated release from the SCN, and exogenous application of little SAAS induces a phase delay consistent with light-mediated cues regulating circadian timing. In other cases, well-known brain peptides are shown to have unique functions. Several additional examples of neuropeptide discovery are described across a range of metazoan life.

(82) **Sensitivity of Carbon-Fiber Electrodes**

Mark Wightman[1]; [1]University of North Carolina at Chapel Hill

Carbon-fiber microelectrodes are frequently used for *in vivo* detection of neurotransmitters and their concentration fluctuations in the brain. The detection schemes are required to have sensitivity that is similar to that of the receptors for the neurotransmitter being detected. Strategies to improve detection limits include extending the anodic voltage limit and increasing the sweep rate. Examples of each method will be described.

(83) **Design of Multifunctional Nanoparticle Probes for Molecular Imaging and Sensing in Single Living Organisms**

Dr. X. Nancy Xu[1], Prakash D. Nallathamby[1], Tao Huang[1], Kerry J. Lee[1], Lauren M. Browning[1]; [1]Old Dominion University

We have designed and characterized multifunctional photostable plasmonic nanoparticle (NP) optical probes for study of molecular events of interest in single living cells and single zebrafish embryos. We have developed new imaging approaches to characterize the sizes of single Ag NPs in solution at nanometer resolution by measuring their size-dependent plasmonic spectra and scattering intensity using dark-field optical microscopy and spectroscopy (DFOMS). Our studies show that single Ag NPs exhibit the high quantum yield (QY) of Rayleigh scattering and resist photobleaching and blinking, allowing them to be continuously monitored *in vivo* for any desired period of time. These unique optical properties surpass primary imaging probes (e.g., fluorophores and semiconductor quantum dots) that are currently available for imaging living organisms. With no need of fluorescence excitation, plasmonic NP probes allow us to effectively avoid auto-fluorescence of living organisms and significantly improve the signal-to-noise ratio and to achieve single molecule detection (SMD) sensitivity. We have applied the single NP probes for study of a wide variety of crucial biochemical and biophysical events of interest in single living cells and embryos.

(84) **Smaller, Cheaper, and More User-Friendly: Are We Really Talking about NMR?**

Timothy Peck[1]; [1]Protasis Corporation

Advances in genomics and health science provide opportunities for new synergistic approaches to experimental design, analytical detection, and data reporting. A methodical sample workflow begins with sample prep and purification, and must employ a multiplicity of downstream detectors. These include optical methods, mass-spectrometry, and more recently, nuclear magnetic resonance (NMR). Quantity, purity, and structure remain principle outputs, although newer applications such as metabolomics necessarily incorporate feedback control to factor organism responses into statistically-based decision-making and analysis. Historically the information-rich value of NMR has been diminished due to awkward attributes: systems tend to be large, expensive, high-maintenance, and relatively difficult to use. Even the format of sample introduction – the NMR tube – represents a workflow disruption when compared to more common media and sample formats (wellplates, microvials) used throughout the rest of

the analytical laboratory. The net result is that NMR is often labeled as a "technique of last resort," and typically not even located near the point of sample origin. Technological advances are helping to mainstream the role of NMR in sample workflow. Materials advances have lead to smaller superconducting magnets that are virtually maintenance-free. Robotic automation platforms provide a means for barcode submission of wellplate- and microvial-based samples and make unnecessary the cumbersome and costly transfer to and from NMR tubes. Probe adjustments (e.g., tuning and shimming) between samples is unnecessary in modern small-molecule applications, and feedback control ("smart") sensors coordinate all aspects of sample introduction, analysis, and recovery as well as system cleanup and maintenance. Advances in computational horsepower and data storage now empower decision-making algorithms to perform at levels hardly imaginable a decade ago, and provide a means of processing larger numbers of samples and more focused direction of human effort. A new generation of compact, economical, "push-button" NMR systems that incorporate these advances are being utilized at the point of sample origin - in synthesis laboratories, next to the reaction vessel, or on the production floor. This talk addresses the principle components of the modern NMR spectrometer and illustrates ramifications of recent technological advances to considerably reduce size and costs, improve ease-of-use, and increase versatility.

(85) **NMR with Small Magnets**

Bernhard Bluemich; [1]RWTH Aachen University

The most prominent uses of Nuclear Magnetic Resonance (NMR) are in Chemistry for molecular analysis and in Medicine for diagnostic imaging. Typically the object is placed inside a huge magnet that is commonly understood to enclose the object and to have high field strength for good sensitivity. While the electronics are shrinking noticeably over the years, the magnets become bigger as higher field strength is realized. This restricts the NMR analysis to the location of the magnet. Recent advances in magnet design have led to compact and miniature magnets from permanent magnet material with field strengths common a few decades ago. Magnets that enclose the sample in the conventional way and magnets that approach the object from one side have been developed for NMR relaxation analysis, imaging, and high-resolution spectroscopy. They are inexpensive and can be moved to where needed. The availability of such magnets makes a diversity of NMR studies possible, which are out of question for high-field super-conducting magets. These are high-thoughput chemical analysis and inline monitoring in the factory, NMR measurements at the site of the object, and investigations in dangerous environments. Some recent advances in compact NMR technology are summarized, and the use of such instruments is demonstrated with applications from materials testing including the non-destructive analysis of cultural-heritage objects, chemial engineering, and biomedicine. Reference: B. Blümich, F. Casanova, S. Appelt, NMR at low magnetic fields, Chem. Phys. Lett. 477 (2009) 231-240

(86) **Bench-Top and On-Line High Resolution Permanent Magnet 60 MHz NMR for Reaction Monitoring and Process Control**

John Edwards[1], Paul Giammatteo[1]; [1]Process NMR Associates, LLC

Permanent magnet 60 MHz NMR systems producing high resolution NMR spectra for chemometric based chemical and physical property prediction have been utilized for process control and optimization in refineries and chemical plants for over a decade. An overview of the permanent magnet technology and the small footprint spectrometer design utilized in these systems will be presented as well as some typical refinery applications. Furthermore, the use of an NMR as a simple flow detector for bench-top reaction monitoring, micro-reactor monintoring, mixing monitoring, dilution monitoring, or conversion monitoring will be described. Up to now the use of NMR as a bench-top or "in the fumehood" analyzer has been limited by the need to bring the "reaction" to the typical "supercon" NMR lab. Small footprint NMR systems that can be used as continuous flow detectors and/or an "*in-situ*" reaction monitoring systems are now available and produce highly stable and reproducible NMR data that brings the power of NMR chemical specificity to the process analytical technology arena. The bench-top NMR system utilizes the same high resolution 60 MHz permanent magnet with a simple flow cell and total system volumes of 2 to 5 ml depending on the length and diameter of the transfer tubing. Further, detection limits of analytes in the 200+ ppm range are possible without the use of typical deuterated NMR solvents. Analysis times of 5 to 20 seconds are also possible at flow rates of 5 to 20+ ml/minute. Reaction monitoring directly in standard 5 mm NMR tubes again using conventional (non-deuterated) reactants, solvents and analytes is also described.

(87) **Capillary Electrophoresis Hyphenated with Slotted Microstrip Nuclear Magnetic Resonance Detection**

Roland Hergenroeder[1], Joerg Lambert[1]; [1]ISAS

A new NMR microprobe based on microstrip technology has been established. Owing to its planar design, the probe is easily adaptable to the size and geometry requirements of the samples. The sensitivity of the probe is by factor of 70000 better than that of a commercial NMR probe. With the detection volume being in the low nanoliter range, the probe is not only well suited for investigations on volume limited samples, but also as a detector for chromatographic or electrophoretic separation techniques. In addition, the design is well suited to the implementation into microfluidic manifolds. In currently available solenoidal microcoil designs, the sample tube in electrophoretic separations must be oriented perpendicular to the external magnetic field. Hence, electrophoretic currents, following Ampere's law, give rise to a magnetic field gradient in the flow direction deteriorating spectral resolution and causes substantial distortions in the NMR spectral linewidths and peak heights. These pitfalls can be completely avoided with the new microstrip probe allowing the sample tube to be oriented in parallel to the external field. Hyphenation of capillary electrophoresis and NMR detection based on a microstrip NMR detector are therefore expected to give enhanced spectral resolution as compared to solenoidal microcoil detection. This outstanding sensitivity advantage will be exploited for metabolic studies on synchronized cell ensembles that pose high demands on the selectivity and sensitivity of spectroscopic techniques and have therefore been out of reach hitherto.

(88) **Mass Limited Sample Detection Using cITP Microcoil NMR**

Christopher Jones[1], Cynthia Larive[1]; [1]UC Riverside

NMR spectroscopy is a useful technique that can yield a vast amount of structural information about a compound without destroying the sample. However, it is often limited by poor sensitivity and for samples containing more than one compound of interest, the spectra obtained can quickly become convoluted, complicating interpretation. This is especially true for samples that contain trace impurities that are structurally related to the primary component, a challenge commonly encountered in the drug development process. To address these problems, our group uses online separation methods coupled with NMR detection. An example of such a separation technique is capillary isotachophoresis (cITP). This is an electrophoretic method that can focus charged analytes by up to 2 or 3 orders of magnitude while

also separating them based on their electrophoretic mobilities. By coupling cITP with the increased mass sensitivity of solenoidal microcoil NMR probes it is possible to separate and focus trace impurities to a similar concentration as the more abundant compound. While NMR spectra for focused analytes are typically collected on a transient basis as they pass through the NMR coil when coupling cITP to NMR, stop flow methods can be used to trap analytes of interest in the active volume of the coil which allows for signal averaging and improved S/N ratios.

(89) **Advanced LC/Microcoil NMR Methods for Structure Determination and Metabolite Profiling**

Daniel Raftery[1], Ravi KC[1], Emmanuel Appiah-Amponsah[1], Kwadwo Owusu-Sarfo[1], Tao Ye[1], G. A. Nagana Gowda[1]; [1]Purdue University

The analysis of complex samples such as biofluids poses a number of analytical challenges for NMR. A currently popular approach is the combination of NMR and multivariate statistics that provides a successful methodolgy to analyze the high concentration species for metabolite profiling. However, in order to delve more deeply into the complex samples and analyze lower concentration species that may be more sensitive or specific to changes in biological states, more advanced methods are attractive. The combination of chromatography and/or fractionation with advanced microcoil NMR methods is very useful to identify low concentration metabolites of sometimes unknown structures that are present in complex biofluids. This approach is very complementary to the more conventional methods of whole fluid analysis and the knowledge gained can be leveraged back for high throughput analysis. The use of microcoil NMR is attractive because of the limited amount of these metabolites present at low concentration. In fact, the number of detectable metabolites in the low uM range far exceeds those at high concentration, but are obscured by overlapping peaks such that their detection is challenging. Strategies to observe these species will be described in the talk, such as a recently developed dual microcoil NMR flow probe. One approach is to perform targeted metabolite profiling as it provides a powerful method to circumvent some of the issues resulting from sample complexity. Along these lines, we have recently demonstrated the use of a simple isotope tagging strategy, in which metabolites with carboxyl groups are chemically tagged with 15N ethanolamine followed by detection by 2D NMR. Similarly, amine containing metabolites are tagged with 13C labeled acetic anhydride or formic acid. This approach showed improved detection (on the order of 100x with LOD of a few uM), with concomitant high resolution and reproducibility. We present an approach involving the use of hydrophilic interaction chromatography (HILIC) to facilitate the resolution of these polar "tagged" metabolites in human urine prior to detection by NMR. Once they are isolated, microcoil NMR can be quite useful in helping to determine their structure.

(90) **Recent Advances in IRIRI of Polymers**

Marek W. Urban, Biswajit Ghosh, Dhanya Ramachandran; [1]USM

Recent advances in stimuli-responsive materials resulted in new analytical challenges of monitoring transient processes at molecular levels. This presentation will focus on new developments of stimuli-responsive polymeric materials that mimic processes occurring in nature and the role of internal reflection infrared imaging (IRIRI) in detecting molecular level events associated with these processes. With a spatial resolution below 1 micron, mechanically induced damages and subsequent self-healing processes in polyurethanes were monitored using IRIRI, revealing molecular events responsible for UV induced autonomous repair. The presence of four and five-member rings within the network provides a source of reactive species capable of crosslinking upon UV exposure. Furthermore, a new family of azobenzene-crosslinked brominated vinyl ester polymer networks that exhibit reversible photochromic and fluorescence properties was developed. Structural and conformational changes resulting from network deformations were monitored using IRIRI, and showed that trans-cis transitions in azobenzene crosslinker lead to conformational changes in the surrounding network and subsequent fluorescence emission and color changes. The crosslinker also serves as an internal molecular sensor capable of detecting minute network deformations.

(91) **Mid-Infrared Imaging of Tissue: Reduction of Confounding Scattering Effects**

Max Diem; [1]Northeastern University

Microscopic Infrared spectral imaging of tissue sections has been plagued by distortions of the absorption, reflection or transflection spectra by reflective band shapes. The pioneering work by Peter Gardner (Manchester, UK) has demonstrated that the reflective band shapes are mixed with absorption features via a process now referred to as resonance Mie (r-Mie) scattering. The effects of the r-Mie scattering are often so severe that multivariate methods such as cluster analysis cannot be applied to the datasets. In this presentation, the origin of r-Mie scattering, and ways to overcome the band distortion by methods of Extended Multiplicative Signal Correction (EMSC) will be discussed, and dramatic improvements of the IR images will be demonstrated.

(92) **Dynamic Chemical Imaging in the Near Infrared**

Patrick Treado[1], Matthew Nelson[1], Charles Gardner[1], Ryan Priore[1]; [1]ChemImage Corporation

Near infrared (NIR) chemical (i.e. hyperspectral) imaging sensors for detection of a wide variety of materials in challenging environments are rapidly maturing. For example, detection of hazardous materials in outdoor environments is accomplished by exploiting measurable optical phenomena with hyperspectral imaging sensor designs that can be made to operate while on the move (OTM) at appreciable speeds. This presentation will emphasize strategies being employed for sensor exploitation, as well as sensor design.

(93) **From Formulating for Performance to Fingerprinting Counterfeits: A Review of the Roles of NIR Chemical Imaging in the Pharmaceutical Industry**

Janie Dubois[1], Linda H. Kidder[1], E. Neil Lewis[1]; [1]Malvern Instruments Inc.

NIR chemical imaging (NIRCI) is a powerful tool for the analysis of coated and uncoated pharmaceutical tablets, granules, extrusion cores, wafers and trans-dermal delivery systems. It enables a measurement of the chemical heterogeneity present within a sample: Chemical heterogeneity can be deliberate and part of the structure that yields a desired performance or it may be caused by process changes and negatively impact performance. There has been a trend to move the use of NIRCI into formulation design, where this analytical technique is widely used in the determination of blending, coating or milling end-points, risk assessment of ingredient selection and changes of suppliers, while remaining a workhorse for trouble-shooting of dissolution failure as well as counterfeit detection and sourcing. This presentation will provide a review of the various measurements made with NIRCI to fulfill this broad role. How does the pharmaceutical industry benefit? Better understanding of processes and products leads to better control, less regulatory burden when changes need to be made, and better profitability. In this regard, NIR chemical imaging is fully aligned with the QbD initiative and provides information that bridges the gap between the quality assurance of starting material and performance verification of finished tablets, oral wafers, granules,

capsules and transdermal patches. In addition, the analysis of intermediates enables the building of a knowledge base on the effects of ingredients and modifications at various stages of the process during formulation development, and further link into the consequences for finished products. In short, NIRCI is a risk assessment tool that provides quantitative data for the determination of critical quality attributes. The knowledge gathered during formulation development can save a lot of time and money when having to deal with a performance failure. However, even when it was not used to collect this important information during formulation development, NIR chemical imaging remains a powerful tool for root-cause analysis of performance failure of finished products and intermediates, enabling the rapid implementation of corrective measures. As such, it can have a significant impact on production efficiency and cost.

(94) **Characterization and Monitoring Dynamic Processes of Nanomaterials by Near-Infrared Spectroscopic Imaging Technique**

Chieu Tran[1]; [1]Marquette University

Near-infrared multispectral imaging (NIR-MSI) microscopy has been used to provide the first direct observation and spectral measurement of individual poly(n-isopropylacrylamide-co-acrylic acid) (NIPAM-co-AAc) hydrogel particles. The high sensitivity and high spatial resolution (~0.9 μm/pixel) of the NIR-MSI microscope, coupled with its ability to measure images and spectra directly and simultaneously, allows the unprecedented *in situ* monitoring of the size, morphology, and spectroscopic properties of individual hydrogel particles, which respond strongly to external stimuli (e.g., changes in temperature and/or pH). Importantly, this novel technique allows, for the first time, the direct measurement of the lower critical solution temperature (LCST) phase transition of individual hydrogel particles rather than that of a collection of hydrogel particles. Furthermore, NIR-MSI measurements reveal that the LCST value is unique for each individual hydrogel particle, depending strongly on particle size, with larger particles exhibiting higher LCST values.

(95) **Enhanced Models for Fourier Transform Infrared (FT-IR) Spectroscopic Imaging of Human Tissue Specimens**

Rohith Reddy[1, 2], Brynmor Davis[1], Rohit Bhargava[1, 2]; [1]University of Illinois at Urbana champaign; [2]Beckman Institute for Advanced Science

Fourier transform infrared (FT-IR) spectroscopic imaging is an emerging technique that provides both chemically and spatially resolved information. The rich chemical content of data may be employed for computer-aided determinations of structure or pathologic state of biological specimens. An exciting emerging avenue is the use of one such technique, Fourier transform infrared (FTIR) spectroscopic imaging for determining structure and disease within tissue (histopathology). An important question in this approach is whether the numbers of spectra, samples and patients employed to construct computer algorithms influences the prediction performance of the developed algorithms. In this work, we demonstrate theoretically and experimentally the effects of these parameters on histologic classification of prostate tissue. Results indicate that a small number of samples, carefully tested and identified with appropriate biologic significance, can be employed to provide robust classification schemes. We also present data visualization techniques that format would help both experts and non-experts visualize complex relationships in data. The spatial heterogeneity of samples, which makes spectroscopic imaging more useful than point spectroscopy, results in spectral distortions. We present models based on rigorous electromagnetic wave theory quantifying these distortions. We observe shifts in peak positions and variation in peak heights at the boundary of two materials primarily due to optical effects. An understanding of these spectral distortions is especially important in performing automated data analysis which is becoming increasingly necessary due to the size of datasets in imaging. We explore methods of extracting underlying information from distorted spectra. In particular, we show that choosing appropriate metrics and classification algorithms in human tissue histopathology can help overcome consequences of these spectral distortions.

(96) **The Don Sting Legacy: Making FTIR Accessible to the World**

John Coates[1], James Rydzak[2]; [1]Coates Consulting; [2]GlaxoSmithKline

This paper kicks off a special memorial session entitled "How One Man Changed the World of FTIR and Infrared Spectroscopy; The Don Sting Legacy." By nature, this session will be very anecdotal, with the lives of all of the participants being directly involved and intertwined with Don Sting. Don Sting passed away quietly with his family by him on Monday, March 29 after a long and courageous battle fighting a brain tumor. Don had a brilliantly sharp mind and it was incredibly cruel for him to have suffered such a debilitating disease. In this special session we will relive the life and contributions of Don Sting and the impact of his work on the world of FTIR. In March 1983 I was standing in the Perkin-Elmer booth at Pittcon and Paul Wilks walked up to me and said, "I want you to meet somebody; I want you to meet Don Sting. Don will change the way that you work with infrared spectroscopy. He may even change your life." Those words have echoed many times, and have meant much more than I could have imagined at the time. Don had formed the company SpectraTech and the products, FTIR accessories, that came out of this company revolutionized and changed FTIR sample handling forever. This made a huge impact on the way industrial measurements were performed. These accessories led to the development of smaller FTIR instruments and with dedicated applications like the ATR Microscope, the TravelIR and the in-process instruments that are known as ReactIR. As a spectroscopist the accessories like the Circle Cell and the Diamond ATR really changed the way that work was done on liquid and solid products alike. In addition, these instruments also made difficult tasks seem easy, such as looking at solid polymorphs under the microscope or determining the active pharmaceutical ingredients in synthesis reactions. Don truly changed the way we do spectroscopy, enabling the expansion of applications of FTIR, and making it easy to do; opening up spectroscopy for use by the common man! This paper will discuss what this really meant to an end-user.

(97) **Making Concepts into Products: From Barnes to A2…the History, the Companies and the Products**

Steven Donahue[1], Robert Messerschmidt[2]; [1]A2 Technologies; [2]Rare Light, Inc.

In 1979, with no background in optics or spectroscopy, Don entered the infrared world by purchasing Barnes Analytical. Spectra-Tech was thus born and the rest, as they say, is history. Spectra-Tech made its name as the premier supplier of infrared accessories. This period saw the widespread acceptance of ATR via commercialization of the CIRCLE cell and horizontal ATR. Perhaps the most notable achievement was the development of the IR Plan, the first high volume infrared microscope. Late in the '80's the Dedicated Solutions group was created, which would be spun out as ASI Applied Systems following the sale of Spectra-Tech to Nicolet. ASI introduced a theme that would become more important as Don's career progressed; make FTIR spectroscopy accessible to the non-spectroscopist. The MonitIR was introduced for QC/QA applications. ASI's most important product, the ReactIR reaction analysis system, continues to this day. The

combination of dedicated spectrometer and application specific software coupled with the development of diamond ATR probe technology put routine FTIR analysis in the hands of organic chemists and chemical engineers. As ASI continued to grow SensIR Technologies was created to find other outlets for the technology and became an independent entity following the sale of ASI to Mettler-Toledo. Continuing to see the benefit of integrated systems Don and the SensIR team developed their first modulator, which would find use in both ASI and SensIR products. SensIR products included the IlluminatIR and the TravelIR. SensIR's success will always be identified with the HazMatID, which truly brought FTIR out of the laboratory. The HazMatID continues to be used by first responders around the world to identify potential hazards. As in the past, the sale of one company was simply the starting point for the next. Following the purchase of SensIR by Smiths Detection, A2 Technologies was created and continues Don's legacy of innovation. Looking to take FTIR further out of the laboratory the PAL / iPAL dedicated analyzers were developed using the latest modulator and the TumblIR transmission apparatus. The first high performance handheld FTIR, the Exoscan, has found use in a variety of applications, most notably in aerospace.

(98) **Don Sting the Engineer, the Innovator and the Inventor**

Scott Little[1], Gregg Ressler[2]; [1]Focal Point International LLC; [2]A2 Technologies, Inc.

From the moment Don Sting started the first of his four companies in the late 70's, Spectra-Tech, through the end of his involvement with his last start up in 2009, A2 Technologies (named after grandchildren whose names start with A), he was required to fill many roles. Deciding to leave big corporate life to pursue a small high technology start up is a culture shock that even some of the most talented leaders fail to make successfully. Perhaps the most elusive attribute requisite in navigating this transition is clear vision. Don's emotional intelligence, leadership, and vision of market possibilities were leveraged via a burning passion for the art of competing. While he was successful wearing many hats through the years, it was clear day one which role he loved the most. His passion was engineering, innovation and invention. But unlike most other entrepreneurs, his passion was not carried out through management and supervision activities. Don was a doer who insisted on being engaged in agonizing detail. The formula Don used for creating new products was the same from Spectra-Tech to A2. He would construct young, creative, aggressive cross functional teams of product developers and insert himself at the center of the design activities. The teams he created were unfazed by the magnitude of development challenges. Faced with problems that had never before been solved, the approach was to continuously try new things with the view that nothing is impossible. Don's process of innovating and inventing was founded on constant questioning, learning, experimentation, and always solving the correct problems bolstered by constant contact with the customer and the application. This process created an electric environment where team members did not want to stop working, and fostered improvisation and creativity that resulted in a myriad of new market creating products. A detailed and sometimes humorous look into his approach as well as his legacy of inventions will be presented.

(99) **FT-IR : Migration from the Lab to the Field**

David Schiering[1], John Seelenbinder[2]; [1]Smiths Detection; [2]A2 Technologies

Ten years ago, the first practical, man-portable FT-IR spectrometer was introduced. Instrumental developments in the field of portable FT-IR instrumentation quickly overshadowed this first instrument which now seems archaic by today's standards. This led to a new chapter in both FT-IR instrumental development and applications. Several portable and hand-held FT-IR models are now available through several companies and are solving very important, practical applications where transport of the sample is not desirable or possible. These portable and hand held systems are used for many applications including identification of unknowns, qualifying raw materials, quantifying contaminants, characterizing geological samples and non destructively testing composites, often by professionals not academically trained in chemistry or spectroscopy. Although it sounds cliché, this is where the rubber hits to road in 'ease-of-use' and 'practical spectroscopy.' In the second half of his career, Donald Sting was at the forefront of these developments. Today, the companies that he founded are still among the major players in hand held and portable instrumentation. This presentation will follow the evolutionary development of portable, miniaturized FT-IR spectroscopy instrumentation and applications, perhaps the most important legacy of Donald W. Sting.

(100) **Bringing FT-IR Spectroscopy and Microscopy Together – Another Don Sting Legacy**

John Reffner[1], Greg Ressler[2], Robert Messerschnidt; [1]John Jay College, CUNY; [2]A2 Technologies

When Paul Wilks received a call from Digilab asking him if he could build a microscope accessory for their FT-IR spectrometer, he referred them to Don Sting. "I am sure Don can do the job and he probably needs the work", were his prophetic words. Infrared microspectroscopy development at Spectra-Tech was a great challenge and its success is a tribute to the leadership and creativity of Don Sting. Don and R. G. Messerschmidt Designed and produced the UMA-100 under a contract with Digilab. This was the first commercial microscope accessory for a FT-IR spectrometer. It was introduction at PittCon-1983. This accessory microscope met Digilab's needs and was considered a success. Don had a larger vision. He wanted a microscope for every sample compartment and an accessory microscope for every FT-IR bench. Work began on the IR-Plan microscope. At PittCon 1986, every major FT-IR spectrometer manufacturer had an IR-Plan microscope in their booth. This was followed by a session of accessory microscopes and a dedicated infrared Microprobe, the IRus. One of the more significant developments was the ATR-objective. This special purpose objective brought internal reflection spectral analysis to the microscope. The spectacular results that were achieved using small ATR elements lead Don to pursue their application in general analysis and process control. This is another chapter in Don Sting's legacy. Don did not just develop instruments; he built a company of dedicated contributors. Above all Don Sting was a true leader. He led by insight and example. He challenged all of us to do more than we felt we were able to do, but his simple smile and faith in us assured success.

(101) **Understanding the Chemistry and the Process: Tools for Reaction Monitoring**

Alan Rein[1], Henry Dubina[2]; [1]A2 Technologies; [2]Mettler Toledo Autochem

As an outcome of work started at Merck in the late 1970's, there was interest in new methods for monitoring biological reactions such as fermentations. These are aqueous based reactions and the common thinking at that time was that mid IR was not a useful methodology for analysis of aqueous solutions. Paul Wilks, a consultant to IBM Instruments in the early 1980's, proposed a device for overcoming the problems of monitoring aqueous solutions based on a new internal reflection flow cell that used a cylindrical shaped ZnSe ATR rod. In the early 1980's, Don Sting, Paul Wilks and scientists at IBM collaborated on the development of the CIRCLE Cell. This novel device was the first of the major new Spectra-Tech sampling accessories. Towards the end of the

1980's, Don decided that Spectra- Tech would develop complete FTIR analyzers for specific markets, and one of those chosen was reaction monitoring. In 1989, a new FTIR analyzer was announced, that was purpose-built for reaction analysis, and this device was named ReactIR. Original versions of the ReactIR technology incorporated ZnSe and AMTIR ATR elements into the base of glass reaction vessels. Though these early systems were large and expensive, some important chemical reactions were studied and the confidence that a market for FTIR reaction monitors existed was developed. In the mid 1990's, the scientists and engineers of Don's new company, ASI Applied Systems, worked with Mettler Toledo mechanical engineers and Merck chemical engineers to incorporate a internal reflectance based FTIR insertion probe into the Mettler RC1 Reaction Calorimeter. The merging of these two technologies provided kinetics, thermodynamics and mechanistic information about chemical reactions. It became clear that a new type of insertion probe was needed with the capability to handle a much wider range of chemistries. Don and Milan Milosevic worked on the development of the first commercially viable diamond ATR probe. Their efforts led to the development of the revolutionary DiComp diamond ATR sensor. The use of diamond with its inherent strengths substantially accelerated the value and use of FTIR spectroscopy for reaction monitoring in the lab and plant environments. The DiComp ATR sensor created a true "paradigm shift" in the FTIR community and it positively affected the application of FTIR-ATR in virtually all markets and applications. After Mettler Toledo acquired ASI Applied Systems and began the Autochem division, the development of FTIR reaction analyzers for both lab and plant applications accelerated and there are now thousands of ReactIR FTIR reaction analyzers in use throughout the chemical and pharmaceutical industry. Current products are far smaller, more robust and utilize other technologies such as fiber optics; however the basic principle of using internal reflection based probes in conjunction with dedicated FTIR spectrometers for monitoring chemistry continues today as a very important application for mid IR spectroscopy.

(102) **Spectroscopic Solutions to Support the Design of an Attribute Based Manufacturing Process**

John Bobiak[1], Dongsheng Bu[1], Dimuthu Jayawickrama[1], Kevin Macias[1], Tim Stevens[1], Boyong Wan[1], Gary McGeorge[1]; [1]Bristol-Myers Squibb Co.

Detection and control of risk-bearing formulation attributes are essential parts of designing a robust drug product manufacturing process. Spectroscopic applications are commonly leveraged in manufacturing process design due to their ability to detect key attributes like blend uniformity, density, and tablet assay. This presentation will highlight the impact of near-infrared (NIR) investigations (online, at-line, and imaging) on manufacturing process design for roller-compacted, low-drug load formulations (<10% w/w active).

The quality-related function and technical considerations for the following methodologies will be discussed:

• Online NIR for estimating blend uniformity;
• NIR imaging of blends to understand scale-dependence of uniformity;
• -line NIR for estimating ribbon density;
• At-line NIR for tablet assay

(103) **Monitoring Stem Cell Cultivations with NIR Spectroscopy**

Francisca Folque[1], Tiago Fernandes[1], Margarida Diogo[1], Joaquim Cabral[1], José Cardoso de Menezes[1]; [1]IBB,Technical University of Lisbon (IST)

Spectroscopy based *in-situ* and multiparametric monitoring tools are reasonably well established in biomanufacturing as part of PAT (process analytical technology) and QbD (quality by design) efforts, but to our best knowledge have not yet been tried in stem cell cultivations where their potential for improved process understanding and control could easily be justified Here we report on the at-line use of near-infrared spectroscopy (NIRS) with an immersion transflectance probe in samples taken from stem cell expansion in a microcarrier-based stirred culture system. Calibrations for important substrates and metabolites will be described together with the fingerprinting capabilities of NIRS. Such intensification on monitoring of defined and more complex critical process parameters offer great promise in terms of process optimization and more consistent and sound bioengineering approaches to stem cell cultivations. Keywords: NIR monitoring, stem cell cultivations, PAT based approaches

(104) **Development and Application of a General NIR Model for Monitoring the Moisture During Fluid-Bed Drying**

Patrick Rameas[1], Antonio Peinado Amores[1]; [1]GlaxoSmithKline

Drying is an important operation in the processing of wet-granulated products, the objective of which is to remove the moisture added during granulation to a level suitable for down-stream processing. However, developing an on-line NIR model to monitor the drying evolution and determine the process end point during commercial manufacturing is a challenging task as the NIR spectra are being collected under conditions that cannot be easily reproduced off-line. For example, NIR to sample interface is dynamic because the material is fluidised and physical changes to the material such as elutriation and attrition may occur affecting the representativeness of the NIR Spectra. On the contrary, the NIR signature of free water is very evident and characteristic and can be easily identified in different formulations. This work describes the steps followed to build a general-use NIR model which is scale and product independent, to monitor the drying evolution of fluid bed drying operations. The general model is intended to be used to monitor the drying kinetic in real time from the first batch of development. It can be also used to assess the influence of process events (e.g. filter shakes, sampling), in the dynamic environment of the fluidized bed. In principle, the general-use model is not intended to be used to determine the drying end point. However, if it is implemented within routinely manufactured batches, it will reliably predict the drying end point with a reproducible off-set.

(105) **The Choice of Validation Level as the Basis for Critical Limits in Raw Material Identification and Process Monitoring**

Frank Westad; [1]Camo Software

Multivariate methods are important tools for identification of raw materials and process monitoring across many industries. Rapid collection of signals with multichannel instruments provides versatile tools for these objectives. The sources of variation in this context are numerous, e.g. the sampling procedure, sample preparation, the amount of impurities, raw material supplier, particle size, season, operator, instrumentation etc. The tradition within the chemometric community is to distinguish between two criteria for outlier detection: 1. The distance from a sample to the model center in the model direction (leverage) 2. The distance to the model (residual variance). There exists an ad-hoc criterion for the leverage as a function of the number of components in the model and the number of calibration samples, but the past years the use of the Hotelling's T2 statistic has become more widespread. The critical limits for the model distance are often based on F-tests of ratio of variances or the so-called Q-residual statistic. Although correction for degrees of freedom adds a conservative flair to the statistical limits it does not reflect the actual validation level in the specific case. Cross-validation at the proper validation level is a way of estimating limits according to the objective. One example is when data have been collected over various seasons; then the

critical limits should reflect the robustness of the model over time. The comparison of calibration and validation based critical limits are shown for raw material identification and process monitoring using end-point models in pharmaceutical applications.

(106) **Application of Multivariate Data Analysis to Assess Scalability and Identify Scale-Dependent Variables of a Yeast Fermentation Process**

Louis Obando[1], Thomas Potgieter[1], Venkata Mangalampalli[1], Charles Miller[1]; [1]Merck

Merck has adopted QbD principles outlined in the ICH guidelines for the development many of its products. This work demonstrates the strong link between QbD and multivariate data analysis (MVDA) as it is used here to analyze and represent the multi-dimensional space that results from a QbD approach. Hundreds of small scale experiments are typically done during the development of vaccines and biological therapies. These start as shake flask experiments and grow in scale to tens of liters to hundreds of liters and finally to thousands of liters at production scale. Range-finding studies are typically done at small scale where hundreds of experiments can be done to identify the Critical Process Parameters (CPPs) and their impact on the product's quality, safety and efficacy. The difficulty arises upon scale-up and in how to demonstrate and assure that the critical parameters found at small scale, have been properly scaled at larger scales. Typically, this is done by univariate comparisons of profiles such as respiratory quotient or by simply comparing the end product's quality parameters. In this work, we have used multivariate data analysis to assess the scale-up from 30 liters to 800 liters. Independent PCA analysis of batches from both scales proved useful in understanding variable relationships within and across scales. Projection of 800L batches into a multivariate space defined by 30L batches showed the scales to be essentially equivalent. Furthermore, partial least squares (PLS) models were used to regress process parameters against product quality attributes such as titer. MVDA was used to construct a product design space based on small scale batches where parameters are intentionally varied and their impact to product quality was understood. The same multivariate models can be updated for continuous process verification at production scale where random variation dominates. This presentation will demonstrate both of these aspects.

(107) **At-Line PAT Methods in Supporting Active Precision Coating End-point Determination and Coating Process Development**

Fan Zhang-Plasket[1], John Higgins[1], Ramasamy Manoharan[1], Charles Miller[1], Louis Obando[1], Bruce Thompson[1], Gert Thurau[1]; [1]Merck Sharp and Dohme Co.

The precision coating process for a pharmaceutical active ingredient is known to give larger variation on the active content uniformity than the conventional compression process. The measure and control for coating end-point is highly desirable due to the coating efficiency variation. Various PAT methods evaluated for the active content uniformity analysis on large number of tablets to support the coating process development and for the coating end-point determination are presented. The implementation of the selected method is discussed.

(108) **Using Mid-Infrared Spectroscopic Imaging Basis Sets for Quantitative Raman Spectroscopy**

Matthew Schulmerich[1], Michael Walsh[1], Matthew Kole[1], Michael Asensio[1], Rohit Bhargava[1]; [1]Univ. of IL., Urbana-Champaign

Fourier transform infrared (FT-IR) spectroscopic imaging is a potentially useful tool for automated tissue histopathology. The underlying data consist of both spectral and structural images, which act as inputs to statistical pattern recognition classifiers to identify epithelial and stromal cells, collectively providing recognition of tissue structure and disease. While Infrared imaging is effective for biopsies, Raman spectroscopy is more suitable for analyses *in vivo*. We explored the possibility of using IR spectral images as basis sets for calibrating a single Raman spectrum collected over the same area. In our approach we collected the Raman signal with a single fiber optic to assess epithelium to stromal volume fractions of biopsies on aluminum-coated microscope slides. This approach can be extended to allow independent evaluation of the Raman signal, collected by each fiber in a hyper spectral data set, to recover the underlying histological information that is otherwise not obtainable. At the same time, insight into the histological spatial distribution of the tissue is gained by correlating the coordinates of each collection fiber in a hyper-spectral Raman system. We explored feasibility using a prostate tissue micro-array containing cancer and normal biopsy sections with a goal of localizing masses of epithelium in prostate tissue. Experiments are still underway and the latest results will be reported.

(109) **Raman Spectroscopy and the Search for Life on Mars: A Biological and Geological Perspective**

Howell GM Edwards; [1]University of Bradford

The survival strategies of extremophilic organisms in terrestrially stressed locations and habitats are critically dependent upon the production of protective chemicals in response to desiccation , low wavelength radiation insolation , temperature and the presence of toxins. The adaptation of life to the prevailing conditions involves the control of the substratal geology ; the interaction between the rock and the organisms is critical and the biological modification of the geological matrix plays a significant role in the overall survival strategy . The identification of the these biological and biogeological chemical molecular signatures in the geological record is ,therefore, a crucial stage in the recognition of the presence of extinct or extant life in terrestrial and extraterrestrial scenarios. Raman spectroscopic techniques have been identified as valuable instrumentation for the detection of life extraterrestrially because of the use of non-invasive laser-based excitation of organic and inorganic molecules and molecular ions with high discrimination characteristics ; the interactions effected between biological organisms and their environments are detectable through the molecular entities produced at interfaces , for which the vibrational spectroscopic band signatures are unique. The combination of Raman spectroscopic and optical data acquisition gives an additional information category which is essential for the description of heterogeneous specimens ; for terrestrial biogeological specimens of significant transparency , the use of confocal microscopy for the spectroscopic analysis of subsurface inclusions in halite crystals is a significant development for the analytical detection of halotrophs . A very important attribute of Raman spectroscopy is the acquisition of experimental data using remote optical flexible probes without the need for chemical or mechanical pre-treatment of the specimen ; this has been a major factor in the adoption of Raman instrumentation on robotic planetary landers and rovers such as the ExoMars programme . The merits of using Raman spectroscopy for the recognition of key molecular biosignatures from several terrestrial extremophile specimens will be illustrated and some recommendations made for the technical requirements of a miniaturised system and its evaluation for Martian exploration . The data and specimens used in this presentation have been acquired from Arctic and Antarctic cold deserts , a meteorite crater , and from hot desert saltpan evaporite locations from which it will be possible to assess the advantages and possible limitations of Raman spectroscopic techniques for the detection of extraterrestrial extremophilic life signatures.

(110) **Transmission Raman Spectroscopy as a Tool for Quantifying Polymorphic Content of Pharmaceutical Formulations**

Jonathan Burley[1], Michael Hargreaves[2], Adeyinka Aina[1], Pavel Matousek[3]; [1]University of Nottingham; [2]Cobalt Light Systems Ltd; [3]Central Laser Facility, STFC

We present the first quantitative study of polymorphic content in a model pharmaceutical formulation using transmission Raman spectroscopy (TRS), and compare the results obtained with those from traditional backscattering geometry. The transmission method is shown to provide a true bulk measurement of the composition, being unaffected by systematic or stochastic sub-sampling issues that can plague traditional backscattering geometries. The accuracy of the quantification of the polymorphs using TRS was shown to surpass considerably that achieved using conventional backscattering mode. For a model-free fit, the TRS method yielded R2 of 0.996 compared to the backscattering value of 0.802; for a partial least squares fit with a single component the TRS method accounted for 98.1 % of the variance in the data compared to 89.7 % for the backscattering method.

(111) **Raman Mapping of Biological Tissue Using Clustering Analysis Based on the Pearson Correlation Coefficient**

Frederic Festy[1], Frances Downey[1], Richard Cook[1], Nic Cade[1], Cheryl Gillett[1], David Richards; [1]King's College London

Recent advances in Raman spectroscopy have generated new interests in biomedical research, especially in the field of optical diagnosis and the characterisation of biological tissues. In the literature, differentiation between cancerous and benign tumours from human patients was shown to be possible, using principal component analysis on the collected Raman spectra in a few cases. However, this simple approach has been limited by a number of factors including the need for adequate controls and the lack of images (e.g. those found in conventional histology). Using the rapid Streamline Raman imaging capabilities of Renishaw's inVia Reflex spectrometer, we have mapped, with high resolution, the chemical signature of a number of thin sections of human breast tissue milk ducts. To extract meaningful chemical information from such large datasets (more than 150,000 spectra), we have developed an automated clustering approach based on the Pearson correlation coefficient. Concentration maps were obtained from fitting each pixel with a set of basic spectra derived directly from the results of the cluster analysis. Such "hands-off" approach ensures high quality fitting which is not possible with spectra obtained from chemicals originating from other sources.

(112) **Scanning Angle Total Internal Reflection Raman Microscopy**

Emily Smith[1,2], Kristopher McKee[1,2]; [1]US DOE, Ames Laboratory; [2]Iowa State University

A scanning angle total internal reflection (SA-TIR) Raman microscope has been developed for automated Raman spectroscopy depth profiling measurements. The instrument is based on a traditional optical microscope platform with added beam steering optics to vary the angle of the incident light on a prism/sample interface. Varying the angle of incident radiation upon a prism/sample interface tunes the depth of penetration of the evanescent wave, and in turn the depth over which Raman scatter is collected. The incident angle can be scanned from the critical angle to 75.5 degrees, and depths up to 1500 nm can be profiled with the current instrument set-up. The instrument provides an order of magnitude improved spatial resolution perpendicular to the focal plane compared to confocal Raman microscopy. The instrument has been used for noninvasive and nondestructive measurements of thin polymer films, biopolymer films and adsorption to ordered nanoporous materials. Since SA-TIR Raman microscopy provides chemical content information as a function of pore depth, the instrument can be used to measure transport and catalytic reactions in nanoporous materials.

(113) **Effect of Laser Angle of Interrogation on Raman Signal**

Phillip Wilcox[2], Jason Guicheteau[2], Ian Pardoe[1], Steven Christensen[2], Darren Emge[2]; [1]Excet, Inc.; [2]Edgewood Chemical Biological Center

Current Raman spectroscopic systems are designed to operate in backscatter geometry where the incident beam is normal to a contaminated surface, but this can be impractical or impossible for many real world situations. In order to address the angle dependence of the Raman return signal, experiments have been conducted where the angle between the laser beam and the surface is varied from 0° to 90°. This allowed for quantification of the defocusing effects of the received laser spot image on the spectrometer entrance slit. The data was compared to a model based on excitation and collection geometry and scattering from smooth and rough surfaces. By performing these measurements, we were able to develop a predictive model for the angle dependence of chemical droplets on various surfaces.

(114) **Spectroscopic Characterization of Natural Fiber Welding**

Hugh De Long[2], Luke Haverhals[1], Zane Fayos[1], Hadley Sulpizio[1], W. Matthew Reichert[1], Matthew Foley[1], Paul Trulove[1]; [1]United States Naval Academy; [2]Air Force Office of Scientific Research

The full dissolution of biopolymers such as cellulose and silk is achieved with ionic liquid solvents. During the dissolution and reconstitution process, the native polymer structure is lost. Our laboratory has shown that robust composite materials are created by partial dissolution of biopolymer based materials; a method we call "Natural Fiber Welding." During welding, the outer polymer sheaths of individual fibers are swelled and mobilized so that they can interact with those of neighbors. This is potentially advantageous because much of the native material structure can be retained in the fiber core. Careful selection of process variables allows tunable control of the amount of dissolved polymer and manipulation of material properties. We will present spectroscopic and imaging data from analyses of materials generated by fiber welding. Raman and FT-IR spectroscopy probe chemical changes to biopolymers as a function of process variables. For example, the welding process converts native cellulose I to cellulose II upon dissolution (and reconstitution) and is monitored as a function of processing time and temperature. Combining microscopy with spectroscopic methods allows chemical changes and associated polymer movement to be spatially resolved. Examples of fluorescently labeled cellulosic materials are imaged to document the movement of polymer under different processing conditions. Results indicate that surface polymer is preferentially mobilized and modified as the ionic liquid solvent penetrates individual fibers. Materials tend to densify as adjacent fibers fuse at their intersections to form an extended hydrogen bonded network. Results also show that a significant and controllable amount of the native material may be retained after the welding process is complete.

(115) **Ionic Liquids in Separations and Mass Spectrometry, A New Frontier**

Daniel Armstrong[1]; [1]UT Arlington

Room-temperature ionic liquids (RTILs), also known as liquid organic, molten, or fused salts, are a class of nonmolecular ionic solvents with low melting points. The accepted definition of an RTIL is any salt that has a melting point lower than ambient temperature. However, "ionic liquid" (IL) is often applied to any compound that has a melting point <100 °C. Most common RTILs are composed of unsymmetrically substituted nitrogen-containing

cations (e.g., imidazole, pyrrolideine, pyridine) with inorganic anions (e.g., $C1^-$, PF_6^-, BF_4^-). ILs are also interesting because of their other useful and intriguing physicochemical properties. Wilkes et al. first reported ambient-temperature ILs based on the 1-alkyl-3-methylimidazolium cation in 1982 (1). Since then, many ILs containing a variety of cations and anions of different sizes have been synthesized to provide specific characteristics. Over the past few years, research and applications of ILs have expanded tremendously. The initial impetuses for this expansion were organic synthesis and the growth of green chemistry. In this presentation an overview of the structure and properties of ILs and a description of their expanding use in various applications in mass spectrometry will be given (2).ILs have proven to be the best liquid Maldi MS matrix since we introduced them as such a few years ago (3). The properties of ILs that make them effective will be discussed. (4-5) Further, the dications developed for high stability IL GC stationary phases have found another novel use in electrospray MS as a reagent for ultra sensitive anion analysis. Different analytical approaches, uses and mechanism of this new method will be considered (6,7).

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(116) **NanoGUMBOS: A Novel Concept for the Design of Nanomaterials**

Isiah Warner[1], Bilal El-Zahab[1], Min Li[1], Susmita Das[1]; [1]Department of Chemistry, Louisiana State University

My research group has recently developed a new approach to the production of nanomaterials using a group of uniform materials based on organic salts (GUMBOS). These nanomaterials are typically produced from frozen ionic liquids by use of a variety of methods employed for creation of relatively monodispersed nanoparticles. However, some of our materials do not follow the strict definition for ionic liquids and thus we have adopted the more broadly based acronym of GUMBOS to accurately describe these materials. In regard to these new materials, we believe that our approach represents a paradigm shift in the approach to producing nanomaterials such that our materials are designed and assembled for specific uses, rather than adapted for uses as is the case with most nanomaterials in use today. This is because our approach to nanomaterial development allows for easy production of a variety of properties which can be exploited for selected bioanalytical measurements and applications. This talk will focus on discussions of these new kinds of nanomaterials, as well as contrast these new materials relative to other currently used nanomaterials. The spectroscopic and magnetic properties of some of our new nanomaterials will also be discussed in detail.

(117) **How Proteins Dance and Fold in Ionic Liquids**

Frank Bright[1], Nadine Kraut[1], Bharathwaj Sathamoorthy[1], Michael Dabney[1], Kiran Singarapu[1], David Parish[1], Gary Baker[2], Thomas Ssyperski[1], Taylor Page[1] ; [1]UB, SUNY; [2]Oak Ridge National Laboratory

Several research laboratories have reported biocatalytic reactions performed within ionic liquids (ILs), with varying degrees of success. A major challenge in this area is associated with controlling and understanding those factors that affect the biocatalytic process. Currently very little is known about the molecular-level behavior of biomolecules in ILs in comparison to their better understood behavior in aqueous media. We have set out to address this shortfall by studying two protein systems in aqueous buffer, 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([C4mim][Tf2N]), 1-butyl-3-methylimidazolium tetrafluoroborate ([C4mim][BF4]), and 1-butyl-3-methylimidazolium hexafluorophosphate ([C4mim][PF6]) as a function of temperature and water loading. The first protein is human serum albumin that we have site-selectively labeled at cysteine-34 (located in loop 1 of domain I) with a single fluorescent reporter molecule (acrylodan, Ac). The second protein is ubiquitin dissolved in [C4mim][BF4]. Our experimental tools are steady-state and time-resolved fluorescence and high resolution NMR spectroscopy. The results of these experiments show that there are changes in protein structure over many length scales within IL systems.

(118) **Photonic Ionic Liquids: Blurring the Line Between Solvent and Probe**

Gary A. Baker[1], Ka Yi Yung[2], Nadine D. Kraut[2], Frank V. Bright[2]; [1]Oak Ridge National Laboratory; [2]University at Buffalo

Given the diversity available in synthetic options, task-specific ionic liquids (TSILs) are coming to the forefront as problem-solving fluids in a number of not-so-obvious areas. Among them, photonic ionic liquids are emerging as intriguing neo-class TSILs for elaborating novel photofunctional fluidic approaches. In this work, we detail several recent examples that bridge the gap or blur the line between designer solvent and probe (or transduction) chemistry by integrating molecular units responsive to photo-stimuli within TSIL architectures.

(119) **Ionic Liquids For and By Spectroscopy**

Chieu Tran[1]; [1]Marquette University

Two aspects of ionic liquids that to date have either no or limited studies will be highlighted in this talk. They are (1) exploitation of unique features of ionic liquids to develop novel spectroscopic methods and (2) development of novel spectroscopic methods for the sensitive and accurate determination of thermal physical properties of ionic liquids. In the first category, we will describe several novel methods which were developed utilizing unique properties of ionic liquids for measurements which are not possible otherwise. They include the sensitive and accurate method to determine enantiomeric compositions of a variety of pharmaceutical products with different size, shape and functional groups. This method is based on the use of a chiral IL which serves both as a solvent and also as a chiral selector. Ionic liquids have also been successfully used to substantially enhance the sensitivity of thermal lens measurements. In the second category, we will describe recent development in which transient grating technique and thermal lens technique have been successfully used for the sensitive, accurate, nondestructive determination of thermal physical properties of ILs.

(120) **Isotope Ratio Analysis of Fast Transient Samples with a Mattauch-Herzog Mass Spectrograph**

Jeremy Felton[1], Steven Ray[1], Roger Sperline[2], M. Bonner Denton[2], Charles Barinaga[3], David Koppenaal[3], Gary Hieftje[1]; [1]Indiana University; [2]University of Arizona; [3]Pacific Northwest National Laboratory

A Mattauch-Herzog geometry mass spectrograph fitted with a Focal Plane Camera (FPC) array detector has been used for the analysis of isotope ratios of fast transient species. The MHMS features an electric and a magnetic sector in a geometry that focuses ions along a plane rather than to a focal point. The FPC detector is capable of collecting the ion distribution along the focal plane continuously and simultaneously. Among the many

advantages of this type of ion detection is the ability to measure the time behavior of entire transient samples. This avoids a phenomenon known as spectral skew, which is an inaccurate signal measurement caused by instantaneous sampling of a transient peak. The acquisition speed of the FPC array allows mass-spectrometric detection of transient signals from the second to microsecond time scale. Isotopic distributions of multiple elements in transient samples as well as isotope ratios of fast transient samples with precision levels of less than 5% RSD have been measured with this system.

(121) **Real Time Monitoring and Determination of Trace Elements in Single Airborne Nanoparticles (ANPs) by Continuous Introduction into ICP-MS**

Naoki Furuta[1], Hikaru Sato[1], Shimpei Hikida, Yoshinari Suzuki[1]; [1]Chuo University

The field of nanotechnology is rapidly expanding and the number of new products containing nanomaterials continuously increases. The toxicity of such nanomaterials to humans is of great concern owing to the specific characteristic of nanomaterials. In the atmosphere we inhale every day, a large number of airborne nanoparticles (ANPs) exist. The origin of the ANPs are considered to be diesel exhaust particles, fly ash originated from waste incineration, worn materials of automobiles (break pad abrasion, tire wear, etc.), and natural plant burning. In my laboratory, since May 1995 airborne particulates were collected separately by size on the roof of the university building in the campus of Chuo University, Tokyo, and the concentrations of major and trace elements in the particulates have been monitored for 15 years. Multi-element analysis for airborne particulates has been performed with ICP-MS after airborne particulates collected on filters were acid digested to convert to a solution. During the acid digestion, the sample was diluted by 1000 to 10000 times. This method requires a long sampling period (hours or days) to obtain sufficient amount of airborne particulates for ICP-MS analysis, and the results can provide us with an average concentration of elements in various airborne particulates during a sampling period. In this paper, ANPs with a diameter less than 1 μm were continuously introduced into the ICP and trace elements in the ANPs were determined by ICS-MS. When ANPs were introduced into the ICP, a gas exchange device was used to exchange gas molecules from air to Ar without any loss of ANPs. Calibration was conducted by using aerosols produced by an ultrasonic nebulizer with a desolvation system, gases of Cr, Mo, and W carbonyl complexes produced by a standard gas generator, and particles produced by a laser ablation system. This method enables us to do real time monitoring of trace elements in ANPs without any sample dilution.

(122) **Agilent Technologies-Growth and Innovations in ICP-MS**

Amir Liba[1]; [1]Agilent Technologies

The past thirty years have been filled with exciting and important innovations in atomic spectroscopy, allowing ICP-MS to become an integral and essential tool in most laboratories. These innovations have resulted in accurate and precise inorganic analysis, yielding vital information in a variety of sample types. While in ICP-MS, analyte ionization is attributed to the inductively coupled plasma (ICP), a variety of mass analyzers exist, offering a wide range of analytical detection and capabilities. Among the available mass analyzers (quadrupole, time-of-flight, magnetic sector), the quadrupole ICP-MS is the most utilized ICP-MS on the market. Agilent, among others, have been instrumental in advancing inorganic analysis by continuously innovating the ICP-MS platform. Since 1987, the Agilent ICP-MS has gone through five different generations of instruments, with each new design further advancing its success in the analytical environment. In this talk we will highlight some of those innovations, focusing primarily on its robustness in analyzing complex matrices, it use of helium for polyatomic interference removal and its ability analyzing high TDS samples.

(123) **An Introduction to the Next Generation of Inductively Coupled Plasma Mass Spectrometers**

Kaveh Kahen[1], Hamid Badiei[1]; [1]Perkin Elmer Sciex

The next generation of inductively coupled plasma mass spectrometry (ICP-MS) instruments, was introduced earlier this year. The instrument uses a bench-top design and is equipped with a robust, 40-MHz, free-running generator and the patented PlasmaLok® technology. A unique feature is the use of a triple-cone interface and an orthogonal ion path which provides exceptional stability while dramatically reducing maintenance frequency. The triple-cone interface design improves the ion/gas ratio by removing the unionized species from the beam and reduces the space charge effects behind the skimmer. This results in higher sensitivity across the mass range and particularly in the low mass, where up to an order of magnitude increase is achieved. In addition, the static quadrupole ion deflector which is positioned behind the triple cone interface turns the positively charged ions by 90 degrees while preventing the neutrals and photons from entering the ion optics. The cell-based version of NexION 300 utilizes the Universal Cell Technology (UCT™). The UCT is capable of operating in three distinct modes: 1) standard mode (vented cell) for interference-free isotopes; 2) the dynamic reaction cell (DRC™) mode with reactive gases; and 3) the kinetic energy discrimination (KED) mode with helium. While the KED mode is particularly useful for complex environmental matrices with unknown interferences, the DRC mode provides ultimately low detection limits for the interfered isotopes. Other features, such as high speed analysis using a fast scanning quadrupole, extended dynamic range, and the automated torch positioning system are also discussed in this presentation.

(124) **Fully Simultaneous ICP-MS**

Dirk Ardelt[1], Willi Barger[1], Ulrich Heynen[1]; [1]SPECTRO Analytical Instruments GmbH

Unlike in optical emission spectroscopy with an inductively coupled plasma (ICP-OES), where instruments capable of registering the full spectrum with every single measurement are available for some time now, in ICP-MS, the sequential measurement of the isotopes of interest is still the standard procedure. Only limited simultaneity is available by either using multicollector instruments (typ. less than 10 isotopes simultaneous) or time-of-flight (TOF) analyzers (limited duty factor). Recently, a fully simultaneous ICP-MS using a double- focusing magnetic sector analyzer in Mattauch-Herzog geometry in combination with a large multichannel semiconductor ion detector, covering the full inorganic relevant mass range, has become available. The presentation will discuss applications specifically benefitting from these features and typical figures of merit.

(125) **Inductively-Coupled Plasma Time-of-Flight Mass Spectrometry: Quo Vadis?**

Steven J. Ray[1], Elise Dennis[1], Alexander Graham[1], Christie Enke[1], Gary M. Hieftje[1], Charles Barinaga[2], David Koppenaal[2]; [1]Dept. of Chemistry - Indiana University; [2]Pacific Northwest National Laboratory

Inductively coupled plasma mass spectrometry (ICP-MS) remains the gold-standard technique for ultra-trace elemental analysis. ICP-MS offers high sensitivity over a very wide dynamic range with low limits of detection, and provides isotopic information for elements across the periodic table. As practiced routinely in most analytical laboratories, however, ICP-MS is not a simultaneous multielemental technique. The vast majority of commercial ICP-

MS instruments are of a sequentially scanning nature and capable of observing only a single m/z ratio at any given time. Thus, the S/N with which each measurement can be made is dependent on the number of isotopes observed, a problematic limitation when sample quantity is limited or when transient signals are analyzed. A number of instruments have been developed to overcome this limitation, perhaps the most successful being the use of a time-of-flight mass spectrometer (TOFMS). A TOFMS is capable of simultaneous multielemental analysis at very high spectral generation rates, generating tens of thousands of complete elemental spectra each second. Thus, the entire atomic spectrum is available for analysis without penalty. Thus far, however, these multielemental strategies have been slow to be adopted. In this presentation, the utility of simultaneous multielemental analysis for routine ICP-MS is critically examined, and those applications on the horizon that require such a capability discussed. The current state of ICP-TOFMS will also be examined, and novel strategies and geometries for operating a TOFMS to conform to the unique requirements of atomic mass spectrometry will be introduced.

(126) **Changing the Perception of LIBS: Fundamentals and Applications**

Richard E. Russo[1,2], Travis Owens[1], Jong Yoo[2], Jhanis Gonzalez[1,2], Alex Bolshakov[2]; [1]Lawrence Berkeley National Laboratory; [2]Applied Spectra Inc.

There is no doubt that LIBS has become a very popular analytical technology over the past several years. The benefits of LIBS are considerable and include no sample preparation, real time analysis, spatial (lateral and depth) resolution, standoff measurements, and now the ability to characterize chemical systems by creating database spectral libraries. LIBS offers these significant advantages to varied applications, but like every analytical technology, a thorough scientific/technical effort is required to specify the figures of merit and define reality. Fundamental studies have promoted advances in the applications, mainly by evaluating parameter space (laser and detection) behavior on plasma conditions. However breakthrough our understanding, each application still requires an empirical assessment of the sample response to the laser ablation process and knowledge of the accuracy of the spectra to the original chemical composition. The talk will provide an overview of key fundamental processes underlying the formation and detection of the plasma, and describe successful applications of LIBS when sample properties are amenable to the technology.

(127) **Laser-Induced Breakdown Spectroscopy (LIBS) for the Rapid Identification and Classification of Pathogenic Bacteria**

Steven Rehse[1], Qassem Mohaidat[1], Sunil Palchaudhuri[2], Hossein Salimnia[3, 4]; [1]Wayne State Univ., Dept of Physics and Astronomy; [2]Wayne State Univ., Dept of Microbiology; [3]Wayne State Univ., Dept of Pathology; [4]Detroit Medical Center Univ. Laboratorie

Laser-induced breakdown spectroscopy (LIBS) is currently undergoing a period of rapid growth as reflected by both the variety of applications to which it has been applied and the number of peer-reviewed articles that have appeared describing the advances in the field. The speed, portability, ruggedness, sensitivity, and selectivity of the technique all suggest that LIBS can provide a rapid point-of-care bacterial diagnostic technology for clinical, military, environmental, or civilian applications. The potential significance of such a technology is global in scale (encompassing billions of dollars of testing and research) and would have a major impact on multiple communities (i.e. clinical diagnosis, the food industry, and water and environment quality control) that touch almost every aspect of human health and safety. For bacterial identification, the intensities of many atomic emission lines within the LIBS plasma spectrum (mostly from trace inorganic elements such as calcium, magnesium, phosphorus, potassium, etc.) provide an immediate and unique spectral atomic emission "fingerprint" which positively identifies the bacteria. Statistical signal-processing techniques (known broadly as chemometrics) allow an unknown LIBS spectral fingerprint to be almost immediately classified against a reference library of pre-existing fingerprints. We have performed a LIBS-based identification of multiple bacteria species, both pathogenic and non-pathogenic, including Gram-positive and Gram-negative bacteria, as well as mycobacteria. A discriminant function analysis (DFA) was used to analyze the LIBS bacterial spectra. All the bacteria tested to date were distinguishable from each other and identifiable on the basis of their LIBS spectrum, although strains of a species possessed highly similar LIBS spectra. This talk will summarize some of the results we have demonstrated for the LIBS-based identification of pathogens, including: strains of E. coli (pathogenic vs. non-pathogenic) can be discriminated and identified; the LIBS spectrum of a bacterium is independent of growth medium; a LIBS-based classification is independent of whether the bacteria are reproducing, dormant, inactivated by UV light, or killed by autoclave; mixed samples of bacteria can be identified at mixing ratios down to 80:20 (more than sufficient for clinical samples); and LIBS spectra can be obtained from approximately 500 bacterial cells. Plans for future work will be described.

(128) **Improving Standoff Sensor Detection Performance for Explosive Residues via Fusion of Shortwave Infrared, Raman and LIBS Imaging Data**

Matthew Nelson[1], Paul Mangold[1], Robert Schweitzer[1], Patrick Treado[1]; [1]ChemImage Corporation

Proliferation of explosive threats is an escalating threat to civilian and military personnel. Sensor systems that can rapidly detect explosives at standoff distances in operationally relevant sensor configurations are in development. Current technologies being investigated for standoff detection of explosives include short-wave infrared (SWIR) hyperspectral imaging, Raman spectroscopy and Laser Induced Breakdown Spectroscopy (LIBS). Each of these techniques, operating alone, has demonstrated potential for standoff detection of explosive agents. When the data from all of these sensor technologies is combined using proven, sensor fusion strategies, the overall system performance can be improved. ChemImage standoff hyperspectral sensors have demonstrated their potential for the detection of explosives in multiple field tests. ChemImage's LightGuardTM is a SWIR hyperspectral sensor capable of wide-area surveillance and detection of explosives. ChemImage's RAMAN-ST sensor is based on fiber array spectral translator (FAST) hyperspectral imaging technology in which Raman scattered photons collected from a scene through a telescope are projected onto the two dimensional end of a FAST bundle that is drawn into a one dimensional, distal array coupled to an imaging spectrograph. Software then extracts the full-spectral / spatial information, which is embedded in a single CCD image frame. The acquired spatial-specific Raman information allows threat materials to be computationally differentiated within a complex mixture of background materials. In this paper, fusion results obtained from ChemImage standoff hyperspectral imaging explosives detectors will be discussed.

(129) **Application of LIBS to Aqueous Solutions**

Scott Goode, Amelia Taylor-Perry; [1]Univ of So Carolina

Laser induced breakdown spectroscopy is used primarily for the determination of the elemental composition of solids. But LIBS can be applied successfully to the analysis of liquids, particularly heterogeneous mixtures that would require extensive sample pretreatment to generate particle-free homogeneous solutions. In this presentation we present and discuss several different methods of treating aqueous samples including direct analysis of liquids,

freezing to form a solid, and absorbing into a solid support. Evaluation metrics accuracy, precision, limits of detection, and ease of use.

(130) **Archaeological Applications of LIBS: An Example from the Coso Volcanic Field, CA Using Advanced Statistical Signal Processing Analysis**

Russell S. Harmon[1], Jeremiah J. Remus[2], Jennifer L. Gottfried[3]; [1]ARL Army Research Office; [2]Clarkson University; [3]Army Research Office

Multi-element chemical analysis is a common means for attributing the provenance of archaeological materials. Artifacts made from CVF obsidian are found throughout the southwestern United States and geochemical sourcing of obsidian based on chemical composition has become an important tool for gaining insight into prehistoric Native American trading patterns. The Coso Volcanic Field (CVF) in California, USA contains at least 38 high-silica rhyolite domes, many of which contain obsidian glass that has been quarried for tools by the indigenous population for more than 12,000 years, with 'Coso-type' obsidian artifacts attributed to sites across the southwestern US. Multi-element chemical analysis is a common means for attributing the provenance of archaeological materials. Artifacts made from CVF obsidian are found throughout the southwestern United States and geochemical sourcing of obsidian based on chemical composition has become an important tool for gaining insight into prehistoric Native American trading patterns. The Coso Volcanic Field (CVF) in California, USA contains at least 38 high-silica rhyolite domes, many of which contain obsidian glass that has been quarried for tools by the indigenous population for more than 12,000 years, with 'Coso-type' obsidian artifacts attributed to sites across the southwestern US. Laser-induced breakdown spectroscopy (LIBS) is a simple atomic emission spectroscopic technique that has the potential for real-time man-portable chemical analysis in the field. Because LIBS is simultaneously sensitive to all elements, a single laser shot can be used to record the broadband emission spectra, which provides a 'chemical fingerprint' of a material. Single-shot broadband LIBS spectra were collected using a commercial benchtop LIBS system for 27 obsidian samples from major sites across the CVF and four additional California sites outside of CVF. Classification of the samples was performed using partial least-squares discriminant analysis (PLSDA), a common chemometric technique suitable for performing regression on high-dimensional data. Provenance identification for the obsidian samples was evaluated using signal processing classification approaches. The Coso obsidian source can be distinguished by statistical analysis of single-shot LIBS spectra from the other regional obsidian sources with a high degree of confidence, whereas obsidian sub-sources sources within the CVF, previously recognized on the basis of ICP-MS analysis, are slightly less well differentiated. The results of this study suggest that LIBS analysis combined with appropriate statistical signal processing has the potential to be a useful tool for in-field chemical analysis and source identification of archaeological artifacts.

(131) **Classification Modeling for Pharmaceutical Applications**

Katherine Bakeev[1]; [1]CAMO Software Inc.

Many problems that we are looking to solve in pharmaceutical applications of spectroscopy entail the classification of samples. For many years we have used classification based on SIMCA, nearest neighbor, or correlation methods. There are additional methods that are now popular, though their full value has not yet been determined (nor have figures of merit for them been established). Unsupervised classification in terms of PCA and clustering will be presented, as will some examples of methods coming from the field of machine including support vector machines (SVM), and linear discriminant analysis.

(132) **Data Fusion for Analysis of Pharmaceutical Systems**

Steven Brown[1], Maureen Lanan[2], Mike Koenigbauer[3]; [1]Univ. Delaware; [2]Biogen; [3]Astra Zeneca

As instrumentation of chemical processes used in pharmaceutical manufacturing and characterization improves, the amount and quality of data is rapidly increasing. Many standard chemometric methods, developed assuming limited data, are ill-equipped to deal with new data formats available to chemists. Data fusion is one way to adapt conventional chemometrics to multiple measurements made on the same sample. Fusion of data from different measurements also opens the possibility of new approaches to data analysis. This talk investigates methods for the fusion of data from several sources. I will discuss some results from wavelet transforms and other fusion methods in calibration, and will show results from a study of polymorphic mixtures in tablets measured by Raman, IR and NIR spectrometry and from the characterization of samples using NIR and NMR spectrometry.

(133) **Wavelets and Genetic Algorithms for Multivariate Calibration of NIR Data of Low Content Drug Tablets**

Barry Lavine[1], Nikhil Mirjankar[1], Mehul Vora[2]; [1]Oklahoma State University; [2]Clarkson University

A two step procedure for analyzing complex near infrared multivariate calibration data sets has been developed. The wavelet packet tree is used to denoise and deconvolute NIR spectra by decomposing each spectrum into wavelet coefficients, which represent the samples constituent frequencies. A genetic algorithm for multivariate calibration is used to identify the coefficients, whose PC or PLS subspace possesses the following property - the samples in this subspace are distributed according to the value of the dependent variable. For a given data point, its 3 or 5 nearest neighbors in the subspace will be identified and the fraction of its nearest neighbors corresponding to the 3 or 5 sample data points that one would expect to lie near it on the basis of the value of the dependent variable for these 3 or 5 samples is computed. Clearly, the criterion used to evaluate the information content of the features is different from the criterion used to evaluate the performance of the calibration model. Furthermore, a least squares projection of the dependent variable onto the PC or PLS subspace is not performed during any phase of the evaluation process thereby eliminating problems that arise from chance or spurious correlations which often occur due to the large number of comparisons made with different subsets of the data. Hence, the problems that have plagued applications of genetic algorithms to feature selection in multivariate regression are addressed. In addition, the GA is able to focus on samples that are difficult to fit as it trains using a form of boosting to adjust the values of the sample weights. Two specific studies illustrating the advantages of this approach in multivariate spectral calibration will be discussed. (1) Near IR spectra of a ternary system made to simulate an analgesic mixture was calibrated for caffeine and aspirin. (2) Multivariate calibration models were developed from transmission NIR data to assess detection and quantitation limits for determination of active principles (0 – 5% w/w) in pharmaceutical tablets.

(135) **Multiple Fluorescent Label Capillary Electrophoresis Detection via Supercontinuum Rapid Excitation-Emission Matrix (ScREEM)**

Timothy Corcoran[1], Christopher Dettmar[1], Jacob Balthazor[1], Phillip Allen[1], Jose Chavez[1], Ivonne de la Torre[1], Alisha Lewis[1], Neda Nouri Nassr[1], Hossein Ahmadzadeh[1], ; [1]California State Polytechnic University

The separation capability of capillary electrophoresis and exquisite sensitivity of laser-induced fluorescence detection are enhanced by greater specificity in detection. The supercontinuum rapid excitation-emission matrix (ScREEM) method employs a white-light laser capable of exciting a broad gamut of fluorophores across the visible range and into the near-infrared, combined with a postcolumn sheath-flow cuvette, hyperspectral detection and careful suppression of Rayleigh and Mie scatter. Excitation-emission matrix (EEM) spectroscopy allows the simultaneous differentiation and quantification of a large number of fluorescent species(e.g., fluorescent-labeled antibodies), even in the presence of strong spectral overlap and noise via parallel factor analysis (PARAFAC). Limits of quantitation are below 10-10 mol/L, corresponding to ~103 fluorophores in an excitation zone. EEM collection rates better than 10 Hz are achieved, faster rates are possible. Initial results with unseparated dye mixtures (e.g., fluorescein, rhodamine B) are shown. This method offers significant new capabilities in bioanalytical technology such as sub-cellular sampling.

(136) **Multivariate Analysis (MVA) Applied to Drug Substance Development**

Susan Barnes[1], Christian Airiau[1], Vern De Biasi[1]; [1]GlaxoSmithKline

The process development of a typical Active Pharmaceutical Ingredient (API) is generally associated with a very large amount of data acquired during the development campaigns and production of clinical trial material. This data originates from a range of sources, from chemical properties, physical properties, process parameters and process analytical technologies. Each data source collected is driven by a very clear rational driven by specific departments: analytical results are required for batch release, process parameters are acquired on plant for process monitoring and safety issues. The pool of data acquired during batch processes could however be harvested further to generate new information for the project team by either combining multiple data sources or using more sophisticate tools such as Multivariate Analysis to extract as much value from the data as possible. A structured approach is presented in the study which decomposes the plant campaign data analysis in two sections: 1. A systematic post campaign review of multiple data sources using simple multivariate methods to identify key patterns in the data 2. A specific implementation of MVA workpackages triggered by Project Teams to generate increased process understanding on a focussed part of the process, either off-line or in real-time. Two case studies are presented to illustrate the benefits generated by each aspect of the proposed strategy. A post campaign review on analytical and physical property data is presented, which highlights atypical batches, as well as campaign-to-campaign and site-to-site variability across the development timeline, as well as its impact on the drug product performance. A specific application of MVA to an API re-crystallisation step is used to identify which exact step of the process is responsible for the variability of the physical properties of the isolated material.

(137) **Determination of Encapsulation Efficiency by Single Vesicle Analysis**

Michael Heien[1]; [1]University of Arizona

Encapsulation of molecules within lipid vesicles has many applications and is an important area of research for a variety of fields such as pharmaceuticals, foods, and cosmetics. Interactions between solutes and the lipids used to encapsulate them can lead to high encapsulation efficiencies; such methods are only effective for certain solute/lipid combinations. In contrast, passive encapsulation (i.e., without attractive interactions) is more general but leads to internalization of solutes at or below their concentration in the vesicle formation solution. Solute size is a key determinant in the efficiency of passive encapsulation, with larger solutes excluded from the vesicle interior such that the internal solute concentration is lower than the external concentration during vesicle formation. Knowledge of solute concentrations in the vesicle interior is important for understanding processes as varied as enzymatic turnover, polymerization, crystallization, or phase separation in this volume. It is also of interest for applications in which vesicles serve as carriers for solute delivery, such as in food formulations or therapeutics. Encapsulation is quantified in terms of the bulk encapsulation efficiency. It is typically determined by removing all of the unencapsulated solute (e.g. via centrifugation or dialysis), and then lysing the vesicles to quantify the remaining (encapsulated) solute. This method does not provide information on variability in solute encapsulation between individual vesicles. The ability to determine encapsulation efficiency for individual vesicles also makes it possible to compare encapsulation between vesicles within a batch, which can vary widely. Here I present a method to determine the encapsulation efficiency for individual submicron vesicles (<200 nm). We have developed a platform to separate, lyse, and quantitatively measure the contents of single vesicles using a hybrid capillary-microfluidic device. This device incorporates a sheath-flow design at the outlet of the capillary for chemical lysis of vesicles and subsequent electrochemical detection. The effect of macromolecular crowding on passive encapsulation of biological molecules within individual 200 nm diameter unilamellar vesicles was investigated. In addition, labeled biomacromolecules were encapsulated during vesicle formation in the presence or absence of dextran, which served as a crowding agent. Encapsulation efficiency of the labeled biomolecules in individual vesicles was determined.

(138) **Segmented Flow for High Throughput Analysis at the Nanoliter Scale**

Robert Kennedy; [1]University of Michigan

We have explored the use of multiphase flow wherein sample is formed into a series of plugs or droplets separated by immiscible liquid as a way to manipulate samples in microfluidic and microscale analytical systems. Methods for formation and manipulation of such plugs on the nanoliter scale have been developed and are increasing in sophistication so that it is now possible to perform many common laboratory functions such as sampling from and reagent addition to plugs in microfluidic systems. We have developed systems that allow droplets to be used for injection onto mass spectrometers and capillary electrophoresis and chromatography columns. The resulting systems can have extremely high throughput because of limited time required for rinsing between samples. We have also explored fraction collection by segmenting column effluent into droplets. In this way, complicated sample treatment and off-line interface to detectors such as mass spectrometry can be performed. Splitting droplets allows collected fractions to be stored and re-analyzed at a later time. This allows, for example, samples to be collected and then analyzed by multiple mass spectrometers. It also enables stopped flow (also known as “peak parking”) electrospray ionization-mass spectrometry analysis. The injection and fraction collection systems have applications for “separations-based sensing”, high-throughput screening and analysis, process analytical technology, and multi-dimensional analysis. In this talk we describe the formation and manipulation of segmented flows, interfaces to microscale separations, and their applications.

(139) **Electrochemical Evaluation of Dopamine and Serotonin Neurotransmission in the Fruit Fly Brain**

B. Jill Venton[1], Trisha Vickrey[1], Huai-fang Fang[1]; [1]University of Virginia

Drosophila melanogaster, the fruit fly, is a model organism widely used by biologists and could be a good model system for studying neurobiology because of its homology with mammals. However, the size of the brain is tiny, only 100 µm wide and 8 nL in volume. We have developed techniques for implanting microelectrodes to make real-time measurements of endogenous neurotransmitter changes in a single CNS from a larval Drosophila. Both dopamine and serotonin have been measured and the regulation of neurotransmission studied. A dopamine D2-like autoreceptor has been identified. We have also developed a method for capillary electrophoresis-electrochemical detection separation of neurotransmitters in a single Drosophila nerve cord. This data provides information about how genetic variants and drugs affect the tissue content of the brain.

(140) **The Past, Current, and Future of Reverse-phase HPLC Method Development in Pharmaceutical Industry**

Shujun Chen[1], Gerald Terfloth[1], Alireza Kord[1]; [1]GlaxoSmithKline

Reverse-phase HPLC remains a horsepower in pharmaceutical development and analysis. Separation and determination of impurities in active pharmaceutical ingredients (APIs), starting materials (SMs), and intermediates (IMs) are important analytical activities which are mainly achieved by reverse-phase HPLC. Reverse-phase HPLC method development methodologies have advanced tremendously thanks to the development in instrumentation, column technology, and computer technology since 1950s. In this presentation, the history, current status, and future of reverse-phase HPLC method development are discussed.

(141) **Peak Tailing Suppression of Phosphoric Prodrug in RP Chromatographic Separation Using Acidic Mobile Phase**

Jin Zhng[1], Qinggang Wang[1], Lydia Breckenridge[1], Brent Kleintop[1]; [1]Bristol Myers Squibb

Phosphate esters have been commonly used as prodrugs in pharmaceutical development to enhance the solubility and bioavailability of the parent drug. A well known challenge for this type of compound in RP chromatographic separations is peak tailing of the API and impurities containing the phosphate ester moiety. The work presented here focused on the effect of residual irons, which are believed to be the major contributor to the peak tailing by forming complex with the phosphate in the literature. It was reported that this type of peak tailing can be suppressed by using basic mobile phase, or adding competitive phosphate ion in the mobile phase. This study focused on the peak tailing investigation using acidic mobile phase. Different acidic buffers, TFA and phospheric acid, were used in the mobile phase. A series of eluent was collected as the mobile phase running through the LC system. The metal contents were measured with ICP-MS to determine the iron level in the eluent as the function of time. It was found although phosphate ions were better scavenger for irons, TFA was more effective in reducing peak tailing. In addition, different stationary phase of LC columns was also evaluated to understand the effect of residual silanols on the peak tailing. The mechanism of peak tailing suppression will be discussed in the presentation. Although not conclusive, the results obtained from this study suggested a general procedure can be adopted to suppress peak tailing of phosphate compounds.

(142) **Application of Preparative Chromatography to Accelerate Drug Development**

Leo Hsu[1], Xiqin Yang[1]; [1]GlaxoSmithKline

Drug development in pharmaceutical industries is facing lots of challenges, which includes a several-fold increase in the number of projects in the development pipeline and shortened development timelines. Preparative chromatography, which was seen as a last resort for synthetic chemists, has now been emerging as a key option for the purification of APIs and intermediates. In this talk, we demonstrate how a process development strategy was followed to execute preparative chromatography projects in a predictable fashion and deliver successful preparative chromatography projects every time.

(143) **The Use of SFC from Method Development to Purification of Hundreds Grams of Racemic Material**

Manon Villeneuve[1]; [1]GlaxoSmithKline

Chiral separation plays a very important role in Drug Discovery. Since pure enantiomers can be difficult and/or expensive to synthesize; chromatography is often used to separate these enantiomers. In the early stage of drug discovery, several milligrams / grams of pure enantiomer is often needed for different assays including activity, potency, toxicity and physico-chemical properties. In our lab, we have been using analytical SFC since 1995. Today the technique is still use for the separation of Chiral and Achiral GSK molecules. Preparative SuperCritical Fluid Chromatography (SFC), has been a method of choice for separations of pharmaceutical compounds at GlaxoSmithKline since 1997. Several advantages like minimum solvent use, better resolution, sharper peak shapes and lower pressure drop, make the technique very attractive for purification. The low viscosity of a supercritical fluid result in a very low pressure drop across a column compared to Prep-HPLC. Due to very low column pressure drop, higher flow rates and lower column particle size can be used. The presentation will show our daily chiral screening process and purifications performed on 3 cm I.D. columns. Tables will show the requests trends for the past years. Due to all the advantages of SFC, example on how large scale purification can be performed even in a Drug Discovery lab. The presentation will also include how it is possible to performed preparative SFC on compounds, which are not all that soluble in methanol.

(144) **Lessons in Implementing Open-Access Chromatography Across the Globe**

Steve Cole[1], Helen Weston[1], Bill Young[1], James Roberts[1]; [1]GlaxoSmithKline

Open access analytical instrumentation has become a mainstay in LC-MS, GC-MS and NMR laboratories. The focus of this talk is to highlight the application of open access HPLC-UV and Fast GC-FID in a chemical development setting for hundreds of chemists at multiple sites. Scaling an analytical method to over 100 instruments that run daily provides a unique opportunity to study chromatographic failure modes and their diagnosis, and to implement high-performing chromatography on-scale for the novice user. Case studies outlining high throughput approach and key learnings will be presented.

(145) **Fast GC for Rapid Solvent Composition and Purity Analysis**

Charles Goss[1], Will Canoy[1], Hamid Shafiei[1], Francis DeMartin[1]; [1]GlaxoSmithKline

The presentation will describe open access “Fast GC” systems we have developed and implemented to facilitate common analytical tasks such as monitoring solvent exchange, reaction kinetics, and

purity determination. The Fast GC systems are based on Low Thermal Mass (LTM) GC instruments that provide rapid column heating and cooling to enable faster methods, higher throughput and simple service. LTM-GC columns are wrapped with a low mass electric heating element and temperature sensor that efficiently controls the column temperature to provide fast heating (>600°C/min), fast cooling (>120°C/min), and uniform temperatures. The columns are mounted in modular assemblies that attach outside the traditional GC oven. This approach eliminates interactions between the column and oven heating programs, enables up to 4 independent columns on a single GC, and facilitates maintenance and replacement. Examples illustrating how LTM-GC can shorten method cycle time compared to conventional GC, improve sensitivity, and enable faster method development compared to conventional GC will be discussed.

(146) **Microfluidic GEMBE of "Real-World" Samples and Nanofluidic Separation of DNA**

Elizabeth A. Strychalski[1], Alyssa C. Henry[2], Henry W. Lau[3], Lynden A. Archer[3], David Ross[1]; [1]National Institute of Standards and Technology; [2]Applied Research Associates, Inc.; [3]Cornell University

Microfluidic GEMBE was implemented for the quantitative analysis of complex samples with minimal sample preparation and contactless conductivity detection. Whole milk, various types of dirt and leaves, coal fly ash, and blood serum were analyzed for dissolved potassium, calcium, sodium, magnesium, lithium, and melamine with the single preparatory step of dissolution or suspension in a buffer solution. Gradient elution moving boundary electrophoresis (GEMBE) uses electrophoretic flow to drive electrically charged analytes into a microfluidic channel or capillary for detection, while opposing electro-osmotic and variable pressure-driven flows prevent the remainder of the sample from entering the channel. GEMBE is a simple, robust analytical technique, well-suited to microfluidic analysis of complex samples containing material that might confound other microfluidic techniques. In another study, a non-equilibrium regime of size-based separation was observed experimentally for DNA molecules with lengths below 1 kbp moving electrokinetically through nanofluidic nanoslit arrays. The breakdown of Ogston sieving and associated loss of resolution was overcome at higher electric fields to recover rapid separation with a reversed elution order and elution times one to two orders of magnitude faster than with Ogston sieving at lower fields. A simple kinetic model was developed to describe the experimental results.

(147) **Electrofocusing in Gradient Monoliths**

Cornelius Ivory[1]; [1]Washington State University

Charged molecules can be electrofocused, i.e., separated and concentrated, in a monolithic column that has an axial gradient in its chemical or physical functionality. The ability to do this is enabled by the recent development of methods for photoinitiated polymerization and gradient functionalization of monoliths combined with new electrofocusing methods. Protocols have now been developed for creating fixed gradients in ion exchange moieties, size exclusion chromatography parameters, and stationary phase pH gradients in monoliths. Our long-term goal is to create a multi-dimensional platform in which two or more orthogonal dimensions of separation focus proteins to a true steady state. If this can be done, then each dimension of separation will be uncoupled from every earlier dimension. This paper will first discuss the underlying principles of electrofocusing in gradient monoliths, their anticipated behavior, and then some of the performance bottlenecks that we have encountered in the lab.

(148) **Gradient and Discontinuous Electrokinetics**

Mark Hayes[1]; [1]Arizona State University

So called 'dirty' and messy complex samples from biological and environmental sources require significant processing to present a simplified and clean fraction to detection elements. In many cases, it is desirable to provide for analysis of multiple analytes from a single sample, such that specific targets are isolated and concentrated away from background and other analytes. Further, there is a significant push to miniaturize and create one step processing based on microfluidic systems. Here we present to interrelated techniques to take real world biological and environmental samples, remove unwanted background particulate debris, and then isolated and concentrate targets in a highly parallelized format. The two techniques are DC insulator-based gradient dielectrophoresis and parallel electrophoretic capture. Preliminary results show the ability to differentially isolated particles ranging from 20 nm to 1 micron using various physical parameters, increasing their local concentration by up to one million times. For molecular targets, small molecules to proteins have been differentially isolated and concentrated in specific sub-microliter volumes demonstrating the utility of the approach across the entire range of targets of interest. We envision being able to uniquely isolate and concentrate particulates (cells, viruses, bacteria, spores, organelles, etc.), process them and execute highly parallelized molecular isolation and concentration on a large number of targets reaching maximum detection limits and dynamic range.

(149) **Evaluation of the Precision of Dual-Opposite-Injection Capillary Electrophoresis and Comparison with Conventional Capillary Electrophoresis**

Joe Foley[1], Donna Blackney[1]; [1]Drexel University

Despite the well-known bias in the resolving power and analysis time that conventional capillary electrophoresis (CE) has for the simultaneous separation of anions and cations, other less-biased approaches to voltage-driven separations in free solution including an alternative approach now known as dual-opposite-injection capillary electrophoresis (DOI-CE) have received little attention, particularly with respect to reproducibility. DOI-CE was introduced in 1998 by Kuban and Karlberg [1] and Padarauskas et al. [2] for inorganic ions and low molecular mass organic ions and extended in 2007 by Weekley and Foley [3] to higher molecular mass pharmaceutical anions and cations. In DOI-CE, electroosmotic flow (EOF) is suppressed and identical samples are introduced at both ends of a capillary, allowing anions and cations to migrate respectively from the cathodic and anodic ends of that capillary to a detector window located near the center of the capillary. Whereas Weekley and Foley quantitatively compared several separation-based figures of merit (migration time, efficiency, resolution, resolution per unit time, etc.) for CE and DOI-CE and described new approaches for eliminating co-detection, neither they nor other researchers have addressed the issue of reproducibility in sufficient depth. In this presentation, DOI-CE is introduced and briefly compared with convention CE from a conceptual perspective. Thereafter, the precision of DOI-CE separations is evaluated and compared with the precision of conventional CE separations for various types of samples in terms of the precision of migration time, resolution, and peak area. [1] P. Kuban, B. Karlberg, Anal. Chem. 1998, 70, 360-365. [2] A. Padarauskas, V. Olsauskaite, V. Paliulionyte, J. Chromatogr. A 1998, 829, 359-365. [3] B. S. Weekley and J. P. Foley. Electrophoresis, 2007, 28, 697-711.

(150) **Using Buffer Additives to Improve Analyte Stream Stability in Micro Free Flow Electrophoresis**

Nicholas Frost[1], Michael Bowser[1]; [1]University of Minnesota

Micro free flow electrophoresis (uFFE) is a separation technique that continuously separates analyte streams as they travel through an electric field applied perpendicularly to the flow in a microdevice. Application of the technique has been limited by the generation of electrolysis bubbles at the electrodes, which results in unstable flow paths through the device. The current paper introduces the use of surfactants and nonaqueous solvents in the carrier buffer as a means of increasing stability of separated analyte streams. Adding surfactant or nonaqueous solvents lowers the surface tension of the carrier buffer, which we hypothesize promotes the formation of smaller electrolysis bubbles. A 6-fold improvement in the standard deviation of analyte stream position was observed upon addition of 10 mM SDS. Likewise, an approximately 12-fold improvement in stability was observed upon addition of 300 uM Triton X-100. Similar stability improvements were found in carrier buffers containing nonaqueous solvents. An 8-fold improvement in stability was found with a carrier buffer containing 50% methanol and a 6-fold improvement was found with a carrier buffer containing 37.5% acetonitrile. Long term use was demonstrated with a carrier buffer containing 300 mM Triton X-100 in which separated analyte streams remained stable for nearly two hours.

(151) **Determination of Inorganic Ions in Mineral Water by Gradient Elution Moving Boundary Electrophoresis**

Paul Flanigan IV[1], David Ross[2], Jonathan Shackman[1]; [1]Temple University; [2]NIST

Gradient elution moving boundary electrophoresis (GEMBE) is a modified capillary electrophoresis (CE) technique first described in 2007 that involves electrophoretic separation in a short-length capillary using a continuous injection. A pressure-controlled, variable hydrodynamic flow combined with the electroosmotic flow (EOF), controls the bulk flow. As the pressure-driven flow is swept from high to low, analytes enter the channel at different times because only species with electrophoretic velocities greater than the counterflow velocity are allowed to enter the channel. Therefore, each analyte is introduced sequentially in order of decreasing electrophoretic mobility, and the resolution is essentially controlled by the pressure gradient. Analytes are detected as step-wise changes in signal and later differentiated into conventional peaks. Little or no sample preparation is necessary for complex samples, as the bulk flow can exclude particulates and complex matrix interferants from entering the separation column. The simplicity of GEMBE allows it to be easily multiplexed. In this application, GEMBE was used with a capacitively coupled contactless conductivity device (C4D) to determine the major inorganic ions in several commercial mineral waters. This application is the first to demonstrate the separation of cations and anions simultaneously using GEMBE. Seven ionic analytes were separated in less than 7 minutes with detection values in the low micromolar to sub-micromolar range with excellent reproducibility. The experimental concentrations showed good correlation to reported concentrations for the mineral water samples. In addition, phosphate and arsenate were separated in less than 2 minutes with limits of detection of 300 and 140 nM respectively.

(152) **Quantum Cascade Laser for Quantitative Analysis in Liquid Phase**

Bernhard Lendl[1], Markus Brandstetter[1], Andreas Genner[1], Wolfgang Ritter[2]; [1]Vienna University of Technology; [2]QuantaRed Technologies

Due to their small size, room temperature operation and high spectral power density mid-IR quantum cascade lasers (QCLs) have the potential to find widespread application in dedicated, robust process analyzers. This presentation focuses on the use of these lasers for liquid phase measurements. Results on achievable signal to noise ratios for a distributed feedback QCL (narrow wavelength range) as well as a broadly tunable external cavity QCL (tuning range 200 cm-1) along with data on long term stability are presented. A distributed feedback QCL is used to develop new method for measuring total petroleum hydrocarbons (TPH) in water, for which an ASTM standard is in preparation. This method covers TPH concentrations in water ranging from 0.2 – 2000 ppm. It is based on a solvent extraction using cyclohexane, sample clean-up by filtration over Florisil and measurement of C-H bending vibrations. An external cavity QCL was used for the simultaneous determination of glucose (0-800 mg/dL) and lactate (0-224 mg/dL) in aqueous solutions. Measurements are carried out in the spectral region from 1030 – 1230 cm-1 using a CaF2 flow cell with an optical path of 130 µm. For simultaneous determinations, a partial least square (PLS) calibration model has been developed based on EC-QCL spectra recorded from over hundred quaternary solutions. Typical RMSEP values are 9.4 mg/dL for glucose and 6.0 mg/dL for lactate respectively.

(153) **Mid-Infrared Absorption Spectroscopy Using Quantum Cascade Lasers**

Adam Erlich[1]; [1]Block Engineering

Block Engineering has developed an absorption spectroscopy system based on widely tunable Quantum Cascade Lasers (QCL). The system rapidly cycles through a user-selected range of the mid-infrared spectrum anywhere between 6 to 12 µm (1667 to 833 cm-1). The system can detect and measure substances on surfaces from a standoff distance of up to 2 feet. It can identify bulk materials and detect sub-micron films based on their absorption characteristics. It can also analyze vapors or liquids, in a single device. The higher power density allows measurements from diffuse and highly absorbing materials and substrates. Other advantages over FTIR include portability, ruggedness, fast analysis, and the ability to work at a distance either through free space or a fiber. The system has been able to analyze trace amounts of explosives at a standoff of 2 feet using an eye-safe laser. This paper will discuss the basic technology behind the system, empirical data on various samples, a comparison of the technology relative to FTIR and Raman, and a review of potential applications.

(154) **Challenges and Opportunities in Biomedical IR Imaging with QCLs**

Rohit Bhargava[1], Rohith Reddy[1], Matthew Schulmerich; [1]University of Illinois at Urbana-Champaign

Discrete frequency infrared (DFIR) spectroscopy can potentially become an attractive alternative to Fourier transform infrared (FT-IR) spectroscopic imaging. In DFIR, only data from targeted wavelengths are recorded and used to extract molecular information. Hence, spectroscopic recording, especially for imaging, can be very rapid and efficient. We first present the biomedical motivation for DFIR. Next, we describe the theoretical underpinnings of DFIR and its comparison with FT-IR spectroscopic imaging. We specifically focus on examining the potential and challenges for DFIR imaging using quantum cascade lasers (QCLs). We examine the issue of image formation in a microscopy system and the use of coherent data recording from the narrowband laser output. A theoretical model is developed to rigorously model the transmission, focusing and sample interactions of light from QCLs using an interferometer for characterization. The home-built interferometer is interfaced to a commercially available QCL and used to record data from well-characterized polymer samples. A low-intensity, broadband thermal source was used in the same setup to provide comparisons with the

high-intensity QCL and the results are reported here. Finally, we present comparisons of experimental data and theoretical predictions.

(155) **Recent Results from Broadly Tunable External Cavity Quantum Cascade Lasers**

Michael Radunsky[1], David Caffey[1], Vince Cook[1], Tim Day[1], Martin Algots[2]; [1]Daylight Solutions; [2]Algots Design

Broadly tunable access to the mid-infrared spectrum (3-12 µm) has largely been the domain of the glow bar and passive techniques. In the last ten years Quantum Cascade gain media have challenged this paradigm by becoming the engine in broadly tunable, coherent, narrowband lasers. The quality and performance of the QC gain media has steadily improved as researchers and manufacturers refine their processes. One area where great strides have been made is in the bandwidth of the gain. Chips that exhibit extremely broad gain bandwidths can be mounted in external cavity resonators to produce close to 500 cm-1 of narrow band tuning. This broad tunability is highly desirable for building lasers that can be used for chemical identification and detection, oncologic diagnosis, and microscopic imaging. Beyond the raw gain of the chip, other essential factors to bring this technology to a point where it is not the target of a research project, but can be used by the researcher as a laboratory tool have been developed. Cavity optimization and flexibility, optical coating, and miniaturization techniques each come into play. Broad tuning chips have traditionally operated best in pulsed mode. But there has been progress here as well since wavelength coverage of cw-operated chips has increased. Furthermore, "daisy-chaining" these broad tuning lasers in an appropriate configuration provide the opportunity to cover from 6 to 12 µm in a single head. Aspects of the design, performance, and application of these broadly tunable lasers will be discussed.

(156) **Nanosecond Time-Resolved IR Spectroscopy in Conventional and Supercritical Fluids Using External Cavity Quantum Cascade Lasers**

Mike George[1], James Calladine[1]; [1]University of Nottingham

Fast Time-resolved IR Spectroscopy, a combination of UV/visible laser flash photolysis with fast IR detection is a very powerful technique for the detection of short-lived reaction intermediates and the elucidation of reaction mechanisms. External Cavity Quantum Cascade Lasers (QCLs) provide a convenient IR source for TRIR instrumentation and can be used to obtain high sensitive nanosecond IR spectra. This lecture demonstrates describe the use QCLs for the construction of a fast time-resolved IR measurements.

(157) **First Application of External-Cavity Quantum Cascade Lasers for Nanosecond Time-Resolved Infrared Detection of Intermediates Generated by Pulse Radiolysis**

David Grills[1], Andrew Cook[1], Etsuko Fujita[1], Michael George[2], Jack Preses[1], James Wishart[1]; [1]Brookhaven National Laboratory; [2]University of Nottingham

In this paper we present the first application of nanosecond time-resolved infrared (TRIR) spectroscopy for the identification and monitoring of short-lived intermediates generated by pulse radiolysis of condensed-phase samples. Pulse radiolysis, utilizing short pulses of high energy electrons from accelerators, is the definitive method for rapidly adding single positive or negative charges to molecules. The resulting species are important in applications such as redox catalysis for solar energy conversion and hydrogen production, and in many other areas such as advanced nuclear fuel processing. Coupled with fast UV-visible detection, pulse radiolysis has become an extremely powerful method for monitoring the kinetics and subsequent reactivity of these species on timescales ranging from picoseconds to seconds. However, the radicals formed are sometimes difficult to identify due to their broad and featureless UV-visible absorption spectra, making mechanistic investigations difficult. In contrast, TRIR spectroscopy is a powerful structural probe of short-lived intermediates, often allowing multiple transient species to be identified and simultaneously monitored. Unfortunately, due to several technical challenges, the coupling of fast (sub-millisecond) TRIR spectroscopy with pulse radiolysis has received only limited attention, being confined to a handful of gas-phase studies. Taking advantage of recent developments in mid-IR laser technology in the form of tunable external-cavity quantum cascade lasers (EC-QCLs), we have recently developed a sensitive nanosecond TRIR detection apparatus for condensed-phase samples subjected to pulse radiolysis at our Laser Electron Accelerator Facility (LEAF) at Brookhaven National Laboratory. EC-QCLs are compact, tunable IR lasers, that emit with a high output power (up to >100 mW), making them ideally suited for use in a pulse radiolysis environment. Using two EC-QCLs we have demonstrated the technique by monitoring the one-electron reduction, and subsequent decay, of catalysts that are useful for CO_2 reduction, such as $[Re(bpy)(CO)_3(CH_3CN)]^+$ and $[Ru(bpy)_2(CO)H]^+$ (bpy = 2,2'-bipyridine). The one-electron reduced forms of such catalysts are key intermediates in photocatalytic cycles, and can be prepared directly by pulse radiolysis in acetonitrile in the absence of sacrificial electron donors. The routine availability of TRIR detection will open up many new opportunities for mechanistic investigations of redox processes with pulse radiolysis.

(158) **A Robust, Reproducible, and Inexpensive Optical Oxygen Sensor for Process Analysis**

Charles Branham[1], Tom Dearing[1], Lauren Hughs[1], Kent Mann[2], Brian Marquardt[1]; [1]University of Washington; [2]University of Minnesota

Vapochromic compounds exhibit distinct changes in luminescence wavelength and intensity due to the reversible incorporation of solvent vapors and simple gases into their loosely-packed crystal lattices. The optical response of vapochromic compounds can be directly related to the identity and concentration of particular analytes. When paired with low cost fiber-optic sensing platforms, the sensitive and rapid vapo-luminescent response is ideally suited to environmental, industrial, and marine applications. Current work has been focused on developing sensitive, small, and low-power fiber optical oxygen sensors based on phosphorescence quenching in both gaseous and dissolved phases. This presentation will focus on the methods used to calibrate the sensor response and evaluate its analytical performance. In addition, methods used to transfer the calibration model from one oxygen sensor to multiple sensors will be presented.

(159) **Laboratory and Process Based Analytical and Sample Conditioning Applications for Modular Sample Conditioning Systems**

Mike Cost; [1]Parker Hannifin Corporation

The use of modular sampling systems has been well accepted in the process analytics industry for process control and environmental monitoring applications. However, the use of these systems has not been progressed extensively in non-hazardous, lab-based environments. Parker has initiated a lab-based analytics initiative to demonstrate feasibility of modular sampling systems for gas calibration and high level analytical measurement (i.e. RAMAN, FTIR, GC, etc.). The flexibility of modular systems accommodates miniaturization of analytics and fluidic controls. This allows the end user flexibiltiy in experimental design and research. The flexibiltiy of such systems and how the use of modular hardware assists redcution in system engineering time, promotes integration of analytics and simplifies overall experimental design will be discussed.

(160) **Process Optimisation in Microreactors Based on Flow Rate Manipulation and Real-Time Non-Invasive Measurements by Raman Spectrometry**

David Littlejohn[1], Alison Nordon[1], Sergey Mozharov[1], Charlotte Wiles[2], Paul Watts[2], Paul Dallin[3], John Girkin[4]; [1]University of Strathclyde; [2]University of Hull; [3]Clairet Scientific; [4]University of Durham

The purpose of this work was to study the applicability of Raman spectrometry with a specially designed optical interface for process optimization in micro-reactors. Two reactions were selected for this study: 1. Baylis-Hillman reaction between methyl acrylate and benzaldehyde. 2. Knoevenagel condensation between ethyl cyanoacetate and benzaldehyde: The first reaction is an example of slow processes that requires 1-3 days to complete. To overcome difficulties associated with the low reaction rate, a special micro-reactor with high internal volume has been assembled. Monitoring development of such a slow process would require continuous operation of the Raman system over a long period of time. We have developed an alternative approach based on extracting location-specific information from steady state. It relies upon accurate flow rate manipulation and rapid Raman measurements allowing the data gathering time for kinetic studies to be cut from possible days to only 10 minutes without any loss of information. With this new methodology it has become possible to simplify the optimisation procedure and save time. It also opens up new opportunities for the use of Raman spectrometry in microfluidic chemistry, particularly in process optimisation and kinetics studies. The second reaction is relatively fast and completes within 0.1-3 hours depending on conditions. It was selected to validate the new method against conventional optimisation procedures. The results confirmed good reliability of the new technique at 10° C.

(161) **Multi-Plasma Laser-Induced Breakdown Spectroscopy (Multi-Plasma LIBS): New Developments and Further Evaluations**

Galan Moore, Douglas Jennings, Michael Carson; [1]Corning Incorporated

Multi-Plasma LIBS is one approach being developed to address the challenges of frequent compositional monitoring in a manufacturing platform, that is representative of the bulk. Multi-plasma LIBS is defined here as dividing a single energetic laser pulse into multiple reduced energy laser pulses in order to form an array of nearly identical laser-induced plasmas. Each plasma contains information about its localized environment, so by increasing the number of plasmas taken simultaneously, a second spatial dimension to describe a material's homogeneity can be obtained. For this article, an extensive study was preformed to evaluate two different ideologies of multi-plasmas, one using a beamsplitter and the other using a microlens array. Both methods used in the creation of multi-plasma conditions were also be compared to conventional single plasma LIBS.

(162) **LED Array Based Light Induced Fluorescent Sensor for Real-Time Monitoring**

Jason Dickens[1], Mervin Taylor[1], Mike Vaughn[1], Mike Ponstingl[2]; [1]GlaxoSmithKline; [2]Custom Sensors and Technology

A small compact novel LED array light induced fluorescent (LIF) sensor has been developed and commercialized to address various real-time monitoring problems. This sensor affords unique detection capabilities by exploiting the fluorescent advantage where the signal response is governed by LED array excitation source parameters. Dynamic excitation control affords versatile analytical capabilities to meet various analytical requirements across applications, which is unrivaled by existing detector gain limited real-time LIF instruments and conventional spectroscopies (NIR, IR, Raman, etc.). That is, this new sensor affords tailored analytical capabilities where conventional LIF systems and spectroscopies are insufficient to meet various sensitivity, detection limit, precision requirements. Flexible detection capability within a sensor platform is particularly advantageous in real-time applications such as process analysis where wide solute concentrations often exist across a manufacturing route. Moreover, lower detection limits are increasingly becoming a more common requirement. Monitoring of various physical states, in particular solids and liquids, is an additional available feature with this optically configurable sensor. Sensors were developed with various excitation-emission combinations across the UV-Vis range consistent with the spectral characteristics of most fluorophores. The combined sensor attributes along with its small self-contained mechanical design, supporting utilities, and smart sensing features afford a flexible, deployable, innovative device that fills an unmet real-time analytics capability gap. The sensor design(s), features, and analytical merits will be discussed within the context of process analysis across the chemical, pharmaceutical, and biochemical manufacturing industries. Example PAT applications including pharmaceutical content uniformity endpoint detection during blending and cleaning verification will also be discussed.

(163) **The Next Generation of Near-Infrared Spectral Sensing Systems**

John Coates[1], Nada O'Brien[2], Fred Van Milligen[2]; [1]Coates Consulting; [2]JDSU

Miniaturized near infrared sensing has been implemented in the extended silicon detector region (700nm to 1100nm). The advantages of the silicon-based solution have been size and cost. However, the shortwave NIR region is often limited by the need for a rather large pathlength that is required to obtain good absorption intensity. Also, the information content in this short wave is primarily linked to the overtone hydrides with very little coupling of the neighboring functional groups within a molecule. When one moves to the traditional NIR spectral region, from 900nm to 3300nm (or there about) one encounters more spectral detail and information content, and one measures with more manageable pathlengths, typically in the 1mm to 10mm range. The difficulty has been finding a suitable technology for miniaturization, and sufficiently low in cost to qualify for cost-effective spectral sensing. The authors have been collaborating on the use of linear variable filter (LVF) technology in combination with low-cost near infrared sensing arrays. An LVF is an optical element that spatially separates wavelengths with distance across the length of the filter. Functionally it operates as a continuously variable bandpass filter and can be described as a solid state Fabry Perot Interferometer component. Around the year 2000 JDSU introduced the first solid state spectrometer, the MicroPac, based on this technology in combination with a standard silicon-based detector, providing a spectral measurement system covering from 600nm to 1100nm. In a current project we are developing a similar concept utilizing arrays that operate in the traditional NIR spectral region. In this paper we will discuss the integration with a new inexpensive room temperature InGaAs detector. This detector provides an ideal format for a low-cost NIR sensing system, initially covering a nominal range from 900nm to potentially 2000nm. The footprint of the room temperature InGaAs is relatively small and the packaging enables convenient interfacing of the linear variable filter. The full NIR range can be considered with the approach, either based on extended InGaAs or lead sulfide. Both are practical, but are more expensive and yield slightly larger integrated solutions.

(164) **Support Vector Regression and Wavelength Interval Selection in Biological Raman Spectroscopy**

Narahara Chari Dingari[1], Ishan Barman[1], Jeon Woong Kang[1], Chae-Ryon Kong[1], Ramachandra R. Dasari[1], Michael S. Feld[1]; [1]Massachusetts Institute of Technology

Failure to frequently monitor blood glucose may lead to both acute (ketoacidosis) and chronic (diabetic retinopathy) health complications making non-invasive monitoring of glucose levels critical. However, despite encouraging results in serum and whole blood samples using Raman spectroscopy, the development of a clinically accurate and robust algorithm capable of prospective prediction in a human population has proven to be difficult. The absence of successful calibration transfer can be attributed to multiple factors including variations in tissue turbidity, autofluorescence levels and differences in glucose concentrations in the blood and interstitial fluid compartments. To develop enhanced robustness in the calibration models and to reduce over-fitting, we propose the application of kernel-based non-linear support vector regression (SVR). This shift from the conventional linear calibration schemes is based on the understanding that the linearity assumption between the spectral and concentration datasets may fail under the influence of fluctuations in the process and system variables. We show that SVR provides a significant improvement over the partial least square models with an increase in correlation between the actual and predicted concentrations by nearly 40% while also enabling clinically accurate predictions in the hypoglycemic range in human subjects. We also discuss the underlying nature of SVR-based improvement by studying tissue phantoms under controlled conditions. Further, we observe that the whole spectrum analysis may not yield optimal predictions due to the presence of uninformative and spurious regions in the spectrum. We perform wavelength interval selection based on a moving window approach and minimization of cross-validation error in the calibration set using SVR. We demonstrate the ability to significantly reduce the number of spectral channels while maintaining nearly constant levels of prediction accuracy in tissue phantom studies. Importantly, we show for the first time the transferability of selected bands across human subject studies. We expect that this will lead to significant miniaturization of clinical Raman devices to portable or even hand-held sensors, primarily driven by the reduction in spatial footprint of the detector components.

(165) **Confocal Raman Microscopy Reveals the Origin of Refractive Index Variation in a Living Cell**

Jeon Woong Kang[1], Niyom Lue[1], Ishan Barman[1], Narahara Chari Dingari[1], Chae-Ryon Kong[1], Ramachandra R. Dasari[1], Michael S. Feld[1]; [1]Massachusetts Institute of Technology

Live cell imaging without staining is a big challenge. Techniques such as phase contrast and Nomarski/ DIC microscopy overcome the problem by transforming the phase information into intensity distribution and thus reveal the cellular structural details. Recently, quantitative phase microscopy based on interferometry has been developed as a new technique for quantitative biology. From the variation of optical path length in a cell, the detailed morphological structure was obtained in 2D and even in 3D. Fast acquisition and full-field quantitative imaging without exogenous agents are the primary advantages of the phase microscopy methods. However, the following fundamental question still remains unanswered: "What causes the refractive index variation in a cell?" Since it was first observed in 1928, Raman scattering has been widely used as an analytical tool in many fields. Small amounts of inelastically scattered light from the sample include "finger print" vibrational information about the sample which can be used for both qualitative and quantitative analysis. Incorporating confocal microscopy, which is extensively used to obtain three-dimensional information, along with conventional Raman spectroscopy provides an exciting research avenue, due to the possibility of obtaining accurate information about the chemical and morphological components with high spatial resolution. This can be further augmented by standard reflectance and fluorescence modalities. Despite its great promise, confocal Raman microscopy has not been widely used for biological research, in comparison to fluorescence imaging, due to its intrinsically weak signals. In this talk, we present the design and development of a new hybrid microscope system, integrating confocal Raman and quantitative phase microscopy. Bright field imaging and confocal reflection modalities are also incorporated. Living HeLa cells are imaged by our hybrid microscope. To investigate the origin of the refractive index variation, two approaches are used. First, we obtained the refractive index map from a quantitative phase microscope and the Raman map from confocal Raman microscope. Coregistered images from both methods are subsequently compared and analyzed. Another approach is to use quantitative phase image to guide where to collect Raman spectra. From these two approaches, the origin of the refractive index variation of a cell is being investigated.

(166) **Advanced Single Cell Analysis by Means of Raman Spectroscopy**

Juergen Popp[1,2], Benjamin Dietzek[1,2], Michael Schmitt[1], Christoph Krafft[2], Robert Moeller[1,2], Petra Roesch[2]; [1]Friedrich-Schiller University Jena; [2]Institute of Photonic Technology Jena

Raman spectroscopy has been recognized to be a powerful tool to study biological cells. One challenge in Raman microspectroscopy of biological cells is the analysis of the spectra. Since Raman spectra of biological samples like cells or tissue are superpositions of the molecular information from all components special chemometrical methods to properly analyze the data are necessary. Here we describe some of our latest results concerning the application of Raman microspectroscopy in combination with innovative chemometrics to characterize biological cells. The first part reports about the great potential of Raman microspectroscopy in combination with modern chemometrics for the identification of single bacterial cells like e.g. pathogens without the need of pure cultures or any cultivation step. In combination with flu orescence staining the presented method can be used even for complex sample matrices like e.g. soil or blood. In addition to a biotic / abiotic differentiation, even a life / dead staining with two fluorescence dyes can be combined with Raman spectroscopic identification. The main focus within the second part of this presentation is concerned with Raman studies on eukaryotic cells for biomedical applications. Due to the larger size of eukaryotic cells, sub-cellular features such as nucleus and vesicles can be resolved using Raman microscopic imaging. In particular we will report about the development of a classification algorithms capable of distinguishing between breast cancer cells (MCF-7) and normal epithelial cells (MCF-10A) by their nuclei information. It is known that major differences between cancerous cells like and benign cells arise from variations in the cell nuclei. The presented algorithm which is suitable for online diagnosis by Raman spectroscopy has accuracy rates above 99%. The algorithm is based on the utilization of two classification steps. In doing so the first step, the so called "Top Level Classifier" identifies Raman spectra which are measured in the nuclei region. This separation of nucleus Raman spectra is done by artificial neuronal network. On the second step the nuclei Raman spectra are classified via a wide range of discriminant models.

(167) ***ex vivo*** **Determination of Breast Tissue Margins Using Spatially Offset Raman Spectroscopy**

Anita Mahadevan-Jansen[1], Daniel Masters[1], Matthew Keller[3], Mark Kelley[2]; [1]Dept of Biomedical Engineering, Vanderbilt Univ; [2]Dept of Surgery, Vanderbilt Univ; [3]Lockheed Martin Aculight

The risk of local recurrence for breast cancers is strongly correlated with the presence of tumor within 1-2 mm of the surgical margin on the excised specimen. Spatially offset Raman spectroscopy (SORS) holds much promise for intraoperative margin analysis. We have characterized the ability of SORS to distinguish between two soft tissue layers and have conducted Monte Carlo simulations to determine the source-detector separation needed to assess 1-2 mm of breast tissue specimens for the presence of tumor signatures. Based on simulation predictions for signal-to-noise ratio differences among varying spatial offsets, a 1st generation SORS probe with multiple source-detector offsets was designed and tested. This probe was initially tested in 35 resected breast tissue specimens *in vitro*. The probe and system is currently under evaluation for assessing the presence of tumor signatures in intra-operative breast tissue specimens in partial lumpectomy patients. Spectra from each detector ring were averaged to create a composite spectrum with biochemical information covering the entire range from the tissue surface to ~2 mm below the surface, and a probabilistic approach was used to classify these composite spectra as having "negative" or "positive" margins. This discrimination was performed with 95% sensitivity and 100% specificity.

(168) ***in vivo*** **Dental Caries Assessment with Polarized Raman Spectroscopy: A Pilot Study**

Lin-P'ing Choo-Smith[1], Mark Hewko[1], Michael Sowa[1]; [1]NRC-Inst. for Biodiagnostics

Polarized Raman spectroscopy (PRS) is being developed for the detection of early dental caries. Over the years, this development has been evolving from lab bench Raman microscopes with large laser sources, to smaller Raman spectrometers with optical fibres for laser delivery from compact lasers and Raman signal collection via handheld fibre probes. The scaling down of the instrumentation is required for PRS to be useful in a clinical dental setting. A volunteer test group to evaluate these developments includes orthodontic patients. Due to the long duration of the treatment and the difficulty in brushing around the orthodontic braces, these patients are at risk of developing early dental caries (white spot lesions). Preliminary results will be presented of a pilot study whereby PRS is used to measure the regions around the orthodontic brackets of patients wearing orthodontic appliances. The challenges, successes and limitations of the pilot study will be presented.

(169) **Transcutaneous Raman Spectroscopy of *ex-vivo* Human Bone**

Francis Esmonde-White[1], Karen Esmonde-White[1], Blake Roessler[1], Michael Morris[1]; [1]University of Michigan

Raman spectroscopy of bone is used for non-destructively assessing bone chemistry. Raman microspectroscopy is now widely used for bone materials studies to complement mechanical, histological and x ray analysis methods.(1-3) Raman spectral features correlate to mechanical properties of bone, including elastic modulus and bone strength, and can be used to predict fracture susceptibility.(4-6) Most commonly, these studies have been performed on *ex vivo* tissue from humans and small animals, or human tissue biopsies. Our lab is currently involved in a blinded 5-year multicenter prospective study to determine the applicability of Raman spectroscopy as a tool for predicting skeletal fragility in humans.(7) Raman spectroscopy is currently of limited use for *in vivo* diagnostics because the proven method (microspectroscopy) cannot be used without taking invasive bone biopsies. To address this limitation, we are developing transcutaneous Raman spectroscopy and Raman tomography methods for *in vivo* use with small animals and humans. In early studies, we have demonstrated transcutaneous Raman spectroscopy using projective optical systems for analysis of small animal and human cadaveric tissues,(8,9) as well as mice *in vivo*.(10) In our current work, we use conformal fiber optic probes to simultaneously collect Raman spectra from many points at the skin surface, repeating the measurements for several different illumination patterns (structured illumination). From the non-invasive transcutaneous measurements, Raman tomographic maps of bone in dog and rat tibia have been reconstructed. The results are encouraging and this process will be extended to human geometries. We have made several improvements in data preprocessing which will be discussed, including a correction for dispersion misalignment in the imaging spectrograph. We developed accurate multilayer tissue phantoms for optimization and validation of transcutaneous measurements, use of these tissue phantoms will be described. Finally, results of non-invasive Raman tomography on human cadavers and the reference exposed-bone measurements will be presented. References: [1] McCreadie, Bone 2006, [2] Carden, Calcif. Tissue Int. 2003, [3] Kohn, Cells Tissues Organs 2008, [4] Timlin, Anal. Chem. 2000, [5] Akkus, Bone 2004, [6] Akkus, Bone 2008, [7] Mandair, Proc. SPIE 2010, [8] Schulmerich, J. Biomed. Opt. 2006, [9] Schulmerich, J. Biomed. Opt. 2008, [10] Schulmerich, Appl. Spectrosc. 2009

(171) **Raman Spectroscopy of Active Molecular Electronic Devices**

Richard McCreery[1,2], Andrew Bonifas[2,3], Lian Shoute[1,2]; [1]University of Alberta; [2]National Institute for Nanotechnology; [3]The Ohio State University

Characterization of molecular electronic devices requires spectroscopy through partially transmitting contacts and sensitivity to molecular layers in the range of 1-25 nm thick. Nonvolatile memory devices based on bias-induced doping of conducting polymers are being investigated for possible applications in microelectronics. Current devices are faster than today's Flash memory, and have much longer retention than DRAM in wide use commercially. We use Raman spectroscopy to both characterize such devices during fabrication, and also to monitor changes in devices structure in response to "write"and "erase" bias pulses. A combination of Raman and UV-Vis spectroscopy in active devices reveals a critical requirement of the conducting polymers which permits fast switching and readout while preserving long retention time.

(172) **Nanomaterial Strategies for Immunodetection**

Marc Porter; [1]University of Utah

The explosion of innovations across biotechnology underscores the importance of ultra sensitive, high-speed diagnostic tests. This presentation will describe efforts to develop a readout methodology that potentially addresses these needs by coupling gold nanoparticle (i.e., spheres and rods) labeling concepts with surface enhanced Raman scattering (SERS) or superparamagnetic nanoparticle labeling concepts with giant magnetoresistors (GMRs). Strategies will be detailed for both the fabrication and readout of chip-scale platforms by examining fundamental dictates for optimal performance. Results from experiments that focus on the use of immunoassays for the ultralow level detection of viral and microbial pathogens, along with challenges central to analytical sensitivity, speed, nonspecific adsorption, and fluidics manipulation, will be discussed.

(173) **A New Technology to Isolate Muscle Stem Cells for Muscular Dystrophy Research and Treatment**

Nicholas Dobes[1], Wei Xu[1], David Detwiler[1], Chris Sims[1], Joe Kornegay[1], Nancy Allbritton[1,2]; [1]University of North Carolina Chapel Hill; [2]North Carolina State University

Existing stem cell assays for intracellular markers via immunocytochemistry have the drawback of necessary cell death as cell perforation is required for the introduction of primary and secondary antibodies for fluorescent labeling. This shortcoming makes expanding target muscle cells for future research a challenge. Other muscle stem cell determination methods exploit the slow adherence properties of stem cells via a multi-step plating technique. The downfall of this method is heterogeneous population of cells in the final plate. The research proposed here circumvents both of these issues with the use of segregated micropallet arrays. Micropallet arrays consist of micron-sized pedestals, referred to as pallets, atop which single cells can attach and grow/divide as they normally would in culture. Segregated micropallet arrays use the same principles and basic design but add a bridge between a pair of adjacent pallets. This bridge allows cells on a pallet to grow across to the adjacent pallet, forming a contiguous colony on the two neighboring pallets and the bridge. The bridge is then removed with a pulse of predetermined energy from a Nd:YAG laser, dividing the colony and leaving one half of the original colony on each of the adjacent pallets. One of the pallets containing cells is then removed from the array using the laser and fluorescence tests are run for stem cell identification. Should cells test positive, the other half of the colony remaining on the array can be removed and cultured for future use.

(174) **Synthesis of Fluorescent Peptide Substrates for Capillary Electrophoresis-Based ErbB-2 Kinase Assay**

Abigail Turner[1], Ryan Phillips[1], Angela Proctor[1], David S. Lawrence[1], Nancy L. Allbritton[1,2]; [1]University of North Carolina at Chapel Hill; [2]North Carolina State University

Abnormal activity of protein tyrosine kinases due to genetic mutation can often lead to inappropriate cell growth and cancer. ErbB-2 (a.k.a. HER2), a transmembrane tyrosine kinase belonging to the EGFR kinase family, is overexpressed in approximately 25% of breast cancers, and is implicated in several other cancers including melanoma and pancreatic cancer. Trastuzumab (Herceptin) is an anti-ErbB-2 monoclonal antibody widely used to treat ErbB-2 overexpressing breast cancer patients. Successful treatment with Herceptin and similar drugs requires accurate identification of patients with abnormal ErbB-2 activity. This can be accomplished either by identifying the presence of mutant genes or proteins, or by directly assaying kinase activity. Methods for gene expression and protein identification are sensitive and well established in the clinic, but they suffer from the limited correlation between mutant gene expression and abnormal kinase activity. Traditional kinase assays directly quantify activity, but require pooling of inconveniently large numbers of patient cells, thereby losing much information about the heterogeneity of the harvested sample. To overcome these disadvantages, we have developed a platform for conducting single-cell kinase assays based on capillary electrophoresis (CE) and fluorescence microscopy. Fluorescent peptide reporters that serve as substrates for ErbB-2 can be synthesized and modified with non-native amino acids to improve affinity, specificity, and resistance to degradation by peptidases. These fluorescent substrates can then be loaded into cells of interest to be phosphorylated by ErbB-2. The cell is lysed and immediately loaded into a capillary, where phosphorylated and non-phosphorylated peptide are separated by CE and detected with laser-induced fluorescence. In the present work, we demonstrate the synthesis and *in vitro* phosphorylation of a fluorescent peptide substrate for ErbB-2, as well as the ability to separate and quantify phosphorylated and non-phosphorylated peptide by CE. This is a first step toward the development of a single-cell kinase assay that significantly reduces the number of cells required for analysis while avoiding loss of heterogeneity due to lysate pooling. Future work will focus on optimizing the peptide reporter for intracellular phosphorylation and peptidase resistance, with the ultimate goal of testing the assay in clinical samples.

(175) **Characterization of Bacterial Endospores Using Fluorescence Spectrometry**

Paul DeRose[1]; [1]NIST

Fluorescence-based assays are being used to detect bacterial endospores, including anthrax. Some of these assays implement fluorescent tags to label spore-specific analytes, while others measure the intrinsic fluorescence from spores. The conditions under which Bacillus spores may be detected using both types of assays will be reviewed, including necessary sample preparation and instrument calibration. Differentiation between strains and tracking germination of spores will also be discussed.

(176) **Identification and Quantification of Human Growth Hormone by MALDI –TOF/TOF MS with On-Target Digestion**

Renee N. Easter[1, 2], Colin G. Barry[2]; [1]Department of Chemistry, University of Cincinnati; [2]Forensic Chemistry Center, U.S. Food and Drug Administration

Human growth hormone (hGH), a 191-amino acid, single chain polypeptide, is used clinically to treat genetic growth disorders in children, as well as a number of clearly defined wasting syndromes in adults. In the body, hGH binds to receptors to regulate body composition, skeletal muscle, bone growth and more. A number of illicit uses are made of this drug in anti-aging and weight management regimens in addition to its role as a performance enhancing drug. The increasing popularity of these off-label uses as well as concerns over diversion, illegal importation and counterfeiting have expanded the need for analytical tools to rapidly identify and quantify this substance. Current analytical approaches include capillary electrophoresis 3,4 and enzyme-linked immunosorbent assay (ELISA) 5. In addition, high performance liquid chromatography coupled to mass spectrometry has been used 1,2 in conjunction with enzymatic digestion to achieve more confident identification at the cost of a relatively long sample preparation and analysis time. This study employs MALDI-TOF/TOF-MS for the identification of hGH based on two experiments. Initially, the intact molecular weight (~ 22 kDa) is determined and, then, a rapid tryptic digestion is conducted on the plate and analyzed to provide sequence information. Five tryptic fragments have been identified which are consistently present following these on-target digestions. For quantification, these five peptides have been synthesized with the introduction of stable isotope labels and these are used as internal standards6-8. 1. Ribela et. al., Curr. Pharm. Anal. 2006; 2:103-26 2. Wisniewski et. al., J. Forensic Sci. 2009; 54(1); 122-7 3. Vinther et. al., Talanta. 1991; 38(12): 1369-79 4. Nielsen et. al., J. Chormatogr A. 1990; 516:99-114 5. Yuki et. al., Biol Pharm Bull. 1994 Jul;17(7):977-9. 6. Gerber et. al., PNAS. 2003; 100(12): 6940-45 7. Kirkpatrick et. al., Nature Cell Biology. 2006; 8(7): 700-10 8. Ciccimaro et. al., Molecular Pharmacology. 2009; 75(3); 658-666

(177) **Analytical Method Development for 3,3',4,4'-Tetrachloroazobenzene (TCAB) in Rodent Mammary Gland Tissue in Support of a Short-Term Toxicology Study**

Franz Thomas[1], James Blake[1], Stephen Cooper[1], Kelly Amato[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

3,3',4,4'-Tetrachloroazobenzene(TCAB) is a known contaminant of dichloroaniline (DCA) and herbicides synthesized from DCA. It

is a widespread environmental contaminant due to its presence in pesticides and can accumulate in crops and lead to potential human exposure. A gas chromatography-electron capture detection (GC/ECD) method that was previously validated for adipose tissue was optimized and the performance of the method was verified for the determination of TCAB in female Sprague-Dawley (SD) rat mammary gland tissue at ng/g levels. An analytical aliquot (~0.500 g) was removed from a homogenized sample, to which was added 25 µL of internal standard solution (PCB-118). The sample was mixed by vortex action and digested at least 3 hours on an orbital shaker after adding 2 mL of 30% KOH solution. After digestion, the sample was kept chilled throughout the extraction process. The sample was extracted three times with a fresh aliquot of methylene chloride each time and the combined extract was passed through a silica gel column. The collected extract was evaporated down to near-dryness and reconstituted in 250 iL of acetone. The concentrated extract was vortexed and transferred to an amber GC vial with a silanized micro-insert for analysis by GC/ECD. The method was successfully applied for the determination of TCAB in ~100 mammary gland tissues over a linear range of 10 to 4000 ng/g and the linearity of matrix and solvent standard calibrations were confirmed by correlation coefficients ≥ 0.99. A limit of detection (LOD) was established at 3 ng/g with an experimental limit of quantitation (ELOQ) at 10 ng/g; Accuracy (defined as Percent Relative error) ranged from -21.7% (at ELOQ =10 ng/g) to 15%; Extraction recovery (Matrix Standard/Solvent Standard) x 100%) ranged from 73.1% (at ELOQ) to 116%; Method Specificity indicated as no discernable response for control (undosed) tissue above the LOD.

(178) **A Highly Fluorescent Conjugated Polymer Nanoparticle for Measuring pH in Acidic Compartments of Living Cells**

Prakash Kandel[1], Lawrence Fernando[1], P. Christine Ackroyd[1], Kenneth A. Christensen[1]; [1]Clemson University

Conjugated polymer (CP) nanoparticles have been used as highly fluorescent labels for live cell measurements. These particles are typically 10X brighter than Quantum Dots and over 1000X brighter than conventional organic dyes with good photostability and no observed cytotoxicity. Using PFPV (poly[{9,9-dioctyl-2,7-divinylene-fluorenylene}-alt-co-{2-methoxy-5-(2-ethylhexyloxy)-1,4-phenylene}]), MEH-PPV (poly[2-methoxy-5-((ethylhexyl)oxy)-p-phenylenevinylene]), PFBT (poly[(9,9-dioctylfluorenyl-2,7-diyl)-co-(1,4-benzo-{2,1',3}-thiadazole)]), and mixtures of these polymers doped with Chromoionophore I, we have developed both single emission wavelength and emission ratiometric doped CP nanoparticle pH sensors for making measurements in living cells. These sensors were also coated with PEG-lipid to increase nanoparticle stability and improve biocompatibility. The hydrodynamic size of these CP pH sensing nanoparticles were characterized by dynamic light scattering to be between 80-120 nm in diameter depending on the polymer polydispersity and concentration. Individual batches of doped CP pH nanosensors were nearly monodisperse with respect to size. Zeta potentials of the nanoparticles were -40 ± 5 mV. *In vitro* assessment of nanosensor performance showed a linear response to pH from 4.5-7.5 and the signal was not perturbed by changes in ionic strength or protein concentration. Hence, these pH sensing nanoparticles are suitable for making measurements in cells. We tested these nanoparticle pH sensors in cells following uptake of the nanoparticles by macropinocytosis in a mouse macrophage-like cell line (J774.A1). CP nanoparticle fluorescence changed as the macropinosomes acidified and eventually fused with low pH lysosomal compartments. We also demonstrated that doped CP nanoparticle pH sensors could be calibrated *ex vivo* by incubating labeled cells with valinomycin and nigericin in buffers of various pH. Finally, we showed that our doped CP nanosensors could easily be visualized and pH measured by fluorescence microscopy at the single particle level.

(179) **Analytical Method Development and Validation for Safrole in Gavage Dose Formulations for Rodent Toxicology Studies**

Gwendolyn McNeill[1], Jennifer Gilliam[1], James Blake[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International.; [2]NIEHS,NTP

Safrole, a naturally occurring substance, has been used as a flavoring agent in drugs, beverages, and foods, and in the manufacture of heliotropin and piperonyl butoxide Safrole is produced by distillation of oils rich in safrole. Potential occupational exposure to workers handling safrole may occur through dermal contact. Health professionals, such as pharmacists, physicians, and nurses may possibly be exposed during formulation, preparation, administration, or clean-up of drugs containing safrole or sassafras. The present work describes the development and validation of a formulation analysis method for safrole in two different gavage vehicles, 0.5% aqueous methylcellulose (MC) and corn oil, for use in toxicology studies. A critical component of any toxicological study is an acceptable analytical method to insure that the correct test article is being administered at the specified dose concentrations. Furthermore, it is also important to confirm the homogeneity and the stability of the dose formulations. To achieve these objectives a capillary GC/FID method was developed and validated for analysis of safrole in both vehicles. Sample preparation involves extraction of the test article form the aqueous methylcellulose formulations using ethyl acetate and a simple dilution of corn oil formulations with methylene chloride. The method was successfully validated over a range of ~1 to ~40 mg/mL with additional dilution scheme to extend the range up to 100 mg/mL in both dosing vehicles. Linearity of matrix and vehicle standard calibrations were confirmed by correlation coefficients >0.99. Average extraction recovery was determined to be >91% for the aqueous vehicle. The experimental limit of quantitation (ELOQ) was established as ~1 mg/mL with a limit of detection (LOD) estimated as 0.1 mg/mL for both vehicles. Results are summarized below.

Method Parameter	Results
Linear range	~1 to ~40 mg/mL for both vehicles
Method verification (ext. range)	Up to 100 mg/mL for both vehicles
Linearity (r)	≥0.99 for both feed types
Precision (as %RSD)	≤6.5%/≤5.2% (MC/Corn Oil)
Accuracy (% Relative Error)	-6.1% to 11% for both vehicles
Average extraction recovery	91.5% (aqueous MC)
Storage stability	Confirmed upto 42 days
Dose simulation stability	Confirmed upto 3 hours

(180) **Apoptosis Detection by Fluorescence Correlation Spectroscopy in an Affinity Microdevice**

Michelle Martinez[1], Randall Reif[1], Dimitri Pappas[1]; [1]Texas Tech University

Fluorescence is one of the most utilized spectroscopic techniques for apoptosis detection. Recently it has been shown that fluorescence correlation spectroscopy (FCS) is capable of detecting apoptotic cells as early as 45 minutes after induction of apoptosis using the fluorogenic probe L-bis aspartic acid rhodamine 110. This probe was also utilized in the detection of apoptosis in a microfluidic device, where cells bound to an anti-CD95 coated surface were induced and the temporal dynamics of apoptosis were observed using fluorescence microscopy. In this work, the FCS method has been applied to apoptosis detection of cells captured in the same microfluidic device. The use of this device will allow cells to remain viable for longer amounts of time, allowing for

more specific information about the timing of caspase activation to be elucidated. In addition, subsequent analysis of the same cell by FCS will allow for caspase activity to be monitored over time with a greater level of sensitivity.

(181) **High Definition sFTIR Imaging of Plaques in Alzheimer Disease Mouse Model Tissue**

Marzena Kastyak[1], Michael Nasse[2], Carol Hirschmugl[2], Kathleen Gough[1]; [1]University of Manitoba; [2]Synchrotron Radiation Center (SRC) University

We are studying brain tissue from TgCRND8 mice, a transgenic model for Alzheimer disease (AD) expressing doubly mutant amyloid protein precursor and developing both dense core and diffuse plaques. Plaques are neuropathological features of AD formed as a result of amyloid peptide (Aβ) aggregation and deposition. Dense core plaques are primarily composed of closely packed amyloid fibrils characterized by β- sheet secondary structure. Aβ aggregates can also form amorphous diffuse plaques. FTIR spectromicroscopy provides secondary structure characterization and is a unique tool for biomolecular imaging in situ. We present results from the new mid-infrared beamline (IRENI - InfraRed ENvironmental Imaging) at the Synchrotron Radiation Center, University of Wisconsin at Madison. While similar to other configurations, being an infrared interferometer coupled to a microscope with Focal Plane Array detection, IRENI combines 12 brilliant synchrotron source beams with multi-element detection for a revolutionary advance in mid-infrared imaging capability. High definition chemical images are obtained at 0.54 micron pixel resolution with excellent signal to noise factor in minutes. Plaques are primarily composed of Aβ, however other tissue components are present even in dense core plaque spectra. Several new peaks characteristic for pure Aβ are now apparent in these well resolved spectra. The amide I maximum of the plaque core was at 1635 cm^{-1}, but also peaks of lower intensity, difficult to detect in IR spectral images acquired with lower spatial resolution, were present. We found that plaques can be imaged based on the area of the peak with a maximum at 1390 cm^{-1}. This peak is located in the spectral region free from scattering artefacts caused by the density of plaque core. We show the importance of this high definition sFTIR imaging for the analysis of the spatial distribution of biomolecules in plaques and adjacent tissue.

(182) **Polymer Fiber-Based Platforms for Measuring Gene Expression**

Kenneth Christensen[1]; [1]Clemson University

We have developed several polymer fiber-based diagnostic platforms that can be used for handling and analysis of minute volumes of fluids with high sensitivity and specificity. These include small bundles of polyvinylidene fluoride (PVDF) nanofibers which have extremely low non-specific binding, micron-sized polypropylene (PP) shaped fibers with outstanding fluid-wicking properties, and the Simultaneous Parallel Channel Diagnostic (SPCD) which consists of a capillary-channeled polymer fiber with more than 10 distinct parallel channels and functions as a simple multiplexed passive microfluidic array. The surfaces of all these fibers can be modified by addition of a "spin finish" during fiber production allowing conjugation of sensing molecules to the fiber surface using simple chemistries. While many classes of molecules, including antibodies, proteins, sugars, and oligonucliotides, can be immobilized on the surface of these polymer fiber substrates, here we report conjugation of molecular beacons or hybrid molecular probes for measuring gene expression. Using molecular beacons or hybrid molecular probes designed for a variety of inflammatory markers including IL-1β, TNFα, IL-6, and IL-10, we have produced fiber-based diagnostics for monitoring inflammation in a macrophage cell model. Detection limits have been measured to be in the fM regime using all platforms. We have measured levels of gene expression from sample volumes as small as 1 pL. Simultaneous detection of all 4 genes was performed in triplicate using µl volumes on a single SPCD. All these fibers can be produced on the km-scale from commodity materials and use very small reagent volumes making these platforms very inexpensive. These fiber-based devices are a potentially useful platform for point-of-use diagnostics in the laboratory or the clinic and represent a paradigm shift from existing microfluidic devices.

(183) **Encapsulated Silver Nanoparticles as Optical Labels for Biomolecules**

Kyle Dukes; [1]Clemson University

The unique optical properties of metal nanoparticles are of great interest for bioanalytical methods. Silver nanoparticles demonstrate the most efficient interaction with visible light but are unstable in physiological conditions. Therefore methods of protection must be developed for their use in bioanalytical methods. Modification of silver nanoparticles was conducted via self assembled monolayer (SAM) of hydroxyl terminated long chain thiols followed by encapsulation in silica via the sol-gel process. The hydroxyl terminated SAM provided an ideal surface onto which a dense silica shell was formed through sol-gel chemistry. The silica shell was densified through hydrothermal treatment at 130oC and characterized by UV-Vis and electron microscopy. The hydrophobic SAM and silica encapsulation provided stability to the silver nanoparticle in physiological medium. This was demonstrated by exposing the encapsulated particles to 1.0M NaCl. Several months of exposure in this solution showed no change in the particles optical properties. Contrarily, unprotected particles aggregate and dissolve instantly when introduced to the same conditions. The silica surface of the nanoparticle was functionalized with an epoxide terminated linker molecule, which is then reacted with a diazide via azide-epoxide coupling. The amine terminated linker molecule provides binding sites for proteins and biomolecules. Neutravidin was bound to the surface amino groups at pH 7.2. The modified silver nanoparticles were bound to cells exposed to biotinolated antibodies. The approach was used for the separation of cells in flow cytometry and imaging cells in optical microscopy. The intense light scattering of the silver nanoparticles allows for a non-fluorescent labeling. In addition, the encapsulated silver nanoparticle produces bright labels that have superior photostability compared to a fluorophore which has a limited lifetime.

(184) **Increasing HPLC Throughput for Analysis of Luciferin, its Intermediates, and Derivatives**

Celine Maravick[1], Leonard Moothart[1], Laurent Bernad[1]; [1]Promega Biosciences

Firefly luciferin derivatives (pro-luciferins) are effective substrates for biochemical analyses using bioluminescence detection. However, the superior sensitivity of this technique can suffer from unacceptable background due to luciferin contamination. HPLC methods are an indispensable tool in the production of pure products with low background. The objective of this project was to reduce method times by at least 50% without significant investment in equipment, yet maintain or even improve resolution. Original methods were approximately 30 minutes long and used a 250mm or 300mm column on an Agilent 1100 HPLC system equipped with a diode-array detector and a quaternary pump. This new approach was developed by modifying the instrument, gradient, and column packings. We will describe the use of shorter columns, modifications to minimize pooling and to better handle elevated pressures and modifications to the detection system to improve sensitivity and resolution. To ensure system was at its optimal

performance, a system suitability mix was instigated. Gradients were created to accommodate the greatest number of different substrates possible, while still providing acceptable separation and detection of intermediates. The goal was achieved in developing an accelerated analysis method, which reduced both cycle times and solvent use by approximately five fold, thus increased the HPLC throughput of the analytical lab.

(185) **Development of Novel Chiral Polymerizable Surfactants: Comparison for Chiral Separations in MEKC and CEC**

Jun He[1], Congying Gu[1], Shahab Shamsi[1]; [1]Georgia State University, Department of Chemistry

Micellar electrokinetic chromatography (MEKC) and capillary electrochromatography (CEC) are two of the major capillary electrophoresis modes that have been interfaced to mass spectrometry (MS) for sensitive and selective analysis of chiral compounds. To achieve chiral separation, chiral surfactant is used as pseudostationary phase in MEKC. On the other hand in CEC, chiral selectors can be either coated or chemically bonded on the stationary phase to form monolithic chiral stationary phase. One important question that will be addressed in this study is the following: Will MEKC and CEC provide similar chiral selectivity using the same chiral selector either present as pseudostationary phase or immobilized as monolithic phase? To answer this question, two novel chiral surfactants with leucine head group and polymerizable acrylamide tail was synthesized and used in both MEKC and CEC. For MEKC, chiral surfactants was polymerized (by 60 Co gamma radiation) into molecular micelle, which was used as pseudostationary phase. On the other for CEC, the same chiral surfactant was mixed with cross linker, porogens and initiator and was thermally polymerized into monolithic stationary phase. Two types of linker (amide and carbamate) with the same chain length were attached to the leucine head group: sodium 12-acrylamidododecanoyl-L-leucinate and sodium 12-acrylamidododecenoxy carbonyl-L-leucinate. Enantiosresolution, efficiency and selectivity of several ephedrine derivatives (e.g., ephedrine, pseudoephedrine, and N-methyl-ephedrine) and beta blockers (such as propranolol, alprenolol, and atenolol) were compared under CEC-MS and MEKC-MS conditions. This comparison will provide insightful knowledge on the difference of chiral recognition mechanism under MEKC and CEC conditions.

(186) **Analytical Method Development and Validation for Di (2-Ethylhexyl) Phthalate (DEHP) in Rodent Feed for Developmental and Reproductive Toxicology Studies**

Charles Crafford[1], Jennifer Gilliam[1], Gwendolyn McNeill[1], Donna Browning[1], Shaun Norton[1], James Blake[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

Di (2-ethylhexyl) phthalate (DEHP) is a ubiquitous environmental contaminant that has shown to cause adverse reproductive and developmental effects in laboratory animals. DEHP is a high production volume chemical that is commonly used as a plasticizer. It is found in many consumer products, including building materials, clothing, food packaging, child products, and certain medical devices. Human exposure to DEHP can occur through ingestion (via food or drink packaged in DEHP-containing plastics) and medical procedures that use DEHP containing plastics. The present work describes a development and validation of a formulation method for DEHP in two different rodent feeds, NTP-2000 and NIH-07, for use in toxicology studies. A critical component of any toxicological study is an acceptable analytical method to insure that the correct test article is being administered at the specified dose concentrations. Furthermore, it is also important to confirm the homogeneity and the stability of the dose formulations. To achieve these objectives a UPLC/UV method was developed and validated for analysis of DEHP in NIH-07 feed and subsequently cross-validated in NTP-2000 feed. Sample preparation includes extraction of dosed feed (~10 g) using methanol and direct analysis by reverse phase UPLC with UV detection at ¦Ë = 225 nm. The validation parameters included linearity, range, accuracy, precision, homogeneity, stability, and detection and quantitation limits. The method was successfully validated over a range of 15 to 1500 mg/kg (ppm) with an additional dilution scheme to extend the range up to 12,000 mg/kg in both feed types. Linearity of matrix and vehicle standard calibrations were confirmed by correlation coefficients ≥0.99. Analyte recoveries were determined to be ≥90% versus solvent standards. An experimental limit of quantitation (ELOQ) was established as 15 mg/kg and a limit of detection (LOD) was estimated as 0.6 mg/kg. Results are summarized below.

Method Parameter	Results
Linear range for both feed types	15 to 1500 mg/kg
Method verification (extended range) for both feed types	Up to 12,000 mg/kg
Linearity as correlation coefficient (r)	r ≥ 0.99 for both feed types
Precision (as %RSD) (NIH-07/NTP-2000)	≤ 2.2%/≤ 2.1%
Accuracy (% Relative Error)	A range of -8.4% to 6% for both feed types
Average extraction recovery (Vehicle standard/Solvent standard) x 100%)	98.4%/91.6% (NIH-07/NTP-2000)
Storage stability/Dose simulation stability	Confirmed for 42 days/7 days

(187) **Investigations on the Extensibility of Electrochemically Modulated Separations (EMS) Using Ion Selective Electrodes Coupled with ICP-MS Detection**

Katy Fordyce[1], Kate Ziegelgruber[1], Michael Green[1], Shane Peper[1], Douglas C. Duckworth[1]; [1]PNNL

Electrochemically modulated separation (EMS) has historically been used to concentrate easily reduced metals for plasma spectroscopy, and more recently, actinide (IV) ions on anodized glassy carbon electrodes [1-4]. The general experimental setup consists of a three electrode flow-by cell which controls the deposition and subsequent stripping of metal analytes. The potentials applied within the cell, as well as the properties of the electrode surface, greatly influence the deposition of analyte ions. Replacing anodized glassy carbon electrodes with ion selective electrodes (ISEs) represents a promising approach in terms of surface modification. ISEs, an established class of electrochemical sensors capable of selective ion extraction, consist here of polymeric membranes applied to the surface of glassy carbon electrodes. The analytes to which an ISE responds can easily be changed by adjusting the components which make up the polymeric membrane. Currently, the following elements have been studied using the EMS-ISE approach: cesium, cadmium, silver, and neptunium. These elements represent both redox active and redox-inactive species over a wide mass range. Cesium in particular was chosen as an analyte because it is monovalent and represents a broad class of elements whose redox chemistry does not suit more general EMS approaches. Here, we report the systematic optimization of the ISE components for multiple elements and characterize the performance of the electrodes when coupled in-line with ICP-MS for the separation, concentration and detection of various analytes from clean and complex matrices. [1] J.R. Pretty and G.J. Van Berkel, Rapid Commun. Mass Spectrom., 12, 1644 (1998). [2] J.R. Pretty, D.C. Duckworth and G.J. Van Berkel, Anal. Chem., 70, 1141 (1998). [3] W.J. Clark Jr., S.H. Park, D.A. Bostick, D.C. Duckworth and G.J. Van Berkel, Anal. Chem., 78,

8535 (2006). [4] M. Liezers, S.A. Lehn, K.B. Olsen, O.T. Farmer, and D.C Duckworth, J. Radioanal Nucl Chem., 282, 299 (2009).

(188) **Simlutaneous Estimation of Silylated Monosaccharides and Bio-Ethanol in Lignocellulose Hydrolysate by Gas Chromatography**

Anju Chopra[1], Dheer Singh[1], Ravi Sahai[1], Ravinder Kumar[1], M.B. Patel[1], A.S. Sarpal[1]; [1]Indian Oil Corporation, R&D Centre

Cellulosic biofuels, the second generation biofuels, made from inedible parts of the plant offer the most environmentally attractive and technologically feasible alternative to fuel. Moreover, the biomass used for the production of second generation biofuel is generally wood residue, saw dust, agricultural residue such as corn stalks and wheat straw, and therefore it does not decrease the amount of biomass available for food. Although, cellulosic biomass can be converted into any type of fuel such as ethanol, gasoline, diesel, jet fuel etc., the conversion route that has attracted the fuel industry most, is unlocking the sugars and their fermentation into bioethanol. The hydrolysis of celluloses and hemicelluloses generates monosaccharides such as C5 / C6 sugars which are further fermented to ethanol. The monitoring of production of ethanol requires estimation of C5 / C6 sugars as well as ethanol in the hydrolysed cellulose / hemicellulose. Since the saccharification and fermentation process produces an aqueous supernatant containing both volatile and non-volatile materials including high concentrations of biomass debris, novel analytical methods are required for estimating the concentrations of these materials. Chromatographic techniques have been widely used by various laboratories for monitoring the bio-ethanol concentration, C5/C6 sugars and other side products such as furfural and hydroxymethyl furfurals. The monitoring of volatile components such as ethanol and furfural is carried out normally by Gas chromatography while the non-volatile monosaccharides such as glucose, fructose, xylose and arabinose are determined by HPLC technique. The use of two different techniques for analyzing the lignocellulose hydrolysate in totality is very time consuming. Therefore, a method has been developed by Gas Chromatography for the simultaneous estimation of ethanol, C5/C6 sugars and furfural in a single chromatographic run. The C5/C6 sugars have been derivatised using N-methyl-N-trimethylsilyltrifluoracetamide (MSTFA) to convert them into the volatile silyl ethers. The separation of C5/C6 sugars, ethanol and furfural has been optimized on a polar Polyethylene Glycol based stationary phase which is compatible with aqueous phase. The quantitation has been performed against isopropanol internal standard using FID detector.

(189) **Portable Microcoil NMR Detection Coupled to CE for the Analysis of Perfluoro Organic Acids**

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Previous research has demonstrated the advantages of coupling capillary electrophoresis (CE), a simple rapid separation method, with an on-line nuclear magnetic resonance (NMR) detection system to separate, identify, and provide structural information of analytes in nanoliter sample volumes. To date, most of the coupling work has been performed in large laboratory scale NMR instruments. New technological developments in electronics have reduced the size of the NMR system and small high-field permanent magnets provide the possibilities of a truly portable NMR which could offer a low-cost, high-throughput and information-rich detection method for substances separated and preconcentrated by CE. The resolution of the current portable NMR system is approximately 2 ppm which is sufficient for the analysis of fluorine containing molecules. One particular application is the analysis of longer chain perfluoro organic acids (PFAs) which are of environmental concern and have attracted attention, due to their detection in human and animal samples along with their persistence in the environment. Perfluorinated surfactants are used in a large number of industrial applications and consumer products because of their unique surface active properties. Despite the widespread use of these compounds, little is known about their environmental long term health effects due to exposure. Therefore, it is important to develop new analytical techniques to both get a better understanding of these substances, to elucidate their structure in different matrices and to monitor products intended for human use as well as the environment for PFAs. In this work, CE is coupled with a portable, briefcase-sized NMR system that incorporates hand-wound microcoils which are placed around a bubble-cell capillary and a 1.8 T permanent magnet to measure 19F NMR spectra. The bubble-cell configuration around the NMR detection coil in the 75 μm ID capillary shows great sensitivity improvements in the NMR spectra for the PFA compounds. We will further present details on the on-line 19F-NMR-CE system with some representative separations of longer chain PFAs. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

(190) **Superheated Water Extraction for the Determination of Aliphatic Hydrocarbons in Source Rock and Its Application in Geochemical Exploration**

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The extraction of hydrocarbons from petroleum source rock by superheated water was investigated using a simplified method and the conditions for maximum yield were determined. The results showed that the temperature and kinetic rates have significant effects. The optimum temperature for the extraction of n-alkanes and isoprenoid hydrocarbons from sedimentary organic rocks was 250 °C. The optimum extraction time for lower and medium molecular weight n-alkanes was 20 minutes, while the higher molecular weight alkanes were exhaustively extracted at 50 minutes. The yields of the analytes were much higher with using the superheated water extraction method than Soxhlet extraction. The recoveries of the n-alkanes for GC-MS analysis from the extractant water by SPME with a PDMS fibre ranged from 90 – 100 % and most of the compounds were above 93%. Application of superheated water in geochemical exploration for petroleum was also investigated. Using Niger Delta samples as a case study, the geochemical ratios and parameters were calculated from n-alkane and isoprenoid hydrocarbon data. The pristane/phytane, pristane/nC17, Ph/nC18 ratios, and carbon preference index (CPI) ranged from 0.90 – 1.29, 0.61-2.30, 0.86-1.44, and 0.88 – 1.88 for the western Niger Delta samples, respectively, and from 0.35-3.49, 1.01-3.03, 0.87-1.98, and 1.00 – 2.15 for the eastern Niger Delta samples, respectively. The geochemical plots revealed that a preponderance of the samples from both western and eastern Niger Delta had mixed organic matter input and a good number of the samples also had contributions from terrestrial organic matter while few samples had strong contributions from marine organic matter. The plots also indicated that samples were sourced by organic matter deposited in more of reducing environments than oxidizing environments. Biomarker data also confirmed that the samples are mainly of terrestrial and mixed organic matter origin. The results of this study agreed with the results of previous studies based on Soxhlet extraction sample preparation. The study showed that superheated water extraction provides a better alternative to Soxhlet extraction as samples preparation procedure in geochemical evaluation of petroleum source rocks because of its environmentally friendly nature.

(191) **The Importance of Protein Phosphorylation in Cerebral Spinal Fluid for the Development of Biomarkers Preventing Strokes**

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Human cerebral spinal fluid (CSF) is a secretion product of several different central nervous system structures. It surrounds the brain, spinal column, as well as the optic nerve up to and including the optic disk. As a relatively undiluted draining system from the brain, it reflects several different disorders of the central nervous system [1]. Metallomics represents a comprehensive approach to study and identify metalloproteins as well as phosphoproteins present in the CSF, and may be utilized to discover disease associated proteins as possible biomarkers to signal further complications from diseases, such as a subarachnoid hemorrhage stroke. The combination of subarachnoid hemorrhage (SAH) with cerebral vasospasm (CV) leads to severe debilitation or death of an estimated one million people worldwide every year. A biomarker that would predict CV after a SAH has yet to be found. A significant difference in the phosphorproteom could be one step towards the discovery of a diagnostic marker that may predict CV after SAH. The significance of phosphorylated proteins as a marker is manifested in the constitutive nature of intracellular signaling involved in the pathological events seen post SAH. The aim of this work is to use capillary liquid chromatography (cap-LC) coupled to inductively coupled plasma mass spectrometry (ICPMS) and nano liquid chromatography-phosphochip-electrospray ionization-ion mass spectrometry (NanoLC-Chip-ESI-ITMS) in order to study phosphorous associated with particular proteins. [1] Cohen, P. Annu. Rev. Biochem., 1989, 58, 453-508

(192) **Investigation of Black Particles in Melt Granulation**

Frances Liu[1], Charles Pan[1], Greg Argentieri[1], Don Drinkwater[1], Ferris Harmon[1], Rosario LoBrutto[1]; [1]Novartis Pharm.

To investigate the nature of black particles found in melt extrusion process, the following tests were performed, X-ray, MDSC, Raman, FT-IR, size exclusion chromatography (SEC)/Nitrogen detector, SEC/PDA, SEC/triple detectors (Refractive Index, light scattering, and viscometer), SEC/MS, ICP/MS, Light microscopy, scanning electron microscopy (SEM), and energy dispersive x-ray (EDS). The crystalline form of DS did not change in black particles. However, an extra crystalline peak was detected by X-ray. The melting point of DS in black particles decreased since the impurities existed. Chemical change of black particle on the tablet was not detected by Raman but did detected by FT-IR. The metals (or metal ions) which related to extrusion machine were detected in black particles by SEM/EDS. Ni, Co, and W contents in the tablets with black particle were higher than those in the tablet without black particle (ICP/MS test). HPC is the major excipient used in this melt granulation. Modified HPC was found in black particles, that contained nitrogen (detectable by a nitrogen detector), conjugated structures with a UV chromophore (detectable by a PDA detector), higher molecular weight (measured by triple detector), and two more different polymers (detected by MS). Four DS degradation products were identified based on MS data.

(193) **Development and Validation of an X-ray Fluorescence Method for Detection of Toxic Metals in Pharmaceutical Products**

Sergey Arzhantsev[1], Lucinda Buhse[1], Benjamin Westenberger[1], John Kauffman[1]; [1]US Food and Drug Administration

Non-western medicine is becoming increasingly popular among the American public. Medication based on so-called “ancient recipes” from China and India can be easily purchased over the internet. In several recent cases it has been found that the use of traditional Chinese and Indian medications resulted in toxic metal poisoning. The FDA has developed a rapid and reliable screening method using portable X-ray fluorescence (XRF) instruments for detection of toxic metals in traditional medicines and pharmaceutical products. The XRF method described in this presentation utilizes the continuous wavelet transform to eliminate background signal that is ubiquitous in XRF spectra. A limit test based on signal-to-noise ratio at element-specific emission energies has been developed to identify materials than contain toxic metals. This approach has several advantages over empirical calibration methods that are commonly used to analyze XRF spectra. For example, the method is insensitive to matrix effects, and does not require the preparation of a quantitative calibration model. The XRF method was validated through a collaborative study which included 6 FDA Laboratories. The statistical results from collaborative study and details of the method will be presented.

(194) **PAT for API Drying Process Understanding and Control**

Charles Goss[1], Susan Barnes[1], Dennis Crowe[1], Brian Crump[1], Erwin Irdam[1], Rahn McKeown[1], Ailette Tobien[1]; [1]GlaxoSmithKline

Drying is a common unit operation for most isolated compounds such as active pharmaceutical ingredients (APIs), intermediates, and starting materials. Online process analytical technology (PAT) monitoring of the drying step can provide process understanding to enable greater control of material properties (e.g. water/solvent content, polymorphic form, particle size, flow characteristics, etc.) and ensure consistent drug substance for formulation. It can also reduce cycle times, minimize sampling and operator exposure, and facilitate process transfer. This talk will focus on the drying of an API hemihydrate that could easily over dry to form dehydrated API if inappropriate conditions were used. The use of several complementary online PAT measurements to monitor API drying will be illustrated: (1) mass spectrometry (MS) to monitor solvent efflux, (2) near infrared spectroscopy (NIR) to monitor API polymorphic form and bound water/solvent content, (3) dryer pressure to help control the drying conditions, and (4) API cake temperature to provide comparative data for monitoring in other dryers. Together, these tools provided a comprehensive view of how the drying process conditions affected the API drying and dehydration steps, and enabled process control options to minimize the risk of dehydration.

(195) **Utilizing Spectral Counting to Quantitatively Characterize the Effects of Abundant Protein Depletion and the ALiPHAT Method in Human Plasma Proteomics**

Christopher M. Shuford[1], Adam M. Hawkridge[1], John C. Burnett, Jr.[2], David C. Muddiman[1]; [1]North Carolina. State University; [2]Mayo Clinic College of Medicine

Biomarker discovery and validation efforts in human plasma are greatly hindered by the large dynamic range of protein concentrations. In a traditional proteomics workflow, lower abundance plasma proteins of biological significance often fail to overcome the detection threshold of the instrumentation because of their low absolute concentration and the suppressive affects of more abundant proteins. To this end we have utilized a set of commercially available abundant protein depletion columns, connected in series, to perform tandem depletions so as to maximize the efficiency of the abundant protein removal. In conjunction with this study, we have for the first time applied our hydrophobic cysteine alkylation strategy, termed ALiPHAT, to the analysis of human plasma in order to evaluate its effects within a complex mixture. During these analyses, exogenous B-type Natriuretic Peptide-32 (BNP-32) was spiked into the plasma samples to serve as a detection benchmark. This protein was chosen

based on its low circulating concentrations in plasma (~ 3 pg/mL endogenous), the fact that it contains two cysteine residues, and its importance as a biomarker for congestive heart failure. Spectral counting was utilized to quantitatively characterize the efficacy and consequences of the depletion steps and hydrophobic tagging. Tandem depletion of 14 abundant plasma proteins dramatically increased the distribution of spectral counts for the majority of remaining lesser abundant proteins. Observation of decreased spectral counts between depleted and un-depleted samples in these experiments suggests that several proteins were unintentionally depleted as a result of antibody cross-reactivity or co-immunodepletion. Interestingly, the use of tandem depletion steps did improve the depth of proteins detected. When applying only one or two depletion columns the exogenous BNP-32 was not detectable; however, when using three columns in series the BNP-32 was detected. Also, the use of a newly synthesized hydrophobic alkylation reagent in place of iodoacetamide nearly doubled the number of observed spectral counts for BNP-32 in this same depleted plasma sample. This same hydrophobic alkylation reagent improved the signal for the BNP-32 tryptic peptides effectively enough such that it was detectable in samples with only serum albumin depleted.

(196) **Characterization of Core-Shell Structured Nanofibers Prepared by Coaxial and Triaxial Electrospinning**

Wenwen Liu[1], Yilin Liu[1], Giriprasath Gururajan[1], D. Bruce Chase[1], John F. Rabolt[1]; [1]University of Delaware

Raman scattering and attenuated total reflectance-Fourier transform infrared (ATR-FTIR) measurements were carried out as a means to demonstrate and characterize the core-shell structure of the electrospun nanofibers. Core-shell structured nanofibers were fabricated by multiaxial (coaxial/triaxial) jet electrospinning with polycaprolactone (PCL), a biodegradable polymer, as the shell and gelatin as the core. ATR-FTIR demonstrated the presence of both PCL and gelatin in the nanofibers. The nanofibers were treated with water at different temperatures to study the extent of the dissolution the gelatin core. ATR-FTIR spectra of the post-treatment samples showed the disappearance of the gelatin component and demonstrated the core-shell structure of the nanofibers. This was further supported by the result of FE-SEM examination of post-treatment samples, showing fracture and break of the nanofibers. Electrospinning is a technique to process polymer solution or melts into continuous nanofibers by the application of a strong electric field. The low toxicity three-component (acetic acid/ethyl acetate/water) solvent was used for gelatin, while either a dimethylformamide (DMF)/chloroform (CF) mixture or 2,2,2-trifluoroethanol (TFE) was used as the solvent for PCL. The relationship between the inner- and outer- flow rates and fiber diameter distribution was studied. The results provide a basis for the further study of the preparation of multistructured nanofibers, and illustrate the potential for the application of vibrational spectroscopy to characterization of nanofibers.

(197) **Glycomic Quantification Using Surface Enhanced Raman Tagging Method**

Dongmao Zhang[1], Karthikeshwar Vangala[1], Michael Yanney[1], Roneasa Garner[1], Andrzej Sygula[1]; [1]Mississippi State University

Glycans play a critical role in bacterial and viral recognition as well as anticoagulation, cell to cell communication and cell metastasis. Glycans are found either bound to protein (glycoprotein) or conjugated with lipids (glycolipids). In addition, there are many glycan-specific diseases, including autoimmune diseases, cancer and schizophrenia. Comparative glycomics, the quantitative comparison of glycan levels in two or more biological samples is essential for biomarker discovery and early disease diagnosis. Current glycomic methods are exclusively mass spectroscopy and fluorescence based, and they have limited quantification sensitivity and/or accuracy. In this talk we will present a novel surface enhanced Raman based glycomic quantification method using the surface enhanced Raman tags we recently developed. Current detection sensitivity of the SERS-tagged glycans is in the sub nM range. In addition to SERS tag design and synthesis, possible ways to further improve glycan quantification sensitivity and accuracy will be discussed together with potential ways to integrate this SERS-based glycan quantification with mass spectrometric glycan identification.

(198) **Sequence Specific Genotoxicity Sensing: An Electrochemical Approach**

Eli G. Hvastkovs, Jennifer E. Satterwhite, Amanda M. Pugh, Allison S. Danell; [1]East Carolina University

Genetic modifications caused by exposure to outside substances (xenobiotics) can cause serious health problems.1 Metabolic processes work to eliminate xenobiotics from the body often generating reactive molecules in the process. These “bioactivated” metabolites can react with DNA in a process called genotoxicity. Genotoxicity can lead to permanent DNA mutations.1 Chemical industries screen for potential genotoxicity, but established assays cannot provide pertinent information. Genotoxicity assays can indicate compound reactivity toward DNA,2 but important disease related questions are answered when the actual damaged DNA site is known.3 This information is gleaned after following complicated, expensive, and time consuming procedures.4 Inexpensive, high throughput technologies that offer a rapid molecular-based DNA sequence damage assessment would be welcome in developmental settings. Electrochemical platforms offer sensitivity, cost, throughput, and miniaturization benefits that make them highly attractive for bioanalysis.5 An electrochemical biosensing platform will be presented that can resolve sequence specific DNA damage due to direct acting and *in situ* generated reactive metabolites. Au electrodes modified with a 21-mer double stranded DNA (dsDNA) sequence of the TP53 gene were used to assay DNA sequence reactivity of bioactivated benao[a]pyrene (BP) and its ultimate metabolite, (+)-anti-benzo[a]pyrene-7,8-dihydrodiol-9,10-epoxide(BPDE). Electrochemical monitoring was accomplished using a di-viologen redox molecule, (CH3(CH2)11V2+(CH2)6V2+(CH2)11CH3 (V2+ = viologen (4,4’-bipyridyl), C12Viologen)) that exhibits characteristic voltammetry in the presence of dsDNA. Exposure to BP solutions resulted in C12Viologen voltammetry changes consistent with morphology alterations by covalent DNA damage. Controls show that the damage is specific for BP on this particular DNA sequence. The sensor response was validated using UV-vis and mass spectroscopy. This platform provides concept proof that genetic hotspot elucidation is possible using electrochemical techniques.

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(200) **Measurements of Sample Heating by a Laser Induced Air Plasma in Orthogonal Dual-Pulse Laser Induced Breakdown Spectroscopy**

Janna Register[1], S. Michael Angel[1]; [1]University of South Carolina

The objective of this research is to temporally resolve the temperature change of a solid sample as it is heated by a Laser Induced Air Plasma and to use this information to determine the

contribution of sample heating (by the pre-ablation air plasma) in Dual-Pulse Laser Induced Breakdown Spectroscopy (DP-LIBS). LIBS is an atomic emission analysis technique in which a high-powered laser pulse (typically 100 mJ with a pulse width of several nanoseconds) is focused to a sub-millimeter spot on or in a solid, liquid, or gaseous sample. LIBS, while having great analytical potential, is restricted by practical limitations like ablative irreproducibility, sample inhomogeneity, and matrix effects. However, the discovery of large emission enhancements by DP-LIBS has renewed general interest in the technique. Although it is generally accepted that sample heating is not the major contributor to enhanced emission in DP-LIBS, quantitative measurements of the degree to which sample heating contributes to enhanced emission and ablation have not been described. The temperature of a LIP is tens of thousands of degrees and is formed millimeters from the sample surface, leading to significant sample heating by the air plasma. However, the LIP only lasts tens of microseconds making temporal resolution of surface temperature difficult to measure. The research presented here employs the use of very small, fast thermocouples. Using an Nd:YAG laser at 1064 nm with a 10-ns, 200 mJ pulse, the smallest thermocouples (12.5 µm in diameter) register temperature changes of several hundreds of degrees when placed 0.5 to 3 mm from the LIP. SP-LIBS measurements of the same sample, heated to comparable temperatures show emission and ablation enhancement of only 2-3 fold, indicating at most a minor contributing role of sample heating to the overall pre-ablation spark, DP-LIBS enhancement.

(201) **Determination of Praziquantel and Fenbendazole by Second Derivative Spectrophotometry**

M. Ines Toral[1], Cesar Soto[2], Romina Otipka[1], Sandra Orellana[1]; [1]Faculty of Science, University of Chile; [2]Univesity of Concepcion

Within the group of antiparasitic drugs, anthelmintic products are widely used in veterinary medicine in cattle and pet. Since the anthelmintic spectral band of most drug used for treatment is limited, combination of more than one active ingredient are required to control mixed helminthic infections effectively. Evaluation of such preparation thus requires the determination of their active ingredient in drug formulation. In this context praziquantel (PZQ) ((RS)-2-(Cylohexylcarbonyl)-1,2,3,6,7,11 b-hexahydro-4H-pyrazino (2,1-alpha) isoquinoline-4-one) and fenbendazole (FBZ) (methyl N-(6 phenylsulfanyl-1H-benzoimidazol-2-yl)carbamate) are used to control parasitic infections in pets, where the pharmaceutical formulation has a 1:10 ratio mass. In this work is proposed a method for the simultaneous determination PZQ and FBZ in pharmaceutical formulation by derivative spectrophotometry. Taking into account a study of solubility, solvent effect and spectral behavior for both drugs, HCl 10-1 mol L-1 in ethanol was selected as solvent because the drugs are completely solubilized and that spectral bands despite are overlapped the second derivates show zero crossing characteristic for FBZ and PZQ. The second derivative spectra were obtained using smoothing factor 16,000, amplification factor 10,000 and the wavelengths, were 248.8 and 226.4 nm, for the determination of FBZ and PZQ, respectively. The detection limits were for FBZ 2.9 x10-7 mol L-1and for PZQ 4.5 x10-7 mol L-1. The determination ranges for both drugs were 9.9 x10-7 to 4.0 x10-4 mol L-1 and 1.5 x10-6 to 2.5 x10-4 mol L-1 for FBZ and PZQ, respectively. Good levels of repeatability (RSD) of 2.0% and 2.4% were observed for FBR and PZQ, respectively. Synthetic samples corresponding to 10:1 mass relation of FBZ:PZQ, a different concentration between 1.0 x10-5/1x 10-6 to 5.0 x10-5/5.0 x 10-6 mol L-1 were prepared from standard solution and it was found that the recoveries were 100.4 ± 2.0% and 99.4 ±3.0% for FBZ and PZQ respectively. The method was applied in FENTEL10kg (Laboratory KUALCOS S.R.L.,Buenos Aires, Argentina), with a content nominally 500 mg of FBZ and 50 mg of PZQ. Following the proposed procedure, FBR presents an average of 497.2 ± 1.2 mg and PZQ 49.2 ±0.5 mg. This work was financed by FONDECYT Project N° 1100103.

(202) **Raman Spectroscopy Using a Spatial Heterodyne Spectrometer**

Nathaniel Gomer[1], Christopher Gordon[1], S. Michael Angel[1]; [1]University of South Carolina

The objective of this research is to develop a spatial heterodyne Raman spectrometer (SHRS) and compare the performance to comparable grating based systems. The SHS is a modified Michelson interferometer, with diffraction gratings used in place of mirrors. The SHS operates in Littrow configuration, where the angle of incidence on the grating is also the angle of diffraction. The SHS produces an interference pattern on a CCD so it does not require moving parts to make a measurement. The throughput of the SHRS should be much higher than a grating based spectrometer because of the SHS increased angular acceptance and lack of an entrance slit. The system we are developing should allow a band pass of ~2500 cm-1, while maintaining a resolving power of 3700, with a spectral resolution of ~5 cm-1.

(203) **Optical Fiber-Based Tools for the Analysis of Zinc in Aqueous Environments**

Steven Kopitzke[1], Peter Geissinger[1]; [1]University of Wisconsin-Milwaukee

Zinc is an essential nutrient required for many organisms but is toxic at elevated levels. Because many manufacturing processes produce zinc as a waste product, continual monitoring of zinc levels in the environment at many locations is essential. However, collecting and processing samples for laboratory analysis is time-consuming and prohibitive for covering large numbers of potential sampling sites. Moreover, monitoring in real-time, which clearly is desirable given the dynamic nature of concentrations of chemical species in the environment. Optical-fiber-based sensors provide an alternative sensing modality that allows for remote monitoring of large areas in real time: many sensor regions can be created on a single optical fiber, where changes in the luminescence of the sensors indicate the presence of substances of interest. These sensor regions can be interrogated in real-time and spatially resolved, when pulsed-laser excitation and time-resolved detection of sensor luminescence pulses at the fiber end is utilized. Specifically, this design utilizes evanescent waves from a laser pulse as a means of excitation for luminescent sensor molecules that are responsive to the presence of zinc and that are attached to the surface of the fiber core. To ensure a rapid response time, the sensor molecules are contained a fiber-cladding material, which contains microscale and nanoscale channels for efficient analyte transport; these channels were created in the cladding using a microsphere templating strategy. This sensor demonstrates the ability to effectively measure concentrations as low as 90 ng/L. This fact coupled with a stable signal being obtained in under 30 seconds makes this sensor design an ideal candidate for remote monitoring in various circumstances.

(204) **Polarized Fourier Transform Infrared Spectroscopic Study on Molecular Orientation in Electrospun Polymer Fibers**

Xiaoqian Ma[1], Bruce Chase[1], John Rabolt[1]; [1]Materials Sci. and Eng., Univ. of Delaware

Polarized Fourier transform infrared (FTIR) spectroscopy was applied to analyze the driving force for the molecular orientation of electrospun poly (vinylidene fluoride) (PVDF) fibers. The effects of applied voltage, working distance and electric field distribution on the molecular orientation were investigated. PVDF fibers were collected between two counter electrodes separated by an air gap under different electrospinning conditions. Scanning electron microscopy (SEM) images confirmed that PVDF fibers were

macroscopically aligned. Polarized FTIR spectra demonstrated PVDF fibers were also aligned at molecular level. Furthermore, both dichroic ratio and total absorbance for three-dimensional fibers calculated from polarized FTIR spectra were evaluated to reveal the relationship between the molecular orientation and the electric field.

(205) **Application of DRIFTS for Direct On-Filter Characterization of Extrathoracic Wood Dust Collected in Working Environments**

Madalina Chirila[1], Taekhee Lee[1], Michael Flemmer[1], James Slaven[1], Martin Harper[1]; [1]Nat. Inst. for Occupational Safety and Health

The current method to assess exposure to wood dust is to collect the aerosol in the workers breathing zone and to determine the mass in a known volume and assume all is wood dust. Since the gravimetric analysis is non-specific, there is interest in separately measuring the wood component from other components of dust. In this study, Diffuse Reflectance Infrared Fourier Transform (DRIFT) spectroscopy has been employed to determine the mass of wood dust. The cup from the diffuse reflection unit was replaced with a motorized translational horizontal stage where a typical 37 mm filter with wood dust was set. Diffuse reflection spectra were collected from across six diameters of the filter to average the signal from most filter surface. Two reflection bands at 1510 and 1595 wavenumber, attributed to symmetric and asymmetric stretching vibrations of the aromatic rings of lignin, were monitored for quantitative analysis. The choice of 37 mm silver metal membrane filter instead of the less expensive but more absorbent glass fiber filter for background and support for the wood dust allows proper identification of the type of wood dust (hardwood or softwood). Separate calibration curves were constructed for standard extrathoracic (particle size between 10 and 100 µm) red oak (hardwood) and yellow pine (softwood). Calibration of DRIFT intensity versus known wood dust mass on the filter using Kubelka-Munk function showed non-linear dependence on mass less than 10 mg (approx. 13 µg/mm2 of wood dust per square millimeter). This mass limit is dictated by workplace samples which typically have total gain of less than 10 mg. The experimental conditions and the small-thickness samples indicate that Kubelka-Munk conditions are not obeyed for samples that have less than 5 mg of wood dust. Alternatively, the pseudo-reflection log (1/R), while still giving non-linear dependence against mass, is closer to a linear dependence.

(206) **Globalization of Spectroscopic Data Access**

David Joyce[1], Steve Best[1]; [1]Thermo Fisher Scientific (Informatics)

In data intensive disciplines such as spectroscopy, collaboration and effective use of information can be hindered by poor data management. Large quantities of data can be rapidly generated and often then resides in silo's either on or near the instrument in question. This leads to problems when the data needs to be analysed or compared with results derived from other reference techniques. The issue is compounded when the data is stored in a proprietary format requiring access to the original data capture software to extract any meaning. When this laboratory scale problem is mapped onto a global organization then it is clear that a system is required to manage this data. We will describe how readily available information technology can be applied to scientific data to address these problems. Desktop data conversion tools such as GRAMS/AI or Open Babel help to unlock the data from proprietary formats making use of data standards such as AnIML, GAML, JCAMP and MZXML to allow data portability. When these tools are combined with modern internet technology this provides portals such as www.chemspider.com and www.spectraonline.com which allow global collaboration on scientific data sets. Ultimately these technologies will be driven down to the organisational level - so that we can soon expect the same ease of access to our scientific data as Google has brought to the printed word.

(207) **Investigation of Excitation / Ionization Processes of High-Power Pulsed Microplasma for Aqueous Sample Analysis**

Yoichi Nagata[1], Yuichiro Takahashi[1], Yuta Negishi[1], Kenji Kodama[2], Hidekazu Miyahara[1], Kuniyuki Kitagawa[2], Akitoshi Okino[1]; [1]Tokyo Institute of Technology; [2]Nagoya University

Inductively coupled plasma (ICP) is widely used as an ionization or excitation source for elemental analysis. ICP has served as the most powerful tools for trace elemental analysis because they offered detections at sub-ppt levels and dynamic range of up to 8 orders of magnitude. In recent years, elemental analysis research has centered on exploration on samples that are small in volume, such as nano-particles or bio-cells. However, conventional ICP system consumes large volumes of test sample (0.5 to 1 mL/min) and so it is not good for these applications. To analyze small sample more efficiently, a high-power pulsed microplasma source has been developed. With this device, a pulsed power generator is developed and used to achieve high-power operation avoiding electrode damage due to overheating. Short-duration high voltage (less than 100 ns, ~2.5 kV) pulses are applied for ignition of the plasma followed by longer duration, relatively low voltage (~10 us, ~0.5 kV) pulses to excite and ionize the analytes. With this system, 40 kW of the peak electric input power can be realized to enhance the emission intensity 1,500 times compared with usual dc operation. In this study, spectrometer with intensified charge coupled device (ICCD) detector is used for high-speed and high-sensitive measurement, which realizes 1 us of time resolution. By introducing 1,000 ppm yttrium solution with an ultrasonic nebulizer, emission intensity of Y I, Y II and Y III is investigated. As a result, Y III takes maximum 4 us after ignition, while Y I and Y II does 5 us after. Thus, from 4 us to 5 us after ignition, Y III ratio decreases while Y I and Y II increase relatively. 5 us and more after, Y II weakens earlier than Y I. These facts show that recombination rate of doubly charged ions exceeds production rate 4 us after the ignition. Then, population of singly charged ions decreases 5 us after ignition, followed by reduction of excited atoms.

(208) **Newly Observed Effects of Anions on the Raman Bending Vibration of Water**

Henk-Jan van Manen, Bojk Berghuis, Aurelie Arrouet, Rob Bloemenkamp, Oscar van den Brink; [1]AkzoNobel RD&I

Quantitative Raman spectroscopy is gaining importance in process analytical technology as a label-free, chemically specific method for the in-line analysis of gases, liquids, slurries, and solids. In aqueous solutions, the Raman bending vibration of water at 1640 cm-1 is often used as an internal standard in order to correct for changes in the properties of the sample and the Raman analyzer. Whereas the effects of dissolved electrolytes on the Raman O-H stretching bands of water have been known since the 1930s, only a handful of reports have detailed the changes in the water bending vibration in aqueous electrolytes compared to bulk water. For a reliable internal standard in process applications, a more detailed understanding of the effects of aqueous electrolytes on the water bending vibration is of crucial importance. This study therefore presents in detail the effect of 9 different anions on the Raman bending vibration of water. First, we will show that the effect of increasing concentrations of anions on the intensity of this band vary from mildly negative (carbonate) to largely positive (iodide). The anions only roughly follow the Hofmeister series, with notable deviations observed for perchlorate and nitrate, confirming recent reports that cast doubt on the notion that the making and breaking

of water structure is crucial to the Hofmeister series. Second, our precise measurements show for the first time that the intensity of the Raman bending band of water increases nonlinearly with concentration for most of the anions, which may be due to overlapping ion hydration shells at higher concentration. Third, using multivariate curve resolution (MCR) and peak fitting, we show that multiple bending vibrations of water are present in iodide solutions, which also has not been reported before. In conclusion, our study has revealed large and some hitherto unknown effects of anions on the Raman bending vibration of water. These results are of fundamental interest, because they shed light on the behavior or water in electrolyte solutions, as well as of practical interest with respect to using the bending vibration as internal standard in process Raman applications.

(209) **Evaluation of a Portable SERS Substrate Reader for Point of Use Analysis**

David Eustace[1], Alastair Mcinroy[1], Bryan Ray[2], Rick Cox[2]; [1]Renishaw Diagnostics; [2]DeltaNu-Intevac Photonics

The development of portable Raman spectrometers has led to their use in many different industries such as pharmaceuticals and law enforcement. Due to the inherent poor sensitivity available for normal Raman methods, current portable systems are exclusively used to identify bulk materials and cannot be used for trace detection. To overcome this poor sensitivity, Surface Enhanced Raman Scattering (SERS) can be employed. The use of SERS substrates could open the door to new applications such as trace detection of pharmaceutical residues during cleaning verification or point of care medical diagnosis. Here, data will be presented showing the application of a portable Raman system for detection of targets for biomarker assays using SERS tags and trace pharmaceutical applications. Data will also be presented comparing a portable Raman system designed for reading SERS substrates to that of a large Raman microscope system in a pharmaceutical cleaning application.

(210) **Investigating the Relationship of Instrument Parameters and Sample Complexity Towards the Analysis of Label-Free Relative Quantification Using Spectral Counts**

Genna Andrews[1], Adam Hawkridge[1], David Muddiman[1]; [1]North Carolina State University

Subsequent to protein characterization by liquid chromatography coupled to mass spectrometry (LC-MS), protein quantification measurements prove significant for proteomic science. Quantification measurements afford protein expression levels resultant from changes in environmental stimuli (e.g., media nutrients, the presence of additional species) or incurred due to genetic makeup (e.g., gene mutations). Increasingly prevalent, label-free spectral counting relatively quantifies by the number of tandem mass spectra from peptides corresponding to a particular protein. Although this approach is merited the most straightforward, several issues hinder the interpretation of accuracy in the measurements. Importance exists in gaining a fundamental understanding of the interplay of proteome coverage and spectral counting as a function of experimental parameters. Here, we comprehensively evaluate proteome coverage and corresponding spectral counts in a model system while controlling instrument parameters. Whereas we can assess the lower limit, one spectral count divided by the number of replicates, the upper limit of spectral counts for a particular sample is currently undefined. Monitoring and systematically altering mass spectrometric parameters, a bioinformatics platform incorporating MASCOT and ProteoIQ affords protein identification and comparative and statistical analysis generating spectral counting data. Initial studies probing the intracellular proteome of two thermophilic bacterial microorganisms in mono and co-culture afford a basis of evaluation of spectral counts and normalization for comparison of protein expression as a function of environmental stimuli. This presentation will discuss the fundamentals of spectral counting affording accurate protein expression assessment over a larger dynamic range than is currently observed.

(211) **Surface-Enhanced Raman Scattering for the Detection of Lipid Mediators Secreted by Mast Cells during Allergic Response**

Audrey Guerard[1], Kyle Bantz[1], Christy Haynes[1]; [1]University of Minnesota

Despite the prevalence of allergies and asthma, the cellular communication mechanism that gives rise to allergic response is poorly understood. During allergic response, immune system cells release chemical mediators diverse in both structure and function, including bioactive lipids such as prostaglandins, leukotrienes, arachidonic acid, and platelet-activating factor. Detailed examination of the dynamic roles played by vasoactive and inflammatory lipid mediators in the production of allergic response could yield insight into the causes of allergies, but traditional methods of lipid analysis require lengthy separation times. Surface-enhanced Raman spectroscopy (SERS), however, facilitates rapid measurement of unique spectra for many molecules, including those with structural similarities. This method can be used to detect the presence of a variety of mediators secreted from immune system cells with minimal sample processing. In this work, cell-secreted lipid mediators, including leukotrienes C4, D4, and E4 (slow reactive substance of anaphylaxis), prostaglandin D2, and platelet activating factor, were detected from immortal mast cell lines using SERS. A self-assembled alkanethiol partition layer on a roughened silver surface has been employed to promote the requisite interaction of lipid analytes with the SERS-active substrate and facilitate detection. To eliminate interference from simultaneously secreted protein mediators, lipid extraction procedures have been explored and optimized, and chemometric techniques have been employed to discriminate the identities of multiple mediators secreted during the 24 hours following induction of allergic response. While detection in complex biological mixtures has unique challenges, preliminary results show promise for the application of SERS to the detection of lipids with roles in cell-to-cell communication processes.

(212) **Detection of Fungal Exudates Using Synchrotron FTIR**

Kathleen Gough[1], Merrill Isenor[1], Susan Kaminskyj[2], Carol Hirschmugl[3], Michael Nasse[3], Rusty Rodriguez[4], Regina Redman[4]; [1]University of Manitoba; [2]University of Saskatchewan; [3]Synchrotron Radiation Center; [4]University of Washington

Fungi play a variety of valuable roles in the environment: saprotrophs decompose dead plants and animals, endophytes reside within plants and can confer stress tolerance via habitat-adapted symbiosis (1), and mycorrhizal associations with plant roots help plants acquire nutrients from soils. However, certain fungi can cause diseases in plants and animals. Fungal plant pathogens are a serious threat to human food supplies and fungal infections in humans are difficult to treat. A greater understanding of fungal growth and cell composition and how each is related to lifestyle is necessary to control fungal activities. Fungi grow by extending long, tubular hyphae; growth occurs only at hyphal tips. Hyphae are typically 2-10 μm in diameter but can be hundreds of microns in length. Cell composition can vary dramatically over just a few microns. During growth, hyphae exude enzymes as a method of nutrient acquisition. Synchrotron Fourier transform infrared (sFTIR) spectromicroscopy is well suited to the analysis of fungal hyphae (2). Cellular components are probed at high spatial resolution and spectral information can be compared to data from other techniques (e.g. optical microscopy, SEM, AFM, fluorescent

staining). The IRENI beamline at the Synchrotron Radiation Center provides 0.54 µm pixel resolution FTIR images, offering even greater detail about cell composition. For the first time, we have been able to detect fungal exudates adjacent to Aspergillus nidulans hyphae on the surface of substrates. FTIR can be used to examine conditions for fungal exudation and potentially identify secreted compounds. This discovery offers key information about fungal interactions with their surroundings and could lead to a better understanding of their responses to different conditions and their roles in the environment. (1) Rodriguez RJ, Henson J, Van Volkenburgh E, Hoy M, Wright L, Beckwith F, Kim Y-O, Redman RS. (2008) The ISME Journal 2: 404-416. (2) Szeghalmi A, Kaminskyj S, Gough KM. (2007) Analytical and Bioanalytical Chemistry 287: 1779-1789; Jilkine K, Gough KM, Julian R, Kaminskyj SGW. (2008) Journal of Inorganic Biochemistry 102: 540-546.

(213) **Determination of Melamine and Cyanuric Acid in Contaminated Pet Food and Milk Products Using Surface-Enhanced Raman Scattering**

Ngee-Sing Chong[1], Sunil Kumar Setti[1], Beng Guat Ooi[1]; [1]Middle Tennessee State University

Melamine and cyanuric acid were found in contaminated pet food which was linked to the death of many cats and dogs in the U. S. in 2007. Later in 2008, these contaminants were found in milk powder in China and were blamed for the death of babies who consumed the tainted baby formula. Subsequently, techniques based on the liquid chromatography-mass spectrometry and gas chromatography-mass spectrometry have been developed for screening contaminated food. The goal of this project is to develop an alternative method based on Raman spectroscopy for detecting these contaminants. A simple and rapid method for preparing stable silver colloids for surface-enhanced Raman scattering (SERS) analysis of melamine and cyanuric acid was developed. The silver nanoparticles were produced by the reduction of silver nitrate solutions with hydroxylamine hydrochloride at alkaline pH and at room temperature. SERS spectra of melamine, cyanuric acid, and melamine-cyanuric complex are significantly different from their respective Raman spectra in terms of changes in the intensities, positions, and shapes of spectral peaks. Based on the SERS spectra of melamine and cyanuric acid at 10 ppm and 100 ppm, enhancement factors of 1.62 x 10^5 and 1.18 x 10^4 are obtained respectively. The signal intensity of melamine and cyanuric acid increases as a function of time elapsed after mixing the silver colloid and analytes. This indicates that the adsorption kinetics of both compounds is important in determining the SERS signal intensities. Melamine and cyanuric acid were spiked into wheat gluten and rice protein concentrate in order to simulate the sample matrix of pet food products. The detection of spiked melamine in these food matrices was possible by using the solid phase extraction cartridges for extracting and pre-concentrating the target compounds prior to SERS analysis. The detection of melamine contamination in the milk was more readily accomplished than the corresponding detection in the complex matrices of pet food. It was found that the Raman technique was applicable to both fat-free milk and whole milk. The preliminary results indicate that Raman spectroscopy is useful for detecting pet food contamination.

(214) **Quantitative Measurements of Biomass-Derived and Other Oxygenate Additives in Gasoline and Diesel Fuels by Infrared Spectroscopy**

Beng Guat Ooi[1], Joe Boachie[1], Ngee-Sing Chong[1]; [1]Middle Tennessee State University

Fuel additives in gasoline or diesel are commonly measured with gas chromatography, which is very reliable but requires frequent instrument calibration and relatively long analysis time. In order to avoid these disadvantages, a rapid and yet accurate method of quantifying oxygenate additives in fuels is developed using infrared spectroscopy. Oxygenate additives analyzed include ethanol, n-butanol, methyl tertiary-butyl ether, ethyl tertiary-butyl ether, 2-methyltetrahydrofuran, diglyme, triglyme, and triacetin. This method will be useful for monitoring the level of the oxygenate additives during fuel blending and verifying the fuel quality at various distribution points. Both Exxon 87 gasoline and Shell D-2 Ultra Low Sulfur diesel were used as base fuels for blending the additives at various levels up to 29.7 % by weight of the oxygen content. Distinctive peaks of all these additives were found in the fingerprinting region of the spectrum. For instance, peaks of ethanol were observed at 880.5 cm-1, 1046.7 cm-1, and 1086.8 cm-1 in concentrations as low as 0.5 % (v/v). The interaction between the additives and the base fuels were considered in the quantitative analysis. Different spectral bands of each fuel additives were ranked according to their suitability for quantitative calibration. The dependence of analytical accuracy and sensitivity on spectral resolution and data acquisition times was studied.

(215) **Confocal Raman Imaging – A New Method for Drug Characterization and Design**

Jiangyoung Yang[1], Ute Schmidt[1], Andrea Jauss[1], Thomas Dieing[1], Fernando Vargas[1], Olaf Hollricher[1]; [1]WITec GmbH

New medical devices, drug-delivery solutions and biomedical applications using state-of-the-art formulations and technologies are constantly being developed. These developments require the most detailed information possible concerning the structure and composition of a device surface to better understand and predict how the device will interact with the human body. As these surfaces often consist of thin, transparent coatings, surface characterization tools which reveal what remains hidden in optical images are required. Confocal Raman microscopy combines high-resolution microscopy with the chemical sensitivity of Raman spectroscopy, thus allowing non-destructive imaging of chemical properties without specialized sample preparation. Due to the confocal principle, depth information regarding the coatings can be easily obtained. Not only can thickness and uniformity measurements be performed, but the degree of mixing or segregation of the ingredients within the coatings can also be determined. In addition to the distribution of various chemical species, the presence of drug polymorphs can be identified using Raman spectroscopy. Even though the Raman spectra of amorphous and crystalline drugs differ only slightly, they can be clearly distinguished with an extremely sensitive confocal Raman setup. Along with drugs for e.g. oral applications, drug release functions must also be developed for lotions applied as thin films directly on the human skin. In this case not only the chemical composition, but also the size and distribution of the drug are of tremendous importance because it must be introduced into the human body through the pores of the skin. Well suited to these investigations is confocal Raman microscopy which can be used to acquire image stacks, leading to a full three dimensional characterization of defined sample volumes. Confocal Raman microscopy can additionally be used for-label free detection of subcellular organells based on their biochemical composition. This opens the door for noninvasive, *in-vitro* studies of cell biological aspects such as dynamics of mitochondrial movement, drug uptake and many more.

(216) **Surface Plasmon Resonance (SPR) Biosensing within Electrokinetic Channels**

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Two novel eletrokinetic (EK) methods Gradient Insulator Dielectrophoresis (GIDEP) and Electrophoretic Capture (EPC) integrated with Surface Plasmon Resonance (SPR) serve as the

isolation, concentration and detection platform for the lab-on-a-chip device. The capabilities of GIDEP and EPC to separate and concentrate target proteins are exploited and characterized. SPR sensing component is designed and optimized for application in biological fluids. Small volume channels are etched into a dielectric substrate where EK methods are employed to condition the sample for SPR quantitative detection. We implement a novel strategy for functionalizing the SPR sensing pads that relies on electrografting of diazonium salts, which enables in-channel referencing. Detection is accomplished by antibodies capture on a traditional Kretschmann configuration SPR spectrometer. We are able to detect protein at physiological levels of a small liquid volume. This sensor will be applied for rapid quantitation of protein biomarkers in whole blood, to diagnose myocardial infarction and stroke.

(217) **Conservation of Historical Pigments on Sultan Baybas Qur'an and a 14th century Mamluk Qur'an by Raman Spectroscopy**

Enrique Lozano Diz[1], Colin Baker[2], Paul Garside[2], David Jacobs[2], Barr Knight[2], Dean Brown[1]; [1]PerkinELmer; [2]The British Library

Purpose of research : To create and publish a data base of the pigments used to illuminate the British Library's Sultan Babays Qur'an and a contemporary 14th C Mamluk Qur'an using Raman spectroscopy to establish the palette for historical research, conservation and preservation and exhibition purposes. Project and collaboration with PerkinElmer This project is a development of earlier research carried out to create a database of the Raman analysis of pigments and inks used on selected items in the British Library Collections. This research has demonstrated how the Raman spectra of media on manuscripts can be obtained in a non-invasive way to determine the composition of inks and pigments. The British Library in collaboration with PerkinElmer has built a large corpus of spectroscopic data from samples and collection items. Applications of Raman in Book and Paper Conservation The identification, analysis and interpretation of materials used in the manufacture of such artifacts can give us an insight into the development and spread of artistic styles, materials technologies, social changes and even the advances in global commerce and trade. The study of pigments and inks is a subject at the interface of art and science, and which attracts the interest of curators, conservators, art historians, and research and museum scientists. This involves the identification of the original materials and possible degradation products to assist in the determination of related conservation problems. Through laboratory simulations and further analysis the potential exists to reverse some degradation processes and to improve the long term stability the material. Where this is not possible accurate identification of the objects chemical structure makes a fully referenced and researched 'digital virtual restoration' a possibility. The information gathered helps to define the historical perspective of the object by providing new information and helping in the future conservation strategies of the items, or confirming or disproving long held suppositions about the materials used in the manufacture of an artifact. While some of this information may be surmised from other sources, evidence from Raman and similar analytical methods can provide confirmation or contradict what was previously believed.

(218) **Utility of Raman Spectroscopy in a Materials Science Environment**

Shawn Mehrens[1], Slobodan Sasic[1], Linda Lohr[1]; [1]Pfizer Global R&D

In a pharmaceutical based materials science group, one of the major remits is characterization of the solid forms being developed, and assessing the optimal methods to use for each compound. Typical questions in this environment may include determining if a salt has been formed, whether a compound is crystalline, methods to identify differences between polymorphs, assessing the utility of tools for crystallization monitoring, or even characterizing small amounts of material from screening experiments. Raman spectroscopy may provide answers to many of the aforementioned questions, while using small amounts of material, providing fast characterization times and being virtually indestructive, so the sample is available for further analysis by other techniques. Given the range of sampling configurations available in Raman spectroscopy, this technique has found significant use within our group for the analysis of compounds in every stage of development, from discovery research up through registration, and even post market launch. Several examples of the flexibility and utility of Raman spectroscopy will be covered in this discussion. Raman microscopy has been used to determine the form of API (active pharmaceutical ingredient) in multiple non-traditional formulations, where previously only PXRD had been used to identify the API form. The PXRD method in these cases required extensive sample preparation, as well as long acquisition times. In contrast, the Raman microscopic method allowed direct analysis of the formulations, and fast determination of the polymorphic form present. In another case, the formation of any contaminant forms during stability of IR tablet formulations was monitored with Raman mapping. FT-Raman was also assessed as a bulk technique to determine qualitatively what levels of these forms could be seen. Raman mapping was used successfully to ensure that low levels (<5%) of contaminant polymorphic forms were not present in the stability samples. Finally, in conjuction with PXRD, the power of *in-situ* monitoring has also been explored by examining the kinetics of hydrate formation of an exploratory compound. It was found that the kinetics of slurry hydrate formation can be altered by the presence of excipients or by differences in concentration, and these changes were easily monitored by *in-situ* Raman spectroscopy.

(219) **Measurement of Trace Atmospheric Pollutants by Broadband Cavity-Enhanced Absorption Spectrometry with an FT-IR Spectrometer**

Ben Perston[2], Cathryn Langley[1], Gus Hancock[1], Wolfgang Denzer[3]; [1]University of Oxford; [2]PerkinElmer; [3]Oxford Medical Diagnostics

Cavity-enhanced spectroscopy is now widespread in the research environment, and several commercial instruments are available. Most work to date has focused on cavity ring-down spectroscopy, requiring a tuneable laser source and fast electronics. Cavity-enhanced absorption spectroscopy uses a continuous source and measurement of the transmitted intensity rather than a decay time. This technique allows the use of a comparatively broadband source for improved detection of species in mixtures with broad absorption features. In this submission, we describe a practical and relatively inexpensive cavity-enhanced absorption set-up using a near-infrared (NIR) superluminescent LED (SLED) source, a 25 cm cavity (with mirrors having $R \approx 0.9998$), and a commercial FTIR spectrometer. With this system, a minimum detectable absorbance approaching 10^{-7} is approachable within a measurement time of only a few minutes. We demonstrate this capability via the sub-ppm detection of the common atmospheric pollutant butadiene.

(220) **Direct Detection of Components and Nanoparticles in Smoke by Time-Of-Flight Mass Spectrometry**

Yoko Nunome[1, 2], Kenji Kodama[2], Kozo Matsumoto[2], Hyunkook Park[3], Sang Chun Lee[4], Kuniyuki Kitagawa[2]; [1]Graduate School of Engineering, Nagoya Univ.; [2]EcoTopia Science Institute, Nagoya Univ.; [3]Korea Maritime Univ.; [4]Department of Chemistry, Kyungnam Univ.

Recently, particulate matter (PM) in exhaust gas from diesel engines have been reduced by the development of particulate filter (DPF) technologies while nano-sized particles (particle diameter:

D_p < 50 nm) passed through filters increase relatively. In this regard, nanoparticles in the atmosphere have received much attention as a target on human health effects. Inhaled nanoparticles of nanometer in size easily deposit on alveolar area of the deep lung. The compositions in nanoparticles and their chemical characterizations should be clarified. The aim of this study is to detect combustion-generated particles by time-of-flight (TOF) mass spectrometry (MS) which allows analysis of nanoparticles in a high mass range. On-line direct analysis of nanoparticles was performed on a TOF-MS (Micromass, Manchester, UK) equipped with a newly developed interface for atmospheric pressure chemical ionization (APCI) in the positive-ion mode. The particles in smoke were collected from a burning mosquito-coil in a desiccator, and directly introduced into the APCI chamber through the interface. The chamber was kept under a slightly negative pressure to draw the gaseous sample which was humidified using a thermostatic bath system at 80°C. The mass spectrum of coil smoke has major peaks at *m/z* 303 and 332 that are assigned to synthetic pyrethroid, *d*-allethrin and *d*-tetramethrin, respectively. The major components are popular ingredients used in formulating conventional mosquito-coils. Ions can be detected up to 1204 Da which corresponds to particles of 1.2 nm in diameter on the basis of graphite density 2.2 g cm-3. The important feature is that specific 74 peak intervals are observed in a high-mass range of *m/z* 535–1204. We assume that the molecular weight of 74 is assigned to triacetylene (1,3,5-hexatriyne) [1] produced during combustion. Furthermore, we are going to apply a soft ionization plasma (SPI) method using glow discharge to detect higher mass of nanoparticles. [1] A.L. Lafleur, J.J. Gagel, J.P. Logwell, P.A. Monchamp, Identification of aromatic alkynes and acyclic polyunsasturated hydrocarbons in the output of a jet-stirred combustor, Energy Fuels, 5 (1988) 709-716.

(221) **Time Resolved Fourier Transform Infrared (TR-FTIR) Studies Employing Micro Fluidic Mixers**

Christoph Wagner[1], Martin Kraft[2], Michiel Vellekoop[1], Bernhard Lendl[1]; [1]Vienna University of Technology; [2]Carinthian Tech Research AG

Nowadays FTIR spectroscopy is a widely used analytical technique, which provides structural information of the target molecule directly in a non-destructive manner. Classical FTIR measurements cannot provide kinetic information on fast chemical reactions. To obtain this kind of information rapid scanning and step-scan techniques can be employed. Both techniques require triggering of the reaction very precisely and the classical step scan experiment can only be applied for cyclic reactions. A promising approach to overcome the triggering limitation is to induce the (bio)chemical reaction by mixing two liquids by diffusion, in a mixing channel that also serves as the measurement area. The actual measurements take place at well defined spots along this channel, corresponding to specific reaction times: moving the measurement spot towards the entry yields shorter reaction times, moving it towards the channel's end gives longer reaction times. A further way to improve TR-FTIR measurements employing micro mixer technology is to combine them with step scanning technology. In doing so the high time resolution of the step-scan technique can also be obtained for non-cyclic reactions. Firstly one reactant needs to be modified to be releasable by a light trigger. Then the two reactants are rapidly mixed and a laser pulse triggers the reaction while the liquids are still flowing through the mixer. Before the next triggering of the reaction occurs fresh samples are pumped into the measurement spot. This ensures the excitation of fresh sample molecules for every step of the step-scan measurement. Our new micro mixer features a four fluid layer design for fast mixing and a total path length of 8µm for measurements in water. In this work we examine the mixing performance with a fast acid base reaction, resulting in a mixing time of ~7 ms, and then take a deeper look into the formalin-sulfite clock reaction with the classical mixing experiment. Additionally we show kinetic data of the photo dissociation reaction of carboxy-myoglobin recorded in the step-scanning mode in a micro mixer. This analysis revealed second order kinetics at the beginning and pseudo first order kinetics for t > 1 ms for the CO rebinding.

(222) **Molecular Spectroscopy Diagnostic: Aluminum Monoxide**

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Diatomic molecular diagnostic is based on use of accurate line strength files for selected electronic transitions of AlO. Measured progressions and sequences of the AlO B to X system are compared with synthetic spectra to infer temperature in an aluminum-particle laden flame. Details of the computation of the emission spectra are presented, including methods of data reduction that include use of modified Boltzmann plot and/or use of Nelder-Mead algorithm. Experimental spectra indicate temperatures in the order of 3000K. In addition, we discuss time-resolved measurements following laser ablation of alumina.

(223) **Analysis of Lanthanide Elements in Molten LiCl-KCl Eutectic Salt Using Laser-Induced Breakdown Spectroscopy**

Dong Hyoung Lee[1], Bong Young Kim[1], Tae Hyeong Kim[1], Jong-Il Yun[1]; [1]KAIST

The spatial distributions of lanthanide elements and molybdenum in molten LiCl-KCl eutectic salts are investigated by laser-induced breakdown spectroscopy (LIBS). Knowledge of electrochemical behaviors of rare earth elements in molten salts is essential for a pyrochemical process of spent nuclear fuel. The LIBS technique provides a simultaneous multi-elemental and space-resolved analysis on the micrometer scale. Laser-induced plasma is created by focusing a Nd:YAG laser pulse (532 nm) onto the surface of samples, and the plasma emission is detected by an echelle spectrometer. A mixture of LnCl3 (Ln: Nd, Sm, Eu, Gd, Tb, and Dy; 99.99 % purity) is dissolved in the molten LiCl-KCl eutectic salt at 773 K, and then samples are cooled down to room temperature in a glove box under high purity argon gas atmosphere (99.999 %). During cyclic voltammetry experiments, the transparent LiCl-KCl (99.99 %, 44 wt.% LiCl) eutectic salt is being contaminated by corrosion of a Mo wire (99.99 %) served as counter electrode. The emission intensity ratio of Mo(I)386.410 nm/K(I)404.721 nm in the lower part of the sample is almost 11 times higher than near wall and in the interior of the sample. This observation reveals that Mo may exist as metallic form and thus a precipitation occurs. Moreover, the spatial distributions of lanthanide elements (0.5 wt.% for each element) in the LiCl-KCl eutectic salt are investigated with their own strong ionic and neutral emission lines. The emission intensities of Sm show almost 5 times differences depending on the sample positions, while the emission intensities of other lanthanide elements in the bottom position are at least 1.5 times stronger than in the upper position. Results may suppose cautiously that such a dissimilar spatial distribution of LnCl3 is ascribed to their different solubilities in the molten LiCl-KCl salt. In addition, a calibration is made for determining the detection limits for LnCl3 in the molten LiCl-KCl eutectic salt.

(224) **Mid-IR spectrOscopy as a Quality Control Tool for Traditional and Herbal Medicines**

Ben Perston[1], Patrick Courtney[1], Chris Lynch[1]; [1]PerkinElmer

The manufacture of traditional and herbal medicines around the world is becoming increasingly strictly regulated, and in many regions manufacturers are now required to demonstrate GMP compliance to the same level as the pharmaceutical industry. Herbal preparations pose different challenges for analysis than

conventional pharmaceutical formulations, and there is much interest in establishing the most useful and cost-effective analytical techniques for identification tests of raw materials and extracts, intermediate analysis between processing steps, and final product quality control. Infrared spectroscopy is widely used in pharmaceutical manufacture for all of these tasks. We discuss its applicability to the manufacture of herbal medicines, and show that it is suitable for distinguishing Coptis chinensis (Chinese Goldthread) from common adulterants and for validating the composition of the common remedy Red Flower Oil, a blend of essential oils.

(225) **Fluorescence Guided Ingredient Specific Particle Sizing of Nasal Suspension Formulations**

Ryan Priore[1], Oksana Olkhovyk[1], Oksana Klueva[1], Michael Fuhrman[1]; [1]ChemImage

Accurate size determination of an individual constituent in a complex formulation has historically been a challenge. Nasal suspensions are typically comprised of multiple excipient materials as well as one to two APIs. Accurate knowledge of the API particle size is critical for determining the ultimate dissolution rate in the mucous membrane of the nasal cavity as well as establishing bioequivalence or sameness between a generic and innovator product. A validated method does not exist for characterizing ingredient-specific drug particle size in nasal aerosols and sprays due to the presence of insoluble suspending agents along with suspended active pharmaceutical ingredient (API) in the formulation. Current methods used for such measurements include Anderson cascade impaction followed by HPLC, laser light scattering and optical microscopy; however, each method either lacks the ability to perform ingredient-specific particle sizing (ISPS) or is error prone. Optical microscopy in particular relies on the experience of an analyst to recognize particles of a specific ingredient. Chemical imaging yields spatially accurate spectroscopic information and is well suited for ISPS of a complicated mixture. Two current method development areas included sample preparation and representative sampling, and an opportunity exists for pre-screening a prepared sample for determining the optimal regions for chemical imaging data collection. This presentation will compare ISPS of a nasal spray suspension in both unguided and Fluorescence guided sampling configurations using Raman wide-field chemical imaging. The total number of API particles, particle size and particle size distribution of free API particles as well as API/excipient agglomerations will be reported for both sampling configurations.

(226) **Development of a Fieldable Sensing System for Rapid Pesticide Exposure Analysis**

Kevin Spencer[1], Susan Clauson[1], Sarah Spencer[1], Jim Sylvia[1], Quirina Vallejos[2], Sara Quandt[2], Thomas Arcury[2]; [1]EIC Laboratories, Inc; [2]Wake Forest University School of Medicine

Despite the recent interest in organically grown foods, most agricultural crops optimize yield through use of multiple pesticides. The health of the active applicator and the passive neighbor may be affected by the spraying. In between these extremes are the farm workers who pick the crops anywhere from days to weeks after application. How significant is the pesticide residue these workers are exposed to during a workday and how much is transferred back to the residence? Despite their low vapor pressures, what is the true pesticide concentration surrounding a person when pesticides adsorbed to particulate matter are included? What is the relationship between the concentration around an individual and the amount adsorbed/ingested? To find a statistically significant answer to these questions in actual field conditions, a portable, fast, inexpensive measurement device is required. We demonstrate herein the capability of Surface-Enhanced Raman Spectroscopy (SERS) to detect >100 organophosphate, organochlorine and carbamate-based pesticides in the vapor phase as well as the ability of SERS sensors to detect a particular analyte in a synthetic urine matrix. We present SERS analysis of CDC quantified urine samples, and present results of real-time SERS field dosimetry and real-time vapor sampling of farm workers barracks. The issue of potential interferences will also be discussed.

(227) **Biofuels: Properties and Contaminants by FT-IR Analysis**

Ben Perston[1], Aniruddha Pisal[1], Dean Brown[1]; [1]PerkinElmer

Biofuels – principally biodiesel and bioethanol – have become increasingly important as renewable fuels with potentially lower carbon footprint than fossil fuels. With numerous national and international standard specifications for fuel quality, and the potential for variability between biofuels produced by various methods from numerous feedstocks, fast and reliable analysis of biofuels is critical. FT-IR spectrometry is particularly well suited to measurement of several important parameters, and in this submission we describe several of these analyses. Determination of impurities at sub-percent levels is demonstrated: methanol, mono-, di-, and triglycerides in biodiesel; and methanol, water, and hydrocarbon denaturant in ethanol. Discrimination between biodiesel samples from various feedstocks such as palm, soy and rapeseed is also shown, as is measurement of biodiesel in diesel at levels from 0.003 to 100 %v/v.

(228) **Explosives Detection in the Presence of Real-World Interferences Using Surface-Enhance Raman Spectroscopy**

Kevin Spencer[1], Sarah Spencer[1], Susan Clauson[1], James Sylvia[1]; [1]EIC Laboratories, Inc

Many techniques have been developed in the last decade for the detection of TNT-based explosives; most work very well under controlled conditions. EIC Laboratories demonstrated the ability of Surface-Enhanced Raman Spectroscopy (SERS) detect TNT-based landmines. Fewer techniques have been developed for detection of RDX, TATP, ammonium nitrate and urea nitrate. The ability of SERS to detect these explosives will be demonstrated in this presentation. We will discuss development of SERS sensors that optimized detection of the these explosives. The ability of SERS to differentiate the explosive salts from other common salts will be shown. We will demonstrate SERS detection of explosives/explosive impurities in the presence of sweat, common fragrances, diesel and commercial exhaust and in the presence of gunpowder residues. A vapor sampling accessory and portable instrumentation for collection of data will be discussed.

(229) **Realistic Resolution Targets for Chemical Imaging of Pharmaceuticals: PEG-embedded Polydimethylsiloxane Devices**

Laura C. Mecker[1], John F. Kauffman[1]; [1]Food and Drug Administration

Metal-on-glass resolution targets may not adequately represent the practical resolution that can be achieved with Raman chemical imaging instruments when imaging pharmaceutical materials that are non-opaque to the excitation source. Subsurface scattering can influence both lateral and depth resolution, and the dependence of resolution on the depth of the signal source is poorly characterized. In previously published work, we prepared resolution targets composed of 10 micron thick polyethylene glycol (PEG) lines on top of a silicon substrate. These targets indicated that the thickness of the PEG lines and scattering within the PEG lines cause some deterioration of resolution, but these targets did not probe the influence of lateral photon diffusion from the PEG lines into nearby material, because the PEG lines were not embedded in a non-opaque substrate. We have now used microfabrication methods to create resolution targets composed of ~10 micron thick PEG lines

embedded within the surface of a ~5 millimeter thick base layer of non-opaque, Raman-active, polydimethylsiloxane (PDMS). A ~20 micron thick PDMS overlayer was sealed over the PEG lines to make the device more robust, and to examine the influence of an overlayer of Raman-active material on chemical imaging resolution. Because PEG and PDMS have similar Raman spectra, chemometric image resolution methods were required to extract chemical images of these two materials. As a result, these devices provide more realistic resolution targets, because both contrast materials are organic and Raman active. Additionally, aluminum oxide scattering agent was introduced to the PDMS base layer or the PDMS overlayer or into both layers at varying concentrations to determine the dependence of subsurface scattering, and subsequently the deterioration in spatial resolution, on the concentration and location of the scattering medium. We will describe the device construction process in detail, and present our results on the influence of signal to noise ratio and the concentration of scattering material on spatial resolution.

(230) **Quantitative Proteomic Analysis of Human Embryonic Stem Cells During Early Stage Differentiation Utilizing SILAC Labeling and High Resolution Mass Spectrometry**

Timothy Collier[1], Prasenjit Sarkar[1], Balaji Rao[1], David Muddiman[1]; [1]North Carolina State University

Human embryonic stem cells (hESCs) are self-renewing cells that can differentiate to become most cell types. This makes hESCs a valuable tool for investigations into developmental biology, pharmaceutical safety, and regenerative medicine. Exploring the hESC proteome and how it changes qualitatively and quantitatively is integral to understanding their overall biology. Herein, we describe the quantitative proteomic analysis of hESCs during differentiation using stable isotope labeling with amino acids in cell culture (SILAC) in conjunction with multiple stages of fractionation. These fractionation methodologies include subcellular fractionation of nuclear, cytosolic, and membrane cellular components, in-solution isoelectric focusing of peptides resulting from tryptic digestion, and one-dimensional gel electrophoresis. Samples arising from any of these fractionation techniques were further separated using reversed-phase nanoflow chromatography detected with a hybrid LTQ-FT-ICR mass spectrometer equipped with a 7T superconducting magnet. The resulting high mass accuracy (MMA < 5 ppm) mass spectral data was searched against the human NCBI proteome database using both MASCOT and SEQUEST search algorithms. The result of data analysis has yielded quantitative information of hundreds of proteins during differentiation from pluripotent stem cells to early stages of endoderm, mesoderm, neuroectoderm and trophectoderm tissue development. We not only quantified relative protein expression between cell types, but also with temporal resolution along three time points at 0, 2 and 4 days post treatment with molecular triggers. These triggers include ligands such as ActivinA, BMP4, Wnt1, FGF2 and inhibitors such as SB431542, Dorsomorphin, Noggin, LY249002, Dikkopf and BIO. Through the comprehensive analysis of these results we seek to describe the dynamics of protein expression in signaling pathways that regulate hESC differentiation.

(231) **Advancements in Low-Frequency Raman Spectroscopy Using Ultra-High Performance Holographic Notch Filters**

Frank Havermeyer[1], Christophe Moser[1]; [1]Ondax, Inc.

Ultra-narrow-band volume holographic notch filters have been shown to enable observation of both Stokes and anti-Stokes shifts down to 10 cm-1. We report performance advancements to previous work and demonstrate a compact spectrometer configuration that provides a low-cost alternative to expensive triple spectrometer Raman systems. Extensions in center wavelength, optical density, bandwidth and transmission efficiency of the filter elements, along with additional wavelength control and filtering components in the system, enable observation of high-resolution, low-frequency spectra. Both Stokes and anti-Stokes spectra of sulfur, L-cystine, and common pharmaceuticals are presented, showing clear low-frequency peaks to within 15 cm-1 of the excitation wavelength, allowing researchers to cost-effectively access differentiating spectral information.

(232) **Reduction of Spectral Interferences in Inductively Coupled Plasma Mass Spectrometry Using a Universal Cell Technology**

Hamid Badiei[1], Kaveh Kahen[1]; [1]PerkinElmerSCIEX

Resolving spectral interferences in inductively coupled plasma mass spectrometry (ICP-MS) has always been a challenging task. Over the past few years, reaction and collision cell-based instruments have gained widespread popularity for their capability in removing or reducing such interferences. Unlike the reaction cell approach, where gas-phase reactions in the cell followed by band-pass tuning play the main role in eliminating polyatomic interferences, collision cells rely on a higher probability of collisions of an inert gas (e.g., He) with the polyatomic interferences based on their relatively larger collisional cross-section. Polyatomic species will therefore lose more energy to the extent that they cannot overcome the positive energy barrier established between the mass analyzer and the cell. Though the detection limits are typically inferior as compared to those obtained by reaction cells, the main benefit of collision cells is that a single set of operating conditions would remove the majority of interferences without the need for prior knowledge of the sample matrix. This approach has proved especially appealing to applications where moderate detection limits are required for multielement analysis in complex matrices. The novel Universal Cell Technology (UCT™) implemented in NexION™ 300 ICP-MS from PerkinElmer can operate in three distinct modes: 1) Standard mode when the cell is vented and acts as an RF-only ion guide; 2) Kinetic Energy Discrimination (KED) mode with a collision gas such as He; and 3) Dynamic Reaction Cell (DRC™) mode with reactive gases such as NH3, CH4, and O2. This presentation discusses the design details of the UCT and contrasts the performance of the UCT in the DRC and KED modes for interference removal.

(233) **Detection of Drugs and Explosives in Large Volume Headspace Using Planar Solid Phase Microextraction and Ion Mobility Spectrometry**

Jose Almirall[1], Wen Fan[1], Mimy Young[1]; [1]Florida International University

Cargo container screening requires a sensitive, high efficiency and high throughput technique in order to detect hidden explosives and controlled substances. A novel planar solid phase microextraction (PSPME) device has been used to extract trace amount of volatile compounds from small amounts (200 mg) of explosives hidden in a 5'x5' LD3 cargo containers. These planar SPME devices offer higher surface area, capacity, and extraction efficiency compared to conventional fiber-SPME devices. PSPME was used as an extraction and preconcentration device for the headspace analysis of volatile signature compounds found in smokeless powders, a common low explosive used for improvised explosive devices. An ion mobility spectrometer (IMS) was used for detection of these volatile chemical compounds offering high sensitivity and fast analysis time. Different smokeless powders were used and the volatile compounds 2,4-dinitrotoluene (2,4-DNT), trinitrotoluene (TNT), nitroglycerin (NG) and diphenylamine (DPA) were successfully detected by IMS with static extraction times of ~2 hours and with ~5 minutes of dynamic extraction. This PSPME

device is readily adapted for use in the ~15,000 IMS instruments currently deployed at security checkpoints throughout the US without further modification.

(234) **Development of Odor Mimics for Improved Detection of Forensic Specimens by Canines and Instruments**

Kenneth Furton[1], Katylynn Beltz[1], Norma Caraballo[1], Lauryn DeGreeff[1], DeEtta Mills[1]; [1]Florida International University

This paper describes ongoing studies involving the identification and quantification of dominant odor signature chemicals that can be used by certified law enforcement detector dogs and instruments to reliably locate forensic specimens including accelerants, biotoxins, currency, drugs, explosives and humans (living and deceased). In the work presented, methods developed using Solid Phase Microextraction - Gas Chromatography / Mass Spectrometry (SPME-GC/MS) have identified the dominant odor chemicals available at room temperature as well as elevated temperatures. The results demonstrate that canines are generally not using the relatively low volatility parent substances but instead use characteristic volatile headspace components to accurately locate specimens. The application of these results to the optimal selection of canine training aids and the tuning of instruments for these compounds are discussed. Ongoing studies involve the identification and quantification of odorants used by certified police work dog teams and some instruments utilizing double blind field trials. Odorants have been identified from the most common drugs including cocaine, marijuana, heroin, methamphetamine and ecstasy. Odorants have also been identified from common explosives and it is shown that that the combination of as few as six odorant mimics can represent a comprehensive explosive odor kit for the majority of high explosives. The levels required for reliable detection by canines will be presented as well as ways to improve the sensitivity and reliability of detection. Studies on the identification of odorants for human remains will also be presented as well as recent studies evaluating the uniqueness and persistence of human scent as well as the optimal methods for the collection, preservation and presentation of human scent for canine and instrumental matching. A method of sampling human scent volatile organic compounds employing SPME-GC/MS has been developed which is demonstrated to be able to produce a distinguish ability of greater than 99% across a population. Direct contact of forensic specimens and non-contact sampling of the headspace of forensic specimens is compared and dynamic headspace samplers such as the Scent Transfer Unit (STU-100) are used to develop non-hazardous odor samples.

(235) **Characterization of Volatile Compound Evolution from Complex Synthetic and Natural Polymeric Materials: Methodologies, Statistical Data Analysis and Applications**

James Lewicki[1], Sarah Chinn[1], Christopher Harvey[1], Cynthia Alviso[1], John Liggat[2], Lorriane Gibson[2], Robert Maxwell[1]; [1]Lawrence Livermore Nat'l Laboratory; [2]University of Strathclyde

The evolution of volatile organic and inorganic compounds from a range of synthetic and natural polymeric materials can be informative as to the underlying aging, degradation chemistry and relative stability of such materials. Gaining an understanding of the nature of volatiles evolution and offgassing is extremely important for lifetime prediction and stability/compatibility assessment in synthetic engineering materials. Additionally, the characterization and quantification of volatile products of degradation is of great importance in the field of conservation and preservation of artifacts of historical and cultural significance. Although the study of VOC evolution potentially offers a non-destructive and rapid technique for the assessment of the stability and 'health' of complex polymeric systems, there are several technical challenges that have to be overcome if a robust and quantitative analytical methodology is to be developed. Notable difficulties in the application of this approach include: The inherent complexity of the 'spectrum' of volatile materials evolved from many typical real world systems - which often contain diverse mixtures of many individual polymers, compounding agents, impurities and additives; Secondary interactions between volatile products and the parent materials, leading to further complication of the observed volatiles spectrum; Identification and quantification key fingerprint compounds at low concentrations, from a complex mixture of (often unknown) chemical species. In addition to the issue of complexity, is the fact that changes in the chemistry of a multi-component system often manifest themselves as subtle alterations in a volatiles evolution spectrum and therefore require a significant analytical effort in order to detect, quantify and interpret correctly. By employing a range of analytical and chemometric techniques, including SPME headspace sampling coupled with GC-MS, cryogenic trapping/separation of volatiles and multivariate dataset analysis we are in the process of developing a comprehensive methodology for sampling, characterization, quantification and interpretation of a volatile product environment. By extracting significant information from a dataset which comprehensively describes the full spectrum of volatile species in a real-world environment, we aim to identify key physical and chemical changes in materials which will allow the stability and 'health' of a system to be tracked and assessed over extended time periods.

(236) **Metabolomic Profiling of Exhaled Breath to Differentiate Between Asthma and COPD**

Michael Schivo[2], Abhinav Bhushan[1], Weixiang Zhao[1], Nicholas J. Kenyon[2], Cristina E. Davis[1]; [1]UC Davis, Dept Mech & Aeronaut Engineering; [2]UC Davis, Dept Int Med, Div Pulm Med

Asthma and chronic obstructive pulmonary disease (COPD) are two airway disorders with a high prevalence, economic and social morbidity, and an increased mortality. A significant proportion of patients do not fit classic historical or spirometric definitions of asthma or COPD thereby eluding a confident diagnosis. This leads to difficulties determining optimal treatment strategies. Metabolomic profiling may provide an adjunct diagnostic strategy by identifying unique exhaled breath signatures in each group. This analysis may also elucidate specific therapeutic signal transduction pathway targets for future studies. We recruited 25 subjects each in asthma, COPD, and normal healthy control groups based on well-characterized clinical and spirometric criteria. Exhaled breath condensate (EBC) has been collected and we are analyzing the chemical content of the EBC using gas-chromatograph mass spectrometry (GC/MS) and gas-chromatograph differential mobility spectrometry (GC/DMS). Results will be processed using direct peak-by-peak comparison between groups, principle component analysis, and individual biomarker profiles. We anticipate that principle component analysis of the GC/MS and GC/DMS data will allow for accurate separation of the three groups. We also anticipate that biomarker signatures will emerge unique to each group from the raw data. Last, we predict that several plausible biomarker chemicals will be identified from the MS data for further study.

(237) **Clues Overhead: GC/MS Profile of VOCs Produced by Algae as Markers of Contaminated Water**

Andrew Callender[1]; [1]Tennessee Technological University

Multidimensional methods such as LC/MS remain the gold standard for detection and quantification of pollutants such as pharmaceuticals and their metabolites, or particulate species such as nanomaterials. However, these methods are seldom field-portable and throughput can be low. While identification of the specific pollutants may require lab-bound techniques, recognition of the local presence of pollutants may provide a useful indication

of where to sample. Naturally occurring algae are known to emit a broad range of volatile organic compounds. The mix of VOCs may change when the algae are stressed, such as by changes in water pH, salinity and temperature, or by the presence of specific pollutants. These changes may be useful as an indirect marker of water contamination. In this work we present the use of headspace GC/MS and multivariate analysis to characterize changes in the VOCs expressed by algae in response to several stressors.

(238) **Analysis of Microbial and Fungal Toxins in Airborne Grain Dust**

Mustafa Selim[1], S. L. Kinney[1], H. D. Patel[1]; [1]East Carolina University

Farmers and farm workers are commonly exposed to complex mixtures of hazardous chemicals including highly toxic and carcinogenic fungal metabolites (e.g. aflatoxin B1), particularly during bin cleanout and concentrated animal feeding operations (CAFOs). Therefore, we are interested in assessing the health risk to farmers and farm workers from exposure of AFB1 during farming activities in North Carolina. Our findings in Iowa show high prevalence of aflatoxin B1 (AFB1) in the respirable fraction of the grain dust particles (£ 10 mm) collected during bin cleanout. Particles < 1 mm in diameter had a higher concentration of AFB1 than particles with a diameter of ³ 7.4 mm. An estimated dose of 1.5 mg/hr, during bin cleanout, is approximately equal to the weekly dose reported in an epidemiological study of a Dutch peanut mill, where workers were found to experience excess cancer compared to the population as a whole. Insufficient data is currently available on AFB1 exposure levels in CAFOs in North Carolina. This presentation will report on our preliminary findings of the types of microbial volatile organic compounds (MVOCs) and fungal of toxins (including AFB1), their concentrations, and distribution in airborne dust samples from CAFOs in Eastern North Carolina. The results will be compared with our previous findings in Iowa to determine potential variability due to regional and weather conditions.

(242) **A Survey of Metal Contamination in Pharmaceuticals and Dietary Supplements**

John Kauffman[1], James Guthrie[2], J. David Rovertson [2, 3]; [1]FDA Division of Pharmaceutical Analysis; [2]University of Missouri Research Reactor; [3]University of Missouri Dept of Chemistry

The current pharmacopoeial method for determination of heavy metal contamination is a sulfide precipitation preceded by a digestion step, and the allowable limits for toxic metals in pharmaceutical materials can be ten or more parts per million. The US Pharmacopoeia (USP) is currently revising both the methods and the limits for heavy metals, metalloids and residual catalysts, and the International Conference on Harmonization of Technical Requirements for Human Drugs (ICH) has initiated a working group to harmonize the limits across the three ICH regions (Europe, Japan and US). These activities are expected to result in revised limits on metals in pharmaceuticals which could be significantly lower than the current limits. Data on the prevalence of metal contamination in pharmaceutical materials is sparse, and as a result there is uncertainty surrounding the impact of the anticipated new limits on pharmaceutical manufacturers and on the availability of drugs. The FDA Division of Pharmaceutical Analysis in collaboration with the University of Missouri has surveyed the metal content of pharmaceutical products including over the counter products and prescription medicines, generic drugs and innovator products, vitamins and other dietary supplements and drugs for chronic conditions. The survey included analysis of the toxic metals cadmium, arsenic, lead and mercury as well as common catalytic metals using inductively coupled plasma mass spectrometry (ICP-MS). The elements examined in this survey include residual catalysts that are covered by the current European Medicines Agency (EMA) guideline and the heavy metals and residual catalysts that are currently under review by the USP. In general it was found that the metal levels for the products in the survey were below the limits that have been proposed by the USP. In this presentation we will discuss the shortcomings of the current USP method and compare and contrast other methods for the analysis of metals in pharmaceuticals. We will review the rationale for selection of products for the survey and we will discuss the results of the survey.

(243) **Regulation of Elemental Impurities**

Mamata De[1]; [1]US Goverment, CDER, FDA

There is currently no harmonized guidance for appropriate control of heavy and trace metal impurities in drug products and ingredients. The major metals of potential health concern found in food, drugs (medicines), and dietary supplements are lead, cadmium, mercury, and arsenic. Other metals such as chromium, copper, manganese, molybdenum, vanadium, nickel, osmium, rhodium, ruthenium, iridium, palladium, platinum and others may be used or introduced during manufacturing and should be controlled in the final article as impurities. The setting of heavy and trace metal limits is appropriate for medicines, and is appropriate for supplements when heavy metals are likely or certain to contaminate a given product. Setting reasonable health-based limits for some of these metals is challenging because of their ubiquity in the environment, limitations of current analytical procedures, and other factors. The ICH Q3A Guideline classifies impurities as organic, inorganic, and residual solvents. Whereas organic impurities and residual solvents are appropriately addressed in ICH Guidelines, inorganic impurities are not. And while the current lack of clear and consistent guidance for the control of inorganic impurities is very problematic for the pharmaceutical industry, the primary concern is ensuring the safety of the patient. The workshop will discus appropriate risk-based approach to ensure control for metals likely to be present in drug products and ingredients, including those resulting from the manufacturing process (metal catalysts and reagents such as Chromium, palladium etc), as well as those due to the material source(e.g. Pb, Hg, As, Cd). This overall goal of this course is to provide an overview of the regulatory and toxicological issues surrounding heavy metals and trace metals in pharmaceuticals. The course will provide an in-depth look at specific metal impurities in pharmaceuticals and its impact on public health. The course will also review and discuss metal impurity limits; methodology, risk assessment, harmonization, and implementation strategies. Followings are the specifics which will be discussed in the Workshop. 1. Discuss the regulatory and toxicological issues surrounding heavy metals and trace metals in pharmaceuticals. 2. Discuss the possible regulatory guidance's regarding limits on heavy metals and trace metals in pharmaceuticals. 3. Explain the impact of heavy metals and trace metals in pharmaceuticals on public health. 4. Discuss the scientific challenges and regulatory issues facing the FDA regarding heavy metals and trace metals in pharmaceuticals.

(244) **ICP-OES Analysis of Pharmaceutical Materials: USP Elemental Impurities**

Timothy Shelbourn[1]; [1]Eli Lilly and Company

Analytical methods have been developed using ICP-OES to quantify Class I and Class II elements in pharmaceutical materials per draft USP chapters Elemental Impurities <232> and <233>. Samples were prepared using either closed vessel microwave digestion or straight dissolution in an organic solvent. Method development and standard compliance strategies are discussed. Figures of merit (accuracy, precision, LOQ, and linearity) for each analytical method are presented.

(245) **Phosphorus and Sulfur as Internal Tags for Pharmaceutical Analysis by ICPMS**

Joseph Caruso[1], Brittany Catron[1], Renee Easter[1], Kirk Lokits[1], Pat Limbach[1]; [1]University of Cincinnati

The use of metal tagging for analyses of organo- and biomolecules is an exciting technique and has emerged since the beginning of the 21st century. Basically, internal or external molecular tags can be used as long as they respond well to elemental analysis by ICPMS. Rare earths elements are often used as external tags, added to the molecule of interest through an appropriate derivatization, where the metal tag is added in a similar fashion to adding a fluorophore, but with much greater spectral resolution and lower detection as results. Since the introduction of collision/reaction cell technology for ICPMS, a great advantage has been added for using 31P and 32 /34S as internal tags. Further, oxygen may be utilized to move the detection m/z to 47 or 48, respectively. These elements are critical to many important biomolecules, such as proteins, DNA and RNA, phospholipids, phosphosugars, etc. Yet, even without the collision/reaction cell technologies, internal tags such as I or Br (very good elements by ICPMS), have been used, for example, for Levothyroxine analysis. This talk will explore the possibilities for pharmaceutical analyses but, not in the sense of trace metal analysis. Rather, the focus will be on internal tags as elemental handles for determining important biomolecules such as Levothyroxine and more recently, phosphorothioate antisense drugs. To some extent S may be used as an internal tag for common antibiotics.

(246) **HPLC-Particle Beam Mass Spectrometry for Metal Speciation and Actives Profiling in Botanical Products**

R. Kenneth Marcus[1]; [1]Clemson University

By their very nature, botanical products present a number of challenges to nutraceutical producers, consumers, researchers, and regulators. Potential analytes range from the obvious target actives to potential adulterants. In addition, many botanicals sequester metals which are both nutritional as well as potential toxins. The shear number o potential analytes presents immense challenges for the analytical chemist. The wide disparity in raw materials, points of origin, processing procedures, product forms makes the job even more demanding. We present here the utilization of a singular analytical platform comprised of an HPLC coupled to a particle beam mass spectrometer system having either electron or glow discharge ionization sources for the comprehensive speciation of botanical products/nutraceuticals. Use of this approach allows for characterization of actives, adulterants and metals (with speciation) in a relatively straightforward manner, rather than use of a multitude of instrumental methods. Practical examples of the methodology will be presented along with the use of the instrumentation in the characterization of new, NIST SRMs.

(247) **Investigations of Fundamental Processes and Ion Chemistry of the Flowing Atmospheric-Pressure Afterglow and Low-Temperature Plasma Probe Ambient Ionization Sources**

Jacob T. Shelley[1], Joshua S. Wiley[2], Carsten Engelhard[1], Ayanna U. Jackson[2], R. Graham Cooks[2], Gary M. Hieftje[1]; [1]Dept. of Chemistry - Indiana University; [2]Dept. of Chemistry - Purdue University

Plasma sources in mass spectrometry are typically viewed as tools for elemental analysis. However, discharges operated at atmospheric pressure, such as corona and glow discharges, have been used for over 30 years for molecular analyses through soft, chemical-ionization pathways. More recently, a variety of plasma-based sources have been developed to perform direct desorption/ionization of analytes from complex matrices, resulting in field termed Ambient Desorption/Ionization Mass Spectrometry (ADI-MS). These ionization sources often yield high desorption/ionization efficiencies and simple mass spectra, typically consisting of only the molecular ion or its protonated counterpart. Though numerous applications of these plasma-based sources have been explored, little work has been performed to fundamentally characterize and compare the discharges themselves and the different ionization pathways they utilize. In the present study, two plasma-based ADI-MS sources, the flowing atmospheric-pressure afterglow (FAPA) and the low-temperature plasma (LTP) probe, were optically and mass spectrometrically characterized. Fundamental plasma parameters, such as rotational temperature and electron number density, were found to be substantially different between the two discharges. This finding was expected as FAPA and LTP utilize vastly different discharges: a direct-current, glow discharge and an alternating-current dielectric-barrier discharge, respectively. However, mass spectra obtained with both ionization sources for a variety of analytes were found to contain analogous fragments and reactive species, implying that the ionization pathways are quite similar. While many polar analytes produce spectra consisting of strictly protonated molecular ions, other analytes, such as polycyclic aromatic hydrocarbons and ionic liquids, yield spectra containing unexpected fragment ions as well as odd-electron species. Furthermore, these mass spectra were found to contrast with those obtained with a similar, commercial ionization source, Direct Analysis in Real Time (DART), which produced predictable molecular ion signals. This discrepancy was attributed to a direct analyte-plasma interaction that occurs with FAPA and LTP analyses. Lastly, this direct plasma interaction was optically evaluated with a time-gated, monochromatic imaging spectrometer, revealing the formation of short-lived plasma bullets.

(248) **Elemental Bioimaging by Pulsed Radio-Frequency Glow Discharge – Monochromatic Imaging Spectrometry**

Carsten Engelhard[1,2], Steven J. Ray[1], Wolfgang Buscher[2], Maxim Voronov[3], Volker Hoffmann[3], Gary M. Hieftje[1]; [1]Indiana University, Dept. of Chemistry; [2]University of Münster, Institute of Inor; [3]Leibniz Institute for Solid State and Materials

Elemental bioimaging in gels and membranes has been successfully performed by laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) after gel electrophoretic separation. This technique provides excellent limits of detection (µg/kg - range) and spatial resolution in the hundreds-of-micrometer range. However, because it is necessary to raster the sample with a laser, imaging of a large sample area can be time-consuming. In contrast,the method to be discussed here permits elemental imaging of large sample areas at much higher speed. In particular, a pulsed radio-frequency glow discharge (rfGD) can provide this capability when coupled to a monochromatic imaging spectrometer (MIS) and gated intensified charge-coupled device (iCCD). In the glow discharge, analytes of interest on a gel or blotting membrane are subjected to sputtering and excitation. The rf energy is applied in short pulses, which limits the sample surface temperature and enables direct use of fragile substrates. More importantly, pulsing the discharge reduces the effect of diffusion of sputtered atoms before they emit and provides spatial resolution in the hundreds-of-micrometer realm. Model proteins were separated by gel electrophoresis, stained with silver-enhanced colloidal gold, transferred to a blotting membrane, and imaged with the rfGD-MIS instrument. Laterally resolved emission maps have been obtained and current analytical figures of merit will be presented. The technique provides femtomole limits of detection (e.g. bovine serum albumin LOD = 0.05 ng / 1 mm2 spot). During early experiments, monochromatic images were found to be distorted because of the MIS grating angle. The ratio between horizontal and vertical image width is a measure of image distortion and found to range from 18±2% at 400 nm to 46±2% at 750 nm. To correct for

this instrumentally introduced distortion, a correction method was developed. This method is based on fixed frequency images, in which calibration points are localized to characterize the distortion at a given wavelength. Image correction was performed computationally by means of bilinear interpolation. The geometric error, i.e. residuals of the interpolation, was calculated for every pixel in the reconstructed image and typically found to be between 0.12 and 0.18 pixel. Distorted emission maps could be successfully corrected by the method presented here.

(249) **Analytical Methods for the Diagnosis of Death-By-Drowning**

Maite Aramendia Marzo[1], Maria Rosario Flórez[2], Martín Resano[2], Michel Piette[1], Frank Vanhaecke[1]; [1]Ghent University; [2]Universidad de Zaragoza

Drowning is the second leading cause of death from unintentional injury, and accounts for more than half a million deaths annually worldwide [1]. However, the post-mortem diagnosis of drowning continues to be one of the most difficult in forensic pathology, the ideal test for this purpose still needing to be established [2]. In this context, an attempt is being made in our research group in cooperation with the Department of Forensic Medicine of Ghent University to develop alternative analytical strategies that give objective information as to the cause of death for bodies found in the water. Diagnosis of death-by-drowning can be done according to different principles, all based on the fact that the water and, thus, the particles, microorganisms and chemical substances it contains, will enter the blood circulation by different processes at the moment of death. As a result, detection of these water components in the dead bodies (where normally they should not be present) can help in arriving at a reliable diagnosis of drowning. In this presentation an overview of the drowning-markers studied so far in our laboratory will be given, with special attention to Al, for which a new analytical method for its direct determination in blood samples by means of Continuum Source-Atomic Absorption Spectrometry has been developed. The figures of merit of this new methodology will be presented and critically evaluated in comparison to other established methodologies. 1. Strontium Levels in Different Causes of Death: Diagnostic Efficacy in Drowning, M.D. Pérez-Cárceles, A. Sibón, M.L. Gil del Castillo, M.A. Vizcaya, E. Osuna, T. Casas, J.L. Romero and A. Luna, Biol. Trace Elem. Res., 126 (2008) 27-37. 2. Drowning: Still a difficult autopsy diagnosis, M.H.A. Piette and E.A. De Letter, Forensic Sci. Int., 163 (2006) 1-9.

(250) **Depth Profile Capabilities of Pulsed RF Glow Discharge TOFMS. How Far are We from SIMS?**

Jorge Pisonero[1], Nerea Bordel[1], Alfredo Sanz-Medel[1], Antonino Licciardello[2]; [1]University of Ovied; [2]University of Catania

The growing scientific and industrial interest in new and innovative materials has resulted in the application of ultra-thin (nm) coated materials in different fields of technology. Moreover, most physical properties of ultra-thin films (corrosion resistance, adhesion, optical and magnetic properties, diffusion issues) are related to their chemical compositions (dopants and major elements) and structures. Therefore, fast throughput high depth resolution techniques with high sensitivity and wide dynamic range are highly required for the characterization of these materials. Pulsed Radiofrequency Glow Discharge Time of Flight Mass Spectrometry (pulsed-RF-GD-MS) is investigated as an alternative technique for fast direct solid analysis with nanometric depth resolution of relatively large sample areas (~mm2).[1] Atomization and ionization processes in GD plasmas are known to be temporally and spatially separated, reducing the matrix effects. Moreover, the GD plasma operates at relatively low pressure (1-10 torr), thus it does not require sampling at ultra-high vacuum conditions and so it facilitates high sample throughput compared to reference techniques such as Secondary Ionization Mass Spectrometry (SIMS). In this work, the analytical potential of pulsed-RF-GD-TOFMS is investigated for fast depth profile analysis of ultra-thin layers. Nanometer depth resolution in pulsed-RF-GD-TOFMS is investigated through the analysis of two series of ultra-thin (~ nm) Nb/AlxCo1-x bilayers and Si/Co bilayers, deposited on Si wafers by dc-magnetron sputtering. RF-GD-TOFMS permits quasi-simultaneous detection of multi-elemental ions along the RF-GD pulse period and thus the observation of the different time domains as result of the different ionization/excitation mechanisms in the plasma. Thus, qualitative depth profiles of the ultra-thin layered sandwich-type samples are determined integrating the ion signals at different time domains along the rf pulse profile. For instance, qualitative depth profiles obtained in the pre-peak, plateau and afterglow domains of the pulsed-rf-GD are investigated and compared. Furthermore, a good agreement with the depth profiles measured using SIMS is obtained, demonstrating the analytical potential of the pulsed-GD-TOFMS system for fast, sensitive and high depth resolution analysis of ultra-thin layers. [1] R. Valledor, J. Pisonero, N. Bordel*, J.I. Martín, C. Quirós, A. Tempez, A. Sanz-Medel “Direct chemical in-depth profile analysis and thickness quantification of nanometer multilayers using pulsed-rf-GD-TOFMS”, Anal. Bioanal. Chem., 2010, 396:2881–2887.

(251) **New Possibilities for Quantitative Analysis in Environmental and Life Sciences Using (Hetero)Element Tags and ICP-MS Detection**

Daniel Proefrock[1], Andreas Prange[1]; [1]GKSS Research Centre

The accurate quantification of hazardous substances in the environment or biologically relevant molecules such as proteins and peptides becomes more and more essential for the monitoring and understanding of biological processes or finally for the assessment of the environment in terms of e.g. contamination levels. Especially the recent developments within the fields of genomics and proteomics enhanced the understanding of cellular processes, however there is still an inherent lack of quantitative information associated with these datasets which might reflect time dependent changes in absolute protein concentrations or the degree of a site specific post translational modifications. This contribution will focus on the recent possibilities in using ICP-MS for the determination of (hetero)elements, which are naturally present in a number of compounds and which can be utilized as “representative” for the accurate and sensitive quantification of these molecules. In addition the challenges and advantages of using miniaturized chromatographic techniques for front end separation and sample introduction as well as the role of molecule specific MS as complementary detection technique will be discussed.

(252) **Application of Labelled Antibodies for ICP-MS Detection**

Charlotte Giesen[1,2], Michael G Weller[1,2], Norbert Jakubowski[1], Ulrich Panne[1,2]; [1]BAM; [2]Humboldt University Berlin

We would like to report on the use of labelled antibodies for an ICP-MS-linked immunoassay. Ochratoxin A (OTA) is a common contaminant of different types of foods and beverages. Results indicate that it is carcinogenic in rats and mice [1]. Therefore, limit values for OTA in a number of foodstuffs have been established in Directive EC 1881/2006 and 105/2010 by the European Union (EU). Limit values for OTA in wine were set to 2 µg L -1. We developed a fast and reliable ICP-MS-linked immunoassay for OTA detection in wine. For labelling, we applied secondary antibodies conjugated with gold nanoparticles. The limit of detection was determined as 0.003 µg L -1 and the range of quantification was calculated as 0.01–1 µg L -1 through the precision profile approach [2]. Labelled antibodies can also be

applied as immunostains for biological tissues in imaging experiments by LA-ICP-MS. First results will be presented. References [1] J. E. Huff, IARC Scientific Publications, 1991, 115, 229-244. [2] R. Ekins, Immunoassays for clinical chemistry, 1983, 76-105.

(253) **Multivariate Analysis of Variance for Forensic Trace Evidence Decision-Making**

Stephen L. Morgan[1]; [1]University of South Carolina

Comparison of a questioned evidence item (Q) (e.g., found at a crime scene) with one or more known evidence items (K) is central to determination of possible associations between victims, suspects, and crime scenes. A major driving force for statistical concerns is the Daubert decision (1993), which established a checklist for assessing the reliability of scientific expert testimony, one that includes whether the rate of error of the technique or theory has been established. Forensic scientists are exposed early and often to statistical inference via calculation of sample means and standard deviations. We are taught to use these concepts in confidence intervals, t-tests, F-tests to make decisions about differences (significant, or not), and to make judgments (match, or not), between various evidence materials under study. In modern forensic analytical chemistry, we are often faced with multivariate data (e.g., comparing polymers samples by their IR spectra, comparing bullets by their multi-elemental compositions, comparing chromatograms by the peak areas or heights of several relevant peaks). Attempts to treat such inherently multivariate data with univariate methods (e.g., performing separate t-tests on each variable, or plotting values of each measured variable separately), can lead to biased and misleading conclusions. Because multiple variables are often not independent of one another, methods that take into account their correlations must be employed. The multivariate generalization of the univariate Student's t test is Hotelling's T2 test for the equivalence of two multivariate means. As in the common univariate assumption of equal variances for the groups, Hotelling's test assumes that within-group sample covariance matrices are equal and can be pooled. As with the univariate t-test, modest deviations from equality are usually not serious; however, test statistics are also available for testing equality of two or more covariance matrices. Starting with a discussion of univariate analysis of variance, this presentation will discuss multivariate analysis of variance and associated methods in the context of forensic decision-making in trace evidence comparisons. The trace evidence examples based on multivariate data in this presentation will provide analytical chemists and forensic scientists with a basic understanding of these powerful data analysis tools.

(254) **Improving Investigative Lead Information and Evidential Significance Assessment for Automotive Paint by Development of Pattern Recognition Based Library Searching Techniques**

Barry Lavine[1], Nikhil Mirjankar[1], Mark Sandercock[2]; [1]Oklahoma State University; [2]Royal Candadian Mounted Police

New pattern recognition techniques for searching infrared (IR) spectral libraries of the Paint Data Query (PDQ) automotive paint database will be discussed. These techniques are used to differentiate between similar but nonidentical Fourier transform infrared (FTIR) paint spectra, and to determine the model, manufacturer, and year of the vehicle from which an unknown paint sample originated. Modern automotive paints use thinner undercoat and color coat layers, which are protected by a thicker clear coat layer. As a result, only a clear coat paint smear, all too often, is left at the crime scene. In these cases, the text based portion of the PDQ database cannot be used to identify the motor vehicle. Based upon our previous experience, clear coat paint layers, like the undercoat and color coat paint layers, exhibit chemical features in their FTIR spectra that are unique to the automobile manufacturing plant at which they were applied. We will demonstrate that clear coat spectra can be used to identify the manufacturer, model, and year of a motor vehicle. Search prefilters for the PDQ database, which are the focus of this presentation, are necessary to extract investigative lead information from a clear coat paint smear.

(255) **Application of Chemometric Methods and Advanced Pattern to Trace Evidence Analysis**

Nicholas Petraco[1]; [1]John Jay College of Criminal Justice; [2]The Graduate Center/CUNY

Given a set of a set of multivariate data constituting evidence in a crime, how likely is it that, when a conclusion of association/non-association is drawn from that evidence, that conclusion is erroneous? With recent challenges to the admissibility of almost all forms of physical evidence analysis, such a question is of paramount importance. In this talk we will show how state of the art methods of statistical pattern recognition (i.e. machine learning) can significantly aid in answering this question. An overview of how we have applied these methods to fire debris analysis (GC-MS gasoline), tool marks (striation patterns), footwear imprints and photocopies will be presented.

(256) **Application of Target Factor Analysis to the Classification of Ignitable Liquids from Fire Debris**

Michael Sigman[1], Mary Williams[1]; [1]University of Central Florida

The summed ion mass spectrum, i.e., the mass spectrum averaged across a chromatographic profile, has been shown to be highly specific for a complex mixture of the type comprising ignitable liquids often found in fire debris samples. This presentation will discuss the use of summed ion mass spectra combined with target factor analysis (TFA) to correctly assign an ASTM classification to ignitable liquid residue in the presence of significant contributions from pyrolysis products found in fire debris. The method has been successfully applied to samples from laboratory tests and large scale burns. Laboratory test samples were prepared by burning typical building materials in the presence of an ignitable liquid. Experiments involved varying the volume of ignitable liquid while holding the identities of the substrates constant and varying the substrate identity while holding the ignitable liquid volume constant. Large scale burns were conducted in 20' x 8' x 8' shipping containers. The containers were finished on the inside with pine stud walls and sheetrock with carpet and padding on the floor. The finished shipping containers were furnished with furniture and typical household contents. The containers were burned with the aid of an ignitable liquid and multiple samples were taken. Samples were analyzed based on the American Society for Testing and Materials (ASTM) E 1412 and E 1618 methods by gas chromatography-mass spectrometry (GC-MS). The set of summed ion spectra from each fire were tested against a library of 450 ignitable liquid summed ion spectra by target transformations of the eigenvectors achieved by diagonalizing the covariance matrix. Performance of the TFA was evaluated by calculating receiver operating characteristic (ROC) curves. The ROC area was greater than 0.92 for gasoline, petroleum distillates, naphthenic-paraffinic, normal alkanes and isoparaffinic liquids. The aromatic and oxygenated ASTM-classes of solvents gave significantly lower ROC areas. This work was supported by the National Institute of Justice, Office of Justice Programs, award 2008-DN-BX-K069. The content of this talk and abstract do not necessarily reflect the position or the policy of the U.S. Government, and no official endorsement should be inferred.

(257) **The Design of an Infrared Imaging System for Blood Stains at Crime Scenes Using a Chemometrics Simulation-Driven Process**

Michael L. Myrick[1], Heather Brooke[1], Megan R. Baranowski[1], Jessica N. McCutcheon[1], Stephen L. Morgan[1]; [1]University of South Carolina

We have designed a prototype portable camera using mid-infrared (IR) imaging through filters composed of polymer films. We present a simulation-driven process to select optimized custom polymer filters based on "molecular factor computing". There are many factors involved in optimizing discrimination by using optical filtering aids, including, but not limited to, the detector response, optical throughput of the system, optical properties of the samples, and optical properties of the materials for sensitizing films/filters. There are nearly infinite possible setups for the system, which means it is neither cost- nor time-efficient to physically test each one. In lieu of this, we developed routines in MATLAB® that simulate the camera output, per pixel, given specific conditions. Beginning with measured spectra of calibration samples or standards, and using an objective function or figure of merit (FOM) to measure simulated performance, these routines evaluate large numbers of combinations of chemical films as filters for discrimination based on linear discriminant analysis (LDA). In this study, the FOM was the Fisher ratio between a neat fabric and one stained with either a polymer film or blood.

(259) **Novel Nanorod Array Substrates as a Platform for SERS-Based Biosensing of Infectious Disease**

R.A. Dluhy[1], J.D. Driskell[1], Y.-P. Zhao[1], P. Rota[2], R.A. Tripp[1]; [1]University of Georgia; [2]Centers for Disease Control

Development of diagnostic methods for rapid and sensitive identification of viruses and other biomedical pathogens is essential for the advancement of therapeutic and intervention strategies necessary to protect public health. Current diagnostic methods for viruses in particular, e.g. isolation, PCR, antigen detection and serology, are time-consuming, cumbersome, or lack sensitivity. We have investigated the use of aligned Ag nanorod arrays, prepared by oblique angle vapor deposition (OAD), as surface-enhanced Raman scattering (SERS) substrates for the identification and classification of viral pathogens. The OAD method of substrate preparation facilitates the selection of nanorod size, shape, density, alignment, orientation, and composition, while the procedure is reproducible and relatively simple to implement. The current talk will address aspects of the fundamental nanostructural design of metallic nanorod arrays and their influence on SERS enhancement, as well as the development of a spectroscopic assay for virus detection based on these unique nanostructured SERS probes. We will also present results of multivariate statistical analyses on the SERS spectra of different pathogenic species that indicate that it is possible to identify, differentiate and classify viruses and other biomolecules based on their intrinsic SERS spectra, even down to the individual strain level.

(260) **Using Spectroscopy to Reveal Dynamics in Self-Assembled Systems**

Gary Blanchard[1], Heather Pillman[1], Monika Dominska[1], Benjamin Oberts[1]; [1]Michigan State University

Self-Assembly is a process that underlies many important processes, ranging from interface modification to the function of living organisms. While there is a great deal of interest in the organization of self-assembled systems, the dynamical processes that mediate their behavior, such as permeability and fluidity, are somewhat less well understood. We have investigated selected lipid bilayer structures because of their importance to life processes, with a focus on the fluidity and permeability of these interfaces. We will discuss the ways in which we have interrogated these systems and our results. One significant conclusion of our work is that remarkably little impurity is required to produce substantial changes in the properties of model bilayer structures.

(261) **Modified Electrodes in the Solid State: Molecules as Circuit Components**

Richard McCreery[1, 2], Adam Bergren[1], Haijun Yan [1, 3], Andrew Bonifas[1, 3]; [1]National Institute for Nanotechnology; [2]University of Alberta; [3]Ohio State University

A molecular junction is essentially a modified electrode with the addition of a second conducting “contact” instead of an electrolyte solution. Electron transport through molecular monolayers in such devices is a function of molecular structure, orbital distribution, and energetics, and can be controlled over a wide range. Molecular junctions based on carbon substrates will be described, including their electronic characteristics as a function of temperature, molecule length, and orbital energies. The main factors controlling electron transport through molecular junctions were determined, enabling the design of devices with particular electronic behaviors. Recent relevant publications include: Yan, H.; McCreery, R. L.; Anomalous Tunneling in Carbon/Alkane/TiO2/Gold Molecular Electronic Junctions: Energy Level Alignment at the Metal/Semiconductor Interface; ACS Applied Materials & Interfaces 2009, 1, 443. McCreery, R. L.; Bergren, A. J.; Progress with Molecular Electronic Junctions: Meeting Experimental Challenges in Design and Fabrication; Advanced Materials 2009, 21, 4303. Bergren, A. J.; Harris, K. D.; Deng, F.; McCreery, R.; Molecular Electronics using Diazonium-Derived Adlayers on Carbon with Cu Top Contacts: Critical Analysis of Metal Oxides and Filaments; J. Phys. Condens. Matter 2008, 20, 374117.

(262) **Holey Zeolites: Chemistry and Biology in Confined Spaces**

Prabir Dutta; [1]The Ohio State University

Zeolites are crystalline aluminosilicates with cages and channels of molecular dimensions. Our research group has focused on various aspects of these materials, including formation of the nano-porous structure, supramolecular arrangement of molecules in the pores, synthesis of zeolite membranes for high temperature gas sensing and the use of zeolites as model systems for understanding asbestos toxicity. Raman spectroscopy of the template molecules necessary for zeolite synthesis has provided information on the mechanism of zeolite formation. Arrangement of polypyridyl complexes of ruthenium along with bipyridinium electron acceptors in zeolite cages has provided for an efficient route for long-lived photochemical charge separation. Pt-loaded zeolite membranes are effective for reducing interferences in detection of total NOx by electrochemical methods leading to sensors for control of high-temperature combustion processes. Finally, the redox chemistry of iron on zeolite surfaces has provided an excellent model system for understanding asbestos toxicity, in particular, and all respirable particles in general. These topics will form the subject of my presentation.

(263) **Optically Transparent Diamond Electrodes for Use in UV/Vis and IR Transmission Spectroelectrochemical Measurements**

Greg Swain[1], Chen Qiu[1], Denis Proshlyakov[1]; [1]Michigan State University

Electrically conducting diamond is an advanced carbon electrode material that is proving to be useful for several electrochemical technologies. In fact, few materials show as much versatility as an electrochemical electrode as does boron-doped, chemical vapor deposited (CVD) diamond. The material can be used in electroanalysis to provide low detection limits for analytes with superb precision and stability; for high-current density electrolysis

(>1 A/cm2) in aggressive solution environments with little microstructural or morphological degradation; and as an optically transparent electrode (OTE) for spectroelectrochemical measurements in the UV/Vis and IR regions of the electromagnetic spectrum. The application of optically transparent electrodes (OTEs) for spectroelectrochemical measurements in the UV-Vis and IR regions of the electromagnetic spectrum represents a new field of diamond research. This new OTE possesses properties that are superior to those of conventional UV-Vis OTEs, like indium tin oxide (ITO). These properties enable its use in measurements and chemical environments where conventional OTEs fail. The growth and characterization of optically transparent diamond electrodes for use in transmission spectroelectrochemical measurements in the UV-Vis and IR regions of the electromagnetic spectrum will be discussed. Diamond-quartz electrodes were used to study the electrochemical and optical properties of aqueous (Fe(CN)6-3/-4) and non-aqueous (ferrocene) redox systems in the UV-Vis region, while diamond-undoped Si electrodes were used to study the electrochemical and optical properties of these same redox systems in the mid- and far-IR. Studies of structure-function relationships of redox proteins (e.g., cytochrome c and myoglobin) using spectroelectrochemical methods will also be discussed.

(265) **Manipulating Biomolecules by Dielectrophoresis**

Alexandra Ros[1], Asuka Nakano[1], Lin Gan[1], Tzu-Chiao Chao[1]; [1]Arizona State University

The high biological complexity on the molecular level demands for powerful separation and fractionation techniques in many fields of biochemical and biological research and analysis, such as in biomarker discovery or clinical diagnosis. Other examples include current analytical challenges such as seeking to understand the subtle differences in disease progress or reveling processes on the single cell level. Such analyses often need to be performed in minute sample amounts, should be fast to perform and ideally use matrix-free approaches. Here, we suggest adding a novel parameter to the separation space by exploiting the polarizability of biomolecules and concomitantly their dielectrophoretic behavior as a selection criterion for biomolecular analysis. We use insulator-based dielectrophoresis (iDEP) to manipulate polarizable particles in inhomogeneous electric fields on a microfluidic platform. These devices are treated with suitable surface coatings to prevent biomolecules to adsorb and thus suppress biofouling. We investigated both DNA in a size range from 3.5 to 164 kbp and selected proteins (bovine serum albumin, immunoglobuline G, phycocyanin and phycoerythrin). For DNA, we could demonstrate DEP trapping, which is size and conformation dependent. We were thus able to develop a chip-integrated method for separation by iDEP in pL volumes, with pM detections sensitivity and short separation times of 200s. Interestingly, we also observed DEP response of the much smaller proteins. In particular, we observe different trapping behavior of proteins when subject to DEP in a zwitterionic and denaturing buffer as compared to buffer conditions without these agents. We correlated these differences with molecular size and aggregation and observed that the monomeric forms demonstrate different iDEP trapping behavior than protein aggregates. Our future emphasis lies on a more detailed understanding of biomolecular DEP in order to apply it to a broad range of separation and fractionation problems.

(266) **Insulator-Based Dielectrophoresis of Particles Employing Extremely Low Frequency Alternating Current Electric Fields**

Javier L. Baylon-Cardiel[1], Nadia M. Jesus-Perez[1], Ana V. Chavez-Santoscoy[1], Sergio O Martinez-Chapa[1], Blanca H. Lapizco-Encinas[2]; [1]Tecnológico de Monterrey; [2]CINVESTAV-Monterrey

Low frequency alternating current insulator-based dielectrophoresis is a novel technique that allows for highly controlled manipulation of particles. By varying the shape of an AC potential applied across a microchannel containing an array of insulating cylindrical structures it was possible to concentrate and immobilize microparticles in bands; and then, move the bands of trapped particles to a different location. Mathematical modeling was performed to analyze the distribution of the electric field and electric field gradient as function of the shape of the AC applied potential, employing frequencies in the 1 Hz range. Three different signals were tested: sinusoidal, half sinusoidal and sawtooth. Experimental results demonstrated that this novel dielectrophoretic mode allowed highly modulated dielectrophoretic particle manipulation. The development of low frequency AC-iDEP as a technique to manipulate bioparticles has great potential for many biotechnological applications. This novel dielectrophoretic mode allows for an extra parameter to modulate dielectrophoretic mobilization of particles and/or groups of particles; since bands of immobilized particles can be manipulated by varying the shape of the applied AC potential. This highly controlled process could be used to improve the separation and fractionation of mixtures of particles, which are common challenges in many biotechnological applications.

(267) **Quantitative Analysis of Erythrocyte Rupturing in an Alternating Current Dielectrophoretic Field as Compared to Theoretical Observations**

Kaela Leonard[1], Adrienne Minerick[1]; [1]Michigan Technological University

Human blood is of incredible diagnostic interest because it is easily obtainable and can provide a plethora of information about a person's general health such as protein, iron, vitamin and electrolyte levels. The goal of this research is to expand blood diagnostic capabilities by quantifying disease indicators, Medical microdevices are used to study differences in dielectrophoretic response of human erythrocytes based on ABO-Rh blood type. This effort is based on the hypothesis that the rupturing of erythrocytes when subjected to a 1kHz alternating current dielectrophoretic field of density 0.03Vpp/micron is ABO-Rh antigen dependent. In the beta-dispersion region, which occurs around 1kHz, a Maxwell-Wagner relaxation of the membrane occurs due to the current passing through two different dielectrics (the cell membrane and the cell cytosol). It is this membrane determined polarization / relaxation cycle that is believed to be the cause of the erythrocyte rupturing. ABO-Rh antigen observed dependence is likely due to the antigen molecule's influence on the membrane polarization. Experiments were conducted with a custom microdevice on an inverted video microscope to capture still frame images of erythrocyte behaviors every 15 seconds for each 15 minute experiment. Still frame images were custom analyzed to recognize the blood cells as individual objects and then quantify the number of erythrocytes in each image. The quantitative cell counts were translated into overall rupturing percentages and compared with theoretical COMSOL simulations of membrane instabilities.

(268) **Dielectrophoresis at Conductive Liquid Interfaces**

Zachary Gagnon[1]; [1]Johns Hopkins University

Dielectrophoresis is a popular technique for cell and particle manipulation at the microscale, and is commonly used to describe the motion of suspending particles or cells under the application of a non-uniform electric field. The DEP force results from electric field induced charge accumulation at the cell or particle surface due to a discontinuity in the electrical conductivity and dielectric constant at the particle-liquid interface. During exposure to an AC electric field, the interface charges much like a capacitor – governed by conductive charging at low (typically < 500 kHz) frequency and dielectric polarization at high frequency. Commonly, small submicron particles and the interior of most cell types have a

higher conductivity and lower dielectric constant than their surrounding media. Hence, the resulting direction of the induced particle dipole often reverses when the applied AC field frequency exceeds the inverse relaxation (RC) timescale of the media-particle interface, known as the DEP crossover frequency (cof). Interfacial polarization, hence, plays an important role in dictating the magnitude and direction of the field induced DEP force exerted on a particle or cell. Typically, DEP has been applied to particles, cells and liquid droplets in suspension. At its most basic element, however, DEP is simply a force that arises from unbalanced charging at an electrical interface. Here, I explore a different electrical interface - that of two or more adjacent liquid streams flowing within the confines of a microfluidic channel network. Due to the laminar flow profile and slow diffusion timescale associated with microchannel fluid flow, two or more liquid streams can flow side by side without mixing. As both the conductivity and dielectric constant of aqueous solution can be readily adjusted with soluble salts and zwitterions, two solutions of different electrical properties can be allowed to flow side by side to generate an electrical liquid-liquid interface. Much like a suspending particle surface, a microfluidic generated liquid-liquid electrical interface can also be polarized with embedded microelectrodes. Here, I characterize the unique phenomena that arise at polarized electrical liquid-liquid interfaces. Furthermore, I apply them to electrokinetic cellular manipulation applications and biological studies to demonstrate the potential for this new and flexible microfluidic platform.

(269) **Ion Excluded Volume Effects on Dielectrophoresis of a Colloidal Particle**

Hui Zhao; [1]University of Nevada Las Vegas

The dipole moment of a charged, dielectric, spherical particle under the influence of a uniform alternating electric field is computed by solving the modified Poisson-Nernst-Planck (PNP) equations (Bikerman's mean-field model) accounting for excluded volume effects of the finite ion size as a function of the double layer thickness, the electric field frequency, the particle's surface charge, and the volume fraction of the ions in the bulk characterizing the excluded volume repulsion. In the limit of thin electric double layers, we carry out an asymptotic analysis to develop simple models calculating dipole moments which are in favorable agreement with the modified PNP model. Our results reveal that excluded volume effects, imposing a maximum on the counterion concentration, reduce the dipole moment at high frequencies and possibly enhance the dipole moment at low frequencies, assuming that the particle bears the same zeta potential. Excluded volume effects often become significant in highly concentrated salt solutions or near highly charged surfaces and the modified PNP model considering the ion size generally improves the theoretical predictions in comparison to experimental data and a possible explanation for such improvement is suggested.

(270) **Dielectrophoretic Manipulation of Particles and Cells in Curved Microchannels**

Xiangchun Xuan[1]; [1]Clemson University

Dielectrophoresis (DEP) is a powerful tool that has been widely used to manipulate (e.g., focus, trap, concentrate, and sort) particles and cells in microfluidic devices. Traditional DEP (also named as electrode-based DEP) arises from the non-uniform AC electric field between pairs of electrodes that are fabricated within a microchannel. This method suffers from several problems such as the need of hydrodynamic pumping of the sample, the increased complexity in channel fabrication, and the surface fouling of electrodes etc. Such problems are not encountered in insulator-based DEP, where both AC and DC electric fields can be applied through the electrodes that are positioned outside a microchannel. Two types of in-channel insulators have so far been demonstrated to produce the dielectrophoretic force for particle and cell manipulations. The first type is the insulating obstacles (e.g., hurdles, posts, and ridges) that are fabricated inside a microchannel to partially block the electric current. However, the locally amplified electric field around the insulating obstacles may cause adverse effects on both the sample and the device due to Joule heating and particle clogging etc. These drawbacks are overcome in the second type of insulator-based approach where the insulating walls of a curved microchannel are exploited to control the particle motion. Due to the variation in path length for electric current, the electric field becomes inherently non-uniform in a curved channel. Thus induced DEP can generate a cross-stream deflection for continuously focusing and separating particles and cells. Our group has done work in both types of insulator-based DEP approaches. In this talk we will present the recent results on the dielectrophoretic focusing and separation of particle and cells in serpentine and spiral microchannels.

(271) **US Department of Homeland Security Counter-IED Detection Programs and Priorities**

Michael Shepard[1]; [1]US Department of Homeland Security

In an effort to protect the United States from the ever increasing threat of domestic terrorism, the Department of Homeland Security, Science and Technology Directorate (DHS S&T), has significantly increased research activities in the field of explosives detection. More over, DHS S&T has established a robust Counter-Improvised Explosive Device (C-IED) Program to Deter, Predict, Detect, Defeat, and Mitigate this imminent threat to the Homeland. The IED threat, whether deployed by as a suicide vest or Vehicle-Borne IED, presents the analytical community with many unique detection challenges. DHS is continuously reaching out to both academia and industry to meet these challenges. This presentation will focus on the challenges to security screening and explosives detection from both operational and analytical perspectives. Current standoff detection capabilities, limitations, and future direction will be presented. Detection priorities and requirements will be presented along with a discussion of funding opportunities within DHS. This presentation will also serve as the introduction to the Emerging Technologies for Standoff Detection for Homeland Security Symposium. The intent of this Symposium in two-fold: (A) To share with the audience some of the analytical challenges DHS faces and (B) to reach out and leverage the analytical community for contributions to these R&D efforts. Scientists from all areas of analytical chemistry, all and application areas, are encouraged to participate. Opportunities for one-on-one discussions, at the conference, with DHS managers will be discussed.

(272) **Vibrational Sum Frequency Spectroscopy for Stand-Off Detection of Chemicals on Surfaces**

William Asher[1], Ella Willard-Schmoe[1]; [1]University of Washington

Many forensic, counter-terrorism, and homeland-security missions would benefit from development of a rapid, stand-off method for detecting trace level contaminants adsorbed on solid surfaces. The second-order nonlinear optical technique of vibrational sum frequency spectroscopy (VSFS) was developed in a surface chemistry context as a non-contact probe for studying the chemical composition and molecular conformation of interfaces, membranes, and solid surfaces. In VSFS, a surface is illuminated with tunable IR photons and photons with a fixed wavelength. Then, photons with frequency equal to the sum of the incident photons are emitted from the surface. If the wavelength of the IR light matches a molecular vibration transition of a chemical adsorbed on the surface, increases in the number of sum-frequency photons generated at the surface of orders of magnitude can be observed.

Therefore, by tuning the IR light, a vibrational "emission" spectrum of the molecules on the surface can be obtained. VSFS is commonly used in laboratory applications, with distances between the transmitting optics, detectors, and sample of at most a few tens of centimeters. Because VSFS represents a general spectroscopic method capable of detecting the presence of a wide range of compounds, it might provide a rapid stand-off chemical detection method for homeland security applications. The results here demonstrate that commercially available VSFS systems can be adapted to a remote sensing configuration with working distances on order of several meters. It is also shown that detection limits at these distances can be as low as a few hundred nanograms per square centimeter.

(273) **Sensitive Standoff Detection of Chemical Agents By Nonlinear Multi-Photon Laser Wave-Mixing Spectroscopy**

William Tong[1], Marc Gregerson[1], Tiffany Neary[1], Marcel Hetu[1], Manna Iwabuchi[1], Jorge Jimenez[1], Ashley Warren[1]; [1]San Diego State University

Multi-photon nonlinear laser wave-mixing spectroscopy is presented as a sensitive remote standoff detection technique for chemical agents in gas-, liquid- and solid-phase samples. Wave mixing has important inherent advantages including excellent sensitivity, small sample requirements, short optical path length, high spatial resolution and remote standoff capability. Since wave mixing can be used for both fluorescing and non-fluorescing analytes, one can detect chemicals in their native form without the use of tags and labels with sensitivity levels comparable or better than those of fluorescence methods. In a typical wave-mixing optical setup, two input laser beams are focused and mixed inside the sample to create dynamic gratings. The incoming photons are then scattered off these gratings to create a coherent laser-like signal beam that has its own propagation direction, and hence, one can use effective spatial filters to minimize optical background noise levels. The wave-mixing signal is generated instantaneously as the two input beams intersect in the analyte of interest and the signal has the same optical characteristics of the incident coherent laser beams. Hence, optical signal collection is efficient and simple. The wave-mixing signal also has a quadratic dependence on analyte concentration, and hence, small changes in analyte properties result in more dramatic changes in the wave-mixing signal in this nonlinear sensor. Unique wave-mixing nonlinear properties offer effective standoff detection capabilities with excellent detection sensitivity levels.

(274) **Photoacoustic Standoff Detection Using Atomic Vapor Filters**

Dimitri Pappas[1]; [1]Texas Tech University

Standoff detection requires high sensitivity and also specificity. Currently LIBS and Raman techniques dominate efforts to develop standoff spectroscopic detection of key compounds of interest. While these techniques have merit, there is a need for new spectroscopic approaches that can compliment or improve standoff detection. One of the most promising techniques for standoff detection and identification is photoacoustic spectroscopy. Unfortunately, current approaches to photoacoustic measurements require a resonator cell to directly detect and amplify the photoacoustic signal, precluding their use in standoff detection. Even parabolic, remote microphones are subject to interferences. We propose a new approach to detecting photoacoustic signals that would enable standoff detection. This approach uses a laser to interrogate the sample—and generate a photoacoustic field—from a distance, and a secondary spectroscopic detection system to detect the photoacoustic field from the same standoff range. This would allow standoff detection without any need for sample preparation, and the standoff distance will be improved as this technology is developed further. The secondary, spectroscopic detection is based on atomic vapor photodetectors developed by our group and others. The frequency shift of the probe laser by the photoacoustic field is detected by Cs atoms, whose subsequent fluorescence is detected by the final readout. This approach will enable the detection of frequency shifted fields from photoacoustically-excited materials, and can also be used for Doppler shift detection and imaging of ultrasonic fields.

(275) **Single Ultrafast Pulse Excitation for Remote Coherent Anti-Stokes Raman Spectroscopy (SUPER-CARS) for Standoff Detection**

Marcos Dantus; [1]Michigan State University

Our group has been exploring a number of strategies involving phase, amplitude and polarization shaping aimed at improving single-beam coherent spectroscopic methods for standoff detection. Results from gas, liquid and solid phases will be shown.

(276) **Differential Laser-induced Perturbation Spectroscopy (DLIPS) for Standoff Detection**

David Hahn[1], Sarah Smith[1], Jonathan Merten[1], Nicolo Omenetto[1]; [1]University of Florida

The development of new smart sensing methodologies that provide improved sensitivity and/or specificity for rapid and accurate sensing is highly desirable for *in situ* and standoff detection of a number of potential threats, including for example explosives and biological pathogens. However, to date many applications of *in situ* and remote sensing schemes operating in actual real-world environments have been limited by the large sample-to-sample variations in optical response (e.g. fluorescence or Raman signals), as well as by the large variation in background signal levels. A novel sensing scheme is presented that holds promise to significantly enhance both sensitivity and specificity as compared to the current state-of-the-art optical-based sensing methodologies. This scheme is based on our recent research showing that many material matrices may be altered by low intensity (i.e., below the ablation threshold) ultraviolet radiation (primarily 193 to 213 nm) such that the intrinsic fluorescence or Raman scattering response is perturbed. We propose a novel sequential combination of optical probing (for example by Raman or fluorescence), UV photochemical perturbation, and repeat optical probing to realize a powerful new spectral dimension based on difference spectroscopy that will be strongly coupled to the local molecular matrix. Since the same targeted material is optically probed both before and after perturbation with the UV light source, the resulting differential response can avoid the major limitation of the current biosensing and security sensing schemes, namely, the significant variations in the absolute optical response, as generally observed in real-world populations. Preliminary data will be presented for a range of organic and biological materials.

(277) **Raman Scattering as a Probe of Structure in Polymeric Fibers**

Bruce Chase[1]; [1]Pair Technologies LLC

When I first started to explore the potential utility of Raman scattering in an industrial environment, one colleague who continually supported me was Bill Fateley. Bill had experienced all the frustrations of Raman scattering ranging from mercury arc lamps to fluorescence in polymers and he still saw the potential. He was always convinced that the most important attribute of Raman scattering was the information available on chain orientation. This has ended up being true. The power of polarized Raman scattering applied to polymeric fibers, both in static measurements and even in real-time measurements in the process environment, is significant. While a full orientation analysis is rarely worth the effort required, there are reduced sets of measurements that can be

used to qualitatively assess the degree of orientation. This will be illustrated with examples ranging from melt spun to solution spun to electrospun fiber systems.

(278) **Effect of Hydrogen Bond Strength on the Vibrational Relaxation of Interfacial Water**

Ali Eftekhari-Bafrooei[1]; [1]Temple University

Time resolved Sum Frequency Generation (tr-SFG) is used to study the vibrational dynamics of interfacial water at silica interfaces with varying surface charge. IR pump-SFG probe reveals that the relaxation rate of the O-H stretching vibration of dilute HDO in D2O at the silica interface is markedly different from bulk water. Compared to the bulk liquid, the vibrational lifetime (T1) of HDO is shorter at the charged surface than in the bulk, but longer at the neutral surface. The vibrational decoupling of the O-H of the HDO species leads to the observation of a frequency dependent T1 of the O-H stretch, that is shorter at the red than the blue side of the hydrogen bonded OH spectral region. This correlates with the redshift of the SFG spectra with increasing charged surface and is consistent with a theoretical model that relates the vibrational lifetime to the strength of the hydrogen bond network. These results increase our understanding of hydrogen bond network dynamics in environments of reduced dimensionality.

(279) **Applications of Nanoscale Imaging and Spectroscopy Using Tip Enhanced Raman Spectroscopic Techniques**

Ira Levin[1], Zachary Schultz[1,2], Taner Ozel[1], Tsoching Chen; [1]National Institutes of Health; [2]University of Notre Dame

The utilization of metal coated scanning probe tips to enhance local electric fields surrounding a probe provides a means for achieving high sensitivity Raman spectroscopic imaging with sub-diffraction limit spatial resolution. Literature reports of biological applications of tip enhanced Raman spectroscopy (TERS) are sparse with no studies having been reported on the imaging of biological systems. Our group has concentrated primarily on TERS nanoscale imaging of biological specimens in which critical parameters involve tip apex size, choice of material, stiffness of the scanning probe tip, acquisition time, signal to noise characteristics and imaging fidelity and reproducibility. In addressing TERS applications, we will focus on the challenges that are encountered in the biological area, as for example, studies involving model and intact cellular membranes.

(280) **FT-IR Gas Analysis: What We've Learned in 25 Years**

Martin Spartz[1]; [1]Prism Analytical Technologies, Inc.

Twenty five years ago only a few intrepid researchers used FT-IR for anything other than solid sample analysis for functionality or qualitative analysis. Due to many advances in the technology, both hardware and software, FT-IR s are now routinely used for both qualitative and quantitative analysis. In fact, many environmental testing firms now rely on FT-IR for most or all of their analytical field emissions testing work. Where chemiluminescence was once the EPA standard for NOx analysis, FT-IR is now a widely accepted alternative and in some cases the preferred analysis technique. This change is due to many subtle but important improvements that have been made to FT-IR systems over the last 25 years. These technical advances now allow transportable FT-IR systems to detect airborne gas concentrations from % down to single ppb levels and below. This presentation will focus on three areas. First, there will be a discussion on the state of technology in the 1980's and some of the underlying issues that existed with the technology. Second, there will be a discussion on where the technology is today and some of the subtle improvements and things learned that allow the technology to measure low levels of compounds with high precision and accuracy. Third, there will be a discussion on current FT-IR applications that demonstrate the importance of these subtle technical improvements.

(281) **Design Considerations and Best Practices, for the Implementation of a Fluorescence Spectrophotometer in Laboratory and Plant Pharma PAT**

Susan Bragg, Paul Davies, Expo Technologies

Monitoring content uniformity of powder blends by NIR is fast becoming an established technique. The limitations, challenges and benefits of this technique are known, the mechanics of plant installation are understood. However, drug development pushes forward with higher potency and lower dosage formulations that offer a new set of challenges for the PAT Scientist or Engineer. Fluorescence spectroscopy is emerging as a technique for both off line and real time content uniformity monitoring, to compliment NIR and offer some improvement in areas where NIR may not be ideal. Light Induced Fluorescence can offer very fast measurement speeds, sensitivity in the order of 100X that of NIR and a much simpler data handling route. This presentation considers the potential of light induced fluorescence as a PAT application together with the design criteria that allow it to move from a laboratory bench to the process environment. Measurement protocols and the approach to validation for routine use are also proposed. Where permissions allow, accounts of process studies and early feasibility work will be shared.

(282) **The Application of PAT to Continuous Processes**

Martin Warman[1], Justin Pritchard[1], Gregory Connelly[1], Aude Legos[2], Trevor Page[2]; [1]Vertex Pharmaceuticals Inc; [2]GEA Pharma Systems

The recent upsurge of interest in continuous processes for Pharmaceutical Development and Manufacturing is significant. Although recent to the Pharmaceutical industry, the conversion from batch to continuous processes is widespread in other industries, many of whom share similar unit operations. For example, continuous blending is commonplace in other industries, and in recent years, continuous blenders which claim cGMP compliance have also become commercially available for Pharmaceutical use. However, the successful use of continuous rather than batch blending is dependent on the recognition and understanding of its primary role: to take the variation in disparate individual feeds (API and excipients) and generate a uniform blend, such that each and every individual unit dose is of appropriate quality. Simply ensuring the continuous blender complies to cGMP maybe a regulatory requirements in the industry, but does not address the primary purpose of the continuous system, Additionally, the primary consideration for any measurement system used to monitor and control continuous blend performance is the scrutiny of scale. In a batch processes, we can statistically sample (spatially or over time) the blend process. In continuous blending, we require a different scrutiny scale. Effectively, the uniformity (or lack of it) is generated dose by dose, and any measurement system needs to be tailored to fit the specifics of the process it trends. This presentation covers, determining application specificity for measurement systems (used for monitoring and control of continuous blending steps) and how this application specificity can be used to ensure an appropriate measurement system is chosen. Data and comparisons from various types of vibrational spectroscopy will be shared, as well as comparing on-line and at-line measurement options.

(283) **Fundamental Studies in Powder Drying Using *in situ* Spectroscopic and Off-Line Particle Size Analysis Techniques**

Peter Hamilton[1], Elana Duff[1], David Littlejohn[1], Alison Nordon[1], Jan Sefcik[1], Paul Slavin[2], Paul Dallin[3], John Andrews[3]; [1]University of Strathclyde; [2]GlaxoSmithKline; [3]Clairet Scientific

In the pharmaceutical industry, drying of an active pharmaceutical ingredient is the final stage of primary manufacturing and takes place before processes such as blending and tableting are carried

out. Therefore, it is the final step to ensure that the particle size, shape and distribution of the active ingredient are correct for proper action of a drug in the body. However, it is known that phenomena such as attrition and agglomeration can take place during drying which directly affect the final product quality and this can lead to the reworking or ultimately scrapping of a batch resulting in a great loss of time and money. It is important to understand how the effects of such phenomena combine to affect drying efficiency and final product particle size, thus ultimately determining the success of the operation. Currently, there are no established on-line techniques to measure *in situ* particle size during drying and its effect on mixing within a dryer. Optical spectroscopy techniques, particularly NIR and Raman, are known to demonstrate particle size effects on spectra, and both have been used in this study. The Kaiser Raman Rxn1 with PhAT probe, and FOSS online 6500 NIR spectrometers were used to investigate the drying process of needle-shaped cellobiose octaacetate in a bespoke vacuum agitated filter dryer. A design of experiments approach was carried out to determine the effects on attrition and drying time of three factors, namely, agitation strategy, percent loss on drying and jacket temperature. Previous studies have demonstrated that particle size effects, observed in both Raman and NIR spectra, are preserved in spectra of wet powders. Both *in situ* techniques, along with off-line particle size analysis, have been valuable in gaining understanding of the drying process.

(284) **On-line Mass Spectroscopy in the Pharmaceutical Industry**

Charles Goss[1], James Rydzak[1], Gregory Gervasio[1]; [1]GlaxoSmithKline

The development of process understanding using real time *in-situ* PAT tools during process development and pilot plant optimization is becoming a more common practice in the Pharmaceutical industry. Mass Spectroscopy is one of the many useful techniques available in the PAT tool kit. Process Mass spectrometers allow continuous measuring of various solvent concentrations in the gas streams over a wide pressure range, making this technique suitable for numerous monitoring applications in the pharmaceutical industry. This presentation will discuss the applications of on-line mass spectroscopy in dryer, distillation and reaction monitoring for process understanding as well an efficiency and safety.

(285) **NIR Monitoring and Control of a Constant Volume Distillation**

Bob Cooley[1], Russ Fitzgerald[1], Delphi Burton[1], Ming Li Lim[2], Jeremy Yeo[2]; [1]GlaxoSmithKline - RTP; [2]GlaxoSmithKline - Jurong

Prior to the isolation of Pharmaceutical Intermediates and Active Ingredients, a solvent exchange is often performed to switch into the optimal crystallization solvent. These distillations are generally performed by either batch processing or constant volume distillation (CVD). When possible, CVD is preferred because it is typically a more efficient process, requiring less solvent, time, and energy. A critical component for implementing constant volume distillation is the determination of the endpoint, commonly achieved by monitoring the solvent composition in real time. This presentation will describe our recent experience using NIR to monitor a constant volume distillation, from development of the PLS model in the RTP lab, through electronic transfer of the calibration model and successful implementation in the Jurong Pilot Plant. It will also discuss the link between vessel and distillate composition via the Vapor-Liquid Equilibrium equation, as well as solvent recycling, a key sustainability goal for this process.

(286) **Combination of PAT and Data Fusion for the Characterization of a Chemical Process**

Thomas Dearing[1], Brian Marquardt[1]; [1]Applied Physics Laboratory

In the past few years governmental initiatives and advances in sampling systems have lead to an increased deployment of analytical technologies to characterize industrial processes. This proliferation of process analytical technology (PAT) has lead to rapid increases in the measurements performed and data generated. Traditional approaches would require the production of calibration and validation models for each analytical method. The information from each model would then be studied independently to characterize the process. This approach can lead to gaps in the data space which in turn leads to greater uncertainty and gaps in the process understanding. Data fusion provides a solution to this problem by fusing all of the analytical measurements into one continuous block of data. This new data block is then used to produce one calibration and one validation model. Data fusion will allow complete coverage of the entire process space significant decreasing uncertainty and providing optimal process characterization. This study has applied the concept of data fusion along with Process Raman, IR and NMR to characterize a series of differing petroleum samples. Process Raman spectroscopy offers information pertaining to the molecular backbone as well as symmetrical non-polar groups. IR spectroscopy yields information pertaining to hydrogen bonding and asymmetric polar groups. To this end each of the spectroscopic methods and the respective data produced are complimentary to one another. Process NMR provides information relating to the proton environments within a molecule. Process NMR is completely orthogonal to both of the other methods employed as it does not rely on a vibrational response from the molecules being examined. After collection, the respective spectra were corrected for baseline artifacts and light scattering effects. They were then scaled and fused together to form one continuous set of data. Results from the experiments using the fused data will be presented with an emphasis on design of experiments and multivariate modeling for the prediction of a series of petroleum process control metrics.

(287) **Utilizing High-Resolution Raman Spectroscopy to Examine Molecular Interactions Within Lithium Battery Electrolyes**

Wesley Henderson[1], Daniel Seo[1], Qian Zhou[1]; [1]NC State University

Raman spectroscopy is a powerful tool for scrutinizing the molecular-level ionic interactions that govern battery electrolyte properties. State-of-the-art electrolytes are composed of mixed aprotic solvents and a lithium salt. For the advanced lithium batteries intended for plug-in hybrid electric vehicles (PHEVs), there is a drive to replace the currently used solvents and salt with new solvents/salts or even to completely eliminate the volatile, flammable solvents by substituting them with ionic liquids (liquid salts). Electrolyte optimization has thus far been done by an Edisonian approach. The molecular-level solvent-ion and ion-ion interactions which govern electrolyte properties (conductivity, viscosity, wettability, volatility, etc.) remain poorly understood. These interactions result in specific solvates forming in solution including solvent-separated ion pairs (SSIPs), contact ion pairs (CIPs) and aggregates (AGGs) consisting of uncoordinated anions, anions coordinated to a single Li+ cation and anions coordinated to two or more Li+ cations, respectively. Utilizing a combination of phase diagrams, crystalline solvate structures and high-resolution Raman spectroscopy, it is possible to fully characterize such electrolytes thus greatly complementing work by other researchers to optimize multi-solvent electrolytes and efforts to understand and control the electrode-electrolyte interface, thereby enabling the rational design of electrolytes for a wide variety of battery

chemistries and applications (low/high temperature, high power, etc.) (i.e., electrolytes on demand).

(288) **Design and Self-Assembly of Surface-Enhanced Raman Scattering (SERS) Platforms: Building an Optical Biosensor**

Betty C. Galarreta[1], Peter R. Norton[1], Francois Lagugne-Labarthet[1]; [1]The University of Western Ontario

Fine tuning of the optical properties of metallic nanostructures is a topic of interest in the development of new sensors. The interaction of light with the conduction electrons of metals at the nanoscale level leads to surface plasmons, which can guide and enhance the local electromagnetic field. The enhancement effect observed at the vicinity of sharp metallic structures can be used in Raman spectroscopy to enhance the local signal of molecules and biomolecules by several orders of magnitude through the surface-enhanced Raman scattering (SERS) effect. Herein, we report the design of hexagonal arrays of gold nanotriangles on glass slides fabricated by electron beam lithography. The samples were both physically and optically characterized and modeled using finite difference time domain (FDTD) simulation. The calculations predict the polarized components of the electromagnetic field distribution around the nanostructures. Such an approach was used to select the optimal SERS platform designed for a specific excitation wavelength [1]. These SERS platforms were then functionalized with alkanethiols to create a self-assembled monolayer, which would act as a biomolecular receptor. The streptavidin-biotin system was selected for the design of bio-assembled surfaces because of its high guest-host affinity, the presence of four binding sites, and their high stability. We prepared a mixed self-assembled monolayer containing biotinalated groups, and then streptavidin was added to the system. These devices were then characterized and used to evaluate the enhancement of the Raman signals and their potential as biosensors. 1. Galarreta, B.C., et al., Phys. Chem. Chem. Phys. 2010, DOI: 10.1039/b925923f.

(289) **Applications of Raman Spectroscopy for Cancer Cells Detection**

Alexandru R. Biris[1, 3], Meena Mahmood[1], Yang Xu[1], Alokita Karmakar[1], Anindya Gosh[2], Ashley Fejleh[1]; [1]University of Arkansas, Applied Science; [2]University of Arkansas, Chemistry Dept; [3]National Institute for R&D, Romania

Raman spectroscopy is an extremely powerful technique that can be used to analyze the interactions between various nanomaterials and biological systems. Given the unique spectroscopic and scattering properties of carbon nanostructures, they can be successfully used as high Raman contrast agents while in the presence of biological backgrounds. Here we show that based on their characteristic G band structure, single walled carbon nanotubes can be used as high sensitivity agents for the successful detection of cancer cells down to single cell level. Furthermore, in the process of NIR laser irradiation exposure, the nanotubes can induce high heating levels, that are high enough for the thermal ablation of cells. A correlation between the species of carbon nanotubes and their corresponding heating properties under NIR laser exposure is presented.

(290) **Raman Spectroscopy of Algae Biopetroleum Hydrocarbons from Botryococcus braunii**

Taylor L. Weiss[1], Hye Jin Chun[2], Jaan Laane[2], Shigeru Okada[3], Tim P. Devarenne[1]; [1]Texas A&M University, Dept. of Biochem./Biophys.; [2]Texas A&M University, Dept. of Chemistry; [3]University of Tokyo, Agr. & Life Sci.

Botryococcus braunii is a colonial, green microalga able to synthesize massive amounts of hydrocarbons (up to 86% by biomass) which accumulate in intracellular lipid bodies and the extracellular matrix. Already a significant contributor to world petroleum deposits and capable of blooming to remarkably high densities, B. braunii could be well suited to large-scale production of high-quality, petroleum-equivalent hydrocarbons. The B. braunii B race synthesizes unique isoprenoid hydrocarbons called botryococcenes. The simplest botryococcene, C30, is synthesized inside the cell. C30 is then repeatedly methylated until it is predominately found as C34 botryococcene in the extracellular matrix, where a majority of botryococcenes are stored (~ 90%). The mechanism of botryococcene synthesis, methylation, and transport remains largely unknown. Our research seeks to understand the synthesis of C34 botryococcene both for its novelty and because methylation yields a superior botryococcene biopetroleum. We have begun Raman microscopy observations of botryococcene localization *in vivo* and are testing our model of botryococcene synthesis/accumulation in three steps. First, the unique Raman wavenumbers for differentially methylated botryococcenes were determined using a closely related, commercially available isoprenoid standard (squalene), botryococcene crude extract, and HPLC purified C30 , C31 , C32 , C33 , and C34 botryococcenes. Second, all botryococcenes, but notably derivatives too sparse for Raman, were used in theoretical Raman calculations. These calculations are supported by our experimental data, but also yield surprising results of their own. Third, the diagnostic Raman wavenumbers determined were used to produce cellular maps of specific botryococcene localizations.

(291) **Coupling Raman and Fluorescence for Confocal Imaging of Biological and Pharmaceutical Samples**

I. Chourpa[1], E. Munnier[1], A. Paillard[1], E. Allard[1], C. Linassier[1], S. Cohen-Jonathan[1], R. Lewandowska[3], E. Lancelot[3], E. Garcion[2], P. Dubois[1]; [1]Universite Francois Rabelais de Tours; [2]INSERM; [3]Horiba Jobin Yvon; [4]UMR

Development of modern approaches in disease diagnostics and therapeutics of cancer is a multidisciplinary domain supported by progress in both biology, nanotechnology and analytical methods. Among the latter, Raman and fluorescence confocal spectral imaging (CSI) occupies an important place since it is recognised as powerful tool for direct analysis of the biochemical molecular events in tissues and in living cells. We demonstrated recently that more detailed and complementary information from cells can be obtained by CSI based on coupling fluorescence[1] and Raman, namely SERRS (surface enhanced Resonance Raman scattering) spectroscopy[2]. Here, we will present the molecular analysis of biologic and pharmaceutic samples by using a novel microscopic tool that combines spectral and band-passing confocal imaging. The analytical advantages of such a coupling will be illustrated with the results of our research, focused on nanocarrier-mediated delivery of anticancer drugs to live cancer cells. The presented data will deal with cellular mechanisms of uptake of different nanoparticles (biocompatible SERS-substrates[3] and/or drug nanocarriers based on magnetic nanoparticles[4,5] or lipid nanocapsules[6]) as well as with anticancer drug distribution/interaction within cellular compartments. These data contribute to better understanding of molecular mechanism of action of the targeted chemotherapy. Acknowledgement We acknowledge financial support of Région Centre, France and of Ligue Nationale contre le Cancer, Comités Indre, Loir-et-Cher, Maine-et-Loire, France References 1. S. Vibet, K. Mahéo, J. Goré, P. Dubois, P. Bougnoux, I. Chourpa, Drug Metab Dispos. 2007, 35(5), 822-828 2. I. Chourpa, F.H. Lei, P. Dubois, M. Manfait, G.D. Sockalingum, Chem. Soc. Rev., 2008, 37, 993 - 1000 3. A. Shkilnyy, M. Soucé, P. Dubois, F. Warmont, M.L. Saboungi, I. Chourpa, The Analyst, 2009, 134, 1868–1872 4. E. Munnier, S. Cohen-Jonathan, C. Linassier, L. Douziech-Eyrolles, H. Marchais, M. Soucé, K. Hervé, P. Dubois, I. Chourpa, Int J Pharm 2008, 363(1-2), 170-176 5. A. Shkilnyy, E. Munnier, K. Hervé, M.

Soucé, R. Benoit, S. Cohen-Jonathan, P. Limelette, M.-L. Saboungi, P. Dubois, I. Chourpa, J. Phys. Chem. C, 2010, 114 (13), 5850–5858 6. E. Garcion, A. Lamprecht, B. Heurtault, A. Paillard, A. Aubert-Pouëssel, B. Denizot, P. Menei, JP. Benoit, Molecular Cancer Therapeutics, 2006, 5 (7), 1710-1722

(292) **Lean Raman Imaging for Rapid Assessment of Homogeneity in Pharmaceutical Formulations**

Stephanie Brown[1], Mike Claybourn[1], Chris Ashman[1]; [1]AstraZeneca

Solid dispersion formulations and drug-in-polymer matrices are increasingly being used by the pharmaceutical industry to enhance the solubility or bio-availability of active pharmaceutical ingredients (APIs). The degree of solubility or bio-availability enhancement, as well as properties such as chemical stability and physical characteristics, will be dependent on the homogeneity of the drug in polymer matrix. This presentation discusses the use of 'lean' Raman imaging to assess two performance-indicating parameters of a drug in polymer formulation, sedimentation of the API within a capsule formulation and the domain sizes of the individual components. The development of a screening method using Raman line mapping to allow rapid measurement of sedimentation of the API is discussed. This method offers significant efficiency gains as well as improved spatial information over wet chemistry techniques. Sample preparation is fast and straightforward and sample handling minimal. A simple statistical analysis of the data provides a measure of the degree of settling within the capsule. In addition, the development of a 'lean' Raman mapping technique using single line scans to assess drug and polymer domain sizes is described. This technique employs a simple peak ratio approach coupled with statistical analysis to provide a measure of the size of the drug and polymer domains without the need for acquisition of high pixel density images or multivariate analysis. This one-dimensional approach allows the critical information to be extracted from a Raman map and removes the complexity of comparing two-dimensional images. The results demonstrate that Raman spectroscopy can be used to provide rapid assessment of the homogeneity of drug in polymer formulations. The Raman mapping data are compared with the dissolution profiles and processing parameters of the samples tested and a strong correlation is shown between formulation homogeneity and dissolution behaviour. While acquisition of a single Raman spectrum is extremely rapid, Raman imaging has traditionally been limited by the long data collection times needed to acquire detailed images. The 'lean imaging' approaches described in this presentation have shown that information about critical parameters such as formulation homogeneity can be obtained rapidly without the need for detailed images or complex data processing.

(293) **Hydrogen Sensing with a Single Palladium Nanowire**

Reginald Penner[1], Fan Yang[1]; [1]Univ. of California, Irvine

Noble metal nanowires have attributes including strength, ductility, and chemical stability that make them attractive candidates for chemical sensing applications. We have developed a new method for preparing arrays of noble metal nanowires that involves the electrodeposition of metals (palladium, silver, platinum and gold) onto lithographically patterned glass surfaces. Under the conditions employed for nanowire growth, metal is deposited within this patterned photoresist layer on a sacrificial nickel electrode leading to the formation of polycrystalline nanowires that are up to 1 cm in length and 5-200 nm in lateral dimension. The palladium nanowires prepared using this method, called lithographically patterned nanowire electrodeposition or LPNE can form the basis for chemical sensors in which the resistance of the nanowire array is modulated by molecules that chemisorb at the surfaces of these metals. One particularly interesting example involves palladium nanowires in the presence of hydrogen. For this system, Pd nanowires respond to H2 exposure by becoming either more resistive or more conductive, depending on the details of nanowire and sensor fabrication. What is the origin of these resistance changes? In this talk, we focus attention on this issue and we describe how highly optimized sensors can be fabricated that detect hydrogen over a range from 2 ppm to 100% with response/recovery times as fast as 1 s.

(294) **Electrochemically-Fabricated Metal/Organic/Metal Junctions for Electronic Switching and Sensing Applications**

Francis Zamborini[1], Radhika Dasari[1]; [1]University of Louisville

In this talk, we will describe the electrochemical synthesis of Ag nano-micorwire/organic/metal junctions and there use in the fabrication of electronic-based switches and chemiresistive sensors. This strategy involves the deposition of organic thin films or monolayers on one electrode and the electrochemical deposition of Ag nano-micro wires at a second electrode. The Ag wire grows across a 5 micron gap, making contact with the second electrode to form the metal/organic/metal junctions. The use of thin film and monlayer deposition techniques allows a wide variety of organic molecules to be incorporated into the junctions with applications as molecular switches and chemiresistive sensors. The method is simple, fast, low cost and reproducible, allowing the fabrication of several devices rapidly and proper statistical analysis of the devices. The nanoscale Ag wire contact also serves to enhance the Raman scattering of molecules within the junction, enabling simultaneous spectroscopic and electrical characterization of the junction. This allows us to correlate the electronic properties with specific molecular changes in the junction for a detailed understanding of what controls the electonic properties.

(295) **Nitric Oxide-Releasing *in vivo* Glucose Biosensors**

Mark Schoenfisch[1], Ahyeon Koh[1], Dan Riccio[1], Bin Sun[1], Yuan Lu[1]; [1]University of North Carolina

Implantable glucose sensors for the management of diabetes continue to be plagued by unreliable performance in the early period after implantation and limited overall lifetimes. Much work has been put forward to study and describe these phenomena. Poor sensor performance is generally attributed to inflammation, capsule formation, sensor movement, and/or biofilm formation around the subcutaneously-implanted device. We are investigating the utility of outer polymeric coatings that release low levels of nitric oxide, a potent antimicrobial and wound-healing promoting agent, to mitigate undesirable poor tissue biocompatibility. Related to this, we are quantifying the effects of nitric oxide release on sensor performance characteristics. Herein, we will describe new methods for storing and releasing nitric oxide from the sensor-tissue interface based on nanotechnology. The use of nanoparticle as nitric oxide release vehicles enables tuning of the nitric oxide payloads and subsequent detailed study of the potential utility of this approach. In addition to introducing the synthesis and characterization of nitric oxide-releasing particles, the fabrication of miniaturized nitric oxide-releasing glucose biosensors and subsequent sensor performance studies will be described.

(296) **Ultrasensitive Electrochemical Detection for DNA Arrays Based on Silver Nanoparticles**

Danke Xu[1], Hui Li[1], Ziyin Sun[1], Hong-Yuan Sun[1]; [1]Nanjing University

Multiplexed DNA assay approaches are important for clinical diagnostics, environmental monitoring, biothreat detection and forensics since more and more genomic information as well as their recognizing partners has been known with the acceleration of genomics discoveries. Electrochemical transducers coupled with arrays of electrodes have often been used for detection of multiple species with different molecular DNA probes due to their high

sensitivity, small dimensions, low cost, and compatibility with micromanufacturing technology. In this work, a novel electrochemical detection approach was developed by using silver nanoparticles as electrochemical labels. Hepatitis B virus (HBV) sequence was employed as a sample model, and we have achieved a detection limit of 5aM (~120 molecules in 40ìL volume), demonstrating ultrasensitive measurement for DNA. The property of the electrochemical process involving silver aggregates was further investigated and the integrative oxidation of the silver tag was observed. Four kinds of capture oligonucleotides, which sequences are related to Epstein-Barr virus (EBV), Herpes simplex virus (HSV), cytomegalovirus (CMV) and HBV were immobilized on gold arrayed electrodes. The samples containing target nucleotides and the reported probes labeled with silver nanoparticles were incubated with the capture probes on the electrodes. The sandwish hybrids would be formed via hybridization and hybridized events were detected by different potential voltammetry. Based on the quantity of electricity of metallic silver oxidation, the amount of targets could be assayed. The presented method shows its advantages such as its rapid, parallel detection as well as high sensitivity. ACKNOWLEDGMENTS The authors acknowledge financial support of National Natural Foundation of China (Grant No. 20575079) and National Basic Research Program of China (973 Program, No. 2006CB910803). REFERENCES [1]Sassolas, A;Leca-Bouvier,B.D.;Blum,L.J. Chem. Rev.2008,108:109 [2]Drummond,T.G.; Hill,M.G.; Barton,J.K.. 2003, .21:1192 [3]Wang, J; Xu, D.; Polsky, R.J.Am.Chem.Soc., 2002,124:4208 [4] Dougan, J A; Karlsson,C; Ewen, S. W, et.al. Nucleic Acids Research, 2007, 35:11 [5] Lee,J.S.; Lyttonjean, A.K.R.; Hurst,S.J,et.al. Nano Lett.,2007,7:2112

(297) **Nanocatalysts for Electrochemical Energy Conversion: The Challenges for Synthesis and Characterization**

Keith Stevenson[1], Anthony Dylla[1], Sankaran Murugesan[1], Sebatien Verret[1], Salome Nagita[1], Corrinne Atkinson[1]; [1]University of Texas at Austin

This presentation will introduce strategies for synthesizing and characterizing the performance of nano-catalysts for electrochemical energy conversion; and will also highlight the development of new analytical tools for studying catalytic activity. Correlating the structure and reaction kinetic properties of catalysts as functions of size, shape, morphology and composition is a major challenge in homogeneous and heterogeneous catalysis. Yet obtaining information of this kind would facilitate the discovery and mechanistic understanding of new catalysts. Template-based syntheses using structure-directing agents such as dendrimers and polyol surfactants have facilitated our ability to more precisely control structural and compositional characteristics. In particular, we will highlight polyol-mediated approaches for preparing monometallic catalysts, multimetallic alloys and core@shell morphologies. Unfortunately, when evaluated in energy conversion processes, these new catalyst architectures are difficult to characterize by ensemble-averaging, bulk experimental methods. In this regard, we will describe the development of a new electroanalytical method for assessing the catalytic activity of water- soluble, colloidal catalysts of different size and shape.

(298) **Vertically Oriented Nanogap Substrates for Surface-Enhanced Raman Scattering**

Kyle Bantz[1], Hyungsoon Im[1], Nathan Lindquist[1], Sang-Hyun Oh[1], Christy Haynes[1]; [1]University of Minnesota

Surface-enhanced Raman scattering (SERS) is a powerful analytical signal transduction mechanism for the detection of analytes in aqueous environments, largely free from interfering water signals and capable of obtaining unique molecular signatures from analytes. Literature work suggests that large SERS enhancements are achieved when the analyte of interest is placed near a noble metal gap or crevice feature. In this work, new nanogap SERS substrates are developed using simple lithography patterning and atomic layer deposition to create vertically oriented nanogaps for large SERS sensing enhancement. The vertically oriented plasmonic nanogaps are formed between two metal structures by exploiting a sacrificial layer of ultrathin alumina grown using atomic layer deposition. Fabrication can be achieved with underlying masks fabricated by e-beam, optical, or simple nanosphere lithography. This technique gives us control over the gap size, shape, and orientation. To date, gap sizes as small as ~5 nm have been demonstrated. Additionally, the nanogap/nanosphere scaffold tunability makes it easy to achieve the desired optical properties and/or feature size for sensing applications. The average enhancement factors (EF) on the new nanogap substrates are at least two orders of magnitude higher than the traditional Ag film over nanospheres (AgFON) substrate. Analysis shows that the local enhancement factor at the nanogap is as large as 109. Overall, the tunabilty, large EFs, and ease of fabrication make these new nanogap substrates an exciting prospect for SERS sensing applications.

(299) **Protein Structure in the ESI Transition Regime: Where Does Solution End and the Gas Phase Begin?**

Ryan Julian[1]; [1]University of California Riverside

Protein structure elucidation via traditional approaches continues to be a time consuming and challenging endeavor, stimulating the investigation into alternative methods. Mass spectrometry and ion mobility have contributed several alternatives, some which intend to examine structure in solution and others purely in the gas phase. Fundamental to either approach is the fidelity by which information can be transferred into the gas phase. In the case of ion mobility, are solution phase structures themselves retained in the gas phase? For solution based methods, are mass shifts truly representative of the solution phase structure? Selective noncovalent adduct protein probing, or SNAPP, is a method intended to examine solution phase structures. SNAPP is well suited for detecting changes in protein structure which may be initiated by a variety circumstances, but does not reveal information that can be readily transformed into a three dimensional structure. Therefore, the principal targets for SNAPP are proteins which are “natively disordered” or systems which undergo structural transitions. Although it is clear that the method works and reveals information about solution phase structure, it is less clear how SNAPP operates. Recent results suggest that SNAPP experiments likely record information about protein structure in the transition regime of electrospray ionization. High fidelity is maintained when noncovalent adducts attach prior to any structural reorganization that may take place in the final gas phase structure. Experiments investigating this transition regime will be presented.

(300) **The Analysis of Protein Carbonyl Modifications from *in vitro* and *in vivo* Oxidative Stress**

Scott Gronert[1], David Simpson[1], Zafer Ugur[1]; [1]Virginia Commonwealth University

Protein carbonyls represent a common oxidative modification and involve the introduction of an aldehyde functional group on an amino acid side chain. These modifications can result from the direct oxidation of lysine, arginine, or proline by free radicals or by the addition of alpha,beta-unsaturated aldehydes (derived from lipid peroxidation processes) to lysine, histidine, or cysteine. The protein carbonyls have been targeted as potential biomarkers for oxidative stress and have been implicated in aging processes. We have developed a robust model system based on *in vitro* acrolein modification and used it to test the efficacy of a number of labeling,

isolation, and concentration tactics. All the tags are based on a hydrazide for binding to the protein carbonyl. In addition to the hydrazide, the labeling groups contain either a biotin unit for affinity chromatography or a hydrophobic group for enhancing ionization efficiency. The effect of linker type (hydrazide/biotin) has also been evaluated, both in terms of detection efficiency and chromatographic isolation/concentration efficiency. Selected approaches have been applied to biological samples.

(301) **When CID ≠ IRMPD and ECD ≠ ETD: Contradictions to Conventional Wisdom**

Gary Glish[1], Alessandra Ferzoco[1], Natalie Thompson[1], Daniel Thomas[1], Takashi Baba[1]; [1]University of North Carolina

There are several techniques to activate ions in tandem mass spectrometry (MS/MS) experiments. The most common is collision-induced dissociation (CID). This is considered a "slow heating" method because internal energy builds up over time from multiple collisions of the parent ion with the collision gas. Another slow heating method is infrared multiphoton photodissociation (IRMPD). Just like CID, internal energy is accumulated over time by sequential absorption of IR photons. Thus, the conventional wisdom is that these two activation technique should give very similar MS/MS spectra. However, we have recently discovered examples where this is not true. Another form of activation is capturing an electron by a multiply charged parent ion. This can be a bare electron via electron capture dissociation (ECD) or an electron transferred from an anion, electron transfer dissociation (ETD). Again, conventional wisdom is that these process are the same and should lead to the same product ions. Recent results have shown this is not always true. In this presentation these contradictions to conventional wisdom will be discussed.

(302) **Structures and Energetics of Transition Metal Cation N-Donor Ligand Complexes from Collision-Induced Dissociation and Theoretical Studies**

Mary T. Rodgers[1], Nalaka S. Rannulu[2], Holliness Nose[1]; [1]Wayne State University; [2]University of New Orleans

The interactions between copper cations in the +1 and +2 oxidations states and several N-donor ligands are probed experimentally by examining the kinetic energy dependence of the collision-induced dissociation (TCID) of copper bound complexes in a guided ion beam tandem mass spectrometer. These experimental studies are supported and enhanced by theoretical electronic structure calculations using two density functional theory methods, B3LYP and BHandHLYP and the 6-31G* and 6-311+G(2d,2p) basis sets for optimization and single point energy calculations, respectively. Our studies include a variety of complexes of the nature $Cu^{n+}L_x$, where L = pyridine, 2,2'-dipyridyl, and 1,10-phenanthroline, and x varies between 1 and 6 (or over the range of values experimentally accessible). These systems allow us to probe the influence of a variety of properties on the binding interactions in these complexes including: the charge, electron configuration, and sd-hybridization of the metal cation, the flexibility and chelation interactions with the ligand, and the extent of ligation. In all cases, the complexes to Cu^+ dissociate via simple CID via sequential loss of intact ligands ($Cu^{n+}L_x \rightarrow Cu^{n+}L_{x-1} + L$). In such cases, analysis of the CID cross sections allows accurate sequential bond dissociation energies (BDEs) to be extracted. In contrast, the complexes to Cu2+ exhibit richer chemistry. Both simple CID via sequential loss of intact ligands as well as dissociative charge transfer resulting in reduction of the copper cation ($Cu^{2+}L_x \rightarrow Cu^{+}L_{x-1} + L^{+}$) are observed. Analysis of the dissociative charge transfer cross sections allows the Coulomb barriers to be determined. The trends in the binding as a function of the charge on the metal cation, the identity of the ligand, and the extent of ligation are examined and discussed. Comparison of theoretical and experimental results also allows evaluation of the accuracy of the theoretical models employed.

(303) **Characterization of Peptide-Metal Complexes: To ESI or Not to ESI**

Allison Danell; [1]East Carolina University

We have found a significant new problem in the ESI-MS of zinc-peptide systems that may contribute to discrepancies in reported dissociation constants (Kd). During the characterization of zinc-peptide systems, we have observed zinc retention inside the stainless steel emitter of a conventional ESI source. Deposition occurs under oxidizing and reducing conditions, regardless of the ESI polarity, making electrochemical deposition of the metal unlikely. In fact, it occurs even with no potential applied. We have shown that using a nanoESI source equipped with a glass emitter to introduce the same zinc-peptide samples dramatically increases the signal of intact zinc-peptide complexes, confirming that the loss of zinc in the conventional ESI source affects zinc-peptide equilibria. This deposition removes a significant amount of zinc ions from the solution, impacting the resulting mass spectral intensities used to quantify the amount of the zinc-bound species and, subsequently, Kd values. This phenomenon may represent a significant obstacle in the ESI-MS analysis of all peptide-metal systems. Hence, we have investigated several peptide-metal complexes under different conditions: systems with a variety of known binding strengths, metal co-factors, and at different infusion flow rates. Phytochelatins and several synthetic peptides designed to bind cadmium, cobalt, copper, and/or zinc have been obtained. Dissociation constants vary from 10-4 to 10-12 M. After infusing the solutions through the ESI emitter, fractions were collected for off-line atomic emission spectroscopy analysis. Preliminary results indicate no significant correlation between Kd value and degree of zinc deposition, but flow rate does play a role. Zinc is deposited to a lesser degree at higher (2.00 mL/hr vs. 0.250 mL/hr) flow rates. Concentrations drop by 10 to 25% in concentration when comparing infused and control zinc samples. Our current protocol produces working solutions with zinc concentrations only 1.0 to 1.5 times the limit of quantitation, so further experiments of pooled zinc-containing samples are being conducted to obtain more reliable concentration determinations. Cobalt does not appear to be deposited, because concentrations of cobalt in infused and control samples were unchanged (within estimated error). Additional divalent metal cations (cadmium, copper) bound to several different peptides currently are being analyzed.

(304) **MS Approaches for the Investigation of Tertiary and Quaternary Structure of Nucleic Acids**

Daniele Fabris; [1]University at Albany

The observation that less than 1.5% of the human genome codes for actual proteins has lead to the realization that sequence information alone is insufficient to elucidate the function of the vast majority of nucleic acids in living organisms. The recent discovery of riboswitches has keenly reasserted the critical role played by higher-order structure in determining the function of non-coding elements. Beyond sequencing, MS-based approaches can provide direct information about base-pairing and long-range interactions, which respectively define the secondary and tertiary structure of nucleic acids. The ability to observe intact assemblies with other nucleic acid elements and cognate proteins enables the investigation of their quaternary structure. For these reasons, we have been exploring both solution and gas phase approaches to support the 3D structure determination of nucleic acids and their protein assemblies. The talk will cover the implementation of electrospray ionization (ESI) with Fourier transform ion cyclotron resonance (FTICR) mass spectrometry to the characterization of recurring modules that mediate known non-covalent interactions in

ribonucleoprotein complexes (RNP's). This application relies on the ability of ESI to transfer relatively weak non-covalent complexes to the gas-phase without inducing unwanted dissociation. The possibility of detecting such modules in unsolved samples will pave the way for their complete structure elucidation by homology modeling strategies. Indeed, once the presence of these recurring motifs is recognized, their high-resolution structures could be employed as modular building blocks to assemble a full-fledged 3D model for the entire substrate of interest. The talk will also illustrate the integration of structural probing with MS detection (known as MS3D) to obtain valid spatial constraints for the modeling RNA structures inaccessible to established high-resolution techniques. Mono-functional probes are employed to identify nucleotides that are not involved in pairing interactions, thus leading to the elucidation of elements of secondary structure. Bifunctional reagents are instead capable of crosslinking regions juxtaposed by the substrate fold, thus revealing the position of long-range interactions. These types of constraints have allowed us to solve the previously unknown 3D structure of the HIV-1 Psi-RNA, the region of viral RNA that mediates the processes of genome recognition, dimerization, and packaging.

(305) **Archaeometry: Combining Analytical Technique with Archaeological Interpretation to Find a Meaningful Relationship**

Michael D. Glascock[1]; [1]University of Missouri

Archaeometry is about the application of techniques from chemistry and other fields of science to seek answers concerning questions about ancient man and his materials. Studies of stone tools, ceramics, pigments, sediments, metals, bones, and other archaeological materials are advancing our understanding of past human behavior. As a consequence of increased collaboration between archaeologists and chemists, we are learning more about prehistoric human migration, resource exploitation, social interaction and exchange, dietary habits, and other choices made by our ancestors. Examples of collaborative research carried out at the University of Missouri Archaeometry Laboratory will be presented.

(306) **XRF Analayis of Elementally Non Uniform Materials**

Bruce Kaiser; [1]Bruker AXS

A detailed theorectical discusion of the physics of x ray flourecsence with be given as it relates to the analysis of elementally non uniform materials insitu. The broad misuse of handheld xrf analysis is a result of researches using the technology without fully understanding the phyiscs behind the measurement technique. This presentation will address the phyiscs and present several example of both misuse and appropriate use of the technology when analysis various materials in the lab or the field.

(307) **Spectroscopic Investigations of Archaeological Sample from the Coriglia, Castel Viscardo Excavation Site, Italy**

Mary Kate Donais[1], Anna Daigle[1], David George[2]; [1]Saint Anselm College Chemistry Department; [2]Saint Anselm College Classics Department

The Coriglia, Castel Viscardo site is 8 km northwest of Orvieto overlooking the point at which the river Paglia turns and widens. The location is important since it links easily with the Tiber valley, the Via Cassia, and the Via Traiana Nova, a few hundred meters from it. It was discovered by agricultural activity in the late 1980s and first explored in 1990–1993 by the Soprintendenza per i Beni Archeologici dell' Umbria. The elemental compositions of various archaeological samples including deposits from inside a water drainage system, lead pipes, lead connective keys, mortars, tiles, and frescos have been determined. Results for *in-situ* analyses by portable x-ray fluorescence spectrometry will be presented. Graphical evaluations of data using Matlab and chemometrics results using The Unscrambler will be discussed. The impact of this archaeometric work on excavation strategies will be highlighted. Our muti-disciplinary approach and involvement of undergraduate students from both natural science and humanistic disciplines will be included.

(308) **Handheld XRF Analysis of the 6000 Year Old Nahal Mishmar Hoard of Copper Alloyed Artifacts**

Aaron Shugar[1]; [1]Buffalo State College

The Nahal Mishmar hoard of copper objects dates over 6000 years old. This fascinating collection of complex copper alloyed artifacts has been studied for years but no complete comparison of their composition has been previously undertaken. Handheld XRF was used to collect compositional data for the entire collection of over 400 artifacts. Issues addressed include surface contamination and corrosion and dealing with a high alloy and potential elemental segregation.

(309) **LA-ICP-MS Microsampling of Human Bones: The Dynamic Interaction between Sample, Introduction Method and Target Data for Organic Minerals**

Ian Scharlotta[1]; [1]Baikal Archaeology Project, University of Alberta

The nature of the relationship between observed analytical data and the target information desired by researchers is an ongoing concern within archaeological research. Interpretation of the provenance of skeletal materials can be altered at a number of stages between deposition and data interpretation. Prevailing wisdom holds that diagenetic changes will leave distinct signatures on the bone chemistry of a sample. A combination of data quality checks and laboratory procedures intended to correct for diagenetic alteration ensures that analytical data produces only the desired target data. Such measures generally do little to examine the microstructure of bones, proceeding via the assumption that laboratory procedures are effectively removing diagenetic, and only diagenetic, overprinting or alterations. Post-depositional chemical alteration will generally proceed via water contact with the skeletal materials, primarily altering (leaching, depositing) portions of bone already open to contact, namely on the surface and through the Haversian system. The nature of long bone formation and the pathways of interaction between bone samples and the burial environment strongly suggest that portions of the bones disconnected from the arterial system are resistant to diagenetic alteration. Preliminary work on the nature and progression of chemical changes in the bone matrix due to microbial attack using laser ablation inductively coupled plasma mass spectrometry have supported the presence of unaltered portions of bone within diagenetically altered bone and that useful data remains accessible. For mobility studies, tooth enamel is the preferred material due to its resistance to diagenetic alterations, leaving bone samples either neglected or regarded with suspicions of providing spurious data. Laser ablation microsampling of long bone samples was conducted to examine the nature of intra-osteon variability of elemental and isotopic (87Sr/86Sr) data as a means of determining the potential for refinements in the usage of bone samples for mobility research in broad terms and the timescales accessible, for portions of bones identified as having diagentic alteration to be used effectively for compositional and isotopic analysis and thus the scope of available materials for mobility research.

(310) **From Phosphorylation to Metalloproteins as Biomarkers for Hemorrhagic Stroke**

Joseph Caruso[1], Karolin Kroening[1], Yaofang Zhang[1], Renee Easter[1]; [1]University of Cincinnati

Modern techniques in mass spectrometry and combinations of these can go a long way to solving complex environmental and biomedical problems. Metallomics today purports to study various

trace metal species and their interactions with each other in animals or plants. A particular type of stroke is known as subarachnoid hemorrhage which occurs in the space between brain and skull. However, a complication of this stroke is often cerebral vasospasm, CV, a debilitating and often deadly result. With combinations of elemental and molecular mass spectrometries we have begun studies in hope of discovering biomarkers that would signal appropriate intervention to preclude the CV condition. Studies are progressing with phosphorylated proteins, metalloproteins and selenoproteins and these studies will be highlighted. It is notable that a large amount of putative metalloproteins have been found utilizing ICPMS and ESI-MS, and beyond this some exciting results showing selenopeptides and protein have been found by LC-MALDI. All of these will be discussed.

(311) **Metallomics for Non-Covalently Attached Metalloproteins: What are the Limitations?**

James Holcombe[1], Isaac Arnquist[1], Haley Finley-Jones[1]; [1]Univ. of Texas at Austin

The importance of characterizing protein-metal relationships has been well established in the literature. However, there currently exists no rapid screening technique that provides information for a large mixture of proteins, such as that found within a cell. Additionally, despite representing a small fraction of proteins, most work has focused on covalently bound metals. This research seeks to develop a rapid technique to determine the metal-protein association for non-covalently attached metals. In order to achieve this goal, three major challenges must be overcome: 1) With thousands of proteins within a cell and ca. 30% of them associated with a metal, a high resolution separation technique is needed; 2) Protein concentrations vary over several orders of magnitude so sensitivity and dynamic range are critical; and 3) The formation constants and complex lability cover a wide range of values, including labile, weak complexation that result in metal loss during the separation. Blue native polyacrylamide gel electrophoresis (BN-PAGE) was utilized as the protein separation technique, and separation running buffers were loaded with metal to retain metal-protein association. The separation was followed by a modified electroblot with chemically modified blotting membranes prepared especially for the purposes of studying labile metalloproteins. We will discuss the possibility of using the electroblotting data to gain information regarding equilibrium constants and binding kinetics. LA-ICPMS is utilized for metal detection directly off the electroblot membrane. A novel tool that allows solution sample introduction with a conventional pneumatic nebulizer, eliminating the uncertainty and quantitative challenges of LA, has been explored.

(312) **Examination of Trace Arsenic Species in Fruit Juices by LC-ICPMS**

Kevin Kubachka[1], Traci Hanley[1], Nohora Shockey[1]; [1]US Food and Drug Administration

Arsenic speciation has been an intriguing area of research for many years. Due to the significant difference in toxicity among arsenic species, it is imperative to know more than just the total arsenic concentration of a food product when assessing its health impact. Recent media attention surrounding arsenic levels in commonly consumed fruit juices, such as grape and apple juice, lends to the importance of understanding arsenic speciation in such juices. Methods have been developed for juice and other similar matrices to speciate various arsenicals using liquid chromatography with inductively coupled plasma mass spectrometry (LC-ICPMS). In the course of analyzing juices and juice concentrates with arsenic concentrations at or above 23 ng As g-1 (in ready-to-drink (RTD) juice), we have encountered samples which contain several unidentified peaks in the m/z 75 trace at low levels. Preliminary research suggests that these peaks are arsenic containing and they have not been reported in the literature for this product type. The purpose of this work is to provide insight to the properties of these compounds by utilizing LC-ICPMS, including various chromatographic methods and sample preparation techniques.

(313) **Metallomics Approach in Diabetes Research: Relation between Metal/Metalloid Status and Some Clinical Parameters, Typically Evaluated in Diabetic Patients**

Katarzyna Wrobel[1,2], Kazimierz Wrobel[1,2]; [1]University of Guanajuato, Department of Chemistry; [2]Metallomics Center of America

Diabetes has been classified as the fourth leading cause of global death by diseases and an estimation has been made that the number of people with diabetes will double worldwide by 2030. It is not surprising though that multidisciplinary research activities aimed at preventing, treating, and curing diabetes are the priority. In particular, there is accumulating evidence that the metabolism of several trace elements is altered in diabetes mellitus and that certain metals/metalloids might have specific roles in the pathogenesis and progress of this disease. It is well established that metal overload promotes oxidative stress which is considered one of the principal factors in diabetes. Within this sense, some elements (Cu, Cr, Fe) undergo redox-cycling reactions, while others (Hg, Cd, Ni) deplete cellular antioxidants (GHS). Furthermore, the deficiency of micronutrient (Se, Zn, Cu, Cr, Mn) promotes glucose intolerance. It should also be mentioned that different chemicals forms of V, Zn, Mo and Se present insulin mimetic properties and have been used in diabetes treatment. Several studies have approached the evaluation of trace element status in diabetic patients. The determinations have been made preferentially in biological fluids, hair and nails. Some studies have focused a single element in order to understand its specific biological role, yet multi-elemental determinations have also been carried out. However, the main objective of the multi-elemental determinations was to compare diabetic versus non-diabetic subjects and the reported results indicate higher serum concentration levels of Cu, Pb, As, Cd, Ni, Al and lower levels of Se, Cr, Zn y Mn in diabetic patients. The purpose of this work was to gain a further insight on the global role of metal/metalloids in diabetes. In such metallomics approach, ICP-MS determination of V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Mo, Hg, Cd and Pb was accomplished in serum and urine samples in two groups of diabetic patients: (1) without and (2) with complications. In parallel, several anthropometric and clinical parameters typically used for the evaluation of these patients, were assessed and the statistic multivariate analysis was performed in search of possible relationships existing among metals/metalloids and these parameters. The results obtained provide new interesting data suggesting that molybdenum might be involved in the progress of disease.

(314) **Application of Elemental and Molecular Mass Spectrometry and qNMR for the Determination of Arsenobetaine**

Zoltan Mester[1], Anthony Windust[1]; [1]National Research Council

Motivation for the speciation of arsenic in natural tissues lies in the need for estimation of risk associated with consumption of food products, the elucidation of metabolic processes and advances in analytical methodology. In this study synthetic deuterium labeled arsenobetaine has been characterized by HPLC ICP MS and HLPC orbitrap MS whit the intent of using it as an isotope dilution spike for the determination of AsBet in biological samples particularly in a candidate reference material. The purity of arsenobetaine standards has also been assed by qNMR. Results obtained by various instrumental approaches will be presented and the advantages and pitfalls will be discussed.

(315) **Measurements of Metals in Microbes - New Metallomics Techniques**

David W. Koppenaal[1], M Liz Alexander[1], Himadri Pakrasi[2], Jana Stockel[2], Charles J Barinaga[1]; [1]Pacific Northwest National Laboratory; [2]Washington University, St Louis

Microbes utilize metals advantageously for a variety of biological processes, and have developed a multiplicity of mechanisms for assimilating, incorporating, transporting required metals and nutrients. Photosynthetic organisms, such as the cyanobacteria, are under study by us, and we are combining proteomic, metabolomic, and metallomic techniques to better understand the dynamics of their membrane structure, function, and metabolism. The cyanobacterium Cyanothece species 51142 has been extensively studied to elucidate its remarkable ability to assimilate carbon by day (photosynthesis) and nitrogen (fixation) by night, through well regulated, temporally- controlled diurnal cycles. We will report the development of new techniques, using inductively coupled plasma and quadrupole time-of-flight mass spectrometries, to characterize the biological roles and interactions of various metals and metalloids in microbial systems. It is clear that new techniques and approaches are needed for such studies and to better understand the biology and associated environmental roles and interactions of metals and their molecular associations.

(316) **What Chemometrics Tells Us About the Design of Optical Instruments**

Karl Booksh[1]; [1]University of Delaware

The field of chemometrics forms the basis of the guiding theory of analytical chemistry. In this presentation we will discuss some counter-intuitive recommendations for the design and implementation of optical instrumentation that are derived from analysis of chemometric methods. First, we will illustrate the interaction between data collection strategies and data analysis methods. Second, multivariate regression vectors will be demonstrated to contain the optimal experimental design for collection of optical data for quantitative analysis. Using the regression vector to determine the data collection schedule is shown to improve the RMSEP by up to 40%. Finally, we will analyze the trade-off between sensitivity and selectivity with the goal of constructing an instrument with optimal resolution and S/N ratio for a particular application.

(317) **Traps and Pitfalls When Applying Chemometrics to Biomedical Problems**

Jerome Workman[1]; [1]Technology Business Associates

Are chemometric approaches, rather than improved signal quality, the primary answer to most analytical applications as we repeat the refrain, “Math is Cheaper than Physics?” Or do robust modeling solutions for challenging spectroscopic applications require a more comprehensive strategy to provide the highest quality signal combined with appropriate preprocessing and calibration steps. A dual approach including hardware modifications combined with chemometric approaches involves altering instrument design characteristics specific to the analytical application requirements. In addition, one must ask if automated expert algorithms must be included for quality and performance monitoring in near real time. Comprehensive strategies for applying chemometric methods include: experimental design optimization relative to calibration, data selection and preprocessing, outlier or ‘non-analysis’ sample detection, upset condition monitoring or mitigation, improved modeling approaches, calibration transfer strategies, and continuous monitoring and quality control metrics. This paper discusses the potential traps and pitfalls when applying ‘standard’ approaches for accurate prediction and monitoring of challenging spectroscopic biomedical applications.

(318) **Nocturnal Hpoglycemic Alarm Based on Noninvasive Near-Infrared Spectroscopy**

Gary Small[1]; [1]University of Iowa

Near-infrared spectroscopy has gained popularity in automated monitoring scenarios because minimal sample preparation is typically required and because rugged, relatively low-cost instrumentation is available that can be installed at the measurement site. In this presentation, near-infrared spectroscopy will be employed in a real-time monitoring scenario, coupled with the use of pattern recognition methods to implement threshold monitoring of the analyte concentration. The application addressed here focuses on the detection of nocturnal hypoglycemia (low glucose levels) in diabetic patients. Since there is no obvious symptom before hypoglycemia, the occurrence of this condition during sleep can lead to serious health consequences for the patient. It is thus desirable to develop a nocturnal hypoglycemic alarm which will wake diabetic patients during sleep if hypoglycemia occurs. Currently, the standard method to monitor blood glucose levels is a test-strip procedure based on the collection of a small sample of capillary blood. However, this approach suffers from invasiveness and intermittence and is unsuitable in a continuous monitoring scenario while the patient is asleep. To address this limitation, we are developing near-infrared spectroscopic methods for this application. For an *in vivo* measurement, application of infrared light to tissue is painless and can be applied continuously. In this research, we are employing supervised pattern recognition methods to identify the occurrence of hypoglycemia from an analysis of the recorded spectra. Data will be presented from both *in vitro* measurements using two different model systems, as well as from direct *in vivo* tissue measurements of laboratory rats. With both types of measurements, near-infrared spectra are collected continuously during glucose excursions designed to simulate the nocturnal profile of a sleeping patient. The presentation will focus on how to develop a robust and stable pattern recognition model that can detect hypoglycemic events and can account for time-based spectral variation.

(319) **Spectrochemical Monitoring of the Chemical Composition of Microalgae in Response to Changing Environmental Conditions**

Frank Vogt[1], Edward Duranty[1], Rebecca Horton[1], Morgan McConico[1]; [1]University of Tennessee, Department of Chemistry

Human activities impact aqueous ecosystems in complex and interrelated ways which makes their assessment non-trivial. Since measurements of a single chemical parameter would not yield a comprehensive assessment of such impacts, innovative approaches are required. Microalgae biodiversity has been observed to chemically adapt to their environments and are potential indicators of chemical shifts. Since most relevant compounds are infrared-active, FTIR spectroscopy in combination with chemometrics has been utilized for identification of algae species. Quantification of chemical changes, however, would open novel pathways for better understanding, assessing and potentially predicting human impacts on marine ecosystems. In a first study, environmental changes were simulated in lab experiments by culturing several different microalgae species under different nutrient sources (nitrogen and carbon) and concentrations. After growing sufficient biomass, algae samples were dried, mixed with KBr powder and pressed into pellets of known algae weight percentages; these pellets were then analyzed by means of FTIR spectroscopy. For first concentration predictions of multiple, selected components, standard Principal Component Regression (PCR) has been applied; calibrations were based on a spectral database compiled in our lab which comprises concentration series of 30+ analytes (approx. 1,500 samples). This database mainly contains representatives of lipids, amino acids, proteins, carboxylic acids, mono- and polysaccharides. However,

solid samples of biological materials impose several challenges regarding calibration and data evaluation. Light scattering on microscopic algae cells embedded in the KBr pellets causes baseline shifts; furthermore, variations in sample thickness (=absorbance pathlength) result in unwanted absorbance fluctuations. Both challenges require innovative data pre-processing methods to correct for such artifacts. A novel method for baseline correction has been derived which corrects each spectrum individually and does not require user input. Furthermore, a pathlength normalization has been developed correcting for fluctuations in sample thicknesses. Comparing concentration prediction with and without corrections clearly demonstrated an enhanced concentration precision. Current investigations determine the feasibility of such a calibration along with methods to minimize impacts from analytes not contained in the calibration, a common challenge when analyzing biomaterials. An approach based on detecting local (in wavelength) inconsistencies between the calibrated model and the unknown data will be presented.

(320) **Classification of Individual Phytoplankton Cells via Imaging Multivariate Optical Computing**

Laura Hill[1], Tammi L. Richardson[1], Timothy Shaw[1], Michael L. Myrick[1]; [1]University of South Carolina

Linear discriminant analysis (LDA) has been used to classify individual plankton into one of three target species (Emiliania huxleyi, Thalassiosira pseudonana, and Synechococcus sp. (pink)) based on the fluorescence excitation spectra for individual cells in the wavelength range 350-650 nm. A goal of our work has been to develop a method for performing the same discrimination using photometer-like instrumentation that would render the measurement feasible in the field. This instrument is based on multivariate optical elements (MOEs), a form of optical interference filter. The optical system used for experimental confirmation of the modeling will be illustrated and the classification of the phytoplankton cells will be shown.

(321) **Detection of Mastitis in Cows during Milking Using DRIFTS and the Wavelet Packet Tree to Mine NIR Data**

Barry Lavine[1], Nikhil Mirjankar[1], Roumiana Tsenkova[2]; [1]Oklahoma State University; [2]Kobe University

A two-step procedure for analyzing spectroscopic data was applied to diffuse reflectance spectral data of milk obtained from healthy and mastitic cows. First, the wavelet packet tree is used to denoise and deconvolute the spectra by decomposing each spectrum into wavelet coefficients that represent both high and low frequency components of the signal. This decomposition process is iterated through successive wavelet packets until the required level of signal decomposition is achieved. Second, a genetic algorithm for pattern recognition analysis is used to identify wavelet coefficients that can classify the diffuse reflectance spectra by the disease state of the cow (mastitis versus normal). The pattern recognition GA utilizes both supervised and transverse learning to identify wavelet coefficients that optimized clustering of the spectra by class in a plot of the two or three largest principal components of the data. The results of this study although preliminary in nature indicate that period of lactation and the quarter from which the milk sample has been obtained do not appear to be significant covariants in this classification problem. However, the identity of the cow providing the milk sample has been determined to be a confounding factor influencing the classification of the data. The use of DRIFTS and pattern recognition analysis as a potential method to detect mastitis in cows during milking is critically assessed as part of this study.

(322) **Raman Scattering as a Probe of Structure Development in Electro-Spun Fibers**

Bruce Chase[1], John Rabolt[2], Giri Gururajan[3]; [1]Pair Technologies LLC; [2]University of Delaware; [3]Connoco Phillips

Polymeric fibers play a significant role across a wide variety of markets, ranging from commodity markets like apparel to high value markets such as high strength ballistic vests. Fibers can be spun in a variety of processes, both solution based and melt based. The spinning process itself has a large impact on the structure/property relationships in the fibers. One of the few probes which are sensitive to molecular structure and orientation is Raman scattering. We are currently focused on a fiber spinning process known as electro-spinning. A high voltage potential applied to small diameter needle containing either polymer solution or polymer melt will result in very fine (sub micron) diameter fibers being produced. This process can produce fibers in metastable structural forms and with variable degrees of chain orientation. Polarized Raman scattering can be used to probe both structure and orientation on static samples as well as fibers during the electro-spinning process. Results from several different polymer systems will be shown.

(323) **Colorimetric-Solid Phase Extraction (C-SPE): Water Quality Monitoring for Crew Health on the International Space Station**

Marc Porter[1]; [1]Nano Institute, University of Utah

Water quality on the International Space Station (ISS) is presently assessed when samples are returned to Earth via the Space Shuttle. Several months, however, may pass between sample collection and analysis, limiting the ability to counter an on-board contamination event and compromising sample integrity. Iodine and silver, which are biocides used in the U.S. and Russian spacecraft potable water systems, must be held at levels that prevent bacterial growth and continually monitored to with respect to crew health. A comparable need exists for the detection of heavy metals, toxic organic compounds, and microorganisms. It is therefore critical that rapid, on-board methods be created to monitor trace quantities of several indicators of quality in spacecraft drinking water supplies. As part of a toolbox to meet these needs, our laboratory, in collaboration with scientists and engineers at NASA's Johnson Space Center, has developed colorimetric-solid phase extraction (C-SPE), which is currently being tested on ISS. C-SPE is a sorption-spectrophotometric platform that entails the selective extraction and concentration of analytes by a membrane impregnated with a colorimetric reagent, followed by quantification of the analyte on the membrane surface using a diffuse reflectance spectrophotometer. We have thus far designed and tested C-SPE methods for monitoring trace amounts of iodine and silver, as well as nickel and lead. We have also devised methods to extend C-SPE to determinations of formaldehyde, pH, cadmium, and arsenic. This presentation reports on these developments, including results from tests in microgravity simulations via KC-135 and C-9 flights and recent ISS data.

(324) **Evolution Trend in Raman Spectroscopy Instrumentation**

Jun Zhao, Juergen Sawatzki; [1]Bruker Optics

The revolution in Raman spectroscopy brought about by the introduction of FT-Raman, charge coupled device, laser rejection filters, and fiber optics continues today. Instead of standardizing to a common platform, commercially significant instrumentation is diverging into several distinct markets. In the portable Raman sector, an explosive growth in recent years was fueled by government demands for use by first responders and the military. In the laboratory, while high end instruments continue to add more capability, the emergence of the Raman microprobe as an attachment to standard optical microscopes is bringing Raman

micro analysis to a broader range of users. These miniaturized Raman microscopes are typically smaller, lighter and less expensive than the side-by-side mounted embodiments, however, they can be considerably less complicated to build and to operate, very photon efficient, while retaining the majority of the important capabilities of their larger cousins, and taking full advantage of the many visualization methods of the optical microscope. Automatic calibration and simplified software user interface make them more user friendly. In the industrial setting, once the spectroscopy proves to work, the success of a Raman analyzer installation often rest on the system robustness and long term reliability of the probes. System robustness can be enhanced with continuous, automated wavelength calibration. In systems employing diode or solid state lasers, simultaneous laser wavelength calibration is necessary to maintain stability.

(325) **Characterizing Mechanisms and Dynamics of Emulsion Free Radical Polymerization by *in-situ* Raman Spectroscopy**

R. Thomas Cambron[1], Ed Grundner[1], Tom Desmarais[1]; [1]Procter and Gamble

Characterization of an emulsion free radical vinyl polymerization by *in-situ* Raman spectroscopy provides valuable insight into the dynamics of this reaction and the relative reactivity of vinyl groups of the monomers. Time-resolved Raman spectra collected during the polymerization reaction were subjected to multivariate spectral analysis combined with two-dimensional (2D) correlation spectroscopy to elucidate the fine details of the complex reaction process. Raman peaks associated with the monomers and polymer was identified using generalized 2D correlation analysis of time-resolved Raman spectra. Cross peaks in the 2D Raman correlation spectra indicate one of the monomers is consumed at a slightly faster rate than the second monomer. Multivariate Curve Resolution (MCR) was utilized to characterize the time-dependent consumption of monomers during the polymerization reaction. MCR significantly improved measurement capability for this application of Raman spectroscopy compared to simple approaches that utilize peak height or peak area for monitoring this reaction. This approach enabled elucidating fundamental kinetic parameters associated with free radical chain propagation and termination. This application of Raman spectroscopy provided valuable insight into the mechanism of cross linking in addition to providing real-time monitoring of the polymerization reaction.

(326) **VCD in Pharmaceutical Discovery**

Don Pivonka[1], Steve Wesolowski[1]; [1]AstraZeneca

In recent years, more than 60% of new drugs reaching the market are chiral. This statistic highlights that the absolute assignment of chiral structure is critical to drug design and development. Historically, absolute stereochemical assignment was often determined toward the end of the discovery phase via chiral selective synthesis or analysis using single crystal X-ray diffraction spectroscopy. X-ray requires that a high quality single crystal be obtained and that a heavy atom (sulfur or larger) be present in the crystal. Vibrational Circular Dichroism (VCD) spectroscopy has the distinct advantage of providing chiral determinations for compounds without X-ray quality crystals or heavy atom requirements. The VCD technology has risen to a status that it is now on par with NMR and MS in terms of the number of research queue project requests for the AstraZeneca Wilmington site. This paper will discuss advances, insight and best practices which have come to light within our last 4 years of practice with the technology.

(327) **Advances in Pharmaceutical Applications of Vibrational Circular Dichroism**

Laurence Nafie; [1]Syracuse University

Infrared vibrational circular dichroism (VCD) is now being used throughout the pharmaceutical industry to address problems involving the structural determination of the absolute configuration (AC) and conformation of chiral drug molecules. Recently, programs have been written to quantify the degree of agreement between measured and calculated VCD for AC determinations. In addition, methodology is being developed to extend the use of VCD to the measurement of solid pharmaceutical samples, including active ingredients, excipients and formulated products. VCD methodology is also being developed for reaction monitoring to follow the percent enantiomeric excess (%ee) of chiral reactants and products as a function of time without the need for extraction followed by chiral chromatography. Progress in all three of these areas will lead to the expansion in the use of VCD across not only the pharmaceutical industry, but to all areas of chemistry where knowledge and control of molecular chirality is important.

(328) **Practical Considerations for Rapid and Accurate Stereochemical Assignments Using VCD**

Feng Qiu, Yingru Zhang, Michael Reily, David Wang-Iverson, Adrienne Tymiak; [1]Bristol-Myers Squibb Co.

The determination of the absolute configuration (AC) of chiral compounds using VCD is based on the fact that enantiomers have rotational strengths with equal magnitude and opposite sign. Therefore, the AC assignment of a molecule is achieved by comparing measured VCD spectra with calculated spectra. However, there are many different interactions, solute-solvent, solute-solute etc, existing in real sample solution. It is practical impossible to consider all these interactions for a real sample. What are the practical considerations for different chemotypes in order to achieve rapid and accurate stereochemical assignments using VCD? Using examples, we demonstrate improved processes that increase the reliability and throughput of AC assignments using VCD, and discuss the practical aspects when we should consider solute-solvent and/or solute-solute interactions.

(329) **A Theoretical and Experimental Study of Solvent Effects on the VCD Spectrum of 1-(2-Methylbenzoyl)-2-Pyrrolidinemethanol**

James Cheeseman[1], Douglas Minick[2]; [1]Gaussian, Inc.; [2]GlaxoSmithKline

The VCD spectra of (R)- and (S)- 1-(2-methylbenzoyl)-2-pyrrolidinemethanol dissolved in CDCl3 and d6-DMSO were measured between 2000 and 935 cm-1. The experimental spectra were compared with theoretical spectra predicted using B3LYP/DGDZVP model chemistry. The overall agreement between the vacuum calculation (gas-phase modeling) and experimental data was relatively poor, largely due to the inability of vacuum calculations to account for condensed phase interactions. In an attempt to overcome this limitation, VCD spectra were calculated using two different solvent modeling approaches: the Polarizable Continuum Model (PCM) and ONIOM, a multilayered computational method, where solvent molecules were treated explicitly. The ability of these methods to improve agreement with experimental VCD data are compared, as well as their computational efficiencies, which can have significant bearing on the application of these methods to larger molecular systems.

(330) **Calculating Confidence Limits for *ab initio* VCD Structural Assignments: A Comparison of Two Approaches**

Dean Phelps[1], Douglas Minick[1]; [1]GlaxoSmithKline, Molecular Discovery Research

Calculation of confidence limits in *ab initio* VCD studies is a measurement of agreement between an experimentally observed VCD spectrum and a calculated VCD spectrum. Two methods for quantification of agreement will be compared. The first method is comparing the intensities of bands in the observed VCD spectrum to the intensities of bands in the calculated VCD spectrum. This method involves interpretation of the VCD spectrum if the frequencies of some bands have been predicted at frequencies different than the observed frequencies for corresponding bands. One advantage to this method is that bands in the experimental spectrum whose frequencies are not accurately predicted in the calculated spectrum can still be used but require interpretation of the experimental spectrum. This method is time consuming and requires a skilled analyst to interpret the data. The second method is the BioTools CompareVOA© software. This method measures agreement between the observed VCD spectrum and the calculated VCD spectrum by comparing band area overlaps. Advantages of this method include its speed, use of band areas instead of intensities, optimized scaling factors, and ability to compare confidence limits against previous VCD studies. One disadvantage to this method is bands predicted at the wrong frequencies are included in the confidence limit unless those bands are intentionally excluded. Both methods will be compared from different perspectives. The confidence limits calculated using each method will be compared. The potential for wrong assignments based on assumptions of the reliability of each method will be assessed. Ideas of which method works best for certain functional groups will be discussed. Using either method can highlight functional groups that require investigation.

(331) **Applications of Raman Spectroscopy in the Identification of Extraneous Particulats and the Analysis of ProteinTherapeutics**

Xiaolin Cao[1]; [1]Amgen Inc

The techniques and applications of Raman spectroscopy in the pharmaceutical industry have made tremendous progress in the past decade. In particular, the importance and unique advantages of utilizing Raman spectroscopy in the biopharmaceutical manufacturing have been widely recognized (1-3). Micro-Raman is used extensively in the biopharmaceutical industry for the identification of foreign particles due to its advantages of in situ detection, minimal or no sample preparation and non-contact mode for measurements. Furthermore, micro-Raman can be used to detect the samples that are inside glass containers or enclosed in other thin transparent films if a visible laser is used for Raman excitation. The improved sensitivity achieved recently in the dispersive Raman and Raman optical activity has allowed the analysis of protein therapeutics in aqueous formulated solutions (2, 4). In this presentation we demonstrate the micro-Raman applications in the case studies of in situ particle identifications and explore several different types of protein therapeutics with Raman and Raman optical activity techniques in terms of their structural differences and side chain conformations.

(332) **Automated Pulse Shaping and Compression Based on Multiphoton Intrapulse Interference Phase Scan (MIIPS) Enables Development of Novel Biomedical Analytical Methods**

Marcos Dantus[1]; [1]Department of Chemistry, Michigan State University

Femtosecond lasers have been used for nonlinear optical excitation and ultrafast ionization since they were introduced almost 30 years ago. However, their cost, complexity, and dubious reliability kept them from analytical applications. The development of solid-state ultrafast lasers and automated pulse characterization and compression has resulted in sources that can be used reliably in applications that push analytical methods beyond. This talk will briefly review technology for automated pulse shaping and compression and then illustrate how these sources are revolutionizing protein mass spectrometry, atmospheric pressure mass resolved imaging and other nonlinear optical imaging modalities.

(333) **New Frontiers for Old Nonlinear Optical Effects**

Garth Simpson[1]; [1]Purdue University

Second-order nonlinear optical imaging of chiral crystals (SONICC) was used for sensitive and selective detection and characterization of protein crystals. Second harmonic generation (SHG), or the frequency doubling of light, is the oldest and simplest nonlinear optical interaction, yet it still continues to offer surprises and opportunities. The unique symmetry properties of SHG result in no coherent signal from disordered media. However, chiral crystals (i.e., crystals generated from unit cells with nonsuperimposible mirror images) all fall into a relatively small subset of materials that are symmetry-allowed for SHG. Applications of SONICC for addressing key bottlenecks in protein structure determination efforts will be described.

(334) **Plasmon-Controlled Fluorescence: A New Paradigm in Fluorescence Spectroscopy**

Krishanu Ray[1], Joseph R. Lakowicz[1]; [1]Univ of Maryland School of Medicine

Fluorescence detection is a central technology in the biosciences. Majority of the fluorescence experiments are performed in the far-field regime. By far-field we mean at least several wavelengths from the fluorophore. In recent years there has been a growing interest in the interactions of fluorophores with metallic surfaces or particles. Near-field interactions are those occurring within a wavelength distance of an excited fluorophore. The spectral properties of fluorophores can dramatically be altered by near-field interactions with the electron clouds present in metals. These interactions modify the emission in ways not seen in classical fluorescence experiments. Fluorophores in the excited state can create plasmons that radiate into the far-field and that fluorophores in the ground state can interact with and be excited by surface plasmons. These reciprocal interactions suggest that the novel optical absorption and scattering properties of metallic nanostructures can be used to control the decay rates, location, and direction of fluorophore emission. We refer to these phenomena as plasmon-controlled fluorescence (PCF). Our studies on the effects of metallic surfaces and nanostructures on nearby fluorophores showed significant increase in brightness and photostability both at the ensemble and single molecule level. We believe including metallic nanostructures and nanoparticles offer unique opportunities to further expand the scope of single molecule detection (SMD) and fluorescence correlation spectroscopy (FCS). We will present a review of the recent work on metal-fluorophore interactions. Recent research combining plasmonics and fluorescence suggest that PCF could lead in new classes of experimental procedures, novel probes, bioassays and devices.

(335) **Reacting Flow Diagnosis Using Ultrafast Lasers**

Sukesh Roy[1], James R. Gord[2]; [1]Spectral Energies, LLC; [2]Air Force Research Laboratory

Advanced measurement techniques that exploit lasers and optics have become well-established tools for characterizing combusting flows. Such noninvasive measurement approaches are often ideally suited for visualizing complex reacting flows and quantifying key chemical-species concentrations, temperature, and fluid-dynamic

parameters. The fundamental information these techniques provide is essential for achieving a detailed understanding of the chemistry and physics of combustion processes. Although traditional combustion diagnostics based on continuous-wave and nanosecond-pulsed lasers continue to dominate fundamental combustion studies and applications in reacting flows, revolutionary advances in the science and engineering of ultrafast (picosecond- and femtosecond-pulsed) lasers are driving the enhancement of existing diagnostic techniques and enabling the development of new measurement approaches. The ultrashort pulses afforded by these new laser systems provide unprecedented temporal resolution for studies of chemical kinetics and dynamics, freedom from collisional-quenching effects, and tremendous peak powers for broad spectral coverage and nonlinear signal generation. The high pulse-repetition rates of ultrafast oscillators and amplifiers allow previously unachievable data acquisition bandwidths for the study of turbulence and combustion instabilities. The applications of ultrafast lasers for optical measurements in combusting flows and sprays, emphasizing recent achievements and future opportunities, will be discussed.

(336) **Mass Spectrometric Imaging at the Cellular and Subcellular Level by Laser Desorption/Ionization**

Edward Yeung[1], DC Perdian[1], Sangwon Cha[1], Sangwon Cha[1], Young-Jin Lee[1]; [1]Ames Laboratory, Iowa State University

Mass spectrometric imaging has been utilized to localize individual astrocytes and to obtain cholesterol populations at the single-cell level in laser desorption ionization with colloidal silver. The silver ion adduct of membrane-bound cholesterol was monitored to detect individual cells. Good correlation between mass spectrometric and optical images at different cell densities indicates the ability to perform single-cell studies of cholesterol abundance. Feasibility of quantification is confirmed by the agreement between LDI MS ion signals and a traditional enzymatic fluorometric assay. Single chloroplasts isolated from plant cells were also detected and quantified in this manner. We propose this approach could be an effective tool to study chemical populations at the cellular and subcellular levels.

(337) **Nanomaterial Design for Quantitative Surface Enhanced Raman Scattering (SERS) Detection**

Amanda Haes[1], Marie C. Pierre[1], Sudip Nath[1], Maryuri Roca[1], Binaya Shrestha[1]; [1]University of Iowa

Control over the composition, shape, size, and local dielectric environment of metallic substrates is vital to consistent surface enhanced Raman scattering (SERS) enhancements. By varying the sizes and shapes of these novel materials, their optical properties can be tuned throughout the visible and infrared regions of the electromagnetic spectrum thereby influencing the magnitude of the SERS enhancement at a fixed laser excitation wavelength. In the presence of salt or target molecules (i.e. drug molecules or biological contaminants), however, the electrostatic shielding between solution-phase nanoparticles may be minimized thereby inducing uncontrolled aggregation of the nanoparticles. If aggregation occurs, the novel size dependent properties of the nanoparticles and the ability to use these structures in quantitative detection techniques are lost. In this work, a novel method which entraps gold or gold-coated silver nanoparticles in thin silica membranes will be demonstrated. The silica membranes are formed via selective silica dissolution and prevent electromagnetic coupling between the nanoparticle cores without blocking the active metal surface for SERS detection. Quantitative SERS signals will be shown to depend on molecular surface coverage, etching time, nanoparticle plasmonics, SERS excitation wavelength, and molecular incubation time. Improvements in understanding what is on the surface of a nanoparticle and how that surface chemistry influences the activity of the nanoparticles will have ultimate implications on the detection of target biological and environmental toxins.

(338) **The Fateley/KSU Years**

Robert M. Hammaker[1]; [1]Kansas State University

The presentation will contain selected examples of the activities of the Fateley/Hammaker research group taken from the 1985-2005 time period. A very brief proposal will be offered to account for the apparent result that the long time Fateley/Hammaker collaboration appeared to operate to the satisfaction of many of those involved. The selected examples will come from two areas: (1) Hadamard transform spectrometry and imaging (HTS and HTI); (2) infrared (IR) studies of volatile organic compounds (VOCs) in the atmosphere.

(339) **Analytical Spectroscopy: Dow-Then and Dow-Now**

J.D. Tate, Paul Chauvel, Anne Leugers, Marianne McKelvy; [1]The Dow Chemical Company

No modern laboratory in the petrochemical industry is complete without some sort of analytical instrumentation based on Spectroscopy. The amount of information derived from its use, both quantative and qualitative is enormous. Further, its applicability to a wide range of applications and samples including those beyond the laboratory makes analytical spectroscopy an invaluable tool for all industries ranging from chemicals to food and beverage to pharmaceuticals. Dow Chemical has used spectroscopy as an analytical tool dating back to before the ' 50s when the first double beam spectrophotometers were commercialized. Bill Fateley was one of Dow's leading spectroscopists in the late '50s. It is interesting to note that even though the technology has made tremendous improvements since then, the approach and its applications are still similar. We will take a look back in time when our friend Bill was a young spectroscopist employed at Dow Chemical, and compare the those tools and applications in Bill's day to those that are practiced today.

(340) **Fabrication of Substrates for SERS and TERS by Electroless Deposition**

Peter Griffiths[1], Przemyslaw Brejna[1]; [1]University of Idaho

Highly reproducible silver and gold substrates for surface-enhanced Raman scattering (SERS) are readily prepared on germanium substrates by electroless deposition. In principle, because of its low standard electrode potential, silicon should be an even better substrate. However, the insoluble oxide layer must first be removed with dilute HF. Silver is usually deposited in a dendritic form; however, when the silver dendrites are left in contact with water or water:methanol mixtures, the morphology of the silver is changed by Ostwald ripening. Surprisingly, even though the surface area is decreased, the SERS enhancement increases by over a factor of five. We have carried out Raman mapping experiments that, in conjunction with transmission electron microscopy measurements, allows insights to be gained as to the presence of SERS hot spots. We have found that silver nanoparticles form preferentially on the edges of silicon cantilevers used for tip enhanced Raman spectroscopy and will show how such tips can be fabricated simply and inexpensively.

(341) **Hadamard to Terahertz**

Jeffrey White[1], William Fateley[2]; [1]Picometrix LLC; [2]Kansas State University

William (Bill) G. Fateley was always trying something new. His many forays into Spectroscopy research included trips into new instrumentation development. The Fateley / Hammaker research group at Kansas State University lead the development of Hadamard Transform Spectrometers (No moving parts!) for on-line

applications in the late 1980's. That interest in research and development to create new instrumentation continues. This presentation will provide a brief review of Hadamard instruments and (somehow) connect the mentoring, interest and leadership provided by Bill Fateley to carry on with the development of another new technique, Time-Domain Terahertz. The instrumentation, characteristics and application examples of Time-Domain Terahertz (TD-THz) measurements will be presented.

(342) Pharmaceutical Applications of Nanoscale Infrared Spectroscopy and Imaging

Curtis Marcott[1], Michael Lo[2], Kevin Kjoller[2], Craig Prater[3], Bernard Van Eerdenbrugh[3], Lynne Taylor[3]; [1]Light Light Solutions; [2]Anasys Instruments; [3]Purdue University

Atomic Force Microscopy (AFM) based nanoscale infrared (IR) spectroscopy is used to probe the miscibility of pharmaceutically relevant amorphous mixtures. Specifically, binary solutions of polyvinyl pyrrolidone (PVP) and dextran with varying molecular weight ranges were deposited on ZnSe prisms. A tunable IR laser (1200-3600 cm-1), generating pulses of the order of 10 ns was used for excitation of the samples. Short duration thermal waves, due to infrared absorption, were studied by monitoring the resulting excitation of the contact resonance modes of the AFM cantilever. The AFM-IR technique enabled collection of spectra from highly localized regions of the sample. By mapping the intensity of a peak characteristic to PVP, it was also possible to generate two dimensional chemical maps with a high spatial resolution. It was observed that mixtures of high molecular weight grades of PVP and dextran were phase separated as expected. For example, a mixture of PVP K90 and dextran 400-500 kDa, showed a PVP rich continuous phase with discrete domains of a dextran rich phase that were of the order of 3 microns in diameter. The chemical images generated thus enabled visualization of the spatial distribution of the different components. These results demonstrate that AFM-IR is a promising tool to complement standard techniques for the study of miscibility, and enables the extraction of high resolution compositional information.

(343) Fast Raman Imaging Using Compressive Sampling Detection

Dor Ben-Amotz[1], Brandon Davis[1], Amanda Hemphill[1], Derya Cebeci[1], Bradley Lucier[2]; [1]Purdue University, Dept. of Chemistry; [2]Purdue University, Dept. of Mathematics

The design and performance of multivariate hyperspectral imaging spectrometer is described. This instrument makes use of a novel compressive sampling strategy which facilitates the collection of Raman images at much higher speeds than conventional micro-Raman instruments. This is achieved using a spatial light modulator to produce spectral filter functions which project chemically relevant spectral information for detection using an amplified photodiode detector at millisecond (or faster) speeds. The system may be viewed as a generalized spectrometer which can either operate as a conventional tunable filter scanning spectrometer or as a full spectral Hadamard transform spectrometer. Moreover, the system can be used to directly measure Raman spectral response functions arising from particular chemical components for high speed chemical imaging applications. Preliminary results demonstrate the system performance and pharmaceutical applications.

(344) Nonlinear Optical Imaging of Organic Crystal Nucleogenesis

Garth Simpson, Duangporn Wanapun, Umesh Kestur, Lynne Taylor; [1]Purdue University

Second order non-linear optical imaging of chiral crystals (SONICC) is investigated as a selective probe for characterizing crystallinitiy in active pharmaceutical ingredient (API) formulations. Second harmonic generation, or the frequency doubling of light, is symmetry forbidden in amorphous media, but allowed for all crystals with a chiral unit cell. Consequently, SONICC provides excellent selectivity for trace crystallinity of APIs and can be performed rapidly over large fields of view for diverse samples. Studies with model compounds (griseofulvin and chlorpropamide) demonstrate detection limits of SONICC for crystallinity better than 1 part in 100 billion by volume, corresponding to a ~9 order of magnitude improvement in % crystallinity compared to existing commonly used conventional methods (e.g., x-ray diffraction). The absence of a background response from disordered media allows the development of simple image analysis algorithms for automated quantification of nucleation rates, crystal growth rates, and activation energies for nucleation from a single set of measurements. Studies with powdered samples demonstrate the ability to easily quantify the residual 0.05% crystallinity remaining after exhaustive cryo-milling (S/N ~1000).

(345) Applications of Two Dimensional Correlation Spectroscopy to Pharmaceutical Drug Interactions

David Heaps[1], Alfred Rumondor[1]; [1]AstraZeneca

Two-dimensional correlation spectroscopy is a powerful tool for investigating molecular interactions. The growth in the number of publication using correlation spectroscopy is a confirmation to this usefulness. Pharmaceutical drugs can form many different interactions some intended to improve the performance and stability of the compound. In order to show improved performance, drug-polymer miscibility in a binary amorphous mixture may dictate the ability of the system to deliver poorly soluble therapeutic compounds. The increase in solubility of the poorly soluble drug is linked to keeping the drug in its amorphous form in a solid dispersion. Felodipine and four other polymers are investigated for this application. Stability improvements are seen in the interaction of another pharmaceutical drug with several other polymers. The protection of key functional groups with a cyclodextrin prevents degradation of another drug. Two-dimensional correlation spectroscopy shows where the interaction of each system is taking place and allows for the explanation of why each system performs successfully.

(346) Tips for TERS: Improving Their Efficiency and Stability

Alexei Sokolov[1]; [1]University of Tennessee, Knoxville

Tip-enhanced Raman spectroscopy (TERS) is promising technique for analysis of surfaces and interfaces with nanometer scale resolution. It presents a combination of scanning probe microscopy (SPM) with Raman (or can be taken broader – any optical) spectroscopy. The key element of this technique is a modified SPM tip that has Plasmon resonance at its apex and provides very strong and local enhancement of the optical signal. Although a few companies are already selling spectrometers with TERS capabilities, the main obstacle on wide commercialization of this technology is the absence of tips with high enough enhancement of the optical signal and long enough life-time. This talk will present an overview of major problems with tips for TERS, from their relatively low enhancements to fast degradation. Ways to significantly prolong the life-time of the tips by improving their chemical and mechanical stability will be also presented.

(347) **Near-Field Raman Spectroscopy for Nano-Scale of Chemical Analysis of Structured Carbons**

Yuika Saito[1], Mitsuhiro Honda[1], Yoshikiyo Moriguchi[1], Kyoko Masui[1], Prabhat Verma[1], Satoshi Kawata[1]; [1]Department of Applied Physics, Osaka University

Nano-scale optical analyses of structured carbons were performed by tip-enhanced near-field Raman spectroscopy (TERS). In this technique, the spatial resolution 30 nm is realized by the near-field probe which acts as a nano-light source. The nano scale analyses of few layers of graphite including graphene were measured. From the intensity change of the Raman band of silicon generated from the near-field probe, we can conveniently estimate the edge boundaries and the number of stacking layers. TERS measurement across the layer edges revels the nano-scale properties of the material as well as presenting the existence of local defects and edge boundaries. The intensity change of the G-band shows the step-like behavior that follows the layer boundary, while two components in 2D peak show more complex behaviors even inside layers. The peak fluctuation in 2D-band also suggested the local stress distribution due to interlayer interactions. Excess charge effect is observed through the correlation between the peak position and the width of the G-band and their nanoscale distribution within a layer is revealed. Besides the vibrational analysis, we successfully perform the estimation of the number of layers in two dimensional imaging by the same experimental platform, which allows us high-throughput nondestructive identification of graphene layers critical for the evaluation of this material especially in future device applications. The photopolymerization of fullerene C60s encapsulated (en-C60) inside single-walled carbon nanotube (SWNT) was also investigated by TERS. In general, a moderately intense laser irradiation invokes complete photopolymerization of non-encapsulated bulk C60 molecules. However, in contrast, we observed that the en-C60 molecules never get completely polymerized, even under long and strong irradiation. In both far-field and near-field Raman measurements, we observed evidence of simultaneous occurrence of polymerization and de-polymerization of en-C60 during the process of irradiation. The results have been discussed through the unique movement of C60 molecules inside nanotube, and through the interaction between C60 molecules with the inner wall of the nanotube in en-C60 sample, which may generate frictional scission of polymeric bonds between neighboring C60 molecules.

(348) **Optical Nano-Crystallography by Tip- Enhanced Raman Spectroscopy**

Markus Raschke[1]; [1]University of Washington

The capability of probing phase transitions, stress, electron-phonon coupling, or doping via their effect on the vibrational structure of crystals has positioned phonon Raman spectroscopy as a powerful tool for the study of semiconductors and dielectrics. In extending the technique to the near-field, the symmetry selectivity of the phonon Raman response allows for optical crystallography on the nanoscale in tip-enhanced Raman spectroscopy taking advantage of the local field enhancement provided by the nanometer size apex of a plasmonic scanning probe tip. The general selection rules that provide the necessary degrees of freedom are derived as a superposition of the crystal Raman tensor, momentum conservation for phonon and light emission, and the symmetry of the near-field tip scattering geometry. The capabilities are demonstrated for the spectrally and spatially resolved identification of intrinsic ferroelectric domains of individual $BaTiO_3$ and $LiNbO_3$ nanocrystals by probing the A_1 TO and E TO phonon modes with nanometer spatial resolution.

(349) **Spectroscopy of Single Semiconductor Nanowires: From Confocal Raman Microscopy to TERS**

François Lagugné-Labarthet[1]; [1]University of Western Ontario

Probing the vibrational signature of nanomaterials with a spatial resolution in the range of 10-100 nm is of tremendous interest to understand the properties of nanoscale materials due to possible confinement effects. Surpassing the resolution limit of conventional optical microscopy by a combination of scanning near-field techniques with confocal microscopy is a challenge that presents many advantages in terms of spatial resolution and acquisition time improvements. We report the study of one-dimensional semiconductor-nanowires of gallium nitride (GaN) and silicon (Si). The dimensions of the wires vary between 90 nm to 200 nm in diameter and several microns in length. For the GaN nanowires, different types of crystalline orientation were studied using confocal polarized Raman measurements. Raman confocal microscopy quickly differentiate the different types of crystalline orientation at the scale of a single nanowire but its spatial resolution is diffraction limited to about 0.5-1 micron. To further characterize such nano-objects with a spatial resolution better than the diffraction limit, we have developed a setup to perform tip enhanced Raman spectroscopy (TERS) measurements in a transmission geometry. In the TERS setup, we are probing simultaneously the topography and the spectroscopic signal of a sample benefitting from enhancement of the local electric field at the focal point. In this technique, a local metallic probe mounted on an AFM is interacting with a sample and induces enhancement of the Raman signal at the apex of the tip. The position in xyz position is controlled precisely by the Raman/AFM communication protocol, analyzing the sample topography and Raman signal simultaneously. The Raman signals are measured when the tip is in close proximity with the sample and when the tip is retracted 2 microns above the sample. The difference between the two experiments allows one to estimate the near-field effect. Herein we compare different results obtained by confocal Raman imaging and Tip enhanced Raman spectroscopy. Due to the nature of the local enhancement along the tip axis, we show that some vibrational modes can preferentially be enhanced in the TERS setup.

(350) **TERS as a Diagnostic Tool in Bioanalytics**

Dana Cialla[1], René Boehme[1], Tanja Deckert-Gaudig[2], Volker Deckert[1,2], Robert Moelle[2], Juergen Popp[1,2]; [1]Friedrich-Schiller University Jena, [2]Institute of Photonic Technology

Raman spectroscopy is an important analytical tool as it allows the highly specific detection and identification of molecules and even biological particles. However, the Raman effect is intrinsically weak Raman signals get efficiently enhanced by the interaction of molecules with a nanostructured metallic surfaces (for example metal nanoparticles, roughened metallic electrodes or metal island films), the so-called surface enhanced Raman spectroscopy (SERS). The combination of SERS with scanning probe microscopy (SPM) results in tip-enhanced Raman spectroscopy (TERS) that allows the detection of high specific fingerprint spectra with a spatial resolution in the nanometer range. [1]

Within this contribution the application of the TERS technique towards various bioanalytical problems is discussed. The identification of viruses at a molecular level is important in many different situations and research topics. However, a clear identification of the strain is usually impossible for single virus particles by using biological and imaging techniques. Here, TERS spectra of a single tobacco mosaic virus are presented and demonstrate the diagnostic potential of TERS. [2] In order to improve the label-free detection of cell wall components we have established supported lipid structures and further streptavidin labelled supported phospholipid film for TERS investigations. The present results at human cells (human dermal derived keratinocyte,

HaCaT) demonstrate the capability of TERS to provide a detailed and fast insight into the composition of the cell surface, even allowing the detection of single components. [3, 4]

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(351) Tip-Enhanced Raman Scattering on DNA/RDNA Strands

R. Treffer[1], X. Lin[1], V. Deckert[1,2];[1]Institute of Photonic Technology, [2]Institute of Physical Chemistry

Tip-enhanced Raman spectroscopy (TERS), combines a conventional Raman setup with a scanning probe microscope and demonstrated its advantages in high lateral resolution and large signal enhancement in the last few years, and is becoming a powerful tool for bio-nano applications. Here TERS experiments on DNA and RNA strands immobilized on different substrates are presented. On the one hand mica is used, a common substrate for DNA or RNA AFM imaging [1, 2]. Secondly, transparent Au nanoplates serve as substrates due to specific properties concerning immobilization and signal enhancement [3]. Both substrates allow illumination and detection through the substrate, which has distinct advantages with respect to the excitation of the nanoparticle and in the collection of the scattered Raman signal. TERS spectra were collected on several positions on a single RNA strand of uracil homopolymer immobilized on a gold nanoplate. The TERS spectra clearly show uracil features in the spectral region ranging from 1200 to 1800 cm^{-1}[4, 5]. Further TERS experiments were performed on single strands of calf thymus DNA immobilized on a mica surface. The results show that in spite of the different standard Raman scattering cross sections of the four nucleobases all bases can be determined in the spectra. A crucial issue concerning the interpretation of the respective TERS spectra is the formation of secondary structures of the DNA strands is. Synthetic DNA strands consisting of alternating blocks of nucleobases ($A_{10}G_{10}$) were immobilized on a mica surface. TERS spectra were collected, starting at the edge of a DNA strand, then stepping closer to and onto the DNA strand. Contrary to the expected DNA immobilization mediated by the phosphate backbone, measured TERS spectra show the strongest intensity for phosphate assigned Raman modes when the TERS probe is placed directly on the strand. However, the DNA bases should be localized closer to the nanoparticle than the phosphate backbone. Our results indicate strong dependence on sample order on the nanoscale. Single stranded DNA is very flexible and tends to form secondary structures, so our results lead to the conclusion that the strand is not immobilized as expected, but probably forms a twist at this position. As it is very difficult to obtain topographic images with the required resolution of DNA strands using a a TERS tip,, the TERS experiments provide important information concerning the orientation of the DNA on the substrate. Considering TERS spectra of single nucleobase crystals [5] and single poly-cytosine strands [2], these results further demonstrate the feasibility of direct and label-free DNA and RNA sequencing.

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(352) **Aerogels as Reaction Platforms – Progress and Pitfalls**

R. Lloyd Carroll[1]; [1]West Virginia University

Rapid-response sensing and catalytic processes for energy generation are similar in that both often occur in the gas phase. This limits concentrations of analytes or reactants and requires kinetically facile reactions, making sensitivity and response challenging. We have developed aerogels for energy generation applications, and we are exploring their potential in other areas, including sensing and other catalytic processes. As porous, very high surface area, low density materials, aerogels present exciting possibilities as a reaction platform, as well as presenting some challenges which must be overcome. These materials provide excellent support for catalytic reactions, including those important in reformation of petroleum resources. The properties and capabilities of these materials may be modified by tuning the porosity, controlling the size and nature of nanoparticles on the support, and varying the reaction conditions. In this work, I will describe our work to produce thermally stable composite aerogels, modified with nanomaterials. The amount of nanoparticles of iron and cobalt within the aerogel have been controlled using novel approaches. The aerogel composites have been extensively characterized through transformative processes by BET, XRD, XPS, and TGA. The magnetic properties of the materials vary with the nanoparticle loading, and these have been measured by SQUID magnetometry. Considering all of the structural, compositional, magnetic, and chemical data , we assess the particle dispersion, size, and reactivity to identify the most promising materials for potential application to energy problems. I will also discuss ongoing work to apply these materials to other areas, and share some of the pitfalls associated with the use of aerogels and how they may be surmounted.

(353) **Bottom-Up Nanofabrication of Organosilane Films for Organic Photovoltaic Applications Prepared by Particle Lithography**

Jayne C Garno, Evgueni Nesterov; [1]Louisiana State University

An important prerequisite for designing more efficient thin-film technologies such as organic light emitting and photovoltaic devices is to control the nanoscale morphology of thin films of conductive organic polymers. The current paradigm for preparing such devices is based on “top-down” strategies for solution-based processing or spin-casting of polymers, which generally provides only modest control over molecular organization and phase separation at the nanoscale. An alternative would be a “bottom-up” approach to prepare surface-grafted thin films of conjugated polymers by surface-initiated polymerization of small-molecule monomers. This enables *in situ* preparation of organic conducting polymer thin-films directly from the monomers, and provides better control over the molecular structure, organization and arrangement of the polymer at the nanoscale. Hybrid strategies for nanolithography such as combining particle lithography with additional bottom-up chemical steps provides practical new approaches for preparing 2D arrays of nanostructures with well-defined geometries and designed surface coverage. For particle lithography, a dried film of monodisperse latex or silica mesospheres is used as either a structural template or as an evaporative mask to spatially define the periodicity and size of nanopatterns. A close-packed, crystalline arrangement of spherical particles is produced spontaneously when samples of monodisperse latex are dried on flat surfaces. Silanes attach to surfaces covalently by successive steps of hydrolysis and condensation; therefore nanoscopic amounts of water are required to initiate the surface coupling reaction. For particle lithography, evaporative masks of latex mesoparticles form tiny residues of water that are trapped in a

meniscus area between the base of the particles and surface, which exquisitely defines nanoscale sites for binding silanes. Billions of regular ring-shaped nanostructures are generated with particle lithography, using only simple chemistry steps (mixing, heating, centrifuging and drying). Self-assembly provides control of the geometry, density, and surface chemistry at the nanoscale. Periodic arrays of regular organosilane nanostructures define passivated areas to further designate the placement of more complex molecular structures. By designing further successive steps of chemical reactions, surfaces can be tailored to present diverse functional groups. Examples will be presented for backfilling organosilane nanopatterns with polythiophenes and photoconductive molecules, as steps towards designing organic photovoltaic films.

(354) **Vibrational Spectroscopy as Probe of Nanoscopic Environments in Polymer Electrolyte Membrane**

Carol Korzeniewski, Chang Kyu Byun; [1]Texas Tech University

Infrared and Raman spectroscopy are being applied to investigate structural properties of fuel cell membrane materials. Hydration and dehydration lead to changes in the nanoscopic environments that form the pore and channel structures through which protons are transported. Vibrational modes of water and of ionomer backbone and side chain functional groups provide insights into these environments. In connection with infrared and Raman spectra of ionomer membrane materials, this presentation will discuss the use of least squares modelling techniques to differentiate the responses of hydrophobic and hydrophilic groups during changes in hydration state, effects of variation in the side chain end group, from sulfonate to sulfonyl imide and phosphonate moieties, and characteristics water at the interfaces inside pores and channels.

(355) **Highly Efficient Nanoparticle Catalyst for Fuel Cell Applications**

Shouheng Sun[1]; [1]Brown University

The need to limit Pt usage in catalysis, especially in fuel cell reactions, has promoted the search for highly active Pt nanoparticle (NP) catalysts or non-Pt NP catalysts with comparable catalytic properties. Using solution phase based reduction chemistry, we have synthesized a series of monodisperse oleylamine-coated NPs of Pt, Pd, and Au with controlled sizes and shapes. These noble NPs could serve as seeds for the production of structurally more complicated core/shell and dumbbell-like NPs. We found that both Pt and Au NPs were an excellent cathode catalyst for oxygen reduction reaction while Pd NPs were active for formic acid oxidation. The activity and durability of these NP catalysts were controlled by the dumbbell and/or core/shell structure. We demonstrate that highly efficient NP catalyst can be made for practical fuel cell applications.

(356) **Electrocatalysis on Facet-Controlled Pt-alloy Nanocrystals**

Shouzhong Zou[1], Hongzhou Yang[1]; [1]Miami University

Particle size, shape and composition are crucial factors in determining the catalytic activity of Pt-alloy nanoparticles. In most of the previous studies, particle shape is not well controlled, and the comparison of activity is therefore complicated by the shape (i.e. surface structure) difference. The development of nanocrystal synthesis enables production of high quality nanocrystals with monodisperse size and shape. In this presentation, electrocatalytic activities of PtxMy (M = Ni, Fe, Co, and Cu) nanocrystals towards oxygen reduction reaction and fuel (methanol and formic acid) oxidation reactions will be discussed. Results from particle structural characterization will be demonstrated to confirm the quality of the nanocrystals. It will be shown that ORR activity on Pt3Ni nano-octahedra is nearly 6 times of that on Pt3Ni nanocubes. ORR activity on nanocubes made of different M with similar particle size will be compared. Fuel oxidation reactions will also be demonstrated to be shape dependent. These studies underscore the importance of controlling particle shape in fuel cell catalyst development.

(357) **Depolarized Scattering from Silver Nanoparticles for Analytical Applications**

John Heckel[1], George Chumanov[1]; [1]Clemson University

Silver nanoparticles (NPs) efficiently scatter depolarized visible light [1]. When Ag NPs are placed between two crossed polarizers and illuminated, only light that is depolarized by the NPs can be detected. This provides high contrast between the NPs and the background. Depolarized scattering represents an ideal method for imaging Ag NPs. An indium tin oxide coated glass substrate decorated with a single layer of Ag NPs was placed between two crossed polarizers and imaged using an inverted microscope. Depolarized scattering spectra in the visible/near- IR spectral regions from individual Ag NPs were recorded as a function of incident polarization angle. The NPs were imaged using scanning electron microscopy and their shape and size were related to their depolarized scattering spectra. It was found that non-spherical Ag NPs scatter depolarized light at different intensities depending on their orientation relative to the polarization vector. Non-spherical Ag NPs can effectively modulate scattered light if rotated relative to a polarization vector. The wavelengths of scattered light can be tuned by changing the Ag NP size. By adding an iron layer to Ag NPs, one can potentially control the orientation of the NPs and modulate scattered light with a rotating magnetic field. The potential for biomedical applications was investigated. Z. Gryczynski, et al. Chem. Phys. Lett. 421, 2006, 189-192.

(359) **Kinetic Modeling of Multivariate Spectroscopic Images**

Paul Gemperline[1], Patrick Cutler[2], David Haaland[3], Erik Andries[2]; [1]East Carolina University; [2]University of New Mexico; [3]Sandia National Laboratories

We report the use of kinetic modeling of temporal hyperspectral fluorescence image data to extract kinetic information and rate constants for reactions of interest to biologists and computer modelers. The goal of this work was to mathematically resolve temporal hyperspectral images and extract reaction rate information of labeled cell signaling proteins in host-pathogen interactions. Two modeling techniques are reported for systems with unknown initial concentrations: direct non-linear (DNL) fitting and separable least-squares (SLS). In the DNL approach, all parameters including rate constants and initial concentrations are estimated with a non-linear solver. In the separable least-squares approach, the inherently linear parameters (concentrations) and non-linear parameters (rate constants) are separated and solved in succession. The SLS method offers significant improvements in computational speed and robustness compared to the DNL method. These two methods are demonstrated and compared for the resolution of photo-bleaching in multicomponent glass beads and in temporal hyperspectral fluorescence images of fixed transiently transfected A549 cells with IKKalpha proteins tagged with Green Fluorescent Protein (GFP) and MAVS proteins tagged with Yellow Fluorescent Protein (YFP). Results showed that our novel kinetic fitting algorithms were capable of mathematically resolving up to seven different components per image, including pure component spectra, temporal decay profiles, and spatial chemical concentration maps. Through kinetic modeling of temporally-resolved hyperspectral cell images we discovered that several different decay models were needed to adequately model each fluorophore in the image. These kinetic results led us to the hypothesis that different decay curves for each fluorescent specie were due to the presence of the fluorophore in different environments within the sample. Thus, a spatial map of

each fluorophore in the various different environments was obtained directly from the kinetic modeling process.

(360) **Chemical Characterization of Micro- and Nanoparticles by ICP Spectrometry**

Kay Niemax[1], Sebastian Groh[2], Ayrat Murtazin[1], Carmen C. Garcia[3]; [1]BAM Berlin; [2]ISAS Dortmund; [3]Institute for Transuranium Elements

A relatively simple new method for accurate measurements of element masses in nano- and microparticles is presented. Airborne particles or particles in monodisperse microdroplets generated from suspensions are introduced into the ICP. The particles are atomized and produce transient element signals which are measured either by optical emission (OES) or mass spectroscopy (MS). Monodisperse microdroplets of known element solutions are used for calibration. The calibration droplets are desolvated before introduction if airborne particles have to be measured or they are injected without desolvation if the particles of interest are introduced in droplets into the ICP. It will be shown that reliable, accurate measurements of particle masses can be performed. Spherical monodisperse SiO2 and Au nano- and microparticles both in suspensions serve as well-characterized samples and Si and Au standard solutions are used for calibration. The potential of the method will be demonstrated comparing spectroscopic ICP measurements with SEM measurements of particle distributions in sufficiently large ensembles.

(361) **Transparent Magnetic Photoresists for MEMS and BioMEMS Applications**

Philip Gach[1], Christopher Sims[1], Nancy Allbritton[1,2]; [1]University of North Carolina at Chapel Hill; [2]North Carolina State University

Microfabricated devices possessing magnetic properties are of great utility in microelectromechanical systems (MEMS) due to their controlled manipulation with external magnets. Current methods for introducing magnetism into such devices include forming magnetic-particle/polymer composites and electroplating or direct sputtering of magnetic materials. These microdevices possess high magnetic susceptibilities, however, the low transparency of the bulk or aggregated metallic materials inhibit visual inspections of biological specimens on the structures. Uniformly transparent magnetic photoresists have been developed for bioanalytical microdevices that require consistent magnetism and real time imaging. Due to their small size inorganic particles tend to self aggregate when mixed into an organic polymer. Colloidal formation of 10 nm γFe2O3 particles were minimized during dispersion into negative photoresists SU-8 and 1002F through organic capping of the nanoparticles and utilization of solvent-based dispersion techniques. Uniformly transparent photoresists at concentrations of 0.01 to 1% γFe2O3 have been successfully developed and still exhibit the advantageous properties of native photoresists such as: good transparency, low autofluorescence, biocompatibility, and low substrate adhesion. Photoresists with γFe2O3 concentrations of 1% have been used to fabricate microstructures with aspect ratios up to 4:1 and with a minimal resolution of 3 μm. Utility of these magnetic photoresists was demonstrated by sorting single cells plated on magnet microdevices and manipulating the magnetic carriers with an external magnet.

(362) **Isolation and Identification of Potential Chemical Attractants from Rudbeckia Inflorescences**

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We aim to identify the volatile compounds in the inflorescences of two Rudbeckia species that may be responsible for the olfactory attraction of the crab spider Misumenoides formosipes to the inflorescences of these plants. In olfactometric bioassays eighty percent of 30 male spiders moved towards olfactory-only cues from R. hirta inflorescences over a water control, a significant outcome relative to chance (P = 0.0007). Our approach to isolating potential attractants was to use ultrasonic extraction to remove the volatile components from the inflorescences and then separate the extract into fractions using flash chromatography with different solvent systems. At each isolation step, we tested the attraction of the male spiders to the test substance using a y-tube olfactometer. Spiders chose the inflorescences, the bulk ultrasonic extract, and those fractions collected using 100% dichloromethane over controls, suggesting that an attractant was present in each of those test substances. Nuclear magnetic resonance and infrared spectroscopy obtained on the 100% dichloromethane fractions indicate 2 primary components. The data suggest that one is a long chain fatty acid ester, probably an aryl conjugated ester, and the other is long chain alkane that may be cuticular wax. Further spectroscopic and chromatographic results will be performed in an attempt to identify the specific chemical composition of the attractant(s).

(363) **The Exploration of Selenopeptides in Cerebral Spinal Fluid Using Complementary SEC-ICPMS and LC-MALDI for Elemental and Molecular Identification**

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Selenium, an essential micronutrient, has numerous health benefits. It plays an important role in thyroid hormone metabolism, antioxidant defense systems and also immune function. T he main role of selenium in the body is to protect against oxidative stress. Selenium deficiency can cause diseases such as Keshan and Kashin-Beck disease as well as possibly contributing to viral infections, AIDS progression and infertility in males1. Selenium is encoded by the UGA codon in the mRNA and leads to selenocysteine (Sec), the 21st amino acid. To date there about 30 identified selenoproteins, 15 of which have been characterized2. One major class of selenoproteins are peroxidases 2, 3 and contains five Sec-containing gluthathione peroxidases (GPx). GPx1 has antioxidant properties and is believed to be able to store Se. In cerebral vasospasm (CV), a complication after subhemmoragic stroke (SAH), elevated levels of selenium and GPx activity has been seen4. SAH is one of two types of strokes and its onset is caused by a blood vessel on the outside of the brain bursting. CV occurs after the stroke and is a narrowing of the vessels of the brain. This study utilizes both elemental (ICPMS) and molecular (MALDI) identification for the exploration of selenopeptides/selenoproteins found in three different cerebral spinal fluid (CSF) samples; a control (CSF-N), SAH stroke patients (CSF-C) and CV patients (CSF-V). Size exclusion chromatography coupled to inductively coupled plasma mass spectrometry was employed to separate different intact selenoproteins from the three samples. Fraction collection was done off line and the samples further separated and spotted using LC-MALDI. MS and MS/MS data was collected and processed using a protein database search engine for protein/peptide identification. 1. Driscoll et. al. Annu. Rev. Nutr. 2003. 23;17-40 2. Brown et. al. Public Health Nutrition. 2001, 4(2b); 593-599 3. Lu et. al. Journal of Bio. Chem. 2009, 284(2);723-27 4. Pyne-Geithman G, Clark JF; unpublished research 2007

(364) **Investigation of Capacitance Effects on Liposomes Containing pH Gradients**

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Liposomes have been widely used to mimic biological membranes in the study of membrane bound proteins, ion transport, energy transduction, cellular and sub-cellular systems. The influence of trans-bilayer pH gradients is of great importance in applications

such as drug loading and delivery as well as in many cellular functions. Therefore a method to quantitatively and qualitatively assess these gradients is applicable to many such biological systems. Here we examine the effects of a trans-bilayer pH gradient on the electrophoretic behaviors of various liposome populations. We have found that the presence of a pH gradient results in a capacitively induced change in the surface charge and a subsequent change in the electrophoretic mobilities of liposomes. Capacitance effect are also examined spectroscopically using voltage sensitive dyes (VSD) embedded within the lipid bilayer in various liposome preparation. Furthermore, the capacitive effects can be predicted and exploited for separation and characterization of mixed pH-gradient and non-gradient populations.

(365) **ATR Imaging of Engineered Cartilage Constructs**

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Articular cartilage is an avascular and aneural tissue which limits intrinsic healing following injury or degradation. Tissue engineering is a promising therapeutic strategy to generate cartilage constructs that mimic the native tissue structure. Porous polymer scaffolds such as polyglycolic acid (PGA) are widely used to deliver the cells to cartilage defect sites. Therefore, assessing the amount and quality of regenerated tissue and scaffold degradation could enable more precise monitoring of growth during tissue culture. Fourier Transform Infrared Imaging (FT-IRIS) has previously been utilized to monitor cartilage repair and scaffold quality over time, but primarily on thin histological sections. Traditionally, paraffin is used as embedding material to acquire the thin sections of engineered tissue, but biomaterials such as PGA can be dissolved to some degree during paraffin processing. Further, preparation of histological sections does not permit further analyses on the tissues. To overcome these issues, we evaluated ATR imaging of intact engineered bovine cartilage constructs grown on PGA scaffolds for two and five weeks and compared data to that acquired from infrared imaging of histological sections of the same tissue. Data were acquired either in transflectance from thin sections of engineered tissues on low-e slides, or using the ATR imaging attachment of the Perkin Elmer Spotlight 400 Imaging system (Perkin Elmer, Shelton, CT) from intact tissues. Images were collected at 8 cm-1 spectral resolution in the range of 4000 to 748 cm-1 using 64 co-added scans for ATR with a pixel resolution of 6.25 micron, and with 2 co-added scans for the transflectance data acquisition. Quantitative analysis of integrated peak areas was performed using ISys 5.0 software (Malvern, UK). PGA, collagen and proteoglycan content were monitored by the integrated area of the 1740, 1660, and 1050 cm-1 absorbances, respectively. PGA decreased and collagen and proteoglycan increased in the 5-week compared to the 2 week constructs as shown by both ATR imaging and transflectance data. We conclude that ATR imaging of engineered tissues is a promising method for assessment of composition of intact engineered tissues.

(366) **Peroxidase Activated Nanoprobe for SERS Imaging**

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A peroxidase-activated surface-enhanced Raman scattering (SERS) nanoprobe has been synthesized. The 13nm citrate stabilized gold nanoparticles was coated with a SERS dye and thiol-polyethylene glycol (PEG). A peroxidase substrate (e.g. tyramine) was then conjugated onto the SERS particles. Upon exposure to peroxidase, SERS signal can be amplified in solution due to particles clustering. In immunohistochemistry, particles deposit in the vicinity of peroxidase in a controlled manner that results in an increased sensitivity than the conventional assay. The SERS nanoprobe has the potential for sensitive peroxidase detection and imaging.

(367) **Development of Spectral Markers Using Infrared Imaging to Access Cardiac Remodeling**

Naomi D'souza[1], Nancy Pleshko[1]; [1]Temple University

Cardiovascular heart disease (CHD) is the leading killer of both men and women of all racial and ethnic groups. Moreover, more women than men suffer from CHD and studies have shown that postmenopausal women are more prone to developing heart disease than premenopausal women. Limited information is available which explains the mechanisms for these observed sex-differences in the clinical outcome from acute CHD. One potential explanation for these observations may be sex-related differences in the remodeling process. Molecular changes in cardiac tissue remodeling and repair have not been adequately studied, in part due to the lack of a quantitative technique to assess myocyte and matrix component alterations. . In the current study we use Fourier transform infrared imaging spectroscopy (FT- IRIS) to evaluate tissue and cell characteristics related to cardiac remodeling in an estrogen-deficient rat model. FT-IRIS data was obtained from unstained 7 micron thick sections of the left ventricular wall of n = 10 animals, 5 sham operated female rats, age 7 months, and 5 OVX female rats with confirmed estrogen deficiency. FT-IRIS data were collected in transflectance mode from sections placed on low-e slides on a Perkin Elmer Spotlight 400 Imaging System (Shelton, CT). Data were collected at 8 cm-1 spectral resolution in the range of 4000 to 800 cm-1 with a pixel resolution 6.25 (μm)micrometer. Quantitative analysis of integrated peak areas was performed using ISys 5.0 software (Malvern Instruments, UK) Collagen content, collagen helical integrity, and collagen maturity based on crosslinking (ratio of mature: immature crosslinks) were correlated with histological data and functional data obtained from isolated Langendorf heart prep to measure diastolic pressure-volume function curves. Analysis of the pressure-volume relation showed altered LV compliance indicating that estrogen deficiency leads to a more compliant myocardium in the OVX group despite increases in LV collagen content. The findings of increased LV compliance despite increases in collagen content suggest that either collagen types, cross-linking or other components of the ECM are altered with chronic estrogen deficiency We can conclude that FT-IRIS derived parameters could be useful in assessing the role of estrogen in the pathophysiology of cardiac remodeling.

(368) **Formulation and Confirmation of Diisobutylphthalate in Rodent Feed Mixes in Support of Developmental and Reproductive Toxicology Studies**

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Phthalate esters are found in a wide variety of consumer and food packing products. Hence there is widespread exposure of the human population to these chemicals. One of many phthalate esters, diisobutylphthalate (DiBP), is used as a plasticizer and in mixtures that is suspected to have adverse reproductive and developmental effects similar to the non-branched analog, di-n-butylphthalate. DiBP may be found in many consumer products, such as food packaging, children's products, and certain medical devices. Human exposure to DiBP can occur through ingestion and the use of medical devices containing plastics. The present work describes a dose formulation preparation method for DiBP in two different rodent feeds, NTP-2000 and NIH-07, and covering a wide dose range for animal toxicology studies. Typical batch sizes required to support such animal studies span a range of 20 to 100 kilos. The test chemical is a semi-viscous liquid which requires

proper introduction, mixing and blending techniques to insure a homogeneous distribution throughout the feed formulation(s). Initially, a feed premix is prepared by utilizing a small portion of blank feed (~1 kilo) and then slowly adding an acetone solution of DiBP directly onto the feed. After properly rinsing all vessels, the remaining blank premix feed is added to the mixing vessel and all of the acetone is evaporated under nitrogen. About half of the remaining unused blank feed is layered in a blender and the premix is layered on top. The premix container is properly rinsed with blank feed and this "rinse" is also transferred to a blender. All additional remaining blank feed is then layered evenly in the blender prior to the final blending step. Test formulations were prepared in both feed types at 25 and 10,000 mg/kg (ppm) and evaluated for homogeneity and stability by using a reverse phase gradient UPLC method with UV detection at 225 nm. Results are summarized below.

Method Parameter	Results
Batch Size	25 kg
Dose Concentrations (ppm)	25 10,000
Homogeneity Results: (%RSD)	Accuracy (% RE) /Precision
NIH-07	103%/1.7% 94.6%/ 4.1%
NTP-2000	96.1%/ 4.6% 97.6%/ 3.7%
Storage stability/Temperature	42 days/Refrigerated at 25 ppm

(369) **A Lab-On-A-Chip FRET Biosensor for Pharmaceutical Applications**

Annadele Herman[1], Hamed Shadpour[1], Jon Zawistowski[2], Klaus Hahn[2], Nancy Allbritton [1, 2, 3]; [1]Dept. of Chemistry, UNC, Chapel Hill; [2]Dept. of Pharmacology, UNC, Chapel Hill; [3]Dept. of Biomedical Eng., UNC-NCSU

Our group recently developed a miniaturized cell array made of biocompatible photoresists such as SU8 and 1002F for selection, enrichment, collection, and expansion of primary cells and colonies. The combination of an *in situ* surface roughening of these arrays followed by micro-contact printing of biomolecules broadens the utility of the array in biomedical studies. Examples are the formation of surface gradients and the measurement of fluorescence resonance energy transfer (FRET) in biosensor-expressing cells. Due to the index of refraction mismatch between the polymer pallet and the surrounding virtual air wall, strong scattering of cellular fluorescence occurs at the pallet edges when cells are located near the pallet edges at the air:pallet interface. Accurate measurement of cellular fluorescence and FRET requires minimization of this scattered light from the pallet edges during biosensor recordings. Surface patterning of the individual pallets with an extracellular matrix (ECM) such as fibronectin was performed using a customized alignment tool. ECM was placed in the centers of the pallets directing cell attachment away from the pallet edges. Thus biosensor fluorescence from the cells was spatially separated from the pallet edge scatter. Qualitative and quantitative studies were performed on H1299 and mouse embryonic fibroblast (MEF) cells cultured on both stamped and control pallet arrays to compare biosensor fluorescence and FRET signals. These results demonstrated the potential of the pallet arrays in identifying cells with novel FRET-based biosensors as well as the use of these sensors for the selection of cells based on signaling properties.

(370) **Development of a FRET-peptide Sensor for Trace and Ultratrace Metal Detection**

Shelly Casciato[1], James Holcombe[1]; [1]The University of Texas at Austin

Monitoring of metals at the trace and ultratrace level in natural or process sources is commonplace. In most instances, atomic spectroscopic methods are employed to achieve selectivity and to reach the ppm-ppb levels that are typically of interest. For *in situ* analysis of environmental waters, collection and transport of grab samples back to a lab is routine. The development of inexpensive sensors that had the elemental selectivity and ppb sensitivity could be a significant enabling tool for a large number of studies, especially if continuous *in situ* monitoring was required. Currently, we are developing a trace metal sensor, incorporating a peptide chelator that utilizes FRET detection. Our lab has developed peptide-based metal chelators which exhibit shape changes (tertiary structure alterations) upon chelation with selective metals. By placing donor and acceptor fluorophores at selected sites on the peptides, these small changes in the peptide structure are then detected using Forster Resonance Energy Transfer (FRET). In brief, FRET involves the transfer of energy from a radiatively excited donor molecule to a nearby acceptor molecule which then fluoresces at its own characteristic wavelength. Ideally, one monitors the acceptor's emission while exciting at the donor's excitation wavelength. In addition to exhibiting a "turn on" (rather than quenching) response, the excitation and emission wavelengths are separated by much larger wavelength distances than utilized in typical fluorescence. This latter fact has a very significant impact on the simplicity of the instrumentation that can be used for excitation and detection. These instrument design considerations as well as examples of sensitivity and selectivity will be presented.

(371) **Phytoremedation of Metals in Soils by ICP-OES**

Joseph Sneddon[1], Carey Hardaway[1], Venkate Salla[1]; [1]McNeese State University

Contamination of soils and waters from metals is a fact of life in many industrial areas around the world. Traditional methods of remediation such as physical removal of contaminated soils is both expensive and may be ineffective. Phytoremedation using plants, bushes and trees has generated some interest with the potential to be low cost, consumer friendly and green chemistry. In this paper we will present studies on the use of a plant native to Southwest Louisiana, namely Spartina alterniflora (smooth cordgrass). The Spartina alterniflora was grown in soil containing spiked metals and a known contaminated (with metals) soil. Controls were added. At certain time intervals, typically three to six months, soil, root, stem and leaves were sampled, digested using microwave technology and determined for metals using inductively coupled plasma-optical emission spectrometry (ICP-OES). Preliminary data shows that the metals were absorbed by the roots. Further results over the ongoing growing season will be presented. Supported, in part by National Oceanic and Atmospheric Administration.

(372) **Application of Dendrimers for Water Purification**

Priyanka Bhattacharya[1], Pengyu Chen[1], Seung Ha Kim[2], Monica H. Lamm[2], Pu Chun Ke[1]; [1]Clemson University; [2]Iowa State University

Here, we address the fundamental understanding of water purification using a dendritic polymer "nanosponge", a highly branched nanoparticle which affords high selectivity, recyclable capacity and has harmless byproducts. We present an accurate description of the binding dynamics between poly(amidoamine) (PAMAM) dendrimers and water pollutants, such as copper and phenanthrene, from single molecule to ensemble level using combined methodologies of UV-vis absorption spectroscopy, fluorescence microscopy, and electron microscopy. An effective binding of dendrimers conjugated with Alexa Fluor® 350 succinimidyl ester dye [acceptor] with phenanthrene (Phe) [donor] – a polyaromatic hydrocarbon (PAH) commonly found in soils, estuarine waters and sediments, and other terrestrial and aquatic sites - was investigated using fluorescence resonance energy transfer (FRET). The anti-correlation between the fluorescence of the donor and that of the acceptor was observed to be the most robust for a dendrimer-Phe molar ratio of 1:1. Due to neutralization

of the PAMAM dendrimer at high pH, adsorption of the hydrophobic Phe molecule was the most favorable at pH 10. Furthermore, it will be demonstrated that using the surface plasmon resonance (SPR) of gold nanowires, the detection limit of copper can be increased to the micro molar range. Labeled PAMAM dendrimers with negative surface charges was incubated with a CTAB-stabilized gold nanowire suspension at physiological pH. Transition metals like copper quench the fluorescence of the dye. This quenching is further enhanced using the SPR of the gold nanowires. In summary, we have devised a sensitive method for the detection of water pollutants at micromolar concentrations, using novel analytical chemistry and spectroscopy. This research offers a strategy for the sustenance of clean water sources and environmental remediation.

(373) **Environmental Studies of Metals by ICP-OES in Southwest Louisiana**

Caray Hardaway[1], Joseph Sneddon[1], Shilpa Vootla[1], Venkatesula Salla[1]; [1]McNeese State University

A number of studies in Southwest Louisiana are currently in progress in this laboratory involving using ICP-OES to determine metal concentrations in a variety of samples including crawfish and contaminated soils. Of particular interest is phytoremediation of soils. Several studies were done to to examine crawfish exposure to contaminated water and the uptake of metals from the water by the crawfish. Two of the studies examined this effect on live crawfish and another study with ground crawfish exoskeleton. The metal analytes included lead, copper, zinc and selenium. Water was spike with with soluble salts of the metal analytes to produce concentrations in the range of 5 – 250 ppm. Live crawfish or ground exoskeleton were placed in the spiked water from times ranging from 1 hour to 30 days. The crawfish, exoskeleton, water were analyzed for changes in metal content. In one study, the crawfish were separated into shell meat and organs for analysis. The analysis were carried out using ICP-OES on samples prepared via nitric acid/hydrogen peroxide, microwave digestion. Results show uptake of the metals by the crawfish and crawfish exoskeleton. The highest concentration of metals were found in the shell and the least in the meat. Results support that the chitin in the exoskeleton is responsible for the metal uptake. Supported, in part, by National Oceanic and Atmospheric Administration.

(374) **A Comparison of Methods for Analysis of Dispersed Oil in Water and Soil by FT-IR Spectrometry**

Ben Perston[1], Aniruddha Pisal[1], Dean Brown[1]; [1]PerkinElmer

Infrared spectrometry has long been a standard method for detecting and quantifying hydrocarbon contamination in environmental samples. The basic principle is to extract the hydrocarbons into a non-polar, IR-transparent solvent such as Freon-113, tetrachloromethane, or tetrachloroethylene, and then to measure the IR absorption due to C-H stretching modes around 3030–2900 cm-1. One alternative method is to use a volatile hydrocarbon solvent for the extraction, and deposit an aliquot of the extract on the crystal of an attenuated total reflectance (ATR) accessory, allowing measurement of the oil film after the solvent evaporates. A third approach is to use a cyclic hydrocarbon solvent (cyclohexane or cyclopentane), which has a window of low absorption around the symmetric alkane C-H deformation band at around 1380 cm-1 (characteristic of C-CH3 groups), allowing a transmission measurement, albeit without the extremely long pathlengths possible with a perhalogenated solvent. We evaluate these three approaches and present a comparison in terms of convenience, robustness and sensitivity.

(375) **Evaluation of Laboratory Productivity for Environmental Applications**

Laura Thompson[1], Paul Krampitz[1], Stan Smith[1], Praveen Sarojam[1], Zoe Grosser[1]; [1]PerkinElmer, Inc.

Environmental analyses are performed on a variety of matrices such as drinking water, wastewater, and solid and hazardous waste materials. The metals of interest can vary and the concentrations can range from trace levels to higher. The methods developed by various environmental programs have differed in the quality control required and only slightly in the analyte list. The use of a dual-view ICP-OES to meet the requirements for both US EPA methods 200.7 and 6010 is described. By coupling a FAST autosampler to this system, laboratory productivity can be dramatically improved. Evaluation of this complete system shows over 100% improvement in sample-to-sample run time when compared to standard sample introduction systems. System stability has been evaluated and a wash out study conducted to prove acceptable performance for environmental applications. The use of ICP-OES to analyze environmental samples is extended to the determination of metal contaminants in industrial effluents. The procedures and quality control protocols set out in the official methods are described and followed in the analysis of several reference materials and actual effluent samples. This work demonstrates the applicability of ICP-OES to the analysis of environmental samples to meet US EPA requirements as well as improvements in productivity with the incorporation of FAST autosampler technology.

(376) **Parallel Extraction of Oxytetracycline and Flumequine from Fish Food and Its Determination by Derivative Spectrophotometry**

M. Ines Toral[1], Jairo Zuniga[1], Sandra Orellana[1], Cesar Soto[2]; [1]Faculty of Science. University of Chile; [2]University of Concepcion

In this work is proposed an extraction method for oxytetracycline (OTC) and flumequine (FLU) from fish food and its determination by derivative spectrophotometry (DS). For the extraction of each drug, were considered structures, solubility in different solvents, the interaction between analytes and matrix, the stability of drugs and their spectral behavior Based on these considerations and preliminary studies, were selected acetonitrile for the extraction of FLU and buffer HPO4-2/H2PO4– 0.1 M (pH 7.2) and EDTA 0.1 M as complexing agent for extraction of OTC. The enriched samples were prepared starting from 20 g of food with 5.0 mg of each one of the drugs, which corresponding to 250 mg/Kg of OTC and FLU. This sample was powdered and homogenized in ultra turrax T-25 for 5 min. In two parallel samples of 5 g were added 50 mL of each extractant and then they were agitated for 30 min. The extracts were centrifuged at 1,395g by 5 min, and the supernatants were filtered to the vacuum. Finally, the spectra of both extracts were evaluated and it was found in both cases a mixture of these drugs. However, the predominant compounds were; OTC in buffer and FLU in acetonitrile. In this context, the derivative technique was used in order to avoid the mutual interference. The same extraction procedure was carried out but the analytical signals were obtained by first derivative, smoothing factor 16,000, amplification factor 10,000 and the wavelengths, were 389.0 nm and 298.4 nm, for OTC and FLU. In this conditions were found that the extractions are near to 100% for both cases. To synthetic sample of OTC and FLU 250 mg/Kg different quantities of food were added in order to obtain solid dilutions of concentrations between 200 and 25 mg/Kg. The enriched foods, with OTC and FLU, were extracted and evaluated using the proposed procedure. In both cases the extraction limit was of 25 mg/Kg. The recovery for OTC and FLU were 96 ± 4% and 92 ± 5%, respectively. This work was financed by FONDECYT Project Nº 1100103.

(377) **Levels of Sulphur Dioxid and the Correlation to the Total Suspended Particulate Matter in Polluted Air Samples**

EL Mukhtar Belgasem, Ramadan Damja; [1]Al-Fateh University

The concentration levels of the common air pollutant sulphure dioxid was investigated simultaneously in parallel to air total suspendedparticulates in the same sampling locations but using sampling train of different design to trap sulphure dioxide in an absorbing solution of potassium tetrachloromercurate followed by colorimetric determination . Sulphure dioxide pollutant was found to range from 0.082 mg/m3 to 3.12 mg/m3 over tweleve months study period , the concentraction of sulphure dioxide pollutent was investigated in relation to the most important meteorological parameters such as average wind speed , average air temperature and humidy in the study area over a study period , there samed to be a correlation of sulphure dioxide concentration to the level of total suspended particulate matter in the study area.

(378) **Analysis of Suspended Particulates for Their Trace Element Contentes**

Ramadan Damja, EL Mukhtar Belgasem; [1]Al-Fateh University

Air suspended particulate matter is one of the most major atmospheric pollutants espcially in industrial areas and the surronding enviroment . The particulates are believed to have advers effect on humman health , live stock , plants and visibility. One reason for their adverse effect could be attribuated to the heavy metal contents of those complex multi-phase particulates. To assess the complex composition of the particulates in relation to their primary source of emission and possible mechanism of particulate formation , three sampling location for collection of the particulates were carefully selected to be of different sources of pollutions , then the concentration of several trace elements in the collected particulates were investigated using (ICP-OES) technique , the results showed that there is a considerable variation in the particulates composition from the lowest value of 20.24 mg/m3 in winter to 537.39 mg/m3 in the summer time . The chemical composition was investigated in relation to location , time of years and the important meteorological parameters.

(379) **Photon Trapping Spectroscopy: Prototype Optimization and Application to Air Monitoring**

John Frost[1], Joseph Aldstadt[1], Peter Geissinger[1], Jorg Woehl[1]; [1]University of Wisconsin Milwaukee

Absorbance measurements of extremely dilute concentrations of analytes, and very weakly absorbing transitions, are an important aspect of analytical and physical chemistry. Photon trapping spectroscopy (PTS) is a new technique related to cavity ring-down spectroscopy (CRDS) but with several important differences. Light enters the cavity via transmission through a highly reflective dielectric mirror in the same way as CRDS, however, unlike CRDS, the cavity exit mirror contains an exit slit and is rotating on an axle. This arrangement has several significant advantages over CRDS. Absorbance data is recorded directly without the need to calculate ring-down times. Compared to CRDS, longer pathlengths and correspondingly lower detection limits are possible because all of the available light is allowed to leave the cavity via the exit slit. Additionally, PTS allows unprecedented control of the intra-cavity resonance times and the ability to tune the pathlength amplification through a sample over several orders of magnitude. We present performance data for this new instrument by monitoring the atmospherically relevant gas, nitrogen dioxide.

(380) **Detection of Diethylene Glycol Impurity in Propylene Glycol by Near-Infrared Spectroscopy: Method Development and Validation**

Xiang Li[1,2], Sergey Arzhantsev[2], John Kauffman[2], Benjamin Westenberger[2], Lucinda Buhse[2], John Spencer[2]; [1]Penn State University; [2]FDA-Division of Pharmaceutical Analysis

Near-infrared spectroscopy (NIR) is a powerful tool for pharmaceutical analysis. Recent progress in technology has resulted in widespread availability of portable and handheld NIR spectrometers. Application of chemometric algorithms to analysis of NIR spectra significantly lowers limits of detection. Dramatic increases in importation of pharmaceutical materials into the US requires new screening processes at the borders to ensure the safety of the U.S. pharmaceutical supply chain. Rapid and reliable screening methods using portable spectroscopic instruments are being developed by FDA to assess the quality of imported drugs and excipients. We have developed a method based on combination of NIR portable spectrometers and a PLS calibration model to detect diethylene glycol contamination in propylene glycol. The method was developed on a single master instrument and distributed to 3 other instruments using piecewise direct standardization as a transfer of calibration (ToC) procedure. The ToC procedure included the measurements of 10 ToC samples before measurements of actual samples. The method was validated through a collaborative study which included 6 FDA Laboratories. The results obtained from the collaborative study were used to establish realistic limits of detection and to design a system suitability procedure. The ToC and analysis of collaborative study results will be discussed in detail.

(381) **Forensics Applications of Vibrational Spectroscopy and X-Ray Fluorescence to Nail Polishes and Their Signatures**

Dale L Perry[2], Shinobu T Heier[1]; [1]ThermoFisher Scientific; [2]Lawrence Berkeley National Laboratory

Not only does the analysis of nail polishes play an important role in the understanding and formulation of their chemistry in the cosmetic industry, but it also is of tremendous importance in the field of forensics. An understanding of details of nail polish can be critical in the solution of crimes committed in which polish has been found at the scene of a crime, many times on the nails of a body. Since modern nail polishes are mixtures of organic molecules such as solvents, metal chelates for color, and nitrocellulose, the determination of an exact match between the polish of interest and its parent commercial material. In the present study, vibrational spectroscopy and X-ray fluorescence have been used to study a variety of different nail polishes. The polishes have been studied with the primary interest of differentiating one polish from another with respect to their spectral signatures and the changes of these signatures in the context of such parameters as loss of parent solvent from their drying, curing, and aging. X-ray fluorescence has been used to monitor heavy metals in the polishes. One of the authors (DLP) wishes to acknowledge support of the U. S. Department of Energy under Contract Number DE-AC02-05CH11231.

(382) **Signatures of Inorganic Materials by Use of Multiple Spectroscopic Approaches**

Dale L Perry[1]; [1]Lawrence Berkeley National Laboratory

The analysis of materials by multiple spectroscopic and microscopic techniques can play an important role in the derivation and development of signatures, molecular-based “fingerprints” of materials that can be exclusively assigned to specific samples of materials. An understanding of details of different sets of analytical data can lead to combinations of these data to define different samples for the same material…but ones containing different micro- and nano-components. During this study, multiple

spectroscopic techniques have been used to detect markers---or signatures---for a wide variety of inorganic materials, signatures that uniquely describe the individual materials. These materials have been studied with respect to parameters such as elemental analyses, elemental ratios, different sets of elements that are unique to each material, and other experimental aspects that, due to the number of permutations of multiple spectroscopic parameters combined with analytical data, define materials from one sample to another. Possible algorithms for determining unique signatures for both molecularly identical and dissimilar materials are presented. The author wishes to acknowledge support of the U. S. Department of Energy under Contract Number DE-AC02-05CH11231.

(383) **Tablet Identification Using an FT-Near-IR Integrating Sphere with Principal Component Analysis**

Frank Weston[1]; [1]Varian, Inc.

After a grueling 10-15 years and an exorbitant $800 million a pharmaceutical company has developed a new drug candidate that has successfully passed all the FDA mandated clinical trials and is ready for prime time to patients in the United States. There should be no question as to why drug companies are eager to manufacture a generic version of a pioneer equivalent once it is no longer under patent protection. For example, in 2006 Pfizer reported over $1.5 billion in sales for its Zyrtec® product line that was losing patent protection in September of that year. Fortunately for the consumer, the Food & Drug Administration (FDA) passed the Waxman-Hatch Act in 1984 that requires generic drugs to maintain the same bioequivalence and pharmaceutical equivalence as their pioneer counterpart while the excipients can vary. Mid infrared spectroscopy, defined as the spectral region from 4000 cm-1 to 400 cm-1 and near infrared from 12,500 cm-1 through 4000 cm-1 each have the capability of identifying active ingredients and excipients. Each region has advantages and disadvantages that will be discussed while highlighting the features of Near-IR with an example differentiating between Zyrtec® and its Cetirizine generic counter parts.

(384) **Module for the Measurement of Photoluminescence in NIR and MIR Spectral Ranges**

Sergey Shilov[1], Michael Joerger[1]; [1]Bruker Optics

Measurement of photoluminescence (PL) in MIR and NIR spectral region is usually difficult because weak PL signal overlays with a strong thermal background radiation. The accessory module has been developed that allows easy discrimination between PL signal and thermal background. PL is exited with the laser which intensity is modulated using Pockels cell. Modulated PL emission radiation enters FT-IR spectrometer and demodulated using lock-in amplifier. Continuous scan and step-scan demodulation techniques will be compared and discussed in details. Applications examples will be demonstrated.

(385) **Determination of Derivatized Compounds in a Supersonic Jet**

Steven Goates[1], Lindsey Mills[1], Amy Felsted[1], Andrew Orton[1]; [1]Brigham Young University

Laser-induced fluorescence of molecules in a supersonic jet expansion has been shown to be a highly selective detection method for determination of specific compounds in complex matrices. Supercritical fluid carriers allow relatively nonvolatile molecules to be entrained in the jet. In this work, we investigate derivatization of steroid-like molecules to make them more suitable for supersonic jet spectroscopy, and we explore whether closely related molecules can be distinguished from each other.

(386) **Analysis of Composite Interphase Degradation**

Christopher N. Young[1], Clive R. Clayton[1], Richard D. Granata[2]; [1]Stony Brook Univ, Dept of Materials Science; [2]Florida Atlantic Univ, Dept Ocean Eng

Carbon fiber / vinyl ester composite materials are of great interest for naval and other military applications. One major weakness of polymeric composite materials is their susceptibility to environmental degradation from sunlight, heat and water. While the degradation properties of polymer resins are generally well-understood, little is known about the polymeric sizing applied to carbon fibers to compatibilize them with the matrix. Through the use of Raman, FTIR and x-ray photoelectron spectroscopy, the proprietary sizing applied to a system of carbon fibers has been observed. The interphase region formed by the sizing and the resin has been studied in fully-formed composite materials. The exposure effects of heat and simulated sunlight on the physicochemical properties of the sizing and interphase region in these composites have been investigated.

(387) **Vesicle-Based Tools for Single Cell Analysis**

Michelle L. Kovarik[1], K. Scott Phillips[1], Hsuan-Hong Lai[1], Nancy L. Allbritton[1]; [1]University of North Carolina - Chapel Hill

At the level of individual cells, both normal and diseased samples show substantial heterogeneity, and chemical analysis of the contents of single cells has revealed unique subpopulations that are obscured by ensemble methods. Single cell analyses, however, face a number of challenges, including small sample volumes and the need for high throughput so that statistically relevant numbers of cells can be analyzed in reasonable time frames. Miniaturized analysis systems address these challenges by decreasing dead volumes and increasing automation, but several obstacles must be overcome before microfluidics are routinely applied to clinically relevant samples. For example, physiological buffers generally make poor separation buffers, and the cell-to-cell variation that makes these analyses worthwhile also complicates the characterization of new analysis systems. We present two lipid vesicle-based tools for overcoming these challenges to on-chip single cell analysis. Small unilamellar vesicles spontaneously fuse with hydrophilic microchannel walls to form a supported lipid bilayer coating. Previous work in our laboratory has shown that these coatings permit electrophoretic separations to be performed in extracellular buffer, eliminating the need for buffer exchange prior to separation. Optimization of these coatings and other separation parameters for use with fluorescent ABL kinase reporter peptides will be presented. Additionally, giant unilamellar vesicles filled with fluorescein and Oregon Green dyes are evaluated as standards for characterizing new chemical cytometry instrumentation.

(388) **Trapping of Proteins by Insulator-Based Dielectrophoresis**

Asuka Nakano[1], Sanchari Bhattacharya[1], Tzu-Chiao Chao[1], Alexandra Ros[1]; [1]Arizona State University

The reliable and rapid separation of proteins is essential for biological research and medical analysis. The present study demonstrates the manipulation of proteins, chicken IgGs, using insulator-based dielectrophoresis (i-DEP) under direct current electric fields. Insulating posts integrated in a microfluidic platform create a non-uniform electric field necessary for DEP to occur, thus trapping of fluorescently labeled IgG occurs in the insulating post arrays. Proteins tend to aggregate, thus preventing the formation of aggregates is critical for protein manipulation. We used denaturants including urea and 3-[(3-Cholamidopropyl)dimethylammonio]-1-propanesulfonate (CHAPS) with varying concentrations to denature proteins. The size distribution of proteins under denaturing condition was determined by dynamic light scattering (DLS). Results obtained from DLS showed that the use of 6mg/mL

CHAPS in buffer solution is best suited for DEP trapping. In parallel, DEP trapping experiments were performed with varying CHAPS concentrations and, as a result, we confirmed that proteins denatured by 6mg/mL CHAPS were observed to have most significant DEP response. In addition, we study the effect of the magnitude of the applied voltage and the post geometry on the dielectrophoretic response of the protein particles. A mathematical model is built with COMSOL Multiphysics software in order to predict the dielectrophoretic forces exerted on the particles for different post geometries and the varying applied voltages. We find maximum trapping forces in the order of 10-16 N. Experimentally, we demonstrate negative DEP trapping of the fluorescently labeled IgG proteins with an onset voltage of trapping of 660V/cm using a triangular post geometry, corresponding to a field strength of 10^5 V/m in the post array. Using this DEP trapping behavior of proteins, we expect to develop a novel method for protein separation in a microfluidic device, dependent on the polarizability characteristics of the involved biomolecules and based on the tailored microstructuration in the separation channel.

(389) **Dielectrophoretic Single Cell Trapping in a Microfluidic Lab-on-Chip Device**

Sanchari Bhattacharya[1], Tzu-Chiao Chao[1], Prof. Alexandra Ros[1]; [1]Arizona State University

Conventional biological studies are carried out with huge cell populations making it difficult to assess cell-cycle dependent states or inhomogeneous responses to external stimuli. However, accessing the information inherent to single cells will allow us to resolve such inhomogeneities and eventually improve our understanding of enduring problems in molecular biology, cancer diagnostics, pathology and therapy. Single cell analysis involves the challenge of handling and detection of 105 molecules or less in pL volumes. Here, we propose a microfluidic approach, capable of navigating and positioning single cells in parallel with the ultimate goal of creating an interface to matrix assisted laser induced desorption ionization mass spectrometry (MALDI-MS). In particular, we demonstrate the design of single cell traps using dielectrophoresis with model colloids and cancer cells. We employ insulator-based dielectrophoresis (i-DEP) for the immobilization of the target cells. In i-DEP, the deformation of the electric field, E, is evoked due to positioning of insulators in the channel path, creating inhomogeneous electric fields over the entire depth of the microchannel. Polarizible particles respond to this electric field gradient either by moving towards regions of high electric field gradient (positive DEP) or by moving away from it (negative DEP). If DEP forces overcome all other acting forces such as bulk flow, particles or cells are trapped. Numerical simulations using COMSOL multiphysics software allow us to investigate the electric field distribution in a microchannel containing insulating posts in detail for various geometric designs. Under DC conditions, we compare the calculated electric field gradients and forces acting on the microparticles with drag forces and determine optimum trapping conditions. Experimentally, we fabricated microchannels with corresponding design in poly(dimethylsiloxane), in which trapping behavior of polystyrene microbeads (10 μm diameter) in 500 μS conductivity near physiological pH was investigated. Our results indicate negative-DEP of the microbeads above 1500 V applied potential corresponding to an electric field of 2.4 × 107 V/cm and of 2.9 × 10-5 V2/m3 at the trapping areas. Ongoing work is dedicated to DEP trapping of cancer cells in these single cell traps for our future goal of a single cell microfluidic platform coupled to MALDI-MS detection.

(390) **Internally-Etched Silica Encapsulated Gold Coated Silver Nanostructures for Improved SERS Detection**

Sudip Nath[1], Binaya Shrestha[1], Amanda Haes[1]; [1]The University of Iowa

Surface-enhanced Raman scattering (SERS) is a highly sensitive and label-free detection technique for both biological and environmental targets. In this work, we synthesize encapsulated gold coated silver (Ag@Au) nanoparticles as efficient, improved, and quantitative SERS substrates versus other solution-phase materials. The optical properties of Ag@Au nanoparticles arise from the localized surface plasmon resonance of the parent metals and are tunable as the molar ratios of the parent components are varied. As with most solution-phase nanostructures, Ag@Au nanoparticles exhibit irreproducible SERS signals because of their inherent instability in solution and changing optical properties. To combat this detection limitation, Ag@Au nanoparticles have been encapsulated in silica shells (Ag@Au@SiO2). These shells offer robust protection of the metal nanoparticles against aggregation thereby preserving the novel plasmonic properties of the noble metal cores; however, the silica shell also prevents target molecules from interacting with the core surface which eliminates the SERS effect. To overcome this problem, the silica shells on Ag@Au nanoparticles have been converted into silica membranes thereby forming internally-etched silica membrane stabilized Ag@Au (IE-Ag@Au@SiO2) nanoparticles. It will be revealed that these nanostructures consist of permeable silica membranes, exhibit stable optical properties in the presence of 2-naphthalenethiol, and as a consequence, show reproducible and temporally stable SERS signals. Nanoparticles' molecular concentration will be shown to systematically influence the SERS response. This demonstration of utilizing solution-phase IE-Ag@Au@SiO2 nanoparticles as reproducible SERS substrates encourages future fundamental studies to understand the SERS mechanisms as well as realizations of applications which require the direct and reproducible detection of biomolecules.

(391) **Use of Sonication Power to Control Length Distributions of SWNTs in Aqueous Suspensions Used for Network Deposition**

Meagan A. Cauble[1], Pornnipa Vichchulada[1], Jihye Shim[2]; [1]University of Georgia; [2]Kyung Hee University, Korea

Despite their enhanced electronic properties, variations in the electronic properties of carbon nanotubes (SWNTs) limit their applications in electronic materials based on individual nanotubes. Therefore, investigations of various methods for forming 2-dimensional networks of SWNTs have recently emerged. In particular, due to the advantages of possible use on flexible substrates and the ability to chemically modify/purify SWNTs prior to network formation, there is great interest in room-temperature deposition methods. Therefore, the development of methods for obtaining control over the concentration and average length of SWNTs in aqueous suspension is of critical importance. A major challenge in the formation of aqueous suspensions of SWNTs is overcoming their insolubility, which is caused by strong inter-SWNT van der Waals attractions. The effect of ultrasonic probe sonication, which was used to disperse SWNT bundles into suspension, on the length and extent of defects on the SWNTs was investigated via atomic force microscopy (AFM) and Raman spectroscopy, respectively. Quantitative information about the suspension concentration and the effect of sonication power on unbundling the SWNTs was observed via UV-Vis and near-IR spectroscopy, respectively. Transmission UV-Vis spectroscopy was used to obtain extinction coefficients for a series of standards. Then, this information was used to determine the concentration of nanotubes in purified suspensions. In order to obtain suspensions of high-aspect-ratio, undamaged SWNTs, the effect of the magnitude

of energy applied during sonication on the average nanotube length and defect density was investigated. In order to form suspensions of individual SWNTs, a non-oxidizing purification method was used to remove any bundles of SWNTs, carbonaceous impurities and catalyst nanoparticles. This presentation will demonstrate that the concentrations of SWNT dispersions are not greatly affected by the initial concentration of SWNTs. However, the sonication power used strongly influenced SWNT dispersion and defect density. Higher sonication powers yield higher concentrations after purification, although a concurrent decrease in the average length of SWNTs was observed. Additionally, the density of defects, as observed by Raman Spectroscopy, increased commensurately with sonication power. Findings regarding the ideal conditions to use for suspension formation will be presented.

(392) **Quantitative Study of Ligand Adsorption onto Metal Nanoparticle Using Surface Enhanced Raman Internal Reference Method**

Siyam Ansar[1], Dongmao Zhang[1]; [1]Mississippi State University

With their unique electromagnetic and chemical properties, metal nanoparticles have found applications in diverse areas that include medicine, biosensing and chemical catalysis. For many of these applications, surface modification is often a necessary procedure to impart nanoparticle necessary functionality, stability as well as target specificity. As such, fundamental understanding how ligand interacts with metal nanoparticle is of great importance in nanotechnologic development. However, among all the methods proposed for studying ligand/nanoparticle adsorption, few of them are quantitative. Important information such as ligand binding constant and ligand binding densities for many important ligand/nanoparticle binding systems are currently lacking. In this talk we will present a ratiometric ligand quantification technique that is based on the isotope encoded surface enhanced Raman internal reference (IESIR) method we developed previously. Using mercaptobenzimidazole as an example ligand, we demonstrated that ligand adsorption isotherm can be readily obtained with this IESIR method, which allows accurate determination of the ligand binding constant as well as ligand binding density on the nanoparticle surfaces. In addition to its high quantification accuracy, this IESIR based technique has a wider applicability as essentially all the nanoparticle ligand are SERS active, thus can be quantified with the IESIR method.

(393) **Whispering Gallery Resonating Nanoparticles**

Zachary Koontz[1], George Chumanov[1]; [1]Clemson University

Whispering gallery resonators are dielectric spherical structures capable of sustaining electromagnetic waves that are strongly confined within the spherical structure. The containment of the waves is due to the total internal reflection that occurs in between two dielectric layers of different refractive index. The proposed whispering gallery resonators are of a core multishell structure that consisted of a silver nanoparticle core followed by a silica shell and a strontium titanate shell. Silver nanoparticles behave as optical antennas, while silica and strontium titanate have strongly contrasting refractive indices, 1.46 and 2.3 respectively. These nanoparticles are investigated for analytical applications.

(394) **Time-Resolved Spectroscopy of Plasmon-Enhanced Luminescence from Rare-Earth Ions**

Jaetae Seo[1], Maria Veronica Rigo[1]; [1]Hampton University

The optical properties of spontaneous emissions and lifetimes of rare-earth (RE) ions of dysprosium, europium, samarium, and praseodymium in close proximity to the metal nanoparticles (MNPs) of Ag or Au are studied for the photonic applications of plasmonic RE-ion displays and lasers. The RE nitrate pentahydrates or hexahydrates are dissociated in aqueous solution under an ultrasonication at room temperature, and Au or Ag nanometals are added to enhance the luminescence of RE ions by interacting with plasmons. The absorption and emission spectra, and lifetimes of RE ions as a function of the concentration of plasmonic nanometals or RE-ions are characterized with high resolution spectroscopy and time-resolved spectroscopy. The plasmonic emissions in the visible spectra from the RE ions are assigned to 4f interlevel transitions of 4F9/2 – 6HJ (J=15/2 and 13/2) for Dy3+; 5D3 – 7F4, 5D1 – 7FJ , and 5D0 – 7FJ (J=1 and 2) for Eu3+; 4G5/2 – 6HJ (J=5/2, 7/2, and 9/2) for Sm3+; and 1I6 – 3F3, 1D2 – 3H4, and 3P0 – 3F2 for Pr3+. The large luminescence enhancement and increase in radiative decay rate of the RE ions are attributable to the local field enhancement and possible energy transfer in vicinity of plasmonic nanoparticles. Polarization-dependent luminescence enhancement provides a clear evidence of the coupling of RE-ion transition dipoles with plasmon modes in nanometals. Time-resolved PL spectroscopy on RE-ions as functions of the concentrations of both nanometals and RE-ions clarifies the temporal dynamics of energy transfers from RE-ions to plasmons in MNPs. The PL decay of plasmonic RE-ions may have fast and slow exponential decay rates of 1) direct energy transfer from RE-ions to MNPs with relatively fast decay, 2) energy transfer between RE-ions which led to concentration quenching, and 3) radiative recombination within RE-ions with relatively slow decay. If the fast decay component becomes dominant than the two slower components, it indicates the PL quenching occurred by fast energy transfer from RE-ions to MNPs. Luminescence spectroscopy on RE-ions as a function of nanometal concentration analyzes the balancing mechanism between enhancement and quenching processes. This work was supported by the National Science Foundation (HRD-0734635 and HRD-0630372).

(395) **Ambient Measurements of Charge Transport with Designed Surface Structures of Cobaltacarborane Porphyrins Using Conductive Probe Atomic Force Microscopy**

Venetia D. Lyles, Wilson K. Serem, Erhong Hao, M. Graca H. Vicente, Jayne C. Garno; [1]Louisiana State University

A considerable challenge is posed for developing reliable and reproducible methods for measurements in nanosized systems, for scaling surface structures to nanometer length scales, and for evaluating the effects of molecular structure on electrical properties. Quantum effects in electronic conduction have been observed as the size of electronic devices approach molecular scales. Advancement of molecule-based electronic systems will require the ability to routinely achieve reliable and precise measurements of conductance for molecular test structures. Our goals are to develop approaches to prepare robust test platforms of nanostructures of functionalized porphryins, with control of the molecular orientation on surfaces. Conceptually, by arranging and orienting porphyrins on well-defined surfaces, local measurements of charge transport can be enabled for different pathways through the molecules. Also, the resulting size-dependent properties for molecular aggregates can be evaluated with reliability and sensitivity. The molecular-level organization of porphyrin films on surfaces is known to greatly influence the conductive, photoemissive and photovoltaic properties of thin film devices such as light-emitting devices and organic photovoltaics. Accurate and precise electronic property measurements will shed insight on the fundamental mechanisms that give rise to properties as a function of chemical structure. Depending on slight changes in protocols for sample preparation, a range of different surface structures can be generated for porphyrins to produce supramolecular assemblies, arrays, aggregates or crystals. Cobaltacarborane porphyrins with four to sixteen carborane clusters per macrocycle were synthesized in excellent yield (90-97%) using a ring-opening zwitterionic reaction. Conductive probe atomic force microscopy (CP-AFM)

measurements are accomplished by applying a bias voltage to the sample and measuring the current with a metal-coated probe. Topography and current images can be acquired simultaneously by scanning the AFM probe at a fixed sample bias using contact mode feedback. Topography frames provide height information of the surface morphology whereas corresponding current images furnish sensitive maps of the conductive domains of the samples. Local current versus voltage spectra can be acquired with CP-AFM for thin films prepared on conductive or semiconductive substrates. Results will be disclosed for changes of conductive properties of surface structures of porphyrins with different numbers of cobaltacarborane substituents.

(396) **Controlling the Surface Density of Organosilane Nanostructures: Particle Lithography Strategies for Preparing Nanostructures with well-Defined Periodicity and Geometries**

ChaMarra K. Saner, Kathie L. Lusker, Zorabel M. LeJeune, Jayne C. Garno; [1]Louisiana State University

High density arrays of periodic nanostructures of organosilanes can be prepared using different strategies for particle lithography, which require only basic bench chemistry steps of mixing, heating, centrifuging and drying. Particle lithography has previously been applied to pattern metals, inorganic materials, nanoparticles, proteins, polymers, and self-assembled monolayers (SAMs). Key advantages of particle lithography are the capabilities to tailor the geometries and spacing for nanostructures. Particle lithography provides a practical way to define spatial selectivity at the nanoscale, since the selection of SAM molecules yields many possible combinations and terminal moieties for further steps of linking nanomaterials to surfaces. Billions of nanostructures can be prepared with relatively few defects and high reproducibility to enable patterning of large areas. The surface density, as well as the size and periodicity of the nanostructures can be tailored by the diameters of templating mesoparticles. The mesoparticle diameter defines the periodicity of the nanostructures; however the sizes of the nanostructures are much smaller, depending on the physical area of contact between the mesospheres and surface. After solutions of latex or colloidal silica spheres are dried on surfaces, a small amount of water persists and forms a meniscus between the base of the particle and the area of contact with the substrate. Silanes attach to surfaces by successive steps of hydrolysis and condensation, therefore nanoscopic amounts of water are needed to initiate the reaction. By controlling the drying parameters, nanopatterns of rings or pore structures can be produced using protocols with vapor deposition, immersion, or contact-printing. Producing high quality monolayer films of organosilanes has proven to be a challenge, since it is difficult to control the cross-linking during the polymerization. The thickness of the nanostructures indicate the packing density of organosilanes, since a densely packed film would have heights corresponding to a fully upright orientation with the molecular backbone aligned perpendicular to the substrate. High resolution AFM characterizations were used to evaluate the quality of the SAM nanostructures. Results will be presented which demonstrate that slight modifications in the protocols for sample preparation can significantly change the outcome for the quality of the nanostructures that are produced.

(397) **Plasmonic Nanometals and Semiconductor Nanocrystals for the Fabrication of Hybrid Optical Material Structure**

Maria Veronica Rigo[1], Jaetae Seo[1]; [1]Hampton University

The spontaneous emission from semiconductor quantum dots (SQDs) can be modified through coupling of excitons with surface plasmon (SP) in nearby metallic nanostructures. The strong couplings of SQDs to SPs have many applications including photonic bioassay, optoelectronics, light-emitting diode, and solar cell applications. The local-field enhancements associated with SPs can increase light absorption or alter the radiative and nonradiative decay rates of SQDs. The interaction between SQDs and metal nanoparticles (MNPs) is revealed as an enhancement or suppression of SQDs emission for a given wavelength and it depends on the SP's density of state at that wavelength. The emission properties as a function of the distance between SQDs and MNPs with various morphologies of nanometal structures and the degree of coupling between the SP resonance bands of the MNP and the excitation/emission bands of the nanocrystals were studied. The hybrid SQD-MNP nanostructure consists of a single array of MNPs, deposited on a flat quartz substrate, and a single layer of SQDs in the close proximity the nanoparticles. In order to prove the distance-depencent plasmon-exciton coupling, a spacer layer was built between these materials using the Layer-by-Layer (LBL) technique. Such spacer layer has a thickness varying from values smaller than the radius of SQDs to larger than the radius of MNPs. The photoluminescence of excitons in the vicinity of plasmons may be readily quenched due to their direct coupling through a fast energy transfer. Main challenges in the hybrid MNP-SQD nanostructures are a precise control of spacing between MNPs and SQDs, and accurate tunings of energy bands of excitons and plasmons. The energy bands of MNPs and SQDs can be tuned accurately by controlling the morphology of bothMNPs and SQDs. The thickness of spacer between MNPs and SQDs is often controlled by the LBL assembly which provides nanometer precision from a few nanometers up to ~100 nm. The accessibility of MNPs onto the SQD surface may also result in photoluminescence quenching. This work was supported by the National Science Foundation (HRD-0734635 and HRD-0630372).

(398) **Detection of SMC1/SMC3 Protein Binding to DNA via Quartz Crystal Microbalance**

Laura Steller[1], Rolf Jessberger[2], Hagen Schmidt[1], Magdalena Laugsch[2], Electra Gizeli[3]; [1]IFW; [2]Inst. of Physiol. Chem.; [3]Biosensors Lab

The interactions of DNA-SMC proteins (Structural Maintenance of Chromosomes) are core subunits of the cohesion complex which is essential for the linkage of newly replicated chromosomes. Each of our cells inherits their genetic information in the form of chromosomes from a mother cell. In order that we obtain the full genetic complement, cells need to ensure that replicated chromosomes are accurately split and distributed during cell division. Yet, little is known so far about the molecular features of DNA-SMC proteins interactions. Our goal address DNA interaction of SMC proteins by a sensitive and quantitative method based on quartz chrystal microbalance (QCM), which allows to measure such interactions in a soluble system, and yield novel data about the DNA/SMC complex proteins interaction.

(398a) **Synthesis and Application of Multishell Silver Core Nanoparticles**

Whitney Snyder[1], George Chumanov[1]; [1]Clemson University

Novel multishell nanostructures with widely tunable optical properties across visible and near infrared spectral ranges are synthesized. The development of these nanostructures as optical labels for bioanalytical applications is presented. These nanostructures consist of a metal or dielectric core coated with repeating dielectric and metal layers resulting in an onion-like nanostructure. The metal cores are composed of small silver nanoparticles grown by hydrogen reduction while the dielectric layers consist of silica or titania. Changing the number, thickness and chemical nature of these metal-dielectric layers allow the optical properties to be tuned. Embedding organic molecules in the dielectric layers allow for optical labeling using surface enhanced Raman and fluorescence. Optical properties of these onion,

nanostructures are characterized using UV-Vis spectroscopy and electron microscopy is used to monitor the growth of the nanostructure.

(399) **Advances in Structural Characterization and High Spatial Resolution Imaging of Lipid Species with Ion Mobility Mass Spectrometry**

Michal Kliman[1], Jay G. Forsythe[2], John A. McLean[3]; [1]Department of Chemistry, Vanderbilt University

The recently commercialized ion mobility-mass spectrometry (IM-MS) couples well to both electrospray ionization (ESI) and laser desorption (LD) based soft ionization sources, and is highly amenable to circumventing multiple lipid analysis challenges. Through collisions with a neutral gas, IM-MS first separates molecular ions on the basis of their apparent surface area or ion neutral collision cross section, then by mass. IM-MS affords rapid (μs to ms) two dimensional size and mass based separations with unique repercussions to the analysis of complex biological samples, Including lipids. Recently, Woods et. Al [1] and Fenn, et. Al [2] have shown tht lipids can be efficiently resolved from peptides, carbohydrates, and nucleotides in the IM dimension of IM-MS based on the differences in their gas phase packing that leads to differences in their apparent gas phase surface area. This attribute of IM not only provides substantial reduction of chemical noise in lipidomic measurements, and a concomitant increase in dynamic range, but it can also exploit an additional characteristic of lipid ions: various types of lipids themselves display differences in gas phase packing [3] detectable by IM-MS that can be further utilized for their enhanced resolution, detection and description of their gas phase behavior for both gas phase structural characterization and lipid profiling and imaging from tissues and cells. The basis for the structural separation of phospholipid species based on molecular dynamics simulations of their gas phase behavior will be discussed and the utilization of this knowledge in lipid profiling and imaging of animal brain tissues and cells using MALDI and nanostructure initiator mass spectrometry ionization will be presented. [1] Woods, A. S.; Ugarov, M.; Egan, T.; Koomen, J.; Gillig, K. J.; Fuhrer, K.; Gonin, M.; Schultz, J. A., Analytical Chemistry, 2004; Vol. 76, pp 2187-2195. [2] Fenn, L. S.; Kliman, M.; Mahsut, A.; Zhao, S. R.; McLean, J. A., Analytical and Bioanalytical Chemistry, 2009; Vol. 394, pp 235-244. [3] Jackson, S. N.; Ugarov, M.; Post, J. D.; Egan, T.; Langlais, D.; Schultz, J. A.; Woods, A. S., Journal of the American Society for Mass Spectrometry, 2008; Vol. 19, pp 1655-1662.

(400) **Quantitative Measure of Soybean Protein and Lipid Content Using Transmission Raman Spectroscopy**

Matthew Schulmerich[1], Michael Asensio[1]; [1]The Beckman Institute; [2]The National Soybean Research Laboratory; [3]Illinois Crop Improvement Association

Reliable measure of the protein, oil, and other economically important soybean grain components is an important concern in the soybean industry. Both buyers and sellers (including soybean producers, grain elevators, co-ops, processors, and distributors) desire reliable measure of grain composition. In commercial trade, uncertainty in analysis of a grain component can cause major losses to grain elevators in recouping premiums paid for a particular seed component. Additionally, soybean breeders attempting to develop and improve grain quality require reliable information about grain composition. Precise and accurate prediction of composition has proven to be challenging and is still an active area of research. The current technology standard for grain analysis in the soybean industry is near infrared (NIR) spectroscopy which provides quick and easy whole grain sample analysis. We explore the use of transmission Raman spectroscopy to provide more robust compositional analysis of the multiple attributes contained in whole soybean grain. Raman spectroscopy inherently has higher chemical specificity than NIR spectroscopy as the NIR spectrum arises from scattering-modified absorbance of vibrational overtones as opposed to absorbance due to fundamental vibrational modes. We have built a transmission Raman instrument for data collection on individual soybeans and acquired data from several soybean varieties. Calibration curves using wet chemistry analysis will be used to deduce concentrations. The precision and accuracy that can be achieved with Raman spectroscopy will be reported and compared to conventional near infrared (NIR) spectroscopy. As this is on going research the number of samples and latest results will be reported.

(401) **X-ray and UV Excited Luminescence of Doped Y2O3 Nanocrystals**

Yan Zhang[1], CV Gopal Reddy[1], Tuan Vo-Dinh[1]; [1]Duke University

Rare earth doped yttria nanocrystals (Y2O3: RE3+, RE=Eu, Tb) were prepared by combustion method and annealed at various temperatures to obtain luminescent nanocrystals. The structure and morphology of doped yttria nanoparticles were characterized by x-ray diffraction, transmission electron microscopy and light scattering. The size of the nanoparticles was in the range of 10-100 nm, and showed a narrow size distribution and high crystallinity. Both X-ray and UV can effectively excite the phosphors and the emission spectra are similar. The particle size and luminescence intensity increase as the sintering temperature rises. This work allows us to understand the phosphors' microstructure on luminescent properties under x-ray and UV photons.

(402) **Modification and Characterization of a Commercial FT-IR Accessory for Surface Plasmon Resonance Spectroscopy**

Nicola Menegazzo[1], Laurel Kegel[1], Karl Booksh[1]; [1]University of Delaware

The Harrick AutoSeagull accessory for FT-IR spectrometers enables use of several sampling techniques, including specular and total internal reflectance, in a highly flexible platform. Compatibility with total internal reflectance is of special interest since it also forms the bases for surface plasmon resonance (SPR) spectroscopy for prism-based configurations. The work presented here discusses the adaptation of an AutoSeagull in order to perform SPR analysis, allowing for easy incorporation into most common FT-IR spectrometers. Surface plasmon resonance (SPR) spectroscopy is most typically used to measure changes in refractive index with high sensitivity near the surface of a plasmon supporting metal (approximately 200 nm), rendering it the ideal tool for thin layer analysis. This has led to its implementation in the determination of binding constants between biomolecules, monitoring of rate and quality of film formation (inorganic, organic and biological) as well as for sensing applications. Assuming all other conditions constant, changes in RI can be recorded as either shifts in incident angle or the excitation wavelength. With respect to the results discussed here, the constant angle approach was employed, meaning that the analytical signal resulted from shifts in the wavelength of SPR coupling. Adaptation of the AutoSeagull to SPR analysis involved the incorporation of slit apertures to minimize the angular throughput reaching the detector, resulting in sharper SPR 'dips' but at the cost of noisier spectra. In addition, discussion of the system's analytical performance includes comparison of dip width as a function of slit size, tailoring of the dip minima location as a function of incident angle and sensitivity to bulk RI changes.

(403) **Standoff Resonance Enhanced Multiphoton Ionization (REMPI) for Detection of Hazardous Materials**

Maria Damian[1], Janna Register[1], S. Michael Angel[1]; [1]University of South Carolina

Resonance Enhanced Multiphoton Ionization (REMPI) is a very sensitive and selective laser ionization technique for measuring molecular vapors, and a REMPI excitation scan provides a molecular "fingerprint". In the REMPI experiment, a relatively low-power pulsed laser at a wavelength of an electronic absorption band of the molecule excites and subsequently ionizes the molecule by a multi-photon process. Previously we have shown ppb detection of chlorinated and aromatic contaminants, and we have used REMPI measurements to follow the degradation of perchloroethylene (PCE) during its conversion to trichloroethylene (TCE), *in-situ*. REMPI can also be used to measure solid materials, using a pulsed laser to both desorb and ionize the analyte. In recent work we have used this approach to measure TNT, RDX and other high explosives (HE) materials. Currently we are investigating methods to measure HE materials remotely and are considering REMPI with microwave scatter detection and photoacoustic detection. This paper will focus on a description of the methods being considered, measurement data for a variety of HE materials and a discussion of the range limitations of the techniques.

(404) **Use of a Handheld NIR Spectrometer to Assess Agricultural Materials**

David Himmelsbach; [1]Light Light Solutions, LLC

Several handheld spectrometers have appeared on the market for the purpose of taking the spectrometer to the sample in the field. These include Raman, mid- and near-infrared systems. Most of them are aimed at providing rapid identification of unknown materials for security issues. Many of them can also be used for quantitation and classification in the same manner as with laboratory based instruments making remote analysis a reality. The application of a handheld near-infrared (NIR) spectrometer for assessing the fiber content of flax stems, the measurement of bio-oil in petrodiesel, and classification of high versus low oleate in shelled peanuts and latex in dandelion roots will be shown.

(405) **Spectral Imaging of Coupled Localized and Propagating Surface Plasmon Resonance in Nanohole Arrays**

Laurel Kegel[1], Karl Booksh[1]; [1]University of Delaware

Nanohole arrays are investigated as fundamental structures that support the interaction of localized and propagating surface plasmon polaritons as well as substrates for potential use in surface enhanced techniques and surface plasmon resonance (SPR) sensing applications. Exhibited advantages of nanohole arrays include greater sensitivity to bulk refractive index than planar gold, localized electric field enhancement, and tunable SPR wavelength. Far field imaging of nanohole arrays and nearby nanostructures or SP active materials elucidates the interaction of the various SP modes. Furthermore, the LSPR arising from the nanoholes and SP propagation through the continuous gold depends on the diameter and periodicity of the nanoholes, which allows for precise tuning of the SPR wavelength. The SPR wavelength may be controlled for optimal coupling to nanostructures. Exploration of coupling the SP modes of nanohole arrays with other SP active materials to further exploit the localized field is of great interest.

(406) **Physicochemical Determination of the Role of Methylene Chloride and Phenol in Paint Strippers**

Christopher Young[1], Kelly Watson[2], James Yesinowski[3], Clive Clayton[1], James Wynne[3], Young Han[4]; [1]Stony Brook University; [2]Science Applications International Corp.; [3]Naval Research Laboratory; [4]Naval Air Systems Command

Chemical paint strippers containing methylene chloride and phenol have been used extensively in military applications to remove polymer coatings from metal substrates. While these solvents are highly effective, they present numerous health and environmental hazards. Substitutes designed to avoid these problems suffer from inadequate performance and increased cost. The degradation mechanisms by which paint stripping solvents function have not been fully determined. The effects of exposure by solvent components of military standard paint stripping solutions have been evaluated on polyurethane topcoats and epoxy primers similar to those currently employed by the Army and Navy. Chemical changes have been examined through Raman, solid-state 1H NMR, ATR- and PAS-FTIR spectroscopy, while physical changes have been characterized through TGA and DSC.

(407) **Raman Spectroscopy of Supported Lipid Bilayers**

Zhorro Nickolov[1], Selver Ahmed[2], Stephanie Wunder[2]; [1]Centralized Research Facility, Drexel University; [2]Dept of Chemistry, Temple University

Room temperature Raman spectra were obtained for supported lipid bilayers (SLBs) of DMPC, DPPC and DSPC, and temperature dependent Raman spectra were obtained for SLBs of DPPC on silica nanoparticles ranging in size from 5 to 100 nm, and compared with small unilamellar vesicles (SUVs) of ca. 100 nm. Spectra were obtained both in the CH stretching and backbone regions. The intensity ratio of the symmetric CH stretch at 2850 cm-1 to the asymmetric CH stretch at 2880 cm-1, often used as a measure of lateral packing of the alkane chains, with lower numbers indicating higher order, was obtained. The order parameter at room temperature for DPPC was found to increase with decreasing nanoparticle size. These results support a model in which the chains become increasingly interdigitated as the size of the lipid approaches the nanoparticle size. Interdigitation occurs in order to increase hydrophobic interactions between the alkyl chains as the free volume between the chains increases with decreasing nanoparticle size, which would occur if the chains packed in the normal lipid bilayer morphology. The same trend has previously been observed for SUVs with added glycerol, also known to induce interdigitation in lipid bilayers.

(408) **Lipid Analysis Using Nanostructure-Initiator Mass Spectrometry**

Jay Forsythe[1], Michal Kliman[1], Joshua Broussard[1], Donna Webb[1], John McLean[1]; [1]Vanderbilt University

In 2007, a new surface-based mass spectrometry method called nanostructure-initiator mass spectrometry (NIMS) was introduced by Gary Siuzdak and colleagues [1]. Unlike matrix-assisted laser desorption ionization (MALDI), NIMS employs a porous silicon scaffold filled with viscous siloxane "initiators" to ionize analytes. Upon laser irradiation, initiator molecules explode out of the pores; as a result, molecules placed on the surface experience desorption and ionization. Since the initiator matrix in NIMS does not apparently ionize, it can offer two distinct advantages over weak organic acid-based MALDI. First, ion suppression due to matrix does not occur, which gives NIMS impressive sensitivity. Second, NIMS is promising in the field of imaging mass spectrometry because it is not limited by the size of analyte-matrix co-crystallization; as a result, spatial resolution is solely dependent on the spot size of the laser. Recently, molecules which traditionally do not ionize well using MALDI, such as cholesterols and fatty acids, have been detected with high sensitivity using NIMS [2]. We have synthesized our own NIMS wafers according to a procedure in the literature [3], and confirm the ability to detect traditionally difficult molecules to ionize using NIMS with low-femtomol sensitivity. In addition, we look to isolate, profile and image lipid signals from cell monolayers using time-of-flight mass

spectrometry (TOF-MS) and ion mobility time-of-flight mass spectrometry (IM-TOF-MS). Sources: 1. Northen, T.; Yanes, O.; Northen, M.; Marrinucci, D.; Uritboonthai, W.; Apon, J.; Golledge, S.; Nordstrom, A.; Siuzdak, G., Clathrate nanostructures for mass spectrometry. Nature 2007, 449 (7165), 1033-U3. 2. Patti, G.; Woo, H.; Yanes, O.; Shriver, L.; Thomas, D.; Uritboonthai, W.; Apon, J.; Steenwyk, R.; Manchester, M.; Siuzdak, G., Detection of Carbohydrates and Steroids by Cation-Enhanced Nanostructure-Initiator Mass Spectrometry (NIMS) for Biofluid Analysis and Tissue Imaging. Analytical Chemistry 2010, 82 (1), 121-128. 3. Woo, H.; Northen, T.; Yanes, O.; Siuzdak, G., Nanostructure-initiator mass spectrometry: a protocol for preparing and applying NIMS surfaces for high-sensitivity mass analysis. Nature Protocols 2008, 3 (8), 1341-1349.

(409) **Infrared Spectral Imaging Analysis of Cartilage Repair Tissue Over Time**

Madhuri Penmatsa[1], Paul West[2], Xu Yang[2], Nancy Pleshko[1]; [1]Temple University, PA; [2]Hospital for Special Surgery, NY

Recent research has focused on developing many new technologies for cartilage repair. The successful application of these strategies is limited in part to lack of techniques to evaluate tissue response to interventions. Assessment of the structural and molecular changes in the primary cartilage components, proteoglycan (PG) and collagen is critical to evaluate progress of the repair tissue. In the present study Fourier transform infrared imaging spectroscopy (FT-IRIS) was utilized to evaluate molecular changes in normal and degenerated cartilage in a rabbit model of repair. Bilateral full thickness osteochondral defects were created in mature rabbits, and the defect and surrounding tissue removed and harvested at 2, 4 6, 8, 12, and 16 weeks post-defect creation. Tissues were processed and sectioned at 7µm thickness and mounted onto low-e slides for IR imaging and on glass slides for histology. FT-IRIS was performed using a Perkin Elmer Spectrum Spotlight 400 imaging spectrometer. A rectangular region of interest that contained the defect and adjacent normal cartilage was selected, and data were acquired at a spatial resolution of 25µm and spectral resolution of 8 cm-1. Imaging data for repair and normal cartilage were analyzed using ISys 5.0 software (Malvern, UK). Collagen and proteoglycan content were monitored by the integrated area of the 1660 and 1050 cm-1 absorbances, respectively. Collagen helical integrity was monitored by the ratio of the integrated area of the 1338 cm-1 collagen side chain absorbance ratioed to the amide II absorbance. Polarization experiments were performed to determine the distribution of the collagen fibril orientation by assessment of, the ratio of the collagen amide I/amide II absorbance. Results: Compared to adjacent normal cartilage, the repair tissue had a lower collagen and collagen integrity in the initial weeks but reached values comparable to normal tissue by 6 weeks. Histological features of the tissue also improved by 6 weeks. PG content remained lower in the repair tissue and there was no zonal stratification of collagen fibril orientation as seen in normal articular cartilage, throughout the 16 weeks of the study. Ultimately assessment of these parameters may yield insight into optimization of the repair process.

(410) **Coating Effects on Fabrid Infrared Reflectance Spectra**

Megan Baranowski[1], Heather Brooke[1], Jessica McCutcheon[1], Stephen Morgan[1], Michael Myrick[1]; [1]Univeristy of South Carolina

Polymer films of varying thicknesses were deposited onto cotton and polyester fabric samples by dip-coating from solution. SEM images of the coated fabric samples were used to evaluate the quality of the polymer coating. The samples were analyzed by infrared diffuse reflectance spectroscopy to determine the relationship between film thickness and the effect of the coating on the spectroscopy of the two fabrics. The effects observed in four limiting cases are examined: (Case I) weak coating absorbance on a fabric with weak absorbance at the same frequency; (Case II) strong coating absorbance in a spectral region of weak fabric absorbance; (Case III) weak coating absorbance in a spectral region of strong fabric absorbance; and (Case IV) strong coating absorbance in a spectral region of strong fabric absorbance. In the first case we observe effects dominated by reduced scattering as the coating is added. In the second case we observe strong coating absorbance at low coverages that plateaus due to depth of penetration effects. In the third and fourth cases, we observe reduced Fresnel diffuse reflectance as the coating is added, consistent with the reduction of scattering observed in the first case.

(411) **Mass Spectrometry-Based Analysis of the Lung Proteome of Mice Infected with Aspergillus fumigatus**

Chengsi Huang[1], Jason McCarthy[2], Marta Feldmesser[2], Vicki H. Wysocki[1]; [1]University of Arizona; [2]Albert Einstein College of Medicine

Invasive aspergillosis (IA) is a fatal pulmonary infection caused by Aspergillus fumigatus, spores of which enter the lung through inhalation. While the immune system of healthy individuals is capable of quickly eliminating the fungus, immunocompromised patients may be severely affected. IA often occurs in patients who have undergone hematopoietic stem cell or solid organ transplantation, received chemotherapy, or who are suffering from late-stage AIDS. Diagnosis involves time-consuming microbiologic or serological tests that have variable accuracy and patients with IA often receive treatment too late. Hence, a fast, sensitive, and less invasive diagnostic technique is critical in being able to correctly and confidently identify disease in patients soon after onset of symptoms. Because A. fumigatus can colonize the airways or result in allergic diseases, including asthma, a test that can specifically identify invasive disease is highly desirable. Mice are used as a model organism to study the protein expression in various organs when infection occurs. Immunocompromised CBA/J mice were divided into three different groups of five mice each: a naïve control group, an asthma-model group and an IA model group. Lung homogenate (LH), bronchoalveolar lavage (BAL) fluid, plasma, and serum were collected from all mice for protein content analysis. In the present study, a bottom-up proteomics approach is being taken to analyze the LH of these mice. The LH samples are being separated via 1D-PAGE, followed by tryptic in-gel digestion. Peptides are being extracted and purified on C18 columns. The samples thus prepared are being separated via reverse-phase liquid chromatography in-line with mass spectrometry (RPLC-MS/MS). The SEQUEST database searching algorithm is being used for protein identification. Our 1D gel results showed several different protein expression profiles in LH of the mice with IA when compared to the naïve and asthma model mice. RPLC-MS/MS analysis has found differences in both host and A. fumigatus proteins among the three groups of mice. Proteins exclusive to mice with IA will be considered as clinical diagnostic targets.

(412) **Frequencies and Absorption Intensities of Fundamentals and Overtones of NH Stretching Vibrations of Pyrrole and Pyrrole**

Yukihiro Ozaki[1], Yoshisuke Futami[1], Yasushi Ozaki[2], Yoshiaki Hamada[3], Marek Wojcik[4]; [1]Kwansei-Gakuin University; [2]Josai University; [3]The Open University of Japan; [4]Jagiellonian University

The frequencies and intensities of fundamentals and first-overtones of NH stretching vibration bands of pyrrole and pyrrole---pyridine complex were investigated by near-infrared(NIR)/infrared(IR) spectroscopy and density-functional-theory (DFT) calculations. NIR / IR spectra were measured for pyrrole, pyridine and pyrrole-pyridine complex in CCl4 solutions.The first-overtone of the NH

stretching vibration band of free pyrrole is observed at 6856.4 cm-1, but that of pyrrole---pyridine complex is missing or extremely weak to be observed. The theoretical calculations elucidated that the transition dipole moment containing the dipole moment function becomes much smaller upon the formation of the complex, resulting in the remarkable intensity decrease in the overtone of the hydrogen-bonded NH group.

(413) **Microsecond Time-Resolved Desorption Electrospray Ionization Mass Spectrometry**

Zhixin Miao[1,2,3], Hao Chen [1,2,3]; [1]Department of Chemistry and Biochemistry; [2]Center for Intelligent Chemical Instrume; [3]Ohio University

Kinetic study is of great important to study the (bio)chemical reaction intermediates further to elucidate the mechanisms. In this study, a novel mass spectrometric method for fast reaction kinetics with time resolution of microseconds was introduced, based on the direct sampling feature of a "flying" liquid jet by desorption electrospray ionization (DESI). The two reactant solutions undergo ultrafast turbulent mixing to produce a free liquid jet which is sampled by DESI spray at different positions corresponding to the different reaction times. The protein folding/unfolding was chosen as examples to validate this method and the rate constants were successfully measured. In this time-resolved DESI-MS, there is no requirement for the reactant/product carrying the chromophoric substrates or radioactive labeling for the optical response and the MS detector has the advantage of high chemical specificity. This method is believed of high value in fast reaction kinetic study and it should have general interest to audience and impact in the field of mass spectrometry.

(414) **Advancing Calibration Strategies for Plasma Spectrometry: Different Operation Modes of a Dual Drop-On-Demand Aerosol Generator for Micro-Volume Sample Introduction**

J. Niklas Schaper[1], Jan Massmann[1], Jan H. Petersen[1], Nicolas H. Bings[1]; [1]University of Mainz, Analytical Chemistry

Conventional pneumatic nebulization is the most common way of introducing liquid samples into excitation sources in atomic spectrometry, but it is still a major bottleneck due to considerable losses. Well-known drawbacks from the use of these pneumatic nebulizers such as e.g. liquid flow rate dependent aerosol formation efficiency and peristaltic pump-based signal fluctuations are limiting further enhancements. This is especially the case if only minute amount of sample material is available and downscaling of the instrumentation becomes necessary. The novel approach outlined in this work is based on the development of a microcontrolled drop-on-demand (DOD) aerosol generator, which employs a modified thermal inkjet cartridge, offering full access to all parameters important for the droplet generation process of minimum sample volumes. As shown before, our developed system is most suitable for external calibration, while providing improved sensitivity compared to established low-flow nebulizers such as the MicroMistTM. The droplet generation (dosing) frequency, though the liquid flow rate is highly variable without compromising aerosol quality. Therefore extremely low flow rates below 50 nL min-1 but also up to 2 ml min-1 were accessible. As the dosing frequency dictates the absolute sample volume transferred per time, a fully new calibration strategy, based on the dosing frequency, is introduced in this poster. This approach provides a calibration strategy throughout dosing only one standard solution at different dosing frequencies instead of a series of standard solutions of different concentrations. The analytical performance of the novel system and the application of the new calibration strategy will be presented. Advantages, complications and tradeoffs will be provided.

(415) **FAIMS as a Filter for Biomarker Detection**

Charles Harrison[1], Alessandra Ferzoco[2], Mark Ridgeway[2], Desmond Kaplan[1], Kevin Dixon[1], Melvin Park[1], Gary Glish[2]; [1]Bruker Daltonics; [2]The University of North Carolina

Field asymmetric ion mobility spectrometry (FAIMS) is a technique that separates gas phase ions based on the ratio of high- and low-field mobility. FAIMS devices can operate as a filter where only select species are allowed to pass to a mass spectrometer. The filtering mode drastically reduces chemical noise without additional sample preparation, and acts on a much faster time scale than liquid chromatography separation. Unfortunately, coupling of most current FAIMS devices to a mass analyzer results in significant ion trasnmission losses due to ion diffusion within the device, and ineffiecient coupling between the dispersed ion beam and the conductance limit of the mass analyzer. A new small, planar FAIMS design is integrated in an ESI capillary and achieves near 100% transmission efficiency. This new design can be used as a filter to improve the limit of detection of plasma biomarkers or many other analytes of potential biological or environmental interest.

(416) **Improving the Accuracy of Calculated VCD Spectra Using Multilayer Computational Analysis**

Douglas J. Minick[1], Dean P. Phelps[1], Randy D. Rutkowske[1], Luke A. D. Miller[1]; [1]GlaxoSmithKline

Vibrational circular dichroism (VCD) is now widely recognized as an established technique for determining absolute configurations of chiral organic molecules. While the intensities and harmonic frequencies of many functional groups are accurately predicted using standard computational methods, these methods fail to predict the vibrational frequencies of several functional groups of key interest as pharmacophores in drug discovery, particularly the sulfonyl group (SO2). This limitation can be overcome by utilizing a higher level theoretical method to predict vibrational spectra for molecules containing one or more SO2 groups, but at great cost computational, making their use generally impractical. Multilayered analysis is a computationally cost-efficient alternative to standard approaches. This method of analysis allows different theoretical levels to be applied to different regions of a molecule. Its utility for analyzing SO2-containing compounds was tested using 1-(p-tosyl-(R)-(-)-3-pyrrolidinol. VCD and IR spectra were calculated by applying a two-layered method to this structure. These spectra are compared here with experimental data and spectral data calculated using standard and higher level single-method approaches. Results indicate that the fidelity of VCD and IR spectra calculated for SO2-containing molecules is significantly improved using the two-layer method without sacrificing computational efficiency.

(417) **Shock Behavior and Analyte Transport in the ICP-MS via the Direct Simulation Monte Carlo Algorithm**

Ross Spencer[1], Steven Schmidt[1], Paul Farnsworth[1]; [1]Brigham Young University

The simulation code FENIX, which takes advantage of the speed and memory capacity of parallel computation, is now being used to study the details of argon and analyte flow through the skimmer cone of the ICP-MS. It is found that the size and shape of the skimmer orifice determines whether or not a secondary shock forms at the skimmer tip. Sharp tips without a cylindrical throat minimize shock formation. The code is also used to study electrostatic expansion effects on analyte ions as they exit the skimmer throat. Analysis and numerical imaging of these effects will be presented.

(418) **The Potential of Autofluorescence Spectroscopy to Detect Human Urinary Tract Infection**

Unnikrishnan Kuzhiumparambil[1], Sandeep Menon Perinchery[1], Subramanyam Vemulpad[1], Ewa M. Goldys[1]; [1]Macquarie University

Urinary tract infections (UTIs) are known to alter the normal urine composition which, in principle, can lead to changes in urine autofluorescence. Here we describes the study of human urine (normal and UTI) by using UV fluorescence excitation/emission matrices and synchronous spectra and proposes a method of diagnosing UTI without any sample preparation. The method is based on excitation in the shorter UV region (250–350 nm) which shows good discrimination between the normal urine and UTI samples. The synchronous scans with an offset of Äë = 90 nm were also able to differentiate between normal urines and UTI samples. These differences were observed even though the two known major urine fluorophores, tryptophan and indoxyl sulfate were present in the normal urine and UTI samples in similar concentration as established by HPLC analysis. Although the identity of substances responsible for the altered autofluorescence in UTI is not established, our study shows that autofluorescence has the potential to differentiate between normal human urine samples and those with UTI.

(419) **Engineered Nanoparticle - Biological Interactions and Their Role**

Kevin Dreher[1]; [1]EPA

Nanotechnology has been referred to as the next industrial revolution due to the potential applications of novel nanoscale materials in the energy, biomedical, electronic, consumer and environmental sectors. However, the environmental, health and safety implications of novel nanoscale materials and their applications remain a significant uncertainty which must be addressed in order for nanotechnology to develop in a responsible and sustainable manner. Information on how engineered nanoparticles interact with the environmental and biological systems is a prerequisite in identify properties which regulate nanoparticle exposure pathways, fate and toxicity. The presentation will provide an overview of the current state of knowledge regarding nanoparticle - biological interactions which regulate their toxicity.

(420) **DNA-Carbon Nanotube Interaction: Fundamentals and Applications**

Ming Zheng; [1]National Institute of Standards and Technology

DNA is the material that Nature has selected for carrying genetic information in all living cells. Its central role in biology and its unique physical-chemical properties have been the constant source of motivation for investigation by different disciplines. Carbon nanotube (CNT) is a relatively new man-made material with beautiful atomic and fascinating electronic structures. It has potential for many technological applications. A few years ago, we identified a strong interaction between DNA and CNT that is dependent on both the DNA sequence and the CNT structure. This finding has prompted not only theoretical exploration of the nature of the interaction, but also technological exploitation of the interaction in areas ranging from electronic devices to rapid DNA sequencing. In this talk, I will show the use of DNA as a powerful molecular tool to solve a recalcitrant problem in the CNT field - separation of a synthetic mixture of single wall CNTs into pure chirality species, and discuss new insight into DNA structural properties derived from the DNA-CNT hybrids.

(421) **Immunological Properties of Engineered Nanoparticles**

Marina Dobrovolskaia; [1]SAIC-Federick Inc

Nanotechnology is finding growing applications in industry, biology and medicine. The clear benefits of using nanosized products in various biological and medical applications are often challenged by questions regarding toxicity of these materials. One area of interest involves the interactions between nanoparticles and the components of the immune system. Nanoparticles can be engineered to either avoid immune system recognition or to specifically interact with components of the immune responses. This presentation will review reported observations on nanoparticle-mediated immunostimulation and immunosuppression, how manipulation of particle physicochemical properties can influence their interaction with components of the immune system, and discuss challenges with immunological characterization of engineered nanomaterials. Funded by NCI Contract No.HHSN261200800001E

(422) **Measuring Affinity between Proteins and Nanoparticles Using Capillary Electrophoresis**

Wenwan Zhong[1], Ni Li[1], Shang Zeng[1]; [1]University of California, Riverside

We reported a simple scheme to probe the protein-nanparticle interaction by using capillary electrophoresis (CE). Ligand-receptor model was applied and cooperative binding were observed for most of the nanoparticle-protein pairs in the present study. Nanoparticle sizes, surface ligands and protein flexibility have been found to affect the binding behaviour.

(423) **Characterization and Detection of Non-Covalent Binding of Single-Stranded Oligonucleotides to Single-Walled Carbon Nanotubes**

Meagan A. Cauble[1]; [1]University of Georgia

Obtaining a fundamental understanding of the ways in which single-walled carbon nanotubes (SWNTs) interact with biological molecules is of great scientific interest. Further, a deeper comprehension of the interactions at the SWNT/ssDNA interface will aid in the development of new technologies for gene delivery, molecular probes, nano-scale biosensors, and molecular labeling of cells. Yet these interactions are poorly understood. Furthermore, stability effects on double stranded DNA bound to SWNTs have not been thoroughly investigated. In these studies, single stranded DNA (ssDNA) molecules were allowed to non-covalently bind to the surface of surfactant-stabilized (sodium dodecyl sulfate, SDS) SWNTs. Dialysis was used to remove the SDS and any un-bound ssDNA. The extent of binding was determined via UV-Vis, NIR, and Raman spectroscopy. A red-shift in NIR absorbance peaks was observed after ssDNA attached to the SWNTs. ssDNA bound to SWNTs was detected in the solution by UV-Vis spectroscopy and unbound SWNTs were detected using UV-Vis and Raman spectroscopy. Atomic Force Microscopy (AFM) was also used to characterize the extent of binding after deposition of ssDNA-SWNT constructs on Si/SiOx wafers. The kinetics of hybridization of complementary DNA strands to the ssDNA-SWNT complexes was also studied by observing changes in the wavelength of peaks in NIR spectra. The rate of change in the concentration of unhybridized complementary DNA strands were used to calculate the rate constant of the hybridization reaction. Then, the rate constant can be determined experimentally with various other DNA strands to determine if the DNA sequence affects the hybridization kinetics. Additionally, the melting temperature of various double-stranded (dsDNA) samples in the presence of SWNTs allows determination of the effect of interactions with nanotubes on the stability of interactions between DNA strands in double stranded DNA.

(424) **Near-IR Spectroscopy of Clay Minerals and Amorphous Silicates: Understanding Aqueous Alteration Environments on Mars**

Elizabeth Rampe[1], Nina Lanza[2], Thomas Sharp[1]; [1]Arizona State University; [2]University of New Mexico

Near-IR spectroscopy is an important tool for identifying the composition of planetary surfaces from orbital remote sensing instruments. This technique has been used to better understand the surface mineralogy of both Earth and Mars. Of particular interest on Mars are hydrous alteration minerals, which are suggestive of the past presence of liquid water. Remote NIR data of the martian surface collected by the Compact Reconnaissance Imaging Spectrometer for Mars (CRISM) and the Observatoire pour la Minéralogie, l'Eau, les Glaces et l'Activité (OMEGA) have been used to identify a variety of hydrous silicates, including clay minerals and amorphous silicates, which form as the result of interactions between water and rock. Exposures of these alteration products are widespread but limited to older Noachian-aged surfaces (~4.6-3.8 Ga). In addition to presenting evidence for liquid water on the martian surface, the suite of alteration products present can also be used to infer the aqueous environments in which they formed, including pH, water-to-rock ratios, and water activity. Understanding the aqueous history of Mars can help identify potentially habitable environments, or locations conducive to supporting life, that are good targets for future lander or rover missions. Four of the martian locales with the highest concentrations and greatest diversity of alteration products are potential landing sites for the Mars Science Laboratory rover, set to launch in October 2011. One statistical technique that may aid in the interpretation of NIR data obtained for these landing sites is principal component analysis (PCA), which is a measure of spectral similarity. PCA of NIR spectra is an established technique in many fields, including agronomy and medical research; however, PCA is not widely used in the martian spectroscopy community. Here, we use PCA to examine NIR laboratory spectra of discrete clay minerals, hydrous amorphous silicates, and mixed-layer clay minerals (a type of clay mineral that forms at elevated temperatures) and NIR spectra of clay-rich regions from Mars to determine the efficacy of this technique for identifying and discriminating the alteration mineralogy of the martian surface.

(425) **The Role of Near-Infrared in Pharmaceutical Counterfeit Prevention**

Frederick Haibach[1]; [1]Polychromix, Inc.

In the past decade technological advances made for the telecommunications industry resulted in dramatically smaller and energy-efficient optical spectrometers. At the same time, the pharmaceutical industry, governments and international agencies have experienced a dramatic increase in the reports and seizures of illegal pharmaceuticals. It is generally believed that current scientific studies provide only coarse estimates for the actual incidence of substandard and counterfeit medicines. The actual prevalence is of interest to public health agencies, international aid organizations and legitimate pharmaceutical manufacturers. To this purpose, these organizations are just beginning to seriously evaluate technologies that provide the potential for being rapid screening tools for identifying the legitimacy and efficacy of individual dosages. Near-infrared has been evaluated for this role as early as 1987 by Lodder, Selby and Hieftje. The practicality of non-destructive large scale screening of using portable instrumentation has further renewed interest in near-infrared. Important to the practical implementation are methods for estimating the likelihood of type 1 (false-negative) and type 2 (false-positive) errors from compositional variability of authentic dosages. We will review and present experimental designs and procedures that enable large scale screening.

(426) **Near- Versus Mid-Infrared Diffuse Reflectance Spectroscopic Analysis of Compostable Utensils and Biochars: Results Can be Surprising**

James B. Reeves III[1], Walter W. Mulbry III[1], Heekwon Ahn[1]; [1]EMBUL, ARS, USDA

Near-infrared diffuse reflectance spectroscopy (NIRS) and more recently mid-infrared diffuse reflectance spectroscopy has been extensively studied and use in the agriculture arena. More recently, there has been growing interest in applying these methods to help solve or understand environmental problems. Two such areas of interest are composting and biochar. For this work, in each case, diffuse reflectance spectroscopy using a Digilab (Now Varian) FTS-7000 Fourier Transform spectrometer equipped for both mid-infrared and near-infrared diffuse reflectance spectroscopy was used to examine samples of materials before and after composting or before and after the addition of biochar to soils. For the composted samples, spectroscopy was used to determine the fate of compostable eating utensils and drinking cups based on polylactic acid. Efforts comparing near- and mid-infrared spectroscopy to composted utensils were surprising, especially with respect to qualitative spectral interpretation, where the NIR was found to be superior. Results were so clear and straightforward using the NIR, that reasonably accurate quantization was possible using only peak height measurements in the spectral region for starch, a filler used with the polylactic acid in eating utensils. Another area of great interest is the production and application of biochar to soils to improve soil quality and sequester carbon. As will be discussed, spectral results were quite unexpected and may have serious consequences for the ability of spectroscopic calibrations to accurately determine soils C levels if biochar is applied to the soil.

(427) **NIR Instrument Calibration and Quality Control Testing Via a Center of Gravity Algorithm**

Mark Henson[1], Kevin Judge[1]; [1]Molecular Biometrics, Inc.

Spectrometers which incorporate array detectors require calibration with reference standards. These standards typically contain well defined features whose observed pixel positions may be recorded from the experimental spectra and then translated into observed wavelengths based on their nominal wavelength positions. Center of gravity algorithms provide a robust method of determining spectral band positions. For NIR instruments incorporating an InGaAs array detector, pixel sizes can lead to variability in the positions of peak intensity maxima of atomic emission lines used in many calibrations. Center of gravity algorithms result in improvements in calibration accuracy and provide an effective method of extracting consistent calibration verification data from spectra of SRM-2065 reference standards, which contain numerous broad absorbance bands in the 900-1700 nm region.

(428) **Development of a Robust Calibration Model for Monitoring Alcoholic Fermentation Process by Using Near-Infrared and Infrared Dual-Wavelength System**

Takuma Genkawa[1], Masahiro Watari[2], Mitsue Satou[2], Mikiko Konta[1], Yukihiro Ozaki[1]; [1]Kwansei Gakuin University; [2]Yokogawa Electric Corporation

To develop a novel monitoring technique for alcoholic fermentation with a near-infrared (NIR) and infrared (IR) dual-wavelength system, performance of calibration models constructed from NIR and IR spectra of fermentation mixture was estimated. Immobilized baker's yeast was added to 5 % glucose solution, and fermentation was carried out at room temperature for 3-4 hours. Through the fermentation, NIR and IR spectra of the solution were measured continuously. The types of NIR and IR probes used were transflectance and attenuated total reflectance (ATR), respectively. The concentrations of glucose, ethanol, and dissolved carbon dioxide during the fermentation were calculated by using

calibration curves constructed previously. During the fermentation, the concentration of ethanol increased from 0 % to 0.7 % while that of glucose decreased to about 3 %. The concentration of dissolved carbon dioxide increased to about 0.8 % and seemed to be in equilibrium. In the prediction of the glucose concentration, standard error of calibration model constructed from the NIR spectra was smaller than that of the model constructed from the IR spectra. Unfortunately, however, the measurement of NIR spectra was interrupted repeatedly during the fermentation because bubbles of carbon dioxide attached on the NIR probe. The bubbles were generated by the fermentation, and were trapped in the gap (about 1 mm) of the NIR probe. Consequently, the predicted curve of glucose concentration discontinued temporarily. On the other hand, the IR spectra were measured continuously through the fermentation because the ATR probe is insusceptible to attachment of bubbles. Therefore, the outlier detection method was employed to improve robustness of calibration model for fermentation process. The calibration model constructed from NIR spectra was generally used, but when outlier value was detected, the calibration model constructed from IR spectra was used as alternated. The result showed that the robustness of the calibration model was improved.

(429) **What Color is Your Yellow Cake? Vis/NIR Spectroscopic Analysis of Uranium Ore Concentrate Samples**

Greg Klunder[1], Paul Spackman[1], Pat Grant[1], Martin Robel[1], Lars Borg[1], Rachel Lindvall[1], Ian Hutcheon[1]; [1]Lawrence Livermore National Laboratory

Uranium ore concentrates from the mining and processing of uranium are commonly referred to as Yellow Cake which is a reference back to early mines when processed uranium samples were yellow. Today the color of the uranium ore concentrates vary from yellow to dark brown and the color can be an indicator of the process procedure. Although the visual evaluation of the sample color is the simplest detector, it suffers from being very subjective and does not provide chemical information. Near-infrared (NIR) spectroscopy in the spectral range from 1000 – 2500 nm can measure C-H, O-H, and N-H vibrational overtone and combination bands, and has the advantage of being non-contact and non-destructive. Observation of OH and NH overtone vibrations can provide chemical information that could be indicative of different processing. In this study, we have measured visible and NIR reflectance spectra from a number of different yellow cake samples. The spectra were evaluated using chemometric analysis and the samples separated into groups with similar chemical features. The results of this study and the applicability of this technique for yellow cake characterization will be discussed.

(430) **Raman, NIR and FT-IR Spectroscopic Quality Control by Hand-Held Instrumentation**

Heinz W. Siesler[1]; [1]University of Duisburg-Essen

Recently miniaturization of Raman, mid-IR and near-IR spectrometers has made substantial progress. While the mid-infrared systems are based on attenuated total reflection (ATR) measurements, near-infrared spectrometers operate in the diffuse reflection mode. However, the reduction in size must not be accompanied by an equal reduction in measurement performance and miniaturization will only have a real impact on chemical quality and process control if Raman and IR spectra of acceptable quality can be obtained with this new type of hand-held instruments. Thus, comparative studies are required to evaluate whether these new developments can effectively replace conventional laboratory spectrometers. In this communication the quantitative results obtained with selected liquid and solid formulations by Raman, FT-IR/ATR and NIR spectroscopy with laboratory instruments and the corresponding commercially available hand-held spectrometers will be compared with the aim to evaluate the measurement performance of the miniaturized systems. All data sets have been analyzed by multivariate PLS procedures and will demonstrate whether in the near future miniaturized spectrometers can substitute conventional laboratory instruments for *in-situ* chemical quality and process control.

(431) **Exploring the Limitations of Spectroscopic Techniques for Tablet Analysis**

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A major assumption in utilizing spectroscopic techniques to quantify ingredients in a tablet is that the materials are mixed uniformly on the particle scale. To date, the available techniques do not investigate the entire tablet. If a non-homogeneous tablet is analyzed, the technique may not detect local composition variation since the entire tablet is not being measured. In some cases, agglomerates of API may go completely undetected or in other cases the predicted composition will be extremely high. Both scenarios give a false sense of quality. For the purpose of testing the limits of each spectroscopy technique, agglomerates were positioned in specific locations within a series of tablets to explore this sensitivity to sub-sampling. A total of six tablets were manufactured, which varied in the size and location of the API agglomerate. Once the agglomerated tablets were prepared, a number of spectroscopic tools were used to evaluate the compositions. The techniques included: near-infrared chemical imaging, Raman in transmission and reflectance modes as well as transmission and reflectance near-infrared spectroscopy. This presentation walks through the results obtained from analyzing specifically designed tablets containing agglomerated API. It was found that backscattering and reflectance techniques readily detect non-homogeneity on the surface closest to the detector. Transmission techniques will detect agglomerates within the area of measurement. For all techniques, the quantified result tends to over-predict the concentration due to normalization only over the measured area. No independent method was identified to detect all of the agglomerates in each of the tablets. This work underscores the importance of on-line blend monitoring and supports additional upstream process controls to ensure product homogeneity.

(432) **The Spectroscopic Options for Real Time Tablet Content Uniformity: A Comparison Study of NIR Reflectance, NIR Transmission and Raman Transmission**

Yang Liu[1], Stephanie Dolph[1]; [1]Pfizer Global Research and Development

Spectroscopic techniques such as near infrared spectroscopy and Raman spectroscopy have become important components of PAT and the enabler of the real time release. The most popular industry option for on-line/at-line tablet core content uniformity analysis is near infrared spectroscopy. There are various research papers and regulatory agency approval cases exist. One of the critical questions to spectroscopic method for content uniformity is the sampling volume. The formulation, tooling and hardness of the tablets impact the penetrate depth. Although NIR transmission mode is often the desired and mature sampling mode, it has its limitation for thicker tablets and often takes longer data collection time. NIR reflectance mode has been successfully applied to tablet analysis when the homogeneity of the tablets is not a concern. Transmission Raman has attracted many interests in recent years for its better sampling volume, less interferences from the physical properties of the tablets and even faster data collection time. A comparison study of these techniques to understand the pros and cons is necessary for PAT implementation tailored towards specific products and the accuracy requirement. In this paper, the NIR reflectance mode, NIR transmission mode and Raman transmission

mode were compared on an immediate release tablet drug product. Specially made calibration tablets and real manufacturing batches at different scales were used for the study. Further experiment and discussion around the penetration depth are presented. The fit for purpose application strategy for different accuracy requirements at different development and manufacturing stages are demonstrated and discussed.

(433) **Multivariate Image Analysis of NIR Chemical Images of Polymeric Gel Strips**

Rodolfo J Romanach[1], Jackeline Jerez Rozo[1], Jose M Prats Montalban[2], Alberto Ferrer[2]; [1]Univ.Puerto Rico, Mayaguez Campus; [2]Polytechnic University of Valencia

It is estimated that over 40% of possible new drug candidates have very low solubility. One possible approach for improving the solubility of these drugs is to disperse them throughout a polymer film, and prevent them from aggregating. NIR Chemical Imaging (NIR-CI) has been performed with these strip films, providing extremely huge data sets that require multivariate image analysis. The assessment of homogeneity and drug distribution in these gel strip films is challenging because the drug is widely dispersed within the polymeric matrix, and do not include discrete particles such as those commonly observed in tablets. Thus, two sets of strip films were prepared. The first set included large agglomerates of the drug, purposely prepared to facilitate method development. The second set was developed with drug particles widely dispersed through the polymeric matrix, with a lack of large drug clusters. The results for both sets of strip films will be presented. Multivariate Curve Resolution (MCR), Classical Least Squares (CLS) and Target PLS were used to develop chemical images related to the distribution of active pharmaceutical ingredient (API) and excipients. These chemical oriented models, mainly MCR and CLS, enable the analysis of high dimensional chemical structures, by imposing certain restrictions (e.g. non negativity in the concentration of each compound at each pixel location, or by using the pure spectra for predicting the amount of the related chemical information). This way, it is possible to obtain more reliable chemical concentration (or distribution) maps related to the different spectra computed by the models. Once the hyperspectral images have been translated into real multivariate images (4 or 5 bands related to the chemical compounds), the latter can be properly analyzed by traditional Multivariate Image Analysis (MIA) techniques, studying the interactions between the chemical compounds and the effect on the spatial distribution for the gel strips, hence further elucidating drug distribution. Furthermore, if quality information is available from the production environment, and proper spatial distribution (texture) variables can be extracted out from the images, MIA-based soft sensors could be developed.

(434) **A Comprehensive Exploration of Imaging Technologies for Pharmaceutical Drug Development**

Pascal Chalus[1]; [1]F-Hoffman-La Roche AG., PTDFA

Technical drug development is getting more and more complex. In order to reach the treatment target drugs have to perform at best. To develop such robust, high performing drug products it is necessary to understand every key parameter of the compound interactions and production process. Simple end quality testing is most often not enough to understand potential issues and to do root cause finding. Imaging technologies are giving access to information that is not reachable via classical wet chemistry or spectroscopic methods. A broad range of these techniques are now accessible and adapted to pharmaceutical products. The distribution of compound in the formulated drug product and for exemple the influence of the coating thickness are typical key parameters that can drastically jeopardize the performance of a potential drug. Some of the imaging technologies offer the additional advantage to be completely non invasive, therefore leaving the sample, complete, intact and available for the classical methods or further analysis. This allows a direct correlation between an observation, a process parameter and a behavior (e.g. dissolution). This presentation is to show how various imaging technologies have been successfully implemented in the drug development in order to support the decision making during the development. These technologies are ranking from the highly chemistry sensitive ToF-SIMS imaging to the ones that are giving more physical information such as the Terahertz Pulsed Imaging, or X-Ray micro computed tomography going through hyperspectral imaging techniques among which Near Infrared Chemical Imaging or Raman Microscopy. Each of these techniques have there advantages and drawbacks, some of advantages can also become drawbacks under certain conditions. The sample preparation and the screening possibilities have also to be considered as well as how the performed analysis is representative of a full batch of drug product. The presentation will cover the pros and the cons of this large range of imaging technologies in the area of pharmaceutical drug development, by this, describing the advantage of using such a broad spectrum of technique allows accessing information that once brought together helps the decision making and makes a more straight forward development.

(435) **NIR Spectral and Imaging Analysis for Support of a Pharmaceutical Manufacturing Process**

Boyong Wan[1], Kevin Macias[1], Gary McGeorge[1], Douglas Both[1]; [1]Analytical R&D, Bristol-Myers Squibb Co.

In order to develop control strategies for the efficient and robust manufacture of commercial pharmaceutical products it is necessary to monitor the product early during the development phase of a product lifecycle. In this presentation we will apply NIR spectroscopy and imaging techniques to monitor the manufacturing process of drug product in order to understand the relationship of the manufacturing process to the spectral/image properties. Online blending analysis was used to monitor blending processes. Offline NIR spectra and imaging data were collected for blends, ribbons and tablets during manufacturing of these batches. By systematic analysis of spectral and imaging data, significant batch-to-batch variations were observed, which indicated a causal relationship between upstream material properties and downstream product attributes. Spectral and imaging analysis of specific tablets demonstrated the existence of intra-die segregation during tablet compression process.

(436) **On the Usefulness of the Spectral Fluctuation Approach in Laser Induced Plasma Spectroscopy**

Nicolo Omenetto, Heh-Young Moon, Daniel Shelby, Jonathan Merten, Benjamin Smith; [1]University of Florida

This investigation was prompted by previous work published on aerosol analysis [1,2] and by recent work focused on processing shot-to-shot raw data to improve precision in laser induced breakdown spectroscopy (LIBS) [3]. The approach discussed here involves the systematic investigation of the noise present in LIBS spectra obtained at different delay times. Plasma background, analyte signals, and their corresponding standard deviation and relative standard deviation are calculated on a pixel-by-pixel basis and plotted as a function of the pixel numbers (wavelengths). Within the validity of several simplifying assumptions, it is argued that the behavior resulting from a series of N single-shot spectra can indeed reflect some characteristic aspects of the measurement. Such aspects can be: (i) the measurement precision and its evolution during the lifetime of the plasma; (ii) the degree of correlation between the plasma background and the analytical signal and its evolution during the plasma lifetime; (iii) even if only approximately, the identification of the limiting noises and their different relevance during the lifetime of the plasma. Note that no

restrictions are placed on the optical thickness of the plasma. Moreover, while being informative of the limiting noise of the measurement, the above considerations can also provide some information about the type of noise present in the measurement. A complementary approach consists in repeating the above described procedures using several samples containing increasing analyte concentrations. From the results obtained on each sample, the log-log plot of the signal to noise versus signal (concentration) will provide the information sought. Spatially integrated measurements are taken with Al-alloys and brass targets ablated with a Nd-YAG laser at 1064 nm. A grating monochromator-ICCD detection system is used for data collection. Both single-pulse and double-pulse (pre-spark and re-heating) experiments are considered. Acknowledgement: This work was supported by National Science Foundation through grant CHE-0822469. [1] L.A. Alvarez-Trujillo, A. Ferrero and J.J. Laserna, J. Anal. At. Spectrom., 2008, 23, 885-888. [2] L.A. Alvarez-Trujillo, A. Ferrero, J.J. Laserna and D.W.Hahn, Appl. Spectrosc., 2008, 62, 1144-1152. [3] J.M. Mermet, P. Mauchien and J.L. Lacour, Spectrochim. Acta., 2008, 63B, 999-1005.

(437) **Imaging the Ion Beam in the Second Vacuum Stage of an Inductively Coupled Plasma Mass Spectrometer**

Paul Farnsworth[1], Nicholas Taylor[1]; [1]Brigham Young University

For the achievement of optimum performance in an inductively coupled plasma mass spectrometer (ICP-MS) it is essential that ions be transported efficiently from the plasma to the mass analyzer. A critical part of that transport is the formation of a well-characterized ion beam from the presumably neutral beam that is skimmed from the supersonic expansion formed in the first stage of the vacuum interface. When the skimmed beam loses its charge neutrality, mutual repulsion of the positive ions works against efforts to form a tightly focused beam. The extent to which this space charge effect influences beam formation and the degree to which it is influenced by changes in sample composition have not been quantitatively measured in a working ICP-MS. We have designed a system that allows us to image the ion beam at the entrance to the mass analyzer in the second vacuum stage of a commercial ICP-MS using planar laser induced fluorescence. I will present initial results from our instrument in which we examine the effects of analyte mass, concentration, and matrix on ion beam formation and space charge in an ICP-MS.

(438) **Laser Ablation Based Chemical Analysis: Macroscale, Nanoscale, Fundamentals and Commercialization**

Richard E. Russo[1,2], Vassilia Zorba[1], Xianglei Mao[1], Jhanis Gonzalez[1,2], Jong Yoo[2]; [1]Lawrence Berkeley National Laboratory; [2]Applied Spectra Inc

Laser ablation involves the use of a pulsed high power laser beam to remove (sample) a small portion of mass from a solid material and convert that mass into an aerosol (vapor and particles). Laser ablation as a chemical analysis approach is gaining in popularity due to numerous advantages. Coupled with optical or mass spectrometry, the technology provides real-time direct solid sample chemical analysis, without sample preparation and requiring only micrograms or less material. The research at the Lawrence Berkeley National Laboratory investigates fundamental processes of ablation that include laser energy coupling into the sample material, time-resolved imaging of shockwaves and plasmas, and particle formation (ejection, nucleation and condensation). Our work also focuses on application specific parameters such as accuracy and precision of ablation for analysis, and spatial resolution – with emphasis on using near field optics to achieve nanometer diameter analysis. At Applied Spectra, we transition this knowledge based into commercial products by developing instruments using both optical and mass spectrometry detection for laser ablation sampling.

(439) **LA-ICP-MS: A Status Report**

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A progress report about recent developments in the field of LA-ICP-MS will be given. In this context, results addressing the OES-based diagnosis of ICPs operated under wet (SN) and dry (LA) conditions as well as the visualization of aerosol particles penetrating through the plasma source will be discussed. Furthermore, a novel sampling strategy for laser-produced aerosols will be presented [1] making LA-ICP-MS analyses without the need of ablation cells possible. [1] R. Kovacs, N. Kohei, U. Keisuke, D. Gunther, J. Anal. At. Spectrom. (2010), 25, 142 - 147

(440) **Time-Resolved Measurements of Ions Produced from Individual Droplets and Particles in Plasmas**

John Olesik[1], Patrick Gray[1], Josh Dettman[1]; [1]Ohio State University

Measurements of ions produced from individual droplets and particles are of interest to further fundamental understanding of the conversion of sample to signals in ICP-OES and ICP-MS as well as for some practical sample measurements. Fundamental understanding of particle vaporization in the ICP is important to understand potential errors in laser ablation ICP-MS as well as matrix effects in both ICP-OES and ICP-MS. We will discuss measurements of signals from individual droplets and particles made with 0.01 to 1 ms time resolution and the insight these measurements provide on particle vaporization, diffusion and ion sampling. From a practical sample analysis standpoint, measurements of signals from individual particles may provide unique capabilities to measure the chemical composition and size of nanoparticles. Measurements of monodisperse nanoparticles with a range of sizes and composition will be discussed. Measured ICP-OES or ICP-MS signals may depend on a number of factors including where and when the signals are measured relative to the point where particle vaporization is complete and the radial location of the particle in the center channel of the ICP. Three different approaches to the introduction of particles can be considered. Particles may be introduced in monodisperse droplets, particles may be introduced in polydisperse droplets that are desolvated before entering the plasma or particles may be introduced in polydisperse droplets that enter the plasma. The impact of each of these approaches on the measured signals and the relationship between particle size and signal will be discussed.

(441) **Field-Flow Fractionation Inductively Coupled Plasma Spectrometry: Status Report 2010**

Ramon Barnes[1], Atitaya Siripinyanond[2], Supharart Sangsawong[2], Juwadee Shiowatana[2]; [1]ICP Information Newsletter Inc; [2]Mahidol University

The detection and characterization of natural and engineered nanoparticles and macromolecules have received considerable attention recently as many household, medical, and commercial products incorporate nanomaterials. Field-flow fractionation (FFF) using flow (FFFF) or sedimentation (SdFFFF) fields provide separations related to particle size and/or molecular weight. Combined on-line with inductively coupled plasma (ICP) spectrometry detection, the elemental and isotopic distributions in separated materials can be quantified. This review will describe recent instrumental developments and applications of FFF-ICP especially for nanoparticle characterization.

(442) **Liquid Chromatography of Biomolecules Using an Alkylammonium Formate Ionic Liquid Mobile Phase**

Neil Danielson[1], Matthew Collins[1], Shau Grossman[1]; [1]Miami University

We have shown that alkylammonium formate (AAF) ionic liquids, specifically, ethylammonium formate (EAF) and methylammonium formate (MAF), can be effective replacements for standard modifier solvents such as methanol (MeOH) for reversed phase liquid chromatography (LC). Plots of log retention factor versus the fraction of MeOH, EAF, or MAF in the mobile phase indicate quite comparable solvent strength slope values in the 2.2 – 2.5 range. The van Deemter plot taken on the Aqua C18 silica column using 40% MAF-60 % water with phenol as the solute surprisingly showed HETP values about 1.5 times lower than a comparable plot using MeOH. When a polymeric PRP-1 column was used, the van Deemter profile was lower for MeOH than MAF as expected. Suppression of silanol peak broadening effects with phenol by MAF is a likely explanation. The antibacterials nitrofurantoin and furazolidone were separated in 10 min using 10% MAF-90% water at 0.5 mL/min; this separation is difficult to do using MeOH. Compatibility of MAF as both a mobile phase additive at the mM level and as a mobile phase modifier at the 5-20% level with LC with mass spectrometry (MS) detection has been done. We have used MAF as a mobile phase additive for the sensitive detection of statin drugs such as simvastatin (SV), pravastatin (PV), and atorvastatin (AV) using positive polarity with electrospray ionization (ESI) for LC-MS/MS. The MS spectra indicated the methylammonium adduct peak [M+CH3NH3]+ of SV and PV were prominent suppressing other adduct peaks including sodium however AV showed a less intense adduct peak with methylammonium. This work has been extended to other oxygenated organic compounds such as warfarin; in addition, the methylammonium adduct of 1,2-naphthoquinone was significant whereas that for 1,4-naphthoquinone was diminished. A fluorescence spectroscopic study based on tryptophan exposure of proteins including cytochrome c, lysozyme, and hexokinase in alkylammonium formate (AAF) ionic liquids and chromatographic organic solvents has been completed. At room temperature, methylammonium formate (MAF) or ethylammonium formate (EAF) solutions can maintain the native structure of proteins in relatively high ionic liquid concentrations (50%-80% AAF/water or AAF/phosphate buffer pH 7.0) in contrast to similar solutions of MeOH or acetonitrile (MeCN) in water or buffer. The reversed phase separation of a protein test mixture is similar using either a MeCN or EAF gradient. To compare denaturation of proteins under reversed phase HPLC conditions to the spectroscopic work, tryptophan as the internal standard will be separated from a test protein(s) such as cytochrome c, lysozyme, and/or hexokinase using a standard buffer to MeOH, MeCN, or EAF gradient.

(443) **CE-LIF Studies to Facilitate Bioprobe Design and Microbe Detection**

Christa Colyer, Xiuli Lin, Tara Massie, Stephanie Rockett, Jennifer Kneezel; [1]Wake Forest University

Analytical tools that can be adapted to a wide range of analyte types and sizes are invaluable in an increasingly interdisciplinary scientific landscape. Such tools must be able to deliver high efficiency and high sensitivity measurements, especially for bioanalytical targets. To facilitate such measurements, it is possible to exploit noncovalent interactions between protein-based analytes and new fluorescent probes for analysis by capillary electrophoresis with laser-induced fluorescence detection (CE-LIF). Specifically, a comparative and quantitative assessment of the noncovalent binding properties of novel squarylium and triazolylcoumarin dyes will be described. By altering the structure of the dye itself and/or the solution conditions, we can optimize conditions for noncovalent binding. However, a compromise between optimal binding conditions and optimal separation conditions must be struck in order to permit the successful integration of derivatization and analysis procedures by CE-LIF. The nature of noncovalent interactions can be characterized in terms of their governing physico-chemical parameters, so that we can devise more purposeful selections of analyte—probe couples for innovative and important applications of these interactions. For example, preliminary studies involving the turnip yellow mosaic virus TYMV have been conducted with the novel asymmetric, squarylium dye "Red-1c." A fixed concentration of dye titrated with increasing concentrations of virus resulted in a linear increase in fluorescence emission. This response mimicked the behavior of the dye with free solution protein, whereby the fluorescence quantum yield of Red-1c was found to increase significantly (from 0.03 to 0.92) upon noncovalent binding to HSA. The reliance on large surface area-to-volume ratios for microbes with surface charges arising from a number of possible charged moieties exposed at their surface, such as amino acids, acidic carbohydrates, organophosphates and sulfates, to effect separations of noncovalently labeled, intact microbes by CE-LIF, along with the demonstration of greater efficiencies achieved through on-column versus pre-column labeling protocols, will be presented. Furthermore, the potential for using boronic acid-based fluorescent probes, designed to selectively target surface glycoprotein moieties of viral and bacterial agents, will be discussed. This work has important implications for analyses in the areas of clinical chemistry, environmental science, homeland security, and forensics.

(444) **Micellar Electrokinetic Chromatography Coupled to Atmospheric Pressure Photoionization Mass Spectrometry for Analysis of Chiral Benzoin Derivatives Using Mixed Molecular Micelles**

Shahab Shamsi[1], Jun He[1]; [1]Georgia State University

Over the past decade, our laboratory has been actively involved in developing new hyphenation modes for separation and quantitation of enantiomers using capillary electrophoresis coupled to mass spectrometry (MS). In this study, simultaneous enantiomeric separation by micellar electrokinetic chromatography (MEKC) followed by atmospheric pressure photoionization (APPI)-MS of four photopolymerization catalysts: benzoin, benzoin ethyl ether, benzoin methyl ether, and hydrobenzoin were performed. A mixed molecular micellar system consisting of two chiral polymeric surfactants (polysodium N-undecenoxy carbonyl-L-leucinate and polysodium N-undecanoyl-L-leucylvalinate) was used as pseudostationary phase. The MEKC conditions, such as voltage, polymeric surfactant concentration, buffer pH, and BGE concentration, were optimized by a multivariate central composite design (CCD). Next, sheath liquid composition (% methanol in the sheath liquid, dopant concentration, electrolyte composition of the sheath and the sheath liquid flow rate) as well as spray chamber parameters (drying gas flow rate, drying gas temperature, and vaporizer temperature) were also optimized with CCD. Models were built based on the CCD results and response surface method was used to analyze the interactions between MEKC separation and APPI-MS factors and their effects on the responses. The final overall optimum conditions for MEKC-APPI-MS were also determined and compared to MEKC-UV.

(445) **Bioseparations Using Self-Assembled Phospholipids**

Lisa Holland[1], Stephanie Archer-Hartmann[1], Ted Langan[1]; [1]West Virginia University

Phospholipid preparations comprised of dimyristoylphosphatidylcholine (DMPC) and dihexanoylphosphatidylcholine (DHPC)possess unique

physicochemical properties that facilitate their use for thermally responsive capillary separations. The material affords rapid and efficient separations of biopolymers as phospholipds are biocompatible, reduce non-specific interaction within the capillary, and are stable at physiological pH. Under different conditions, these preparations have been incorporated in capillary electrophoresis for improved protein, peptide, drug, and glycan analyses. Phospholipid additives are particularly useful for the separation of complex glycans, including glycans with subtle structural differences. This separation methodology is ideal to address the complexity in monomer composition, branching, and positional linkages inherent in glycan analyses. The properties and separation performance of the material are reported. Innovative separation and detection strategies for glycans are presented and the promise of a wide range of applications is discussed.

(446) **Incorporation of Guanosine Compounds into Sieving Gels for DNA Separations in Capillary Gel Electrophoresis**

Linda McGown[1], Yingying Dong[1]; [1]Rensselaer Polytechnic Institute

A major challenge in DNA profiling has been the resolution of single-stranded DNA fragments of similar or identical length based on differences in sequence. Examples include mutations, polymorphisms and DNA from different organisms in microbial communities. Existing techniques such as single-stranded conformation polymorphism (SSCP) or heteroduplex analysis (HA) rely upon differential migration through a sieving gel due to small differences in conformation of single-stranded DNA fragments or of exact vs. slightly mismatched duplex DNA formed with a complementary strand; however, these small conformational differences become increasingly insignificant with increasing strand size and the high density gels also limit the size of the fragments that can be accommodated. We are working to overcome these limitations through the use of guanosine compounds in combination with sieving gels in capillary gel electrophoresis (CGE). Guanosine compounds exhibit reversible self-assembly to form aggregates of hydrogen-bonded guanine tetrads. They allow unprecedented separation of DNA fragments based solely on sequence rather than strand length or conformation. We will describe the application of these mixed phases for metagenomic profiling of microbial communities such as biofilms.

(447) **Monitoring Anti-Cancer Drug Metabolism with Capillary Electrophoresis**

Amanda Jones[1], Varuni Subramaniam[1], Amanda Haes[1]; [1]The University of Iowa

Acute lymphoblastic leukemia (ALL) is the most common cancer among children. The treatment of ALL utilizes anti-cancer drugs that are metabolized by intracellular enzymes, but the products have poor selectivity and non-specific toxicity. Currently, fluorescence and radioactive labels are used to track targeted anti-cancer drugs and metabolites. The labels provide general spatial information within tissue which may correspond to either the drug or its metabolites. For instance, the anti-cancer drug 6-mercaptopurine (6-MP) forms three specific metabolites from three different enzymes resulting in active, inactive and toxic forms of the drug. The enzyme, xanthine oxidase (XO), which forms the inactive product, is known to be overexpressed in tumors. As a result, the minimization of XO activity on 6-MP metabolism is an important study to increase the overall effectiveness of the drug. In this presentation, capillary electrophoresis will be utilized to directly monitor 6-MP metabolism by XO. Varying enzyme activity and drug concentration reveals information relating to the rate of formation of the metabolite. The optimum enzyme activity and drug concentration at which the least amount of the inactive drug is formed will be discussed. In-capillary mixing of XO and 6-MP will be performed to study a sample mimic in matrices representing biological systems. Enzyme kinetics will be compared between the premixed and in-capillary mixing experiments. We expect that a better understanding of enzyme kinetics associated with the anti-cancer drug will provide important insights into the effective enzyme threshold levels for proper dosages and generalized improvement of current cancer treatments.

(448) **Comparison of Infrared and Raman Spectral Imaging to Study Stem Cell Differentiation**

Max Diem[1]; [1]Northeastern University

Monitoring the development of oocytes, and the early differentiation of stem cells and embryoid bodies has enormous implication in developmental biology. Most microscopic techniques do not have the sensitivity to follow biochemical changes within the target cells, but rely on morphological changes, or staining procedures, to reveal the desired information. In contrast, microscopic techniques based on vibrational spectroscopy can reveal real changes in biochemical composition at the microscopic level. Raman microspectral imaging, which offers spatial resolution similar to confocal fluorescence microscopy, has been found to be a particularly useful method to follow the earliest differentiation of stem cells into embryos. Recent results indicate that individual cells can be detected both in an embryoid stem cell colony and in embryonic bodies. Temporal and spatial spectral changes allow the detection of chemical changes accompanying differentiation.

(449) **FTIR can Detect DNA Conformational Changes in Cells and Tissue in Response to Hydration: Implications for Disease Diagnosis**

Bayden Wood[1], Donna Whelan[1], Keith Bambery[1], Don McNaughton; [1]Monash University

We demonstrate for the first time the conversion of B-DNA to A-DNA in a cell following dehydration using synchrotron FTIR spectroscopy in a single chicken erythrocyte. The next step was to see if we could induce the A-DNA conformation to form the B-DNA conformation from air-dried cells. This required the use of a purpose built dual-chamber transmission cell specifically designed for hydration experiments. The main features observed in the spectra are from protein and DNA and include the strong amide I and amide II bands from proteins at 1652 and 1544 cm-1, respectively, along with the major DNA bands at 1715, 1085, 1051 and 970 cm-1. The DNA bands dramatically increase as the cells become hydrated and also show the large shift in the asymmetric phosphodiester stretching vibration from 1241 to 1227 cm-1. We compare these spectra with those of isolated nuclei and DNA also as a function of hydration. The spectra show similar spectral changes to those observed with the chicken erythrocytes demonstrating that the major changes occurring in the spectra of the chicken erythrocytes are from DNA conformational change. Such changes were first reported in humidity studies performed on isolated DNA in the late 1950s and later distinct conformations of DNA were correlated with X-ray data but hitherto no one has detected this change in eukaryotic cells. We demonstrate the advantage of analysing cells in the hydrated state in a pilot study that compares averaged spectra from patients with Acute Myeloid Leukaemia (AML) (32 patients) and non-Hodgkin's Lymphoma (NHL) (9 patients). The difference spectrum calculated by subtracting the average spectrum of AML from NHL gives a spectrum identical to that of DNA in the B-conformation. The results demonstrate the diagnostic potential of analysing cells in the hydrated state using FTIR spectroscopy.

(450) **Infrared Microscopy of Cells and Tissue: Disentangling Scattering from Absorption**

Peter Gardner[1], Paul Bassan[1], Achim Kohler[2]; [1]University of Manchester; [2]Nofima Mat

Infrared spectroscopy of single biological cells could potentially be invaluable in the field of spectroscopic cytology or in monitoring drug-cell interaction, associated with personalised medicine or new drug development. However, in order for the technique to be successful, practitioners must be able to extract reliably a pure absorption spectrum from a measured spectrum that often contains many confounding factors. The predominant problem is that associated with resonant Mie scattering [1,2]. We have developed a resonant Mie Scattering (RmieS-EMSC) correction algorithm that corrects for the scattering contribution contained in the spectrum and leaves the remaining undistorted absorption spectrum [3]. This algorithm can be applied equally to single cells, where scattering is very strong, and tissue sections where the scattering is generally weaker except at tissue edges. The latter is particularly important in highly glandular tissues as found in prostate biopsies. [1] P. Bassan, H. Byrne, J. Lee, F. Bonnier, C. Clarke, P. Dumas, E. Gazi, M. D. Brown, N. W. Clarke, and P. Gardner "Reflection Contributions to the dispersion artifact in FTIR spectra of single biological cells" Analyst, 2009. 134: 1171-1175. [2] P. Bassan, H. Byrne, F. Bonnier, J. Lee, P. Dumas and P. Gardner "Resonant Mie scattering in infrared spectroscopy of biological materials – understanding the dispersion artefact" Analyst, 2009. 134: 1586-1593. [3]. P. Bassan, A. Kohler, H. Martens, J. Lee, H. J. Byrne, P. Dumas, E. Gazi, M Brown, N. Clarke, P. Gardner. Resonant Mie Scattering (RMieS) Correction of Infrared Spectra from Highly Scattering Biological Samples, Analyst 2010, 135 268-277

(451) **IR Reflectance Microspectroscopy of Particles on a Mirrored Surface: Tools for Estimating Absorbance and Optical Properties**

Michael Myrick[1], Heather Brooke[2], Burt Bronk[3]; [1]University of South Carolina; [2]Naval Research Laboratory; [3]Army Research Laboratory

For several years our research group has studied the spectroscopy of particles (bacterial spores and polystyrene spheres) after filtering them through Au-coated Anodisc™ membranes. Spectra are more reproducible and quantifiable than some other common methods for IR reflectance microspectroscopy, but are complicated by interference and Mie-like scattering effects. In this presentation I will give a short overview of the method and typical data. I'll then describe our effort to compensate for interference and scattering in the calculation of optical properties of the particles, concluding with current work to apply the Mie-derived model of light scattering from particles on a surface developed by Videen to the interpretation of the spectra.

(452) **An Empirical Study to Understand Optical Anomalies in Infrared Microspectroscopy: A Step Forward in Disease Detection**

Heather Gulley-Stahl[1], Andre Sommer[2]; [1]Lexmark International; [2]Molecular Microspectroscopy Laboratory

Infrared microspectroscopy is currently being employed for the detection of disease states in medical research. Typically, a thin section of biopsy is studied using a transmission or transflection sampling method. A drawback to the use of these methods in the clinical setting is that the structure of the sample (voids, mineral inclusions, high contrast edges) can modify the spectral output in various ways making spectral interpretation difficult. Several groups have taken the approach to model these anomalies and correct for them using computer algorithms. However, this approach is successful only when one anomaly is dominant. When several effects are operating, modeling becomes difficult to implement. If the use of infrared microspectroscopy is to progress to the clinical setting, the combined effect of these optical anomalies must be understood or an alternative sampling method is warranted. This presentation will investigate the role that size, shape, refractive index and absorption index contribute to the various optical anomalies exhibited when studying isolated particles or small spatial domains in much larger samples. From these results, guidelines can then be formulated as to which anomaly/anomalies dominate under certain conditions. In addition, an alternative approach to overcome these anomalies will be presented.

(453) **Modeling Distortions in Infrared Spectroscopic Imaging**

Rohit Bhargava[1], Paul Carney[1], Rohith Reddy[1], Brynmor Davis[1], Anil Kodali[1]; [1]University of Illinois at Urbana-Champaign

Infrared (IR) microspectroscopy and imaging are now widely employed for spatially localized spectral analyses. A comprehensive theoretical model for spectral-spatial data recording, however, has only recently been proposed. We present here rigorous theory is for IR absorption microspectroscopy by using Maxwell's equations to model beam propagation through microspectroscopy/imaging systems. Focusing effects, material dispersion, and the geometry of the sample are accounted to predict spectral response for progressively heterogeneous samples. Predictions are validated experimentally using specially fabricated structures and tissues. The results emphasize that meaningful interpretation of IR microspectroscopic data must involve an understanding of the coupled optical effects associated with the sample, substrate properties, and microscopy configuration. Simulations provide guidance for developing experimental methods and future instrument design by quantifying distortions in the recorded data. Distortions are especially severe for transflection mode and for samples mounted on certain substrates. Spatial structure has a profound influence on the recorded data and can be modeled. Last, the model generalizes to rigorously consider the effects of focusing. The distorting effects are shown to be larger than noise levels seen in modern spectrometers. Hence, the model provides a framework to quantify spectral distortions that may limit the accuracy of information or present confounding effects in IR microspectroscopy.

(454) **Projection Two-Dimensional Correlation Spectroscopy**

Isao Noda[1]; [1]The Procter and Gamble Co.

A useful tool in the simplification of often highly congested 2D correlation spectra encountered in practice is described. Projection 2D correlation takes advantage of the fact that correlation spectra can be expressed in terms of simple matrix multiplications of spectral data. Thus, matrix-based projection and null-space projection operations can be applied as an effective filtering tool to transform spectral data to generate new 2D correlation spectra with much more simplified features. Projection matrices used in this approach can be generated from various sources, including a part of original spectral data, other spectra of different origin, perturbation variables, or even arbitrarily chosen mathematical functions. Furthermore, by linearly combining the projected and null-space projected spectra, application of a series of oblique projections to gradually attenuate or augment select features is also possible. Several illustrative examples are used to demonstrate the ability of this technique to obtain pure component 2D spectra, 2D spectra based on contributions of a reduced number of species, and various 2D spectra with specific features selectively accentuated, to assist the unambiguous interpretation of complex and highly overlapped spectral data.

(455) **Two-Dimensional Correlation Spectroscopy of Poly(3-hydroxyalkanoate)s in Terahertz Frequency Region**

Hiromichi Hoshina[1], Yusuke Morisawa[2], Harumi Sato[2], Isao Noda[3], Yukihiro Ozaki[2], Chikoi Otani[1]; [1]RIKEN Advanced Science Institute; [2]Kwansei Gakuin University; [3]The Procter & Gamble Company

Terahertz (THz) absorption spectra of poly(3-hydroxybutyrate) (PHB) and its copolymers were measured by using a terahertz time domain spectrometer and terahertz Fourier transform spectrometer. Absorption peaks due to low frequency vibrational modes of crystalline PHB are observed in 0.3-20 THz. The orientation direction of the transition dipole moment was investigated by the polarization spectra, and peak at 2.92 THz was assigned to a vibration of helical structure along the c axis (fiber axis), and the peak at 2.49 THz was attributed to a vibration due to the hydrogen bonding interactions between helix structures. The temperature dependence of the THz spectra was also examined, which reflects the change in the hydrogen bonding distance. For the analysis of the observed spectra, two-dimensional correlation spectroscopy was applied. The detail of the melting process of crystalline PHA was investigated.

(456) **2D Correlation Raman Spectroscopy for Kinetic Studies of Polypeptide Folding and Aggregation**

Igor Lednev, Vitali Sikirzhytski, Natalya Topilina, Seiichiro Higashiya, John Welch; [1]University at Albany, SUNY

Protein misfolding is often a precursor to the aggregation of proteins associated with human disease. It is known that over 20 global, structurally and functionally unrelated proteins, associated with various neurodegenerative diseases and systematic amyloidoisis, form structurally similar fibrillar structures. Intrinsically disordered proteins can also be involved in the amyloid diseases. Genetic engineering facilitates examination of folding and fibrillation mechanisms by probing the influence of the primary polypeptide sequence on kinetic and equilibrium properties of the protein. We utilize genetic engineering in the study of the mechanism of fibrillation of large biopolymers that are excellent models for intrinsically disordered proteins. The folding mechanism was probed using deep UV resonance Raman spectroscopy, a novel method for acquisition of quantitative information on the peptide backbone conformation of large fibrillar aggregates. Two-dimensional correlation UV resonance Raman spectroscopy was used to probe the fibrillation mechanism at various stages in terms of the sequential order of structural changes and their characteristic times. The evolution of individual secondary structural elements was established through the correlation between Amide I, Amide III, and C_{α}-H bending Raman bands. The temporal order of tertiary and individual secondary structural changes was probed through the cross-correlation of tyrosine and Amide Raman bands. Both the sequential order and the characteristic times of tertiary and secondary structural changes allowed for reconstructing the molecular mechanism of polypeptide structural changes at all stages of fibrillation. The 2DCoS analysis of the Raman data indicated that tyrosine local environment precedes structural rearrangements of disordered elements of the polypeptide backbone followed by the formation of β-sheet. The overall fibrillation mechanism of initially disordered polypeptide is discussed.

(457) **2D-COS Applications of Vibrational Optical Activity in Proteins**

Laurence A Nafie[1, 3], Soo Ryeon Ryu[2], Young Mee Jung[2], Rina K Dukor[3]; [1]Syracuse University; [2]Kangwon University; [3]BioTools, Inc.

The applications of vibrational optical activity (VOA) using 2D correlation spectroscopy (2D-COS) have been growing in recent years. Recently, we have monitored the kinetics of insulin protein fibril formation and development with infrared vibrational circular dichroism (VCD) leading to a better understanding of the association and order of development of five enhanced VCD bands in the amide I and II regions that report on the supramolecular chirality of fibril association, growth and braiding. In a second application, we have undertaken 2D-COS studies of the globular proteins alpha-lactalbumin (ALA) and beta- lactoglobulin (BLG) using Raman scattering and Raman optical activity (ROA). For ALA the agent for correlation change is pH while for BLG the ratio of solvent composition of water to trifluoroethanol (TFE) was changed between zero and one. The results of these studies reveal new information about the dynamics of protein aggregation and conformation change in the response to pH and solvent environment stimulus.

(458) **Self-Modeling Mixture Analysis in Combination with Correlation Spectroscopy**

Willem Windig; [1]Eigenvector Research, Inc.

The combination of correlation spectroscopy in combination with self-modeling mixture analysis is a tool to resolve data of mixtures into chemical components. A variety of techniques have been developed. A technique developed by Windig (1) enables the user to interactively resolve heterospectral correlation spectra, based on mathematically determined sets of pure variables (e.g. wave numbers), where each set describes a variable that is characteristic (pure) for the x data (e.g. Mid-IR) an a variable that is pure for the same component in the y data (e.g., Near-IR) (1). The pure variables are used as concentrations estimates to resolve the mixture data sets. Another technique (2) visually selects pure variables from the location of asynchronous peaks as a starting point for self-modeling curve resolution (SMCR). A problem with self-modeling techniques is that a range of solutions is possible. This presentation will discuss an approach for 'self-modeling correlation spectroscopy', which enables the user to the select the solution range to obtain (a) maximum contrast in the resolved spectra or (b) to obtain the maximum contrast in the resolved contributions ("concentrations") through an angle constraint used in MCR. It will be shown for which experiments the maximum contrast in spectra is the best choice and for which experiments the maximum contrast in contributions is the best choice. 1) W. Windig, D.E. Margevich, W.P. McKenna, Chem. Intell. Lab. Syst., 28 (1995) 108. 2) Y.M. Jung, S.B. Kim, I. Noda, Appl. Spectrosc. 57 (2003) 1376.

(459) **Two-Dimensional Correlation Spectroscopic Analysis of Concentration-Dependent Solvent-Solvent Interactions**

Heinz W. Siesler[1]; [1]University of Duisburg-Essen

Concentration-dependent interactions of different chemical functionalities in solvent mixtures by hydrogen bonding or dipole association can significantly change the corresponding solubility properties and FT-IR spectroscopic parameters (e.g. band position and band shape) of such systems. In most cases a detailed characterization of the involved structural phenomena and their synchronization with concentration can only be obtained by 2D-correlation analysis of the spectroscopic data. In the present investigations we have analysed the FT-IR/ATR spectra of mixtures of different N,N-dimethylamides, ethylacetate and different alcohols and their deuterated analogs by 2D-correlation spectroscopic analysis in order to elucidate in more detail the changes in interaction occurring for different chemical functionalities and as a function of concentration variation.

(460) **Advanced Combustion Diagnostics for Large Scale Fired-Equipment**

J.D. Tate[1], Linh Le[1], Trevor Knittel[2], Jie Zhu[2], Alan Cowie[2]; [1]The Dow Chemical Company; [2]Yokogawa Corporation of America

Energy management and sustainability is one of the hottest topics in the industry at the moment. Volatile natural gas prices in North America as well as volatile oil prices pose an ongoing challenge to U.S. global competitiveness in the petrochemical industry. Pyrolysis of feed stocks (such as naptha, crack gas, etc.) into basic chemicals (e.g., ethylene, VCM, etc.) remains a primary process used in the petrochemical industry. These processes consume enormous amounts of energy and present an opportunity for the industry to lower its energy consumption and improve its sustainability. One of the prevalent unit operations associated with these industries is the "furnace" where the combustion of fuel is used to generate large amounts of heat/radiation required to convert the feed stock into the desired products. Best practice in the industry usually relies on very few analytical measurements to optimize and control these furnaces. In practice, most process control schemes for furnaces rely solely on two analytical measurements for optimization: excess O2 and BTU value of fuel. However, the location of these measurements in proximity to the combustion process and/or their slow relative response results in sub-optimal efficiency of a modern furnace. In addition to these inherent weaknesses, the measurements of other important combustion by-products (such as CO and NOx) are usually ignored in the control schemes adopted by the industry (except for regulatory compliance- where the measurement objectives are very different). We will review a join-effort with the US Dept of Energy and Yokogawa to develop advanced combustion diagnostics that address major weaknesses with existing measurements and best practices in the petrochemical industry.

(461) **Combustion Diagnostics Using *In-situ* Tunable Diode Laser Spectroscopy**

Alan Cowie[1], Jie Zhu[1]; [1]Yokogawa Corporation of America

In recent years, there has been significant advancement in the real-world implementation of advanced process analytical Tunable Diode Laser Spectroscopy (TDLS) technology. One area of industry that is set to benefit significantly from these advancements is combustion control and diagnostics. For both large and smaller scale combustion, the potential for not only improved efficiencies exists but also improved safety and diagnostics. By implementing the use of *in-situ* TDLS, measurements can be made real time at process conditions for gases such as oxygen, carbon monoxide, methane and inferred moisture levels.

(462) **Ambient Air Monitoring Applications for Fluorocarbon Production Units**

Troy Francisco[1]; [1]DuPont

Process analytical technology (PAT) is a major part of an effective effort to improve sustainability and total life cycle of industrial processes. This presentation will focus on ambient air monitoring as a specific example of how PAT can positively impact corporate goals centered around environmental stewardship and safety. Within the company, ambient air monitoring can be implemented as stationary monitors for personnel protection and leak detection, stationary analyzers for industrial hygiene (IH) monitoring or portable devices for on-demand monitoring. DuPont has a long history of being proactive in this field, and a great majority of the systems installed 20+ years ago are now being replaced by new technology that provides much more capability. This forward-looking commitment to ambient air monitoring has paid off by allowing the company to be ahead of the curve with respect to the changes in the EPA Clean Air Act policies that are now requiring additional monitoring, and more complicated data analysis, for sites that produce or handle fluorocarbon products. Three examples that illustrate the DuPont effort will be presented in this forum. First, an general set of applications for stationary monitors for personnel protection and leak detection will be used to introduce both the chemical processes, analyzer system requirements and data reporting schemes. Second, a very specific application of IH monitoring will be presented to show how changing technology necessitates analytical development in order to be able to monitor at low levels with high specificity. Third, a portable analyzer application will be presented to show how the EPA requirements for leak detection and fugitive emission detection fit with existing/standard methods, and how DuPont is working to identify and validate alternative technology that is a better analytical fit for fluorocarbons.

(463) **PAT an Enabler of Sustainability**

Darryl Ertl, Sean Sisk, Bob Cooley, Charlie Goss, Susan Barnes, Greg Gervasio; [1]GlaxoSmithKline

From a sustainability perspective, it is clear that understanding when to take a sample and minimizing the number of off-line samples are key elements in reducing process energy usage, which can be looked at in terms of electricity / fuel, plant time, and resources such as people or materials. This presentation will discuss various applications of PAT and their relationship to obtaining more efficient and sustainalble processes.

(464) **"Green" PAT in Pfizer Manufacturing Plants**

Hiwot Isaac[1], Bronwyn Grout[1]; [1]Pfizer Inc

Process Analytical Technology has been implemented extensively in manufacturing facilities in the Pharmaceutical industry to enhance process knowledge, enhance quality and drive business benefits. "Green" science and environmental sustainability are additional key drivers for PAT. Within Pfizer manufacturing facilities, PAT and process analytics have been deployed for such areas as; increasing solvent recycling efficiency, waste water treatment monitoring and for reducing waste throughout the process and laboratories. This paper will provide an overview of PAT activity in this area in Pfizer Global Manufacturing.

(465) **Temporary On-Line Spectroscopic Analysis for Process Troubleshooting**

Serena Stephenson[1], Wendy Flory[1], Lamar Dewald[1], Roger Gagnon[1]; [1]Dow Chemical Company

Within chemical processes stream compositions are often known only by modeling calculations or the occasional grab sample analysis. However, in some cases, such as when minor stream components cause system fouling and plant upsets, direct and continuous stream composition analysis is required for troubleshooting purposes. Due to the information-rich nature of optical spectroscopic techniques, they are uniquely suited for temporary on-line process stream troubleshooting. Discussed here are example applications where FTIR and/or FTNIR systems were installed to analyze for process impurities in a couple of different Dow Chemical manufacturing businesses. Such impurities were responsible for plugging and corrosion issues resulting in millions of dollars a year in lost productivity. The spectroscopic information enabled validation of process improvements, setting product specification limits, and/or updating process control models.

(466) **Nanoparticle Arrays Tunable in Size and Gap Distance for SERS Detection of Explosives**

Jean-Francois Masson[1]; [1]Université de Montréal

SERS substrates were developed based on gap-tuned film over nanospheres (FON) to detect the presence of trace amount of explosives in water. The SERS substrates are prepared using nanosphere lithography (NSL) in combination with plasma etching

of the spheres to create a tuneable gap between nanospheres. Controlling the etch time provides a method to fabricate well-ordered arrays of nanoparticles with spacing favourable for Raman amplification. Raman was performed at 632 and 785 nm to assess the potential of these substrates at each excitation wavelength. The Raman and LSPR results will be presented for nanospheres of 220, 360, 450, 520, and 650 nm, with various etch times. Appropriately tuning the gap enhances Raman response of nitrobenzenethiol and cysteamine bound to Au or Ag films by a factor of up to 5-10 times in comparison to unetched films (traditional FON). Generally, optimal amplification is obtained with films etched for 1.5 to 4 min, depending on the sphere diameter. AFM/SEM images of the film shows general maintaining of the order of the film, and roughening of the surface with etching. Reproducibility of the sample to sample is better than 20 % for the detection of the Raman response. Modification of the surface with a chemical layer will be demonstrated towards the detection of nitroaromatic compounds in water.

(467) **Controlled Formation of SERS Hot Spots in Gold Nanoclusters**

Rene Lopez[1], Kristen Alexander[1]; [1]University of North Carolina at Chapel Hill

Since its first observation in 1970, surface enhanced Raman scattering (SERS) has been regarded as a promising new tool in the field of sensing technology. Unfortunately, reproducibility issues have made it difficult for scientists to devise experiments that accurately characterize SERS-active nanostructures. The key to this problem lies in the ability to elicit control over the small nanostructure spacing required to attain large enhancement factors. In this research, we present two approaches to regulate the formation and spacing between nanoparticle clusters. The first involves direct nanoassembly of line clusters via a meniscus deposition force and the second controls the interparticle spacing by utilizing the stretching properties of an elastomeric substrate. By building our nanostructures on a flexible substrate and varying the amount of strain applied, nanostructures can be moved relative to one another. Because this technique scales with percent strain, we are able to achieve the nanometer-scale precision necessary to experimentally test the predictions of theoretical simulations.

(468) **Rapid, Sensitive TNT Detection Using SERS Nanotags**

Michael Natan[1], Brad Brown[1], Becky Golightly[1]; [1]Oxonica Materials Inc.

Oxonica Materials Inc. has developed a series of novel, patented nanoparticulate optical quantitation labels based on surface enhanced Raman scattering (SERS). The architecture of these SERS nanotags is simple: a gold (Au) nanoparticle core (typically between 30 and 150 nm in diameter, depending on the application and excitation wavelength), an adsorbed organic molecule ("the reporter"), the SERS spectrum of which is used to both identify the particle type and quantitate the amount of label, and an encapsulating layer of silica, typically between 10-100 nm in diameter. The glass coating renders the particles exceptionally robust to changes in pH, temperature, and ionic strength, and yet enables subsequent chemical and/or biochemical functionalization (e.g. with antibodies). Moreover, because the particles are excited and detected in the near-IR, they are perfectly suited for use in complex, dirty matrices or materials like tissue that would confound results from traditional optical labels. We have developed a series of assay formats and applications that are designed to be used with small or handheld Raman readers. In this presentation, we will highlight results related to rapid quantitation of TNT using a magnetic no-wash format. High-sensitivity detection can be accomplished in two minutes or less, and the receiver-operator characteristic (ROC) are sufficiently rectangular to suggest real-world implementation is feasible.

(469) **SERS Multilayer Substrate Optimization Using SAM Dielectric Spacers**

Charles Klutse[1], Brian Cullum[1]; [1]University of Maryland, Baltimore County

Surface-enhanced Raman scattering (SERS) has become a powerful tool for intracellular analyses due its minimally invasive nature, and its capability to provide molecular structural information about the analytes of interest without the need for exogenous labels. Our group has exploited these unique attributes of SERS to develop and demonstrate a generic procedure for fabricating SERS-based immuno-nanosensors for the detection of cellular signaling species inside individual living cells. Recent results have shown that the sensitivity of the procedure can be improved by an order of magnitude through optimization of the SERS enhancement factors (EFs) of the SERS active nanoparticle substrates used as the sensor supports for the immuno-nanosensors. So far, multi-layer SERS substrates (alternating layers of metal films and metal oxide dielectric spacers on nanostructures) have shown that SERS enhancements can be increased through systematic fabrication of the SERS substrates. To further understand the effect of dielectric spacers on the multi-layer SERS enhancement, self assembled monolayers (SAM) have been used as spacers, thereby enabling systematic variation of spacer thickness and dielectric constant. This talk discusses the characterization and optimization of the SAM-based multi-layer SERS substrates. Monolayers of mercaptoundecane with different terminating functional groups have been systematically sandwiched between layers of metal films to demonstrate dielectric constant dependent SERS enhancement. By caping the SAM with varying amount of metal film overlayer, the metal film overlayer effect on the SERS EF has been evaluated. To evaluate the number of layers ideal for optimal SERS EF, layers of alternating metal films and dielectric spacers were increased systematically and the corresponding SERS EF assessed. Careful choice of optimal SAM and the amount of metal film deposited on SAM has shown that SERS EFs can be further improved by 6 fold compared to previously optimized SERS substrate. This paper will also explore the functionalization of the SAM multi-layer SERS active nanoparticles with appropriate antibodies to develop SERS-based immuno-nanosensors.

(470) **Optimization of Strain Promoted Azide-Alkyne Cycloadition for the Development of Glycan Microarray Technology via Surface Enhanced Raman Spectroscopy**

Sharon Martin[1], Richard Dluhy[1], Jun Guo[2], Gert Jan Boons[2], Yiping Zhao[3]; [1]UGA Department of Chemistry; [2]UGA Complex Carbohydrate Research Center; [3]UGA Department of Physics and Astronomy

Strain promoted azide alkyne cycloaddition for the development of microarray technology was examined by Surface Enhanced Raman Spectroscopy (SERS). Surface Enhanced Raman Spectroscopy is a biosensing technique which has ultra sensitive detection limits and provides intensity enhancement factors of 106 or higher under well defined conditions. In this approach strain promoted azide-alkyne cycloaddtion is used to construct a platform to immobilize biotin to gold or silver nanoparticles. PEG based linkers functionalized with azides are attached to the surface of nanoparticles. Then a biotin complex with a cycloctyne containing spacer will be reacted with the azides attached to the surface. Each step of the surface modification procedure is monitored for completion by SERS. Binding of analytes will give a SERS spectrum that has a unique molecular fingerprint for each analyte. Here, we report studies that were designed to optimize strain promoted click chemistry

reactions. These studies will pave the way for the development of label free detection of microarrays via SERS.

(471) **Nanonstructure Junctions for High Sensitivity Raman Detection and Imaging**

Zachary D. Schultz, University of Notre Dame

Increased understanding regarding the origins of the electromagnetic enhancement in surface enhanced Raman scattering (SERS) has led to tremendous advances in high sensitivity Raman detection. In particular, junctions and crevices between noble metal nanostructures are known to give rise to large SERS signals. To facilitate high-speed acquisition in near-field Raman microscopy experiments, we are studying enhancements obtained from junctions created between metal nanostructures. In tip enhanced Raman (TERS) experiments, interactions between a metallic AFM tip can create a junction with an isolated nanostructure generate a substantial enhancement. Wide ranges of nanostructure arrays, similarly, generate high Raman signals associated the presence of junctions. The combination of SERS and TERS offers tremendous potential for high sensitivity Raman imaging. Results obtained indicate that both the magnitude and the direction of the electric field are important considerations for generating the maximum Raman enhancement.

(472) **Terahertz Measurements of Nonwoven Products**

Jeffrey White[1], John Riccardi[1], Irl Duling[1], David Zimdars[1], Greg Fichter[1]; [1]Picometrix LLC

Nonwoven materials (spunlaid, spunlace, airlaid, hybrid) encompasses a large market of manufactured products. The measurement of a product's basis weight (grams per square meter, gsm) and moisture are critical product attributes. Additionally, many nonwoven products have a coating added to the nonwoven substrate. The detection of the amount and position of the coating on the substrate is useful process information. Time-Domain Terahertz (TD-THz) has demonstrated the capability to make very high speed simultaneous measurements (1000 Hz) of product basis weight and moisture. The single THz source can measure gsm values from below 10 to above 150,000. The measurement spot size can be less than 1 mm allowing measurements of spatial variations in basis weight (i.e., product formation). The measurement sensor operates in reflection (single sided) and is very insensitive to sample positioning and movement and nearly all environmental factors. As opposed to nuclear gauge or X-Ray based measurements, THz measurements are completely safe and do not require any operator warnings. The addition of an external reference structure (rear reflector behind the sample) allows the simultaneous measurement of caliper thickness which permits the direct calculation of density. Again, this measurement can be made with high spatial resolution.

(473) **Nondestructive Evaluation of Sol-Gels Using Terahertz Time-Domain Reflectance Spectroscopy to Sol-Gel Aging**

Gilbert Pacey[1], Anita Taulbee-Combs[2], James Cox[1]; [1]Miami University; [2]University of Dayton Research Institute

Terahertz (THz) time-domain reflectance spectroscopy is evaluated as a technique for nondestructive analysis of sol-gels over the first week of aging without directly contacting or disturbing the sol-gels. In the sol-gels analyzed, tetramethyl orthosilicate (TMOS) is the precursor and polyamidoamine (PAMAM) dendrimers are incorporated into each of two sol-gel sample groups; a third control group contains no dendrimer. The study reports data acquired during sol-gel aging in contrasting humidity and ventilation conditions and determines statistically whether the inclusion of a particular dendrimer and/or the humidity and air circulation of the environment produce significant differences in the THz reflectance intensity observed throughout the first week of sol-gel aging. The results of this study are correlated with previous studies of the same three species analyzed using AFM, impact testing and nitrogen adsorption. The correlations are used to interpret the THz reflectance intensity differences between the sol-gel groups studied using the previously reported results from established methods of analysis regarding the influence of each dendrimer on polymer density, pore size and distribution, and homogeneity of the resulting amorphous silica monoliths.

(474) **High-Speed Terahertz Imaging**

Jeffrey White[1], Irl Duling[1], David Zimdars[1], Greg Fichter[1], John Duquette[1], Chris Megdanoff[1]; [1]Picometrix LLC

A High-Speed Terahertz scanning system and imaging results will be presented. The umbilical connected scanner unit generates linear scan data, over a 6 inch wide band, up to a 10 Hz rate. The scanner is designed to be rolled over large area objects (e.g., storage tanks, radomes) to quickly build up a large area image. Alternatively, the scanner could be mounted in a fixed position over a moving web to generate images. Coupled with a High-Speed (1000 Hz) Terahertz system, the scanner is able to collect a 150 mm by 150 mm size image, with a 2.5 mm pixel size, in less than 10 seconds. Application examples include measurement of defects, voids, water intrusion in multilayer laminate radome or other large area structures. Measurements such as basis weight (grams per square meter, gsm) on moving web products such as paper, nonwovens, building materials and other multilayer products are also presented. Inspection of conveyor packaged goods through packaging cardboard and other protective layer is also presented.

(475) **Nonplasmonic Nanoarrays in Laser Assisted Desorption and Ionization Mass Spectrometric Imaging**

Lin He[1]; [1]North Carolina State University

One of life science's primary challenges is to understand and define the roles of a vast number of biologically relevant molecules associated with various biological events. Yet, the task becomes increasing difficult when most of these species are present in low abundance and are often obscured by the presence of housekeeping proteins at much higher concentrations. Development of effective tools to isolate and enrich such species, followed by accurate identification and quantification, becomes increasingly important. We outline here the development of a novel profiling tool in which a location-specific chemical readout means is used in conjunction with nanoparticles for class-specific enrichment of biologically important molecules. Built on the multiplexing capability of self-addressable solid phase extraction (SPNE) and chemical/position deconvolution capability of MSI, this platform provides an enabling technique to monitor and semi-quantify many crucial molecular species involved in the development and progression of diseases simultaneously.

(476) **Flow-Through Plasmonic Nanosensors Using Embedded Annular Nanoband Electrodes**

Paul Bohn[1], Sean Branagan[1]; [1]University of Notre Dame

This talk describes the construction and characterization of an integrated nanofluidic architecture capable of functioning as an addressable fluidic-switch while accomplishing the linked tasks of detection, identification and chemical transformation. The functional unit is a nanocapillary array membrane with an embedded annular nanoband electrode (EANE), that supports technically diverse, but inter-related, functions, comprised of: (a) flow control, (b) detection of small deviations in the dielectric function of a fluid, (c) highly-specific identification through enzyme-linked bioelectrochemistry and (d) chemical transformation of the detected species. The plasmonic behavior is mediated by the array of nanopores threading the free-standing Au films interposed between two microfluidic channels. The EANEs

exhibit extraordinary optical transmission, mediated by surface plasmon coupling to the regular pore array and propagation through the structure. In this talk we will describe the construction of the device and its optical and electrochemical characterization.

(477) **Nanoholes on Metals and Their Application in Bioanalytical Chemistry**

Alexandre Broloa[1]; [1]University of Victoria

Nanohole arrays milled in thin films of noble metal show an increase of light transmission at certain wavelengths. This phenomenon is called extraordinary optical transmission, and is due to the excitation of surface plasmons (SPs) by grating coupling. The conditions for surface plasmon resonance (SPR) depend on the dielectric constant at the interface. The adsorption of molecular species then shifts the resonance condition forming the principle for the SP-based sensors. In this work, we will report on our progress to the development of an integrated microfluidic system containing nanohole arrays as sensor elements. These arrays are fabricated by focused ion beam milling and they are being explored for detection of cancer markers in serological fluids. The idea is to develop an assay capable of detecting several cancer-specific (ovarian and prostate) antibodies simultaneously and in real time. In this contribution we will also show how these arrays of nanoholes can be used for the screening of antibody producing cells. SPR detection based on nanohole arrays provides the outstanding advantage of a very small sample volume, which is required for ultra-sensitive chemical sensing.

(478) **Sensing Properties of Au, Ag, and Au on Ag Nanohole Arrays**

Jean-Francois Masson[1], Marie-Pier Murray-Méthot[1], Mathieu Ratel[1]; [1]Université de Montréal

Analytical and optical properties will be presented with various nanohole arrays configurations. Nanohole arrays exhibit enhanced transmission at a specific wavelength in resonance with the surface plasmon of the metallic network of nanoholes. The wavelength at which this enhanced transmission occurs depends on the refractive index near the surface, rendering these materials attractive for biosensing. The nanohole arrays with various periodicities, nanohole diameter and metal composition (Au, Ag, bimetallic layers) were prepared using a modified nanosphere lithographic technique of etching the nanospheres with oxygen plasma prior to depositing the metal layer. Thereby, the nanohole arrays are assessed for their analytical properties in terms of sensitivity, spectral resolution and the excitation wavelength. Initial results demonstrated that the sensitivity of Au nanohole arrays is constant for various diameters, while Ag nanohole arrays exhibit improved sensitivity with smaller nanohole diameters. Spectral resolution is improved with nanohole of smaller diameters, providing a more intense and narrower plasmonic band. Integration of surface chemistry and a biosensor will demonstrate the potential of this plasmonic template in detection of proteins.

(479) **Plasmonics for SERS Sensing: Platform Design, Fabrication and Applications**

Tuan Vo-Dinh[1]; [1]Duke University

This presentation provides an overview of the design, fabrication and applications of plasmonics nanoprobes and nanostructures. Plasmonics involves enhanced electromagnetic properties of metallic nanostructures that produce ultrasensitive and selective detection technologies. We describe the development of unique metallic nanoprobe and nanochip structures for surface-enhanced Raman scattering (SERS) detection. The development of nanoparticle-based sensors and large-area nanochip platforms is critical for a wide variety of applications in chemical sensing and biosensing.

(480) **Detecting Vitamin D with Localized Surface Plasmon Resonance Sensors**

Amanda Haes[1]; [1]University of Iowa

Precise control over the size, shape, and local environment of nanoparticles is vital for the development of new technologies based on these materials and their novel properties. This is especially important in understanding how these size-dependent properties impact the detection of biological and chemical targets. For instance, improvements in sensors for the detection of potential (trace) disease biomarkers could revolution how and when disease are diagnosed. This work focuses on the synthesis and modification of gold nanoparticles with antibodies for the detection of vitamin D – a clinical biomarker for nutrition and general health state. Changes in the localized surface plasmon resonance (LSPR) spectra of the nanoparticles will be used to quantify the magnitude of the sensor response. Detection sensitivity and specificity will be shown to depend on the nanoparticle size and surface chemistry homogeneity. This detection approach offers new and promising methods for the rapid, selective, and sensitive detection of biomarkers.

(481) **Low Flow Small Sample MC-ICP-MS Analysis of U and Pu Using Electrochemically Modulated Separations**

M. Liezers[1], D.C Duckworth[1], G.C. Eiden[1], G. Gill[1], M.S. Good[1], G.J. Posakony[1], B.E. Watson[1]; [1]Pacific Northwest National Laboratory

Handling small sample volumes 100ul or less for analysis can be challenging especially where some analyte levels may be very low (attograms) and some degree of elemental separation is required prior to analysis. Conventional off-line methods like column chromatography for separation involve the use of many different reagents that raise risk of analyte contamination, require multiple time consuming processing steps that can also lead to analyte loss. By adopting an on-line miniature electrochemical approach for MC-ICP-MS analysis of U and Pu many of the potential problems highlighted can avoided. Electrochemical Modulated Separation (EMS) employs a tiny electrochemical cell with a glassy carbon working electrode that depending on the potential applied can selectively absorb U or Pu efficiently from an injected solution. The isolated U or Pu can be released back into solution for MC-ICP-MS analysis simply by changing the potential applied to the working electrode with the entire process conducted only in 2% HNO3. As the release of U or Pu back into solution is very rapid and simply triggered by a change in cell potential then at high flow rates (50ul/min) the large preconcentration factor gained can be exploited for very low level measurements. When analyte levels are large, operating at lower flows (1-5ul/min), allows virtually 100% of the analyte to be nebulized directly into the plasma. Analysis times can be extended to improve precision. U and Pu isotope ratios measured on the same tiny sample aliquot using the EMS MC-ICP-MS approach will be presented.

(482) **A New System for Automated Analysis of Micro Volume Liquid Samples by ICPMS**

Daniel Wiederin[1], Kyle Uhlmeyer[1], Cory Gross[1], Patrick Sullivan[1], Nathan Saetveit[1]; [1]Elemental Scientific

Automated analysis of µL and nL samples for trace element and isotopic composition offers the potential for improved detection limits in cases where sample volume is limited and to expand new fields of studies, such as the determination of elemental composition of single cells. This paper explores the use of ICPMS for automated replicate elemental analysis of liquid samples with volume of 1 µL to 50 µL using sample introduction rates in the range 100 nL/min to 10 µL/min. Analyte transport efficiency approaches 100%. In the nL/min range signal intensity decreases almost linearly with flow rate, but detection limits are improved

compared with diluted sample analysis due to minimization of contamination from the diluent. Application to clinical and other micro volume samples is presented.

(483) **Heat-Assisted Argon Electrospray Interface for Low-Flow Rate Liquid Sample Introduction in Plasma Spectrometry**

Ryan Brennan[1], Savelas Rabb[1], Michael Winchester[1], Gregory Turk[1]; [1]National Institute of Standards and Technology

The demands for new or replacement reference materials having lower certified concentrations of trace elements is becoming quite common, e.g., for clinical materials and bioanalysis. These can pose significant certification measurement challenges where traditional analytical methods may no longer be appropriate. To address these challenges, new measurement approaches must be developed, such as the argon electrospray interface reported herein. The aim of this work is to address key challenges in the analysis of biological samples by developing an innovative and efficient sample introduction approach for inductively coupled plasma (ICP) spectrometry that will enable analysis of samples of limited quantity at reduced sample flow rates while achieving maximum sensitivity. A heated (≈ 90 °C) laminar flow interface has been designed to assist in the development of an argon electrospray sample introduction system to meet these requirements. Previously, the stability and robustness of the ICP were compromised by the entrainment of air, N2, or gas mixtures (e.g., Ar-N2) from the electrospray source. Also, more concentrated organic solvents (e.g., 50 % (v/v) methanol-water), typically introduced by electrospray, could generate carbon deposits that obstruct the entrance lens to an ICP optical emission spectrometer (ICP-OES) or the sampler/skimmer cone interface in an ICP mass spectrometer (ICP-MS), decreasing analyte sensitivity. With the new interface design, a stable spray of 5 % (v/v) methanol-water in a pure argon environment is achieved, eliminating the aforementioned problems. The turbulence and the consequent droplet loss caused by high gas velocity around the electrospray capillary are mitigated by using a laminar-flow gas with the aid of a flow diffuser. The argon electrospray interface has been successfully installed on an ICP-OES and an ICP-MS for the first time.

(484) **Potenital of a Novel Low-Flow Drop-On-Demand Aerosol Generator for Plasma Spectrochemical and Speciation Analysis**

J. Niklas Schaper[1], Jan Maßmann[1], Jan H. Petersen[1], Nicolas H. Bings[1]; [1]University of Mainz, Analytical Chemistry

In the field of inorganic trace and species analysis liquid samples are commonly introduced into plasma excitation and ionization sources mainly as an aerosol, which is produced by pneumatic nebulization. It is well known that this aerosol shows a relatively broad droplet size distribution which prevents the complete evaporation of the sample and thus causes serious disturbance of the plasma. Therefore, only small aerosol droplets with a diameter far below 10 µm are suitable for the introduction into the excitation/ionization source. Various spray chamber designs – e.g. optimized for maximum sensitivity or minimum dead volume and wash-out times – serve to overcome this problem, allowing only the small-sized droplets to pass to the plasma source. However, this might also result in an unfavourable loss of sensitivity. Especially when modern low flow separation techniques are hyphenated to ICP-MS the efficient aerosol generation is indispensable. Commonly, an additional make-up solvent flow has to be added to the eluent flow to meet the specifications of conventional nebulization systems for sample introduction into the plasma source. A novel approach of generating aerosol will be presented. The new drop-on-demand aerosol generator based on thermal-inkjet technology, is suitable for the generation of aerosol from very small liquid samples. A micro-controller device has been developed to control individual nozzles of a common printer cartridge regarding droplet diameter and total volume flow rate. Also, the frequency of droplet generation is tuneable over a wide range down to the generation of isolated droplets. The analytical characteristics of this novel nebulizer such as sensitivity and stability will be compared to a common low-flow nebulizer system. Also, its potential for flow injection and speciation analysis will be outlined and the coupling with standard liquid handling devices (e.g. autosamplers) for easy integration into analytical procedures will be presented. The authors kindly acknowledge the financial support within the Interdisciplinary Research Training Program "Trace Analysis of Elemental Species: Development of methods and applications" from the German Research Foundation DFG.

(485) **Development of a New Torch/Fastening Design for Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Low Argon Consumption**

Wolfgang Buscher[1,2], Thorben Pfeifer[1], Michael Sperling[1,2], Rasmus Janzen[1]; [1]Analytical Chemistry, University of Muenster; [2]EVISA; VI for Speciation Analysis

The so called UMAS torch for ICP-MS (Universal Mass Spectrometric Detector for Speciation Analysis) operated at only 1-2 L/min argon consumption is presented in a further developed technical set up. A tool has been constructed that allows the three dimensional adjustment of the injector tube position with respect to its distance from the plasma zone and its angle of the sample carrier gas injection into the central channel of the plasma. During operation the UMAS torch is directly connected with the modified tip of the MS sampler cone, forming an almost closed plasma-MS-Interface system. The analytical figures of merit of the new ICP system are demonstrated. Furthermore, new results from hyphenation of the UMAS detector to gas and liquid chromatography are presented. Particularly when used as speciation-analytical detector after chromatographic separation of metal-containing (bio)molecules the argon consumption per analyzed sample can be reduced heavily due to the long waiting phases in chromatography.

(486) **Interfacing Field Flow Fractionation Techniques to an ICP–MS for the Characterization of Naturally Occurring Particles and Engineered Nano-particles**

Trevor Havard[1], Soheyl Tajiki[1], Thorsten Klein[2]; [1]Postnova Analytics USA; [2]Postnova Analytics Germany

There is has been a recent increase in researchers interfacing Field Flow Fractionation Techniques with ICP-MS for the characterization of naturally occurring nanoparticles to determine the nano toxicological effects of metals within these particles on health and the environment. This paper will describe the different types of Field Flow Fractionation used to interface to ICP-MS. Field Flow Fractionation techniques commercially separates by size (diffusion) and mass (sedimentation), from a few hundred Daltons (0.5nm) up to 100 micron. The system requirements needed to successfully interface these separation devices with ICP-MS and examples of which system best suits the application will be also be discussed. Other examples will include how engineered nanoparticles can also be measured using this technique. And what technological advances are happening now with the separation technique and detections methods.

(487) **Fundamental Properties of Non Equilibrium Radiofrequency Plasmas used for Material Analysis**

Philippe Belenguer[1], Philippe Guillot[2], Thomas Nelis[2], Abdelattif Zahri[1], Laurent Therese[2]; [1]LAPLACE, CNRS, Toulouse; [2]DPHE, CUFR, Albi

Fundamental properties of non equilibrium plasmas for material analysis Abstract Non equilibrium low temperature plasmas are widely used in industrial applications, for example in

microelectronics industry for etching or deposition of materials, lamps, plasma display panels, plasma thrusters, in medical or biological applications to name just a few of them. In this paper will will focus on non equilibrium plasmas used for material analysis as in glow discharge spectrometry. We will present some fundamental properties of such plasmas through numerical models and experimental measurements. Densities, electric field distribution, electron or ion energy distribution functions , ionization mechanisms will be investigated and presented.

(488) **Spatial Distribution and Temporal Dependence of the Optical Emission in a Pulsed Radiofrequency Glow Discharge**

Nerea Bordel[1], Rebeca Valledor[1], Jorge Pisonero[1], Thomas Nelis[2], Alfredo Sanz-Medel[1]; [1]University of Oviedo, Spain; [2]CFUR J. F. Champollion, Albi. France

Glow discharge (GD) atomic spectrometric techniques, such as glow discharge mass spectrometry and glow discharge optical emission spectroscopy, are commonly used as analytical techniques able to provide fast and sensitive direct chemical analysis as well as high resolved depth profile analysis of multilayer materials. Traditional GD-OES instruments allow end-on viewed of the GD plasma, and the emission intensities are in general collected with a point focused lens so they are integrated over the whole plasma. However the observation of the GD plasma plume along its axis can provide useful information about the spatial distribution of the different species, since different discharge processes take place at different distances from the sample. For instance, one of the possible applications of these fundamental studies could be the optimization of the interface in glow discharge mass spectrometers, by evaluating the optimal point for ion extraction. In this work, the spatial distribution of argon and copper population in continuous and pulsed radio frequency GD plasma is investigated for a modified Grimm-type source, previously designed for analytical purposes [1]. Emission spectra have been taken along the plasma plume and 1 mm spatial resolution have been achieved by using a system of lenses and optical fibers, which can be displaced parallel to the axis discharge with micrometric precision. Depending on the argon flow rate and pressure, and for a pure copper sample, two different plasma regimes have been observed and characterized: at certain flow rate and pressure conditions the plasma shows a violet color corresponding to the Ar (discharge gas) emission but at the same flow rate but higher pressures the plasma takes a green color corresponding to a more enhanced copper emission. Furthermore, the emission in the green regime is more confined in the proximity of the anode, since all the emission intensities decrease faster than in the violet regime. Moreover, time resolved study of the optical emission in the three temporal domains (prepeak, plateau and afterpeak) present in pulsed rf-GD has been carried out. [1] J. Pisonero, J.M. Costa, R. Pereiro, N. Bordel, A. Sanz-Medel, J. Anal. At. Spectrom., 2001, 16, 1253-1258.

(489) **Characterisation of Nanostructured Materials by Plasma Profiling Ion Mass Spectrometry**

Agnès Tempez[1], Lara Lobo[2], Abdelhak Bensaoula[3], Nunzio Tuccitto[4], Jorge Pisonero[2], Antonino Licciardello[4], Chris Boney[3], Nacer Badi[3], Nerea Bordel[2], Patrick Chapon[1]; [1]Horiba Jobin Yvon; [2]University of Oviedo; [3]University of Houston; [4]University of Catania

A plasma source fed with pure Ar and created under a pulsed RF potential is coupled to a time of flight mass spectrometer (TOFMS) for depth resolved compositional analysis. This plasma profiling ion mass spectrometry (P²IMS) technique is well suited for profiling ultra thin to thick conductive and non conductive layers with nm depth resolution. There is a perfect fit between the fast erosion rate of the plasma and the ultra-fast detection and quasi-simultaneous acquisition of all mass ions of the TOFMS. The orthogonal TOFMS configuration also allows extraction of temporal plasma source characterization. This is all the more important as signals are largely enhanced in the plasma extinction phase (once RF is turned off) in the so called afterglow region. Sample ion signals are then created through ionisation from the Ar metastables called Penning Ionisation. This region in which molecular ions are detected is also critical for characterising polymer materials. We will specifically present examples of P²IMS analysis of nitride layers for photovoltaics, photonics, and high temperature dielectrics capacitors; such as the stability of BON/Ti on Si capacitors in the range of 500°C to 600°C, boron interfacial diffusion in BON – Si and SiO2 – Si layers using B10 isotopes as a marker, and In% determination over the whole compositional range, their Mg doping levels and Al contamination at the GaN-InGaN interfaces. We will also show results on the application of P2IMS to multilayered polymeric structures.

(490) **Making Lemonade from Lemons: Using Metal Oxide Ions in GDMS for Materials Speciation**

Fred King[1], Jennifer Robertson-Honecker[1], Na Zhang[1], Megan DeJesus[1], Guodong Gu[1]; [1]West Virginia University

For many years analytical glow discharge mass spectrometry was plagued by the presence of various polyatomic species that complicated its use for elemental analysis. Considerable effort went in to optimizing conditions to minimize the spectral contribution of such species or to use hardware and software approaches to resolve the problem. In more recent times, with the redirection of the elemental mass spectrometry community toward chemical speciation, the effort has gone in to utilizing polyatomic ions to gain insight into the chemical environment of the elemental entity. This report will focus on the utilization of metal oxide ions and cluster ions to determine the oxidation state of an analyte material. In this work, plasma operating conditions are optimized, ironically, to favor the production of these cluster ions in order to achieve speciation. Results from studies of the impact of pressure, sampling distance, and discharge power pulsing on such cluster ion production will be presented. The use of characteristic ions and ion ratios to achieve speciation will be described for various transition metal oxides. To date these strategies have proven successful for oxides of chromium, manganese, and most recently iron. The work provides us with insight into this new area of application for GDMS.

(491) **Distance-Of-Flight Mass Spectrometry: A New Instrumental Concept for Elemental Mass Spectrometry**

Alexander W.G. Graham[1], Steven J. Ray[1], Christie G. Enke[2], Charles J. Barinaga[3], David W. Koppenaal[3], Gary M. Hieftje[1]; [1]Department of Chemistry, Indiana University; [2]Department of Chemistry, New Mexico U; [3]Pacific Northwest National Laboratory

A proof-of-principle glow discharge—distance-of-flight mass spectrometer (GD-DOFMS) has been constructed in the Hieftje laboratory in collaboration with Pacific Northwest National Labs and the University of New Mexico. Distance-of-flight mass spectrometry is akin to time-of-flight mass spectrometry (TOFMS) in that both methods separate ions of different mass-to-charge (m/z) by means of their mass-dependent velocities. However, in DOFMS the position of an ion at a given time, rather than its flight time to the detector, is measured to determine its m/z. DOFMS offers a number of advantages over TOFMS. First, because DOFMS separates ions in space, fast timing and digitization electronics essential to TOFMS are obviated. In addition, charge detection arrays that demonstrate broad dynamic range and high sensitivity can be employed, thereby extending the upper mass limit available to the spectrometer. In order to measure the position of ions of several m/z simultaneously, the ions must be all in focus at a single

time—at which their positions are measured. The parameters for focusing ions at a single time were established by Enke and Dobson in 2007 and have been implemented in our instrument. In our DOFMS instrument, an ion packet is accelerated to a constant momentum, propagates through a field-free region, and is energy-focused by means of a linear-field reflectron. This ion-flight pathway yields a single time at which ions of all m/z are focused and at which DOF is inversely proportional to m/z. At the detection time, ions are electrostatically pushed onto a spatially selective detector positioned orthogonal to the ion packet's path. Currently, an MCP-phosphor assembly is used for detection. Resolving powers of around 800 for a suite of elements from 27Al to 208Pb have been demonstrated with our first-generation instrument coupled to a reduced-pressure glow discharge. Design considerations, first DOFMS data, and implications of this new mass spectrometer for elemental analysis will be discussed.

(492) **Particle Beam/Hollow Cathode Optical Emission Spectroscopy (PB/HC-OES) as a Tool for the Study of Metal Binding of Proteins**

R. Kenneth Marcus[1], C. Derrick Quarles, Jr.[1]; [1]Clemson University

The determination of metal distributions in serum proteins is one of the largest challenges in biochemistry. Many proteins are well known to control the trafficking of metals throughout the body. In fact, metals are present in fully one-third of all proteins, as they affect fundamental processes such as redox chemistry within cells. Metal partitioning determinations require quantitative measurement and identification of the metal and the proteins in question. We present the use of the PB/HC-OES methodology as an effective tool in metallomics, having the ability to quantify metals and their stoichiometry within proteins without the need to quantify the actual protein content; in a single measurement. The method uses the optical response of the metal to set its concentration in the sample, and the ratio between it and the non-metals (C, H, N, S) of the proteins to assess stoichiometry. Examples will be presented regarding the loading of multiple metals in the iron-transport protein, transferrin. Results of competitive binding experiments will also be presented to illustrate the utility of the method.

(493) **The Analytical Capabilities of FAIMS: Back to the Basics and More**

Gary Glish[1], Mark Ridgeway[1], Alessandra Ferzoco[1], Alice Pilo[1], Desmond Kaplan[2], Melvin Park[2]; [1]University of North Carolina; [2]Bruker Daltonics

Field asymmetric ion mobility spectrometry (FAIMS) is a technique that separates gas phase ions based on the ratio of high- and low-field mobility. FAIMS devices can operate as a filter where only select species are allowed to pass to a mass spectrometer. The filtering mode drastically reduces chemical noise without additional sample preparation, and acts on a much faster time scale than liquid chromatography separation. Unfortunately, coupling of most current FAIMS devices to a mass analyzer results in significant ion transmission losses through ion diffusion within the device, and inefficient coupling between the dispersed ion beam and the conductance limit of the mass analyzer. A new planar FAIMS design is coupled to an ESI capillary and achieves up to 100% transmission efficiency. This new design can be used as a filter to improve the limit of detection of plasma biomarkers or many other analytes of potential biological or environmental interest.

(494) **Applying Digital Waveforms to Mass Spectrometry**

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With the advent of direct digital synthesis (DDS), waveforms can be produced with phenomenal frequency resolution (up to 48 bit). Our work has shown that the DDS technique can be rapidly manipulated and applied to the production of arbitrary waveforms. Now arbitrary waveforms can be produced so that the temporal spacing as well as the amplitude can be precisely varied within the arbitrary wave. This development has subtle, yet profound, implications for the trapping, manipulation and mass analysis of ions. Consider the rectangular waveforms typically used in digital ion trap mass spectrometry. The temporal placement of the transitions from high to low and low to high can be defined within the temporal jitter of the frequency-variable clock used to readout the waveform. Using off-the-shelf DDS test boards, this jitter was shown to be approximately 20 ps. This value can be reduced to approximately 6 ps. It defines the temporal error of the rectangular wave, ΔT. The period of the rectangular wave then is T and the resolution of the waveform is $T\Delta T$. This has phenomenal implications for the achievable mass resolution of digital ion traps (DITs). Our development also allows the duty cycle of the rectangular waveforms to be varied with up to 48-bit precision. Changes in the waveform can be done in as little as 4 ns. We have shown through simulation (SIMION) that the rectangular waveforms applied to a simple 4 rod quadrupole with grounded end caps can be manipulated to trap and collect ions of any mass and then axially eject them on demand simply by temporal manipulation of the applied rectangular waveforms. The manipulation of the applied waveforms allows the axially ejected ions to be focused or collimated. The kinetic energy of the ejected ions can also be adjusted down to a few electron volts while maintaining their focus. This development has exciting implications for quadrupole time-of-flight and digital ion trap mass spectrometry.

(495) **Metastable Atom-Activated Dissociation of Glycopeptides, Nitrosylated Peptides and Non-Peptidic Analytes**

Glen P Jackson[1], Shannon L Cook[1]; [1]Ohio University

We are presenting our most recent results of a relatively new type gas-phase fragmentation method, which uses metastable atoms of noble gases as the source of energy or electrons (depending on precursor charge state). Extensive backbone fragmentation is observed of singly- and doubly-charged peptides through the interaction of isolated precursor ions with a high kinetic-energy beam of argon or helium metastable atoms. The fragmentation spectra indicate that MAD occurs through similar radical chemistry pathways as electron capture (ECD), electron transfer (ETD), electron ionization (EID), and electron detachment dissociations (EDD). This novel dissociation method therefore appears to be an interesting and complementary alternative to ECD, ETD, and CID, especially for singly-charged precursor ions. We have shown that metastable atoms are effective electron vehicles for peptide dissociation. Extensive fragmentation, in the form of a-, b-, c-, d-, v-, w-, x-, y-, and z-ions, for singly- and doubly-charged peptide ions have already been demonstrated. In addition, we have achieved phosphorylation identification and localization as well as isobaric amino acid differentiation in both cationic and anionic peptides. Several cleavages on the N-terminal side of proline were also detected, resulting in a-, b-, w-, x-, y-, z-type ions. We have successfully shown that MAD-MS can result in over 100% sequence coverage with MAD fragmentation efficiencies approaching 4% for cationic peptides and 7% for anionic peptides and provide a variety of a-, b-, c-, x-, y-, and z-type ions with frequent side chain fragmentation. The structural characterization of other post-translated peptides, such as nitrosylated and sulfated peptides, and biologically relevant non-peptidic species are currently being explored. We will also show results from MAD

activation of singly-charged lipids such as sphigomyelin (SM) and phosphatidylcholine (PC).

(496) **Incorporating a Surface-Induced Dissociation Device into an Ion Mobility-Tandem Mass Spectrometer for Structural Analysis of Protein Complexes**

Chengsi Huang[1], Mowei Zhou[1], Anne E. Blackwell[1], Eric Dodds[1], Ünige Laskay[1], Vick H. Wysocki[1]; [1]University of Arizona

Surface-induced dissociation (SID) is an ion activation method implemented in mass spectrometry. It is a collisional activation method similar to the more commonly used collision-induced dissociation (CID). However, due to the higher center-of-mass of the surface, SID results in higher energy deposition into analyte molecules than does CID. When studying large systems such as protein complexes, the higher energy deposition of surface-collisions allows additional fragmentation pathways to be accessible, yielding product ions that give more useful structural information than CID. Successful SID requires steering and accelerating precursor ions into a surface, and collecting the product ions that result from the collision. While SID has been successfully implemented in various mass spectrometers for the study of a multitude of ions, much research is needed for a complete understanding of the mechanisms and energetics of ion activation and dissociation by SID. Studying the structure of ions after collision with a surface could lend insights into the mechanism. Ion mobility (IM) is typically used to determine the collisional cross sections of gas phase ions. IM of SID fragments could provide information on the structures of unfragmented precursor ions and product ions after activation. Meanwhile, placing the SID device behind the IM cell will allow fragmentation of shape-selected precursor ions that carry similar mass-to-charge ratios. Based on a previous design, we have designed and implemented an SID device with appropriate dimensions for incorporation into a quadrupole time of flight mass spectrometer coupled with ion mobility (Waters Synapt G2®). Ion motion in the SID device in both transmission mode and surface collision mode was simulated using SIMION®. For preliminary testing, the homemade SID device was placed in a quadrupole time of flight instrument (Waters Q-TOF2), similar in configuration to the Synapt G2®. CID and SID of leucine enkephalin (a small peptide) and sHSP 18.5 (a homogeneous, non-covalent protein dimer) were demonstrated in Q-TOF2 with this device in place. For installation in the Synapt G2, the transfer guide was shortened to accommodate the SID device. With the SID device placed behind the IM cell in the G2, both CID and SID have been tested in the new IM-SID-TOF setup. The systems studied include cesium iodide clusters peptides such as Leu-enkephalin, desR9- bradykinin, and the reverse sequence peptides GRGDS and SDGRG, and protein complexes of C-reative protein and transthyretin. When comparing SID to CID, difference in fragmentation patterns and efficiency were observed for each of the systems studied.

(497) **A New Segmented Rectilinear Ion Trap with Modified Smalley Nozzle for Creation of New Catalysts and Dielectric Materials**

Guido Verbeck[1]; [1]University of North Texas

This is a multidisciplinary project, where chemistry meets electrical engineering, and has the ultimate objective of developing novel dielectrics, new catalysts, and other electronic materials with preparative mass spectrometry technologies for the manufacture of "next generation" materials and films. The principal aims of the project were therefore to develop softlanding processes based on stoichiometrically controlled metal-carbides, nitrides, oxides, and phosphides and the new chemistry of volatile organometallic complexes of Zr, Hf, lanthanides and Titanium and to assess the physico-chemical and electronic properties of the resulting materials. Once ions of interest have been synthesized and mass-selected in the gas-phase, it is essential to control the subsequent deposition process. The approach used for deposition will be soft-landing, that is, direct ion deposition from the gas-phase onto a solid surface without fragmentation and significant structural distortion. This has been accomplished in one of two ways, direct deposition and employing the use of a rectilinear ion trap. The main approach to soft-landing employs a rectilinear ion trap mass selective device with Smalley-Gerry-Walker expansion source, and post plasma source for surface processing. This instrument layout is necessary on two fronts. Most ions will not softland intact at high energy deposition. Therefore, the RIT, with the new RF dampening electronics, thermalizes the ions before deposition with no chemical reaction. An example is C60, which can be softlanded at

(498) **New Concepts in Ion Mobility-Mass Spectrometry Instrumentation**

Jody C. May[1, 2, 3], Sevugarajan Sundarapandian [1, 2, 3], John A. McLean [1, 2, 3]; [1]Department of Chemistry, Vanderbilt University; [2]Vanderbilt Institute of Chemical Biology; [3]VIIBRE

In ion mobility-mass spectrometry (IM-MS) instrumentation, analyte sensitivity depends on ion transmission efficiency, while analyte selectivity depends on the spectrometer's resolving power. It is always desirable to have the best of both in order to maximize the amount of information gleamed from the chemical analysis. Fundamental improvements in the sensitivity, resolving power, sample throughput and overall versatility of IM-MS instrumentation is currently being pursued in our laboratory through three different instrument development projects. (1) For improving IM sensitivity, we are developing electrode geometries for a new generation of ion mobility drift cells which utilize the principle of alternating higher and lower DC only electric fields which periodically refocus ions to the center axis of the drift cell. Due to the ion's forward migration, the periodic DC field serves an equivalent effect as a temporally dynamic RF field. We will present ion trajectory simulation results for several periodic drift cell electrode geometries, as well as compare these results to the more conventional uniform field drift cell utilizing an electrodynamic RF ion funnel at the back end. The tradeoffs between sensitivity and resolving power will be discussed. (2) For improved IM resolving power, we are developing an extended length drift tube (ca. 1 meter) which is designed to operate at high voltage (>5 kV), intermediate pressures (1-10 Torr) and low drift gas temperature (down to ca. 80 K). The theoretical considerations will be presented, including predicted analytical performance and current progress will be discussed in light of the technological challenges associated with the instrument's design. Finally, (3) we are pursuing the development of a multi-channel (x8) IM-MS capable of conducting multiple experiments simultaneously, which is designed to significantly improve sample throughput and experimental versatility. Design considerations and the latest status of this instrument will be presented. The prospects of these compartmentalized instrument development projects will be discussed in terms of a global analysis platform approach which we are beginning to develop.

(499) **Single Particle Tracking Analyses of Hyperspectral Fluorescent Images**

Patrick Cutler[1], Michael Malik[2], Fang Huang[2], Diane Lidke[1], Keith Lidke[2]; [1]Department of Pathology, University of New Mexico; [2]Department of Physics and Astronomy, UNM

Single particle tracking (SPT) is an important technique for monitoring protein dynamics in living cells. Semiconductor nanocrystals, or quantum dots (QDs), are often used as molecular tags in SPT due to the photostability and high brightness needed for long-term observation. Another advantage of QDs is their wide

excitation but very narrow emission bands, which permit simultaneous excitation of spectrally distinct QDs with a single excitation wavelength, facilitating multiple color labeling and imaging. We have developed a hyperspectral line scanning microscope (128 spectral channels) with the potential to acquire spectral images with single QD sensitivity at 30 frames per second. Typically in SPT, a 2 dimensional Gaussian estimate of the microscope point spread function is used to localize a single fluorophore with precision a factor of ten or better than the diffraction limit. However, precise localization suffers when particles overlap at distance comparable to the diffraction limit. Hyperspectral images provide additional spectral information that can be used to improve the estimate of fluorphore position and distinguish between spectrally distinct but spatially overlapping particles. Due to the narrow and characteristic emission spectra of QDs, we are also using a Gaussian curve to estimate the spectrum of single QDs. To incorporate spectral information into the modeling of the point spread function, we use a 3 dimensional Gaussian to estimate the spatial position, emission intensity, and spectral characteristics (peak position and width) of single QDs with an iterative maximum likelihood method. Implementation of this technique on graphics processing unit (GPU) hardware allows for fast and efficient single molecule localization in hyperspectral images. To further improve localization, background corrections are made using estimates of background spectra from multivariate curve resolution techniques. We demonstrate this technique on QD images acquired with our newly developed hyperspectral line scanning microscope.

(500) **Integrating Physics with Chemometrics for Enhanced Vibrational Spectroscopic Imaging**

Rohit Bhargava[1], Anil Kodali[1], Matthew Schulmerich[3], Xavier Llora[1], Rohith Reddy[1]; [1]University of Illinois at Urbana-Champaign

Spectroscopic imaging is proving increasingly useful for biomedical applications when used with chemometrics to recognize diverse cell types and diseases in complex tissue. The automated recognition, in turn, facilitates high throughput, low variability and high confidence in diagnoses or scientific understanding of molecular processes. While the molecular basis of chemometrics is well established, integration of the emerging understanding of the physics of spectroscopic signals and image formation can significantly augment chemometric analyses. Here, we first present a systematic approach to including light propagation within structured materials and spectral images acquired from such samples. The nature of spectral distortions due to heterogeneity of tissue samples is discussed and the sources of variance in data from spectral images are identified. Next, the improvements in quality and fidelity of chemometric data analysis through such models are demonstrated. Last, we discuss unique structures for surface enhanced Raman spectroscopic probes to realize enhanced vibrational spectroscopic imaging. In sum, the potential of described approaches in providing an enhanced spectroscopic imaging solution for analyses is illustrated using examples from prostate, breast and colon cancer pathology.

(501) **Autonomous Hyperspectral Imaging in Real-Time**

Patrick Treado[1], Robert Schweitzer[1], Arjun Bangalore[1]; [1]ChemImage Corporation

Hyperspectral imaging sensors for the detection of challenging targets in complex environments are maturing. Hyperspectral imaging sensors generate significant volumes of data that needs to be reduced to a manageable form on a timescale that's relevant to its intended use. This presentation will discuss some representative sensor architectures, algorithmic approaches and example applications where autonomy and real-time operation are critical.

(502) **Hyperspectral Imaging of Microalgae Using Two-Photon Excitation**

Howland Jones[1], Michael Sinclair[1], Ting Luk[1], Bryce Ricken[1], Thomas Reichardt[1], Omar Garcia[1], Jerilyn Timlin[1]; [1]Sandia National Labororatories

A considerable amount research is being conducted on microalgae, since microalgae are becoming a promising source of renewable energy. Most of this research is centered on lipid production in microalgae because microalgae produce triacylglycerol which is ideal for biodiesel fuels. Although we are interested in research to increase lipid production in algae, we are also interested in research to sustain healthy algal cultures in large scale biomass production farms or facilities. The early detection of fluctuations in algal health, productivity, and invasive predators must be developed to ensure that algae are an efficient and cost-effective source of biofuel. Therefore we are developing technologies to monitor the health of algae using spectroscopic measurements in the field. To do this, we have proposed to spectroscopically monitor large algal cultivations using LIDAR (Light Detection And Ranging) remote sensing technology. Before we can deploy this type of technology, we must first characterize the spectral bio-signatures that are related to algal health. Recently, we have adapted our confocal hyperspectral imaging microscope at Sandia to have two-photon excitation capabilities using a chameleon tunable laser. We are using this microscope to understand the spectroscopic signatures necessary to characterize microalgae at the cellular level prior to using these signatures to classify the health of bulk samples, with the eventual goal of using of LIDAR to monitor large scale ponds and raceways. By imaging algal cultures using a tunable laser to excite at several different wavelengths we will be able to select the optimal excitation/emission wavelengths needed to characterize algal cultures. To analyze the hyperspectral images generated from this two-photon microscope, we are using Multivariate Curve Resolution (MCR) algorithms to extract the spectral signatures and their associated relative intensities from the data. For this presentation, I will show our two-photon hyperspectral imaging results on a variety of microalgae species and show how these results can be used to characterize algal ponds and raceways. Sandia National Laboratories is a multi-program laboratory operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Company, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

(503) **Micoralgal Biodiversity as Novel Indicator for Marine Ecosystems - Combining Spectroscopy, Imaging and Prior Information through Bayesian Statistics**

Frank Vogt[1], Morgan McConico[1]; [1]University of Tennessee, Department of Chemistry

Environmental analytical chemistry often involves investigations of numerous, complex and interrelated effects on ecosystems. To study these impacts, novel chemical sensing strategies are required which yield a more comprehensive picture of chemical/biological trends compared to what can be derived from a single chemical parameter. One innovative approach is to use biological entities themselves as novel *in-situ* probes. For instance, microalgae cells have been reported to adapt quickly and sensitively to changes in marine ecosystems. On a longer time scale, the biodiversity of microalgae (biodiversity = species' types and abundances) undergoes shifts along with chemical modifications of individual cells. We report on progress in utilizing microalgae and their biodiversity as a novel probe to investigate the chemical/biological state of marine ecosystems. Biomaterials contain many characteristic, infrared-active components and thus, FTIR spectroscopy is a promising technique. It was hypothesized that a relation between the cells' spectroscopic signatures, their

biodiversity, and an ecosystem's chemical state can be established ('calibration'). Microalgae were cultured under different growing parameters namely concentrations of the nutrient sources carbon (via CO3-) and nitrogen (via NO3- and NH4+). Monolayer films of cells were investigated by FTIR imaging regarding chemical changes in each cell and the overall microalgal biodiversity, both as functions of environmental conditions. The biggest challenge for sample characterization is the large number of possible classes, i.e. number of species times growing conditions, which must be identified in mixtures and their magnitude quantified. In order to enable high classification rates, algorithms based on Bayesian statistics were developed. Bayesian classification gains from incorporating –on top of spectroscopic data– physical and chemical background information like (here) temperature, light, pH, salinity and the microalgae cells' geometric properties (footprint); the latter one utilizes the species' different cell shapes. In order to extract this geometric information from spectroscopic imaging data, novel algorithms for image analysis were designed. Thus, spectroscopic imaging enables the acquisition of chemical and geometric information about the cells and probes enough cells within a sample to measure microalgal biodiversity. Through Bayes' statistics, these spectroscopic analyses can be augmented by additional chemical information about the samples' origin.

(504) **Passive Standoff Detection of Solid Explosive Residues on Soil via Infrared Hyperspectral Imaging**

Neal Gallagher[1], J. F. Kelly[2], T. A. Blake[2]; [1]Eigenvector Research, Inc.; [2]Pacific Northwest National Laboratory

Hyperspectral images of Quincy soil with residues of sodium hypochlorite and ammonium nitrate explosives were recorded using a commercial longwave infrared imaging spectrometer at a stand-off distance of 3 m. Images were also recorded after coating the explosive residues with thin layers (0.1-2 mm) of soil. Anomaly detection methods principal components analysis (PCA), principal autocorrelation factors (PAF) and principal difference factors (PDF) clearly showed target analytes – even when coated with soil. Subsequent estimation of target spectra with multivariate curve resolution (MCR) showed good correlation between the sodium hypochlorite spectrum and reflectance spectra measured in the laboratory. Results suggest that, contrary to conventional wisdom, target analytes may be observable on, and below, soil surfaces using passive infrared spectroscopy at least for thin covering layers up to about 1 mm.

(505) **Rational Design of Nanostructured Probes for Surface Enhanced Raman Spectroscopy**

Anil Kodali[1], Matthew Schulmerich[1], Xavier Llora[1], Rohit Bhargava[1]; [1]Univ of Illinois at Urbana Champaign

Surface enhanced Raman scattering (SERS)-based probes can enable simultaneous and quantitative imaging of tens to hundreds of molecules down to ultra-low concentrations. Here, we present a rational design strategy to obtain reproducible probes using nanospheres with alternating metal and reporter-filled dielectric layers. An optimal strategy is presented to provide a facile route to tuning the response of these structures to be optically flat, resulting in a uniform enhancement for multiple excitation wavelengths and for different shifts. First, we present theoretical calculations that open door for a set of reproducible and robust probes with controlled sensitivity for molecular sensing. Next, we experimentally demonstrate the contribution of electromagnetic enhancement in simple nanosphere structures and compare the enhancements to the theoretical predictions. Last, we show the modeling, fabrication and a statistical validation of signals derived from preliminary probe structures. The strategy proposed here is expected to provide for methods to devise a palette of molecular probes.

(506) **Correction for Physiological Dynamics Significantly Improves Spectroscopy Based Transcutaneous Blood Glucose Predictions**

Ishan Barman[1], Chae-Ryon Kong[1], Narahara C. Dingari[1], Jeon-Woong Kang[1], Ramachandra R. Dasari[1], Michael S. Feld[1]; [1]Massachusetts Institute of Technology

Non-invasive blood glucose measurement techniques have been extensively investigated due to its important implications for diabetes management and therapeutics. Near infra-red (NIR) Raman spectroscopy has previously provided successful predictions of glucose at physiologically relevant concentrations in serum, whole blood and even in human volunteers. However, the development of a clinically accurate and robust algorithm capable of prospective prediction has proven to be challenging. In particular, correction for the presence of a physiological lag between blood and interstitial fluid (ISF) glucose has been difficult. The lag time introduces systematic errors in calibration algorithms, due to the mismatch between the reference blood glucose concentrations and the acquired tissue spectra that predominantly probe ISF glucose. Here, we propose a novel spectroscopic calibration scheme based on dynamic concentration correction (DCC), which is based on a two-compartment mass transfer model of blood and ISF glucose. The proposed formalism allows the transformation of glucose in the concentration domain, ensuring consistency with the acquired spectra in the calibration model. Taking Raman spectroscopy as a specific example, we demonstrate that the prospectively predicted glucose concentrations using DCC-based calibration model closely match the measured glucose concentrations, while those generated with the conventional calibration methods show significantly larger deviations from the measured values. In particular, the resulting improvement in blood glucose estimates improves the spectroscopic ability to correctly determine hypoglycemia and even predict impending hypoglycemia based on the rate of change in glucose concentration. Additionally, we provide an analytical formula for a previously unidentified source of limiting uncertainty arising from a lack of knowledge of glucose kinetics in the prediction samples. A clinical study with human volunteers undergoing glucose tolerance tests indicate that this lag uncertainty, which is comparable in magnitude to the uncertainty arising from noise and non-orthogonality in the spectral dataset, can be reduced substantially (approximately six-fold) by employing DCC in spectroscopic calibration. Further clinical studies, currently underway, will also provide an understanding about changes in vasculature due to the onset of diabetes and the ability of spectroscopy to diagnose such conditions.

(507) **Vibrational Spectroscopy and Microspectroscopic Imaging:Applications to Skin Pharmacology and Wound Healing**

Richard Mendelsohn, Carol Flach; [1]Rutgers University

Vibrational microscopy and imaging are now poised to address important biomedical and pharmacological issues, including the diagnosis of pathological states. A major advantage of these technologies, not widely utilized to date, is the availability of direct molecular structure information inherent in both IR and Raman spectroscopy. When coupled with spatial and temporal changes, unique characterizations of complex biological processes are acquired. The current presentation will illustrate recent extensions of vibrational spectroscopy and imaging to diverse areas of skin science. Recent applications of these approaches from our laboratory will be discussed as time permits, and will be selected from the following:

1) The sensitivity of IR spectra to the molecular structure and supramolecular organization of lipid molecules in demonstrated in two ways:

i. Quantitative evaluation of conformational disorder in biomembrane models.
ii. Spectra-structure correlations established for purified ceramides are used to evaluate an orthorhombic-to-hexagonal packing transition in human stratum corneum as well as to monitor the kinetics of skin barrier reformation following thermal perturbation.
2) Pro-drugs are used to enhance the delivery of therapeutic agents into skin, where they are undergo enzymatic hydrolysis and converted to the active form of the drug. We have demonstrated the feasibility of tracking and spatially imaging, via confocal Raman microscopy, the prodrug-to-drug interconversion for a derivative of 5-fluorouracil, a well-known anti-cancer agent.
3) Proper wound healing entails a complex series of events. Preliminary vibrational microscopic imaging measurements provide evidence for the temporal expression and spatial location of a variety of collagen and keratin species that appear during the healing process. In addition, we observe the spatial and temporal disposition of conformationally both ordered and disordered lipid populations. Our goal is to correlate the phenotypes we observe with the particular sequence of activated genes that characterize the healing.

(508) **Detection of Viral Infection by Spectral Cytopathology**

Max Diem; [1]Northeastern University

First-line diagnoses for disease are presently carried out using methodology developed 60 years ago, using microscopic visual inspection, of individual stained cells (cytopathology). Visual diagnostics is subjective, extremely difficult and fraud with unacceptably low levels of accuracy. The advent of microspectroscopy has opened new avenues for objective, machine-based diagnostic methods. The combination of microspectroscopy with suitable methods of multivariate data analysis will henceforth be referred to as spectral cytopathology (SCP).. SCP demonstrated that disease can be recognized spectrally before it manifests itself in cell morphological changes. Furthermore, a progression of spectral changes from normal, pre-cancerous and cancerous cells was established that may be useful in diagnostic and prognostic applications. Viral infections were found to affect spectral patterns of cells as well. Thus, SCP can be used for screening applications in cytology, where between 50 and 100 million of tests are performed annually in the US alone.

(509) **Applications of ATR-FTIR Spectroscopic Imaging to Biomedical Samples**

Sergei Kazarian[1], Andrew Chan[1]; [1]Imperial College London

The FTIR imaging enables one to make spatially resolved chemical snapshots of microscopic objects with high spatial resolution and this could be fully exploited in the study of biological objects such as cells and tissues. Micro ATR-FTIR imaging allows one to obtain chemical images of cross-section of hair or a histology cancer tissue section, to detect presence of small clusters of cholesterol esters within atherosclerotic lesions, to analyse live cancer cells in the natural aqueous environment by ATR-FTIR without recourse to a synchrotron source. Macro ATR-FTIR imaging offers complementary approach for chemical imaging of biomedical samples with greater fields of view and for dynamic studies of such samples in contact with aqueous solutions as a function of time. Recent developments in applications of these two versatile FTIR spectroscopic imaging approaches to biomedical samples will be reviewed.

(510) **Turning ATR Imaging Upside Down: A New Microscope Specifically Designed for Pathological Investigations**

Andre Sommer[1], Craig Damin[1]; [1]Miami University

The majority of commercial microscopes possessing ATR capabilities have the hemispherical internal reflection element (IRE) oriented such that its base is positioned toward the base of the microscope. Due to the fact that most IRE materials are not visibly transmitting, the sample is positioned using visible light and the IRE is rotated or dropped into position and contacted for subsequent analysis. Perfect registration between visible viewing and ATR imaging can be problematic. In order to accommodate both visible imaging and ATR imaging we propose to separate light paths by inverting the IRE. Visible imaging is conducted from above in epi- illumination and ATR imaging is conducted from below. In this case, the microscope functions as a variant of an inverted biological microscope. The benefit is that visible structural details in a thin tissue section can be correlated directly to the molecular information obtained with ATR imaging. A similar approach has been taken by Zhao et al for the chemi-luminescent detection of single cells in a micro-fluidic system [1]. This presentation will highlight the design of the microscope and demonstrate its capabilities specifically for applications in the histopathology of kidney disease. Reference 1 Zhao, S., Li, X., Liu, Y., Anal. Chem. (2009), 81 (10), 3873 – 3878.

(511) **Exploring the Potential of Vibrational Spectroscopy for Determination of Malignant Lymph Nodes**

Nick Stone[1], Jonathan Horsnell[1], Linda Orr[1], Martin Isabelle[1], Jenny Smith[1], Keith McCarthy[1], Jonathan Christie-Brown[1], Neil Shepherd[1], Charlie Chan[1], Hugh Barr[1]; [1]Gloucestershire Hospitals NHS

The use of Raman and FTIR spectroscopies in the detection and classification of malignancies within lymph nodes from a number of areas in the body have been evaluated. These include head and neck nodes covering metastatic and primary malignancies; axillary nodes with metastatic breast cancer; mediastinal lymph nodes with metastatic oesophageal cancer. Studies outlining the exploration of the techniques for automated molecular diagnosis of disease within these nodes will be discussed. Currently histopathology is considered the diagnostic gold standard. A majority opinion from three expert histopathologists has been obtained and spectral diagnostic models developed by correlation with their opinions. Initially spectral mapping studies have been used to compare and distinguish between diseased and healthy nodes; novel multivariate analytical approaches have been developed to aid decision making. Furthermore, the sampling methodologies have been extended to demonstrate the principles of using Raman fibre probes in a theatre environment to provide immediate results on diseased states within nodes local to tumour resection. This would potentially enable the removal of infiltrated nodes in a single procedure and remove the need for repeat procedures.

(512) **Evaluation of FTIR Imaging Spectroscopy-Derived Parameters on ACI Treated Human Repair Cartilage**

Arash Hanifi[1], Sally Roberts[2], James Richardson[2], Nancy Pleshko[1]; [1]Temple University; [2]Keele University

Autologous chondrocyte implantation (ACI) is a method utilized to facilitate human cartilage repair. Evaluation of molecular components of cartilage repair tissue enables assessment of whether the regeneration process leads to optimized tissue composition. In recent studies several parameters have been elucidated by Fourier transform infrared imaging spectroscopy (FT-IRIS) that enable evaluation of molecular and compositional changes in cartilage with progressively severe OA, and in repair cartilage. The aim of this study is to evaluate spectroscopic parameters of post-ACI human repair cartilage and to assess whether there is a correlation among FT-IRIS derived properties of human repair cartilage and clinical outcome. Osteochondral biopsies were obtained from knees of ACI treated patients at 1, 2 and 3 years post-surgery (N=16) and frozen until use. Ten micron sections were crysectioned onto low-e slides and infrared images

collected on a Perkin Elmer Spotlight 400 Imaging Spectrometer at 8 cm-1 resolution in the range of 4000 to 800 cm-1 using 2 co-added scans and a pixel resolution of 25 microns. Quantitative analysis of integrated peak areas and peak height ratios were performed using ISys 5.0 software (Malvern, UK). The primary component of cartilage, collagen and proteoglycan (PG) content, were monitored by the integrated area of the 1660, and 1050 cm-1 absorbances, respectively. Collagen helical integrity was monitored by the ratio of the integrated area of 1338cm-1 collagen sidechain absorbance ratioed to the amide II absorbance, and the collagen maturity by the ratio of the 1660/1690 cm-1 baselined peak height. Polarization experiments were performed to determine the distribution of the collagen fibril orientation by assessment of the ration of the collagen amide I/amide II absorbance. Clinical outcome of the patients was assessed by the Lysholm score. Results showed that in the majority of biopsies, the collagen orientation was primarily random, and therefore dissimilar to normal articular cartilage. Improvement in Lysholm score was however correlated to the collagen helical integrity (1st year: R2=76%, 2nd year: R2=70% and 3rd year: R2=51%), but to no other parameters at any timepoint. These data indicate that infrared spectral parameters could be useful in assessment of therapeutics for cartilage repair.

(513) **2D Correlation Spectra Based on Higher Order Moments for Correlation Analysis with Constituent Concentrations**

Jun Uozumi[1]; [1]Hokkai-Gakuen University, Faculty of Engineering

Generalized 2D correlation spectroscopy is a powerful tool for analyzing correlation properties between same or different spectra at different wavelengths. The 2D correlation spectrum of spectra A and B is defined by an average of the product of DA=A-MA and DB=B-MB with respect to an external variable t that is controlled by a certain manner, where MX stands for the mean of X. In many cases in NIR spectroscopy, 2D correlation peaks are required that reflect correlations exclusively with a certain constituent for which a calibration is to be established. Since it is not easy to control the constituent concentration as the external variable, we consider, as an alternative approach, correlations defined by higher-order moments involving DA, DB and Dp, where p is the concentration of a constituent of interest, the average being taken over many samples having different concentrations. As a reference, the fundamental correlation R between DA and DB is also considered. The first idea would be the third order correlation R3 involving these three factors, since this function seems to have high values where the three variables vary all in phase. The next candidate is the fourth order correlation R4 involving the factor of (Dp)2. These three types of correlations were examined for near infrared spectra of rice flour for which the concentrations of moisture, protein and amylose are known. The 2D correlations were actually evaluated in their normalized forms by adequate individual factors. It was shown that R3 does not work, as is seen from the fact that any odd order moment of zero-mean Gaussian variate vanishes. Meanwhile, R4 was successful, giving auto- and cross-correlation peaks in some wavelength bands that can be assigned to the constituent examined. This approach was applied to two types of extended versions of derivative spectra, fractional derivative (FD) and fractional absolute derivative (FAD) spectra. The FD extends the derivative order to any positive real number, while FAD suppresses peak shifts associated with FD. The 2D correlation spectra exhibit interrelation of correlation peaks between the same or different spectra of the original, FD, FAD, and those of different derivative orders.

(514) **Use of Infrared Correlation Spectroscopy to Characterize Polymer Materials**

Georgia Arbuckle-Keil[1], Frank Weston[2], Isao Noda[3]; [1]Rutgers University; [2]Varian, Inc; [3]The Procter & Gamble Company

Dynamic infra-red (IR) spectroscopy provides unique insights into the rheo-optical properties of polymers. For example, phenyl and phenylene modes in poly(2-phenoxy p-phenylene vinylene) (PO-PPV) were distinguished by the application of correlation spectroscopy to the dynamic IR spectral response of the precursor and conjugated PO-PPV material. Results from this poly p-phenylene vinylene (PPV) derivative as well as others will be presented. The biodegradable hydroxybutyrate copolymer, Nodax(TM) is of interest as a transparent, biodegradable packaging material. The bulk mechanical properties of these polymers will be compared with the rheo-optical results obtained by dynamic infra-red linear dichroism (DIRLD).

(515) **A Depth Profiling and Heterogeneity Investigation of Polymers, Including Multi-Laminate and Conductive**

Frank Weston[1], Georgia Arbuckle[2], Isao Noda[3]; [1]Varian, Inc.; [2]Rutgers University; [3]Procter & Gamble

FT-IR Imaging with a focal plane array detector can be used to provide the ultimate surface analysis with high spatial resolution & speed for a variety of applications when determining chemical heterogeneity. The imaging technique (whether micro-ATR, macro-ATR, transmission, or reflection) can be used as both a surface analysis technique and a depth profiling technique with sample modification or preparation. Coupling the surface analysis capability of FPA Imaging with the depth profiling ability of Photoacoustic Spectroscopy (PAS) can be a powerful analytical tool for the investigation of many types of samples, including polymeric materials. Digital Signal Processing (DSP) with a step-scan FT-IR spectrometer continue to maximize the efficiency of the PAS technique by eliminating the need for any additional hardware and demodulating the PAS signals from the fundamental phase modulation frequency up to and including the 9th harmonic while maintaining a linear penetration depth into the sample. The analytical techniques and theory will be presented by a case study approach including a multilayer laminate; a conductive polymer; and a biodegradable polymer, P&G's Nodax®. Asynchronous correlation contour data will be presented to compare phase rotation and phase spectra as related to depth profiling.

(516) **Multiple-Perturbation Two-Dimensional Correlation Analysis of Cellulose by Attenuated Total Reflectance Infrared (ATR IR) Spectroscopy**

Yukihiro Ozaki[1], Hideyuki Shinzawa[2]; [1]Kwansei Gakuin University; [2]Advanced Industrial Science and Technolo

Multiple-perturbation two-dimensional (2D) correlation analysis for multi-dimensional perturbation for two independent perturbation system is described. Sets of time-dependent attenuated total reflectance infrared (ATR IR) spectra of water and cellulose mixtures were collected during the evaporation of water from finely ground cellulose. The system exhibits complex behaviors in response to two independent perturbations, i.e., evaporation time and grinding time. Multiple-perturbation 2D analysis reveals a specific difference in the rate of evaporation of water molecules when accompanied with crystallinity change of cellulose. It identifies subtle differences in volatility of water, which is related to the crystalline structure of cellulose.

(517) **Recent Trends in Multiple-Perturbation 2D Correlation**

Hideyuki Shinzawa[1], Yizhuang Xu[2], Yuqing Wu[3], Isao Noda[4]; [1]Advanced Industrial Science and Technology (AIST); [2]Peking University; [3]Jilin University; [4]The Procter & Gamble Company

Recent trends in multiple-perturbation two-dimensional (2D) correlation spectroscopy are reviewed in this presentation. Multiple-perturbation 2D correlation analysis is to prove complex responses of system induced by combination of perturbations. For example, in multiple-perturbation 2D correlation scheme, sample system is perturbed by more than one type of perturbations, e.g., time and temperature in combination, to induce mixed responses to the multiple stimuli. An intriguing possibility arises when multiple perturbations are applied. Stimulation of a system with multiple perturbations may lead to an interesting opportunity to prove complex responses of the system induced by the combination of perturbations, as well as possible synergistic interaction between them. In addition, even the system responses themselves may also interact with each other, resulting in additional characteristic behavior of the system, including nonlinear responses. In this presentation, general scheme of multiple-perturbation 2D correlation will be presented first. In addition, some of interesting family of multiple-perturbation 2D techniques, orthogonal sample design (OSD) and round trip scan, will be reported as pertinent examples of applications.

(518) **2D Correlation Spectroscopy As A Tool for Spectral Interpretation**

Franklin Barton[1], James de Haseth[1]; [1]Light Light Solutions, LLC

Near Infrared Spectroscopy has always been hampered by a lack of "classical" spectral interpretation. In the mid infrared the fundamental vibrations of organic molecules have been well studied and numerous texts have been written for the student of spectroscopy. However, the near infrared does not contain any of the fundamental vibrations, only combination bands and overtones of the fundamentals. Twenty years ago two-dimensional (2D) spectroscopy techniques were developed that allowed the comparison of different spectral regions and correlated the responses. The technique was needed to help with the interpretation of NIR spectra and chemometric models. Classical spectroscopists were skeptical of wavelengths chosen which did not appear at the top of a band and the inclusion of wavelengths which did not appear to relate to the analyte. The technique has been used to describe the process of digestion , the de-ligninification of cell walls by white rot fungi and the effect on carbohydrates and the difference between hard red winter and hard red spring wheat which removed instrument differences. Calibration transfer and instrument differences and similarity have been a topics of great interest throughout the past forty years of the use of Near Infrared Spectrometry (NIRS) to determine the properties of agricultural and food products. This paper describes the general 2DCOS work used for interpretation and other studies which show the differences and similarities of instruments and a graphical representation of calibration transfer potential. In all 2D correlation spectroscopy is an aid to interpretation of any region of the spectrum.

(519) **Real-Time *in situ* FTIR Analytics as a PAT Tool for Batch and Continuous Processes**

Wes Walker[1]; [1]METTLER TOLEDO

Real-Time *In situ* FTIR instruments are powerful tools used to bring principles of process analytical technology (PAT) and quality by design (QbD) to bear on the development and production of drug substances in the pharmaceutical industry. Attenuated total reflectance spectroscopy is particularly useful for this work, as it can be used for reaction volumes ranging from less than a milliliter to thousands of liters. In this presentation, strategies for implementing *in situ* FTIR and several industrial case studies will be discussed.

(520) **From Batch to Continuous – The GSK Continuous Hydrogenation Workflow**

Robert Yule[1], Gary Kelly[1]; [1]GSK

Catalytic hydrogenations are often used in commercial processes to reduce a wide variety of functional groups. These reactions are an extremely useful tool, but often only batch mode operations are evaluated. Sometimes lack of heat transfer capacity, long residence times, safety concerns and economies at scale can derail an otherwise promising batch hydrogenation process. Continuous hydrogenation processes may avoid these pitfalls. This presentation will illustrate the workflow GSK uses to evaluate hydrogenations for continuous processing.

(521) **NIR Monitoring of Hot Melt Extrusion Processes**

Brandye Smith-Goettler[1], Robert Meyer[1], Neil MacPhail[1], Colleen Gendron[1]; [1]Merck Sharp and Dohme Corp.

Process Analytical Technology (PAT) and Hot Melt Extrusion (HME), commonplace in the food and polymer industries, are becoming increasingly deployed in the pharmaceutical industry. Hot melt extrusion maximizes efficiency by combining several processing steps and enabling continuous manufacturing. This manufacturing platform is ideal for poorly soluble drug substances and utilizes a pharmaceutical grade polymer and high temperatures to convert the drug substance from a crystalline to an amorphous state. PAT, in general, yields numerous manufacturing advantages but two specific to HME are as noted. PAT, as applied herein, eliminates the need for sample preparation. This is advantageous for obvious reasons, but more specifically the introduction of polymer to the sample matrix can complicate active pharmaceutical ingredient (API) extraction for drug product quality assessment. Process monitoring better enables continuous manufacturing as any quality attribute deviations are identified in real-time. For example, suspect hot melt extrudate can be diverted to waste until the real-time quality attribute is within specification as opposed to having to discard the entire batch. Herein details the application of in-line, transmission mode, Fourier-Transform Near-Infrared spectroscopy (FT-NIR) to the hot melt extrusion manufacturing platform for two Merck drug products. In both circumstances, NIR and Partial Least Squares (PLS) models were developed for real-time drug loading (%) predictions. These predictions were used for fault detection and real-time troubleshooting of the respective processes. The NIR/PLS output were used for release of the intermediate drug product.

(522) **Determining the Efficacy of Rapid Spectroscopic Techniques for *in-situ* Characterisation of Polymorph Contamination**

Michelle C Hennigan[1], Yun Hu[1], Alan G Ryder[1]; [1]School of Chemistry, NUI Galway

Active pharmaceutical ingredients (APIs) are frequently delivered to the patient in their solid form by inclusion in tablets, capsules and spray dried powders. APIs can exist in various solid state forms including polymorphs, solvates, and hydrates. In the case of polymorphs, each form can possess its own unique physical properties and chemical stability which in turn can directly affect performance characteristics such as bioavailability and solubility of the API. Thus the accurate characterisation and quantification of the precise solid state form(s) present is of vital concern for safety and regulatory affairs. Here, we demonstrate the efficacy of several rapid characterisation methods (Raman spectroscopy and IR spectroscopy) in combination with Chemometric methods for the analysis of polymorph contamination in model tablets. We have developed a model system which comprises of a model API, 5-methyl-2-[(2-nitrophenyl) amino] -3-thiophene carbonitrile (ROY),

with several excipients (e.g. microcrystalline cellulose and magnesium stearate). ROY exhibits extensive polymorphism with ten known polymorphs. Tablets containing 2.5 to 30 % by weight (increments of 2.5%) of the stable yellow polymorph of ROY, were generated with four different tablet thicknesses; 750, 1000, 1250 and 1500 microns. From this data, calibration models were developed to which different pre-processing and multivariate methods were applied for spectral processing and compared to find the best chemometric model. Low levels of polymorphic impurities were incorporated in subsequent tablets and the efficacies of the analytical techniques at detecting these low levels of contamination were compared. Acknowledgements Funding received from the Solid State Pharmaceutical Cluster, a Science Foundation Ireland funded Strategic Research Cluster, is gratefully acknowledged.

(523) **Controlling the Crystalline State of Electrospun Nylon 6 by Varying the Solvent Evaporation Kinetics**

Carl Giller[1], Bruce Chase[1], John Rabolt[1], Christopher Snively[2]; [1]University of Delaware, Dept. Mat. Sci./Eng.; [2]SCHOTT North America, Inc.

The effect of solvent evaporation on the crystalline state of electrospun Nylon 6 fibers was examined by electrospinning into a closed chamber filled with varying concentrations of solvent vapor. It was found that the thermodynamically thermodynamically stable alpha form became increasingly present in Nylon 6 fibers electrospun out of both 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP) and formic acid as the vapor phase solvent concentration increased. It is believed that the formation of the metastable gamma form is due to the fast solvent evaporation kinetics associated with the electrospinning process. By varying the rate of solvent evaporation during electrospinning, we were able to control the resulting crystal structure of the electrospun Nylon 6, as evidenced by XRD and Raman and FTIR spectroscopies.

(524) **Accelerating Drug Development Using Flow/Continuous Processes**

Sandeep Kedia, Thomas Lovelace; [1]Chemical Development, GlaxoSmithKline

Chemical Development routinely receives synthetic methods and processes for active pharmaceitucal ingredients from discovery which are not conducive to scale up because of limitations in conventional batch style plants. In some cases, the use of flow chemistry (continuous or batch) can facilitate the production of larger amounts of these materials without redesigning the synthesis thereby reducing the resource needed to progress projects through the pipeline. Several examples of applying flow chemistry in this fashion will be discussed.

(525) **Engineering SERS Tags for Cytometry**

John Nolan[1]; [1]La Jolla Bioengineering Institute

Multiparameter and multiplexed measurements are key tools in the study of cells and cell systems. Fluorescence is the most widely used optical approach to single cell measurements, and cytometry instruments employ multiple excitation wavelengths and detectors to simultaneously measure many fluorescence signals from individual cells. Flow cytometry has pushed this approach the farthest, with commercial instruments enabling the high speed measurement of nearly 20 fluorescence signals per cell. It will be difficult to significantly increase this number, however, because the broad emission spectra of available fluorescent dyes fill the visible wavelength range accessible by current instruments. Raman scattering, and surface enhanced Raman scattering in particular, offer a potential solution to this limitation because of the higher information density that can be encoded into the narrow spectral features of Raman scattering. We have developed the first Raman flow cytometers, which are capable of measuring SERS spectra from individual cells at high analysis rates. A critical component of this approach is SERS tags that are bright, uniform, and reproducibly prepared. We have evaluated these properties for a number of SERS tag formulations, including aggregated colloids, nanorods, and nanoshells. Single nanoparticle analysis is an important tool in this effort, and we have developed a nanoparticle flow cytometry approach that enables the high speed analysis of single particle Rayleigh and Raman scattering. We have found that a composition of layered shells composed of gold, Raman tag, and silver over a silica core provides a reproducible path to bright and uniform SERS tags.

(526) **Plasmonics Nanoprobes and Nanochips for Environmental Sensing and Medical Diagnostics**

Tuan Vo-Dinh; [1]Duke University

This presentation provides an overview of the development and applications of the plasmonics and surface-enhanced Raman scattering (SERS) nanoprobes and nanostructures for environmental sensing and medical diagnostics. Plasmonics refers to the research area of enhanced electromagnetic properties of metallic nanostructures that produce ultrasensitive and selective detection technologies. We describe the development of unique metallic nanoprobe and nanochip structures for SERS sensing. The development of large-area nanochip platforms having controlled nanostructures exhibiting plasmonics-active properties is critical for a wide variety of applications ranging from chemical detection to biosensing. Our methodology employs a hybrid approach integrating deep UV lithography and controlled epitaxial growth of silicon germanium on silicon nanostructures to form diamond-shaped nanowire structures. This unique methodology provides the scaling process bridging the gap between nanoscale requirements of plasmonics and macroscale regimes of practical nanochip-based sensors. Applications in environmental sensing and medical diagnostics are discussed to illustrate the usefulness and potential of plasmonics nanoprobe and nanochip technology.

(527) **Integrated SERS Sensors Based on Modified Particles Stabilised in Polymer Gels**

Steven Bell, David Jones[1], Colin McCoy[1], Maighread McCourt[1], Alan Stewart[1]; [1]Queen's University of Belfast

There is a significant difference between the challenges for quantitative SERS analysis carried out within specialist laboratories and methods which will be suitable for a general analytical laboratory or in the field. For the former, emphasis is often placed on developing materials and methods which push the limits of detection lower, typically within samples which are pure solutions of the target analyte. These methods can be extremely successful but transferring them to non-specialist users and to real world samples, which may be contaminated by a huge range of interfering constituents, is extremely challenging. At the same time, the introduction of affordable hand-held instruments has meant that there is real potential for carrying out SERS measurements outside the laboratory, for environmental monitoring and homeland security. In effect, SERS measurements can become a viable alternative to existing trace detection methods in much the same way that conventional Raman measurements have already begun to supersede existing methods for routine QA, QC and process monitoring. We have now started to address the remaining barriers to widespread adoption of quantitative SERS. These are associated both with increasing the stability, affordability and robustness of the enhancing media and improving detection selectivity. We have concentrated on nanoparticles because their ease of preparation gives them a huge cost advantage over solid substrates prepared by "top down" techniques. Although methods for preparing many different particles sizes and morphologies are now known we have been careful to compare their effectiveness under standardised

conditions and can report data for variously sized and shaped Au and Ag particles. These particles have been stabilised within polymer gels which protect them during storage but release them when required for analysis. Finally, the surfaces of the particles have been modified to increase binding by the target analytes while rejecting interfering materials using surface modifiers found through a novel high throughput screening approach. This overall approach has allowed us to work toward sensors which have the levels of selectivity and sensitivity required for forensic/security applications but are packaged as disposable single use "peel, test and discard" pads.

(528) **Sensitive SERRS Detection of DNA by Novel Signal Amplification Based Approach**

Jennifer A. Dougan[1], Kristy McKeating[1], Duncan Graham[1], Karen Faulds[1]; [1]University of Strathclyde

Surface enhanced resonance Raman scattering (SERRS) is an analytical technique which offers advantages over competitive techniques in terms of improved sensitivity and multiplexing. Nevertheless, fluorescence spectroscopy dominates the detection technologies employed with different bio-diagnostic assay formats. Highly selective and specific detection of oligonucleotide sequences of interest is of pivotal importance in the detection of genetic mutations, infectious disease management and gaining a comprehensive understanding of the results of the human genome project. Here we demonstrate a novel assay format which relies on the generation of a measureable signal following sequence specific hybridisation achieving high sensitivity and specificity through signal, rather than target amplification techniques i.e. PCR. The assay has been developed by exploiting the digestion criteria of the processive enzyme λ-exonuclease. Judicious development of probe strands that contain both the required 5'-phosphate for enzyme action and a suitable label can, upon hybridisation, generate a detection signal. λ-Exonuclease acts on the 5'-phosphate probe strand digesting each nucleotide and ultimately liberating the label for analysis. Since the target is then returned to a single stranded state it is free to hybridise with another probe strand rendering a double stranded complex for further digestion. By this method, more SERRS label is liberated and the detection signal is amplified. A number of detection strategies have been investigated for this assay system – nanoparticle-based plasmonics, fluorescence and SERRS. In addition, sequences of social interest have been targeted such as Chlamydia trachomatis and methicillin-resistant staphylococcus aureus – synthetic, PCR-product and genomic.

(529) **Continuing Development of Standoff Chemical Sensing and Concealed-Threat Detection with Millimeter-Wave and THz Radiation**

Michael Gord[1], Anita Taulbee-Combs[2], David Hufnagle[2], Gilbert Pacey[2], Carla Benton[3], Douglas Petkie[3], Satya Ganti[3], Jason Deibel[3], Michael Moulton[4], James Gord[4]; [1]Dayton Christian High School; [2]Miami University; [3]Wright State University; [4]Air Force Research Laboratory

Many current and future applications require new techniques for detecting and identifying hazardous chemicals and other potential threats. Such applications include the Global War on Terror, homeland and portal security, medicine, industrial process monitoring, and atmospheric and environmental studies, including exploration of greenhouse gases and global warming. CBRNE (chemical, biological, radiological, nuclear, and explosive) threats are of particular interest. Millimeter-wave and terahertz (THz) radiation is ideally suited for many such applications. Eye-safe radiation in this spectral region has been used with techniques that include absorption spectroscopy, interferometry, and frequency-modulated continuous-wave (FM CW) radar to identify various classes of gaseous and solid chemicals and to detect concealed threats. Standoff measurements at safe distances have been demonstrated using radiation beams and remotely operated vehicles (ROVs) as well.

(530) **Using Terahertz Pulses in the Process World**

Philip Taday[1], Mike Evans[1], Axel Zeitler[3], Yaochun Shen[2], Dipankar Dey[4]; [1]TeraView; [2]Liverpool; [3]Cambridge; [4]Oystar-Manesty

Over the years we have reported first that it is possible to measure the coating thickness on solid dosage forms using terahertz pulsed methods and then more recently we have shown that it is possible to correlate this to dissolution of products. These measurements where undertaken in a well controlled environment with a robotic system used to acquire as much data from a tablet as possible. This talk will present will discuss the application of a terahertz probe placed inside a coating drum to monitor the build-up of coating on a solid dosage form in real-time. Data is acquired at a rate-of-greater-than 100 tablets per minute over the coating run of 5-6 hours.

(531) **Terahertz Computed Tomography Measurements**

Jeffrey White[1], David Zimdars[1], Greg Fichter[1], Chris Megdanoff[1], John Duquette[1], Irl Duling[1]; [1]Picometrix LLC

Computed Tomography (CT) Terahertz (THz) was first demonstrated in 2004. The ability of Terahertz radiation to transmit through a wide range of solid materials makes the technology a promising candidate for further CT applications. CT THz appears ideally suited for very low index materials (e.g., foams) and other materials not suited for X-Ray inspection. With the increases in Terahertz waveform collection rates (e.g., 1000 Hz), the use of THz systems to collect relatively rapid CT data and subsequent image generation has been demonstrated. Most measurements have been made with a confocal transmission imaging. Sample materials studied include foams and thermal protection materials (tiles, Kevlar cloth) for NASA applications. As opposed to X-Rays, or many other radiation sources for CT, THz energy is completely safe and does not require any warnings or special exposure concerns for the operator or samples. Benign materials, such a water or IPA, can be used as contrast agents to find thin cracks or other defects.

(532) **Distinguished Service: Becoming an Oxymoron?**

Alexander Scheeline[1]; [1]University of Illinois at CU

Universities have as their mission teaching, research, service, and (of late) economic development. Fiscal and other societal pressures on science and education place the service component of that mission under considerable stress. While all mission components can be evaluated subjectively, areas other than service have an objective measure with which they may be assayed: cash flow. The very notion of service implies that benefit flows from the provider to the recipient. Because thermodynamics requires that there be dissipation to generate benefit, financially-stressed institutions frequently find it easier to reward success in obtaining external resources (grants) than in providing community service. This talk will explore the message that was heard by the Baby Boom generation when selecting science as a career, and will give examples of how that message differed from reality then and now. The consequences of changes in institutional priorities, for science and society, during the last 40 years, will be discussed. Most importantly, the centrality of serendipity in all aspects of the academic enterprise and life as a whole will be noted. Happily, stubborn adherence to a standard of balanced participation in the many missions of universities results in significant benefits to society, although at the expense of grant cash flow and research group size. As the author is interested in oxidative stress, does that mean his service work has made him an oxy-moron?-

ABSTRACTS

(533) **Advanced Sensor Technologies for Enhanced Mid-Infrared Diagnostics**

Boris Mizaikoff[1]; [1]University of Ulm

Optical chemical sensors operating in the spectral range of 3-20 µm (mid-infrared) are gaining importance in a diversity of application areas ranging from process analysis, environmental monitoring, and security/surveillance applications, to the biomedical and diagnostic field. The perspective of robust optical sensors providing inherent molecular specificity renders mid-infrared technology among the most promising sensor platforms for addressing complex samples in demanding measurement environments. Given the current advancements in light source technology (e.g., room-temperature operated broadly tunable quantum cascade lasers) and detectors, it appears of paramount importance developing appropriate mid-infrared transducers and waveguides to the same level of maturity for facilitating continuous and highly sensitive direct molecular analysis in the gas phase and in the liquid phase. Consequently, the advent of appropriate waveguide technology including mid-infrared transparent optical fibers, planar semiconductor waveguides, and novel hollow waveguide structures in combination with either protective or molecularly responsive surface coatings (e.g., diamond-like carbon, sol-gels, polymers, etc.) promises advanced infrared diagnostics applicable at extreme conditions (e.g., deep sea), for label-free biomedical analysis (e.g., *in-vivo* surgical monitoring and breath analysis), and for next-generation multifunctional analytical platforms (e.g., in combination with atomic force microscopy) enhancing cell physiological studies [1-12]. Selected application examples and novel technological developments will highlight recent advances in miniaturized mid-infrared sensor technologies for next-generation diagnostic sensing platforms.

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(534) **Transforming SERS into a Reliable, Ultrasensitive Analytical Tool**

Martin Moskovits; [1]University of California Santa Barbara

Surface Enhanced Raman Spectroscopy is a technique discovered some 35 years ago. It's unusually high sensitivity – good quality Raman spectra involving a single molecule are obtainable under favorable circumstances – is acknowledged without question. This together with its ability to report good quality vibrational spectra even in aqueous media should make it a valuable analytical tool for low concentration applications such as explosive and environmental hazard detection. However, the sentiment that SERS reliable, reproducible, prescriptively-manufacturable substrates were hard to come by, and the myth that the mechanism of SERS is poorly understood so that an engineered approach to designing SERS substrate may not be at hand, have impeded the development of SERS as a tool for chemical analysis. The presentation will summarize both the facts and the myths about SERS and discuss the engineering principle for making reliable, sensitive SERS substrates and illustrate their application.

(535) **Development and Application of an UPLC/UV Method for Determination of Resveratrol in Rat Urine at Parts-Per-Billion Levels**

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A liquid chromatographic method with UV detection (UPLC/UV) was developed and applied for the determination of resveratrol in Wistar Han rat urine at ng/mL levels for use in support of toxicology studies. An aliquot (~100 µL) of rat urine was transferred into separate Mini-UniPrep syringeless filter devices and 10 µL of internal standard solution, 10 µg 2',4'-dihydroxy¬propiophenone/mL methanol, was added to each sample or standard and vortexed completely. A 100 µL aliquot of methanol was then added to each sample. The filter piston was inserted and the contents further vortexed. The filter piston was pressed through the sample and the resulting filtrate was analyzed by UPLC using UV detection at 305 nm. A gradient elution at a flow rate of 0.28 mL/min was used with a Waters Acquity UPLC HSS T3 (2.1 x 100 mm, 1.8 µm) column with Waters Vanguard T3 guard column at ambient temperature. The mobile phases consisted of 5 mM ammonium acetate in water with 5% 2-propanol, and methanol with 5% 2-propanol. The method was successfully verified over a range of 20 to 5050 ng/mL with an additional dilution step extending the range up to 226000 ng/mL. The linearity of matrix standard calibrations were confirmed by correlation coefficients > 0.999 using $1/x^2$ weighting. The method has been successfully used to determine the concentration of resveratrol in Wistar Han rat urine samples from animals dosed at 78 mg/kg and 1250 mg/kg.

(536) **Development and Validation of a New LC/MS/MS Bio-Analytical Method for the Determination of Curcumin in Human Plasma Samples**

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The objective of current investigation is to develop a method that can be suitably applied for analysis of curcumin in human plasma. There is no bio-analytical method reported in literature for curcumin yet. Hence it is of prime importance to develop a method for estimating curcumin in biological matrix with highest level of sensitivity for better estimate of pharmacokinetic parameters like AUC(0→α) (Area under curve from zero to infinity) and kel (elimination rate constant) of drug in patients. A fast, sensitive and specific LC¨CMS/MS bio-analytical method has been developed and validated. Betamethasone was used as the internal standard (IS). We achieved not only high sensitivity of 10.08 ng /mL as the lower limit of quantification (LLOQ) but also showed dynamic linearity ranging from 10-500 ng/ml (r2=0.9991) with acceptable precision, accuracy and recovery (as per limits set by USFDA). Our novel method involves minimum extraction steps achieving an excellent recovery for the analyte as well as Internal Standard (more than 90% with coefficient of variation less than 10 %). Sufficient sample stability was shown, to allow for the completion of sample analysis in clinical trials. This validated rugged method led to analysis of large number of plasma samples in a very short span of time (due to lower retention time of 4-5 minutes) obtained for pharmacokinetic and bioavailability or bioequivalence studies.

(537) **Microbeam Radiation Therapy of Cells on Microchips**

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Microbeam radiation therapy (MRT) is a strategy to target tumors by using discrete narrow parallel beams of high-dose radiation. Compared to current broad beam radiation therapy, MRT shows selectivity for tumors over normal tissue and greater survival in animal models. Although MRT reflects a promising preclinical alternative to current therapeutic strategies, little is known about the basis for tumor selectivity. To investigate the mechanism of MRT, an ultrahigh dose rate electron microbeam system based on carbon nanotube (CNT) field emission was modified for MRT applications. To study the effects of MRT on the small population of cells that are directly exposed to the microbeams and how these cells influence neighboring cells, a microfabricated cell array platform, termed a micropallet array, was engineered using 1002F photoresist to analyze, sort and collect viable cells from a mixed population while the cells remain adherent to their growth surface. Cells cultured on micropallet arrays are then inverted onto the CNT irradiator for MRT experiments. A three micron-thick barrier of Mylar is required to both minimize beam attenuation and protect the CNT irradiator from the culture medium. The micropallet array was fabricated to position the cells in near contiguity with the Mylar film by placing micron-sized spacers on the array at varying intervals to prevent compression of the cells onto the Mylar. Spacer heights varied from 5 to 30 microns by utilizing a two-step fabrication process to place both the pallets and spacers on the same array. Spacer height and shape were optimized and characterized using confocal microscopy and surface profiling methods. To test if the chemical/optical properties of 1002F photoresist changed in response to high dose irradiation, micropallet arrays were also subjected to high dose irradiation using a standard cesium irradiator. By measuring absorbance, fluorescence and x-ray photoelectron spectroscopy (XPS) post-irradiation, we confirmed that high dose irradiation does not change the properties of the array material. These advances in microplatform design will allow us to investigate the effects of microbeam radiation on tumor and normal cells to understand the mechanism of MRT.

(538) ***In vitro* Monitoring Vitamin C in Human Urine by Flow-Injection Chemiluminescence with Luminol-Dissolved Oxygen System**

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Vitamin C, also known as ascorbic acid, plays an important part in human body. Vitamin C participates in the synthesis of neurotransmitters, steroid hormones, carnitine, and collagen in connective tissue immune response. It also plays an important part in wound healing, therapeutic purposes and converting cholesterol into bile acids. Moreover, being a biological reducing agent, Vitamin C is used to the prevention of degenerative diseases, such as cataracts, some types of cancers, cardiovascular diseases and viral myocarditis. Several methods have been reported for the determination of Vitamin C such as high-performance liquid chromatography, electrochemistry, titrimetry, spectrophotometry. There is no report of the *in vitro* monitoring of Vitamin C in human urine after taking of tomatoes and Vitamin C tablets by luminol and dissolved oxygen chemiluminescence system. A sensitive chemiluminescence method, based on the inhibitory effect of vitamin C on the chemiluminescence reaction between luminol and dissolved oxygen in a flow-injection system, was proposed for the determination of vitamin C. The decrement of chemiluminescence intensity was linear with the logarithm of vitamin C concentration over the range from 0.8 to 100.0 ng mL-1, with the detection limit of 0.3 ng mL-1 (3ó) and relative standard deviation of lower than 3.0% (n = 5). The proposed procedure was successfully applied to the determination of vitamin C in human urine after taking of tomatoes and tablets respectively. The total elimination constant k and half-life time t1/2 of VC after taking 420 g tomatoes were 0.2082 and 3.33 h, and after administering VC tablets orally were 0.3044 and 2.28 h respectively.

(539) **Development of a Quartz Crystal Microbalance (QCM) Immuno Sensor for Sesame Protein Detection in Complex Food Matrices**

Fatima Tazeen Husain[1], Margit Cichna-Markl[1], Romana Schirhagl[1], Franz Ludwig Dickert[1]; [1]University of Vienna

Food allergies continue to pose an increasing health problem. In the past, sesame (Sesamum indicum) allergy was only common in Eastern countries. However, recent studies indicate that allergic reactions to sesame have become more frequent world wide. Numerous papers report the severity of allergic reactions caused by the sesame seed consumption, including life threatening anaphylaxis. The only preventive strategy for an allergic patient is to stringently avoid food containing the allergen. Sensitive and selective analytical measurements have to be carried out to verify if the food products are labelled in accordance with the regulations. The present paper therefore deals with the development of a quartz crystal microbalance (QCM) immunosensor to detect traces of sesame in food. Antibodies were obtained by immunizing a hen with a protein extract of sesame (1 mg/mL) and isolating the antibodies from egg yolk by ammonium sulphate precipitation. The antibodies were immobilised onto a 10 MHz quartz coated with gold paste via its sulphur group and mounted onto a cell. After an incubation time of 30 min, 180 µL of the sample extract were injected and an antigen antibody complex was formed. After a washing step, the change in the signal was observed which corresponded to the concentration of the sesame protein injected. After destroying the antigen-antibody complex with guanidine hydrochloride the sensor could be re-used. The sensor did not show cross reactivity with nuts, legumes and several other seeds that

were tested. The limit of detection (LOD) of the immunosensor was found to be 7 µg of sesame protein per mL corresponding to 35 µg of sesame protein per gram food.

(540) **Tip Enhanced Raman Spectroscopy and Imaging of Lipid Membranes**

Tsoching Chen[1], Ira Levin[1]; [1]National Institutes of Health

The combination of an AFM scanning probe and Raman spectroscopy provides Raman scattering that is not only at several orders of magnitude, but also with sub-diffraction limit spatial resolution. As a promising technique for studying nanostructures, the application of Tip-Enhanced Raman Spectroscopy (TERS) on biological samples remains challenging. Using model lipid membranes consist of binary phosphatidylcholines, we demonstrate the prospect of TERS for illuminating chemical and physical features of biological systems in nanoscale. Strong tip-enhanced Raman scattering is achieved and discriminated from the far-field Raman signal by preparing lipid membranes with the presence of TERS-favorable substrate in the near-field. The approaches to TERS images of high fidelity are addressed by examining factors such as sample preparation, substrate selection, image acquisition time, and noise reduction.

(541) **Infrared Imaging Analysis of Skin Mineralization in the Genetic Disease Pseudoxanthoma Elasticum**

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Pseudoxanthoma elasticum (PXE), caused by the Abcc6 gene mutation, is characterized by pathological mineralization of the connective tissue, cardiovascular system, skin and eyes. PXE is very difficult to diagnose in early stages and has no current treatment. Most importantly, the mechanisms causing the calcification of the elastic structures and collagen fibers are not clear and needs further examination. An animal model, the Abcc6 deficient mouse, has been used for investigations in this disease. An earlier study confirmed the mineralization of vibrissae, heart and kidneys by von Kossa and alizarin red stains. In this study, Fourier transform infrared imaging spectroscopy (FT-IRIS) was utilized to test skin samples from the previous study to investigate mineral formation at different ages. Data were acquired in transflectance mode from thin sections on low-e slides using a Perkin Elmer Spotlight 400 Imaging System (Perkin Elmer, Shelton). Images were collected in the mid-IR range (4000-748cm-1) at a spectral resolution of 8cm-1 and spatial resolution of 6.25 Micrometers. The bands associated with the mineral vibrations (phosphate at 1200 to 900 cm-1) were evident at the connective tissue capsule surrounding the sinuses of vibrissae of all samples. The area under the mineral peak at each pixel was ratioed to the collagen peak area (amide I at 1598-1710 cm-1). Mean values of the mineral/collagen were 0.165±0.005 for 3 months, 0.6 ±0.1 for 6 months and 0.335 ± 0.035 for 24 months old tissues. Comparison to standard mineral phases showed that the mineral in the 6 month old tissues was primarily a mixture of carbonated hydroxyapatite, and brushite. For 3 and 24 month old samples the mineral was primarily carbonated hydroxyapatite and amorphous calcium phosphate. For all ages, the mineral phase is typically more amorphous in the center and more crystalline at the edges of the hair shaft. FT-IRIS data such as these can provide additional information on the mineralization mechanism caused by PXE and might serve an important role in early diagnosis and the development of treatment.

(542) **Infrared Spectroscopy to Quantify Collagen in Infarcted Myocardium after Targeted VEGF Treatment**

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Introduction: Every year, 1.5 million people in the United States suffer from myocardial infarction (MI). This condition causes adverse cardiac remodeling, in which necrotic heart muscle tissue is replaced with fibrous collagen. Collagen prevents efficient pumping of the blood, which can cause congestive heart failure. Previously, we have shown that a low dose of vascular endothelial growth factor (VEGF) targeted via anti-P-selectin immunoliposomes to the infarcted heart improves cardiac function and vascular perfusion. The aim of this study is to assess molecular changes in post-infarction myocardium using Fourier Transform Infrared Imaging Spectroscopy (FT-IRIS) and examine the role of targeted VEGF in preventing collagen deposition, and subsequently, cardiac remodeling after an MI. Methods: An MI was induced in rats, and treated with anti-P-selectin conjugated immunoliposomes containing VEGF (0.12 µg/kg), or left untreated. After four weeks post-infarction, hearts were excised, 9 µm cryosections cryosections were mounted on low-e slides and scanned with a Perkin Elmer Spotlight 400 spectrometer to obtain FT-IRIS data. Sections of the same hearts were stained for collagen using Gmori's Trichrome and Picrosirius Red staining protocols. Analysis: The absorbance band centered at 1338 cm-1 arises from the amino acid side chain vibrations in collagen. Peak integration mapping of this absorbance band at each pixel in the infrared image represents the quantity of collagen at that pixel, and was used to create a map of collagen quantity distribution in the tissue. These data corresponded well with the Gmori's Trichrome Stain of the same tissue which indicates the feasibility of FT-IRIS to separate the collagen from the rest of the myocardium matrix. Discussion: Histochemical staining results show that the length of the collagen scar, as well as overall collagen deposition through the anterior wall decreases in treated animals. FT-IRIS analysis also shows that treated hearts had reduced overall density of the collagenous tissue. FT-IRIS has the advantage of providing more information about the molecular structure of the tissue. Our ongoing detailed investigation of FT-IR images, such as quantification of collagen content and assessment of collagen integrity and crosslinking, will provide a better understanding of the molecular mechanisms of cardiac remodeling after MI.

(543) **Using Contact Printing Method to Modify Surface of Arrayed Microstructures**

Wei Xu[1], Yuli Wang[1], Jonathan Clark[1], Christopher E. Sims[1], Nancy L. Allbritton[1]; [1]University of North Carolina

A novel surface modification method was developed by utilizing contact printing of a sacrificial layer of polyacrylic acid (PAA) to selectively modify the upper surfaces of arrayed microstructures. The method was characterized by printing polystyrene onto SU-8 microstructures to create an improved substrate for a cell-based microarray platform. Cell attachment and proliferation experiments on arrays composed of SU-8 microstructures modified with polystyrene and fibronectin demonstrated improved growth of NIH 3T3 (93% vs. 38%), HeLa (97% vs. 77%), and HT1080 (76% vs. 20%) cells relative to that of the previously used surface modification method using adsorbed fibronectin alone. In addition, use of the PAA sacrificial layer permitted the printing of a layer of other materials onto the arrays in a facile manner, which would otherwise be challenging to microfabricate, such as functionalized polystyrene, carboxylate polystyrene nanospheres, and silica nanospheres. Finally, a high concentration of extracellular matrix materials (ECM), including collagen (5 mg/mL) and gelatin (0.1%), was contact printed onto the array structures using as little as 5uL of the ECM reagent and without the formation of a continuous film

bridging across the microstructures. Murine embryonic stem cells cultured on arrays printed with gelatin-hydrogel remained in an undifferentiated state indicating an adequate surface gelatin layer to maintain these cells over time.

(544) **Development of "Protectides" to Prolong the Lifetime of Peptide Reporters for Intracellular Abl Kinase Activity**

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There is an increased interest in using peptides with consensus site motifs as reporters to assess kinase activity in living cells for cancer research. However, the application of these reporters is often challenged by the short lifetimes of these peptides due to hydrolysis by peptidases within the cell. Since the majority of intracellular peptidases possess catalytic sites buried deep within a cavity, the linearized peptide can access these spaces from the N-terminus and are degraded subsequently. Here, appending a bulky group to the N-terminus of a linear peptide is proposed to overcome this issue. This bulky group might block the access of peptide to sterically hindered peptidase catalytic sites and improve the lifetime of peptide within cells. Previous work demonstrated that small folded motifs based on beta bends were resistant to peptidases *in vitro*. These small bend with typically less than 12 amino acids were termed "protectides" due to their ability to resist hydrolysis by peptidases. Several designs of protectides are presented in this work: two non-crosslinked beta-bend peptides; two crosslinked beta-bend peptides and a FlAsH-tetracysteine beta-bend complex These protectides are linked to the N-terminus of abl kinase substrate peptide via a polyethylene glycol linker. The fluorophore labeled protectide-peptide constructs were characterized in Ba/F3 cell lysate and with purified intracellular peptidase prolyl oligopeptidase . Capillary electrophoresis with laser-induced fluorescence detection was used to quantify peptide breakdown. Preliminary studies demonstrated that the lifetime of control substrate peptides were 10 minutes in a cell lysate, while substrate peptides with an attached protectide were stable for up to 50 minutes. The studies also demonstrated the protectide-peptide constructs remained a substrate for the target kinase. Thus, attachment of a bulky group to the N terminus of a peptide can provide "protection" to that substrate from cytosolic peptidases. A future goal is to use these protectide-peptide constructions as reporters of kinase activity in single intact cells.

(545) **Spectroelectrochemical Characterization of Polymerized Hemoglobins**

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Oxygen carrying proteins such as the hemoglobins from Lumbricus terrestris (earthworm) and Arenicola marina (marine worm) serve as model systems for developing artificial oxygen carriers (blood substitutes) in humans. These proteins are gigantic polymeric heme proteins with molar masses of approximately 4x106 daltons. Because of their relatively positive formal reduction potential, these extracellular hemoglobins strongly resist oxidation. In mammals, hemoglobin is normally protected within the reducing environment of the red blood cell. However, an artificial oxygen carrier must be able to function outside of a red blood cell since this is how it will be administered. Bovine hemoglobins were investigated using spectroelectrochemistry (SEC) and Nernst plot analysis to examine the effect of polymerization on the formal reduction potential (E°′). The average E°′ value for native bovine hemoglobin was -123(±10) mV vs Ag/AgCl. The formal reduction potential for HBOC-201, a glutaraldehyde polymerized bovine hemoglobin, was found to be -116(± 12) mV vs Ag/AgCl. T-state stabilized polymerized bovine hemoglobin and R-state stabilized polymerized bovine hemoglobin had reduction potentials of -148(±13) mV and -130(±25) mV, respectively. These findings question the hypothesis that polymerization is the major factor responsible for the numerically positive reduction potentials found in natural hemoglobin polymers extracted from Lumbricus terrestris (+73 mV) and Arenicola marina (+65 mV).

(546) **Polymer Blend Characterizations Utilizing FRET and Multivariate Fluorescence Correlation Spectroscopy**

Carol Roach[1], Sharon Neal[1]; [1]University of Delaware

Polymer blends are extensively used in products such as plastics, and extended release drugs. Of particular concern in creating polymer blends are issues such as diffusion, co-diffusion, viscosity, and polymer interaction. Several examples of use of fluorescence correlation spectroscopy (FCS) and fluorescence resonance energy transfer (FRET) as independent methods to characterize polymer diffusion and polymer interaction exist, however no examples of simultaneous collection of FRET and FCS for polymer characterization exist. Here, we will discuss the use of FRET and multivariate FCS (mvFCS, a variation of the FCS technique) as a means of characterization of the physical properties of a polymer blend specifically related to diffusion and polymer interaction.

(547) **Development of a Spectroelectrochemical Assay for Serum Bilirubin**

Paul Flowers[1], Megan Alexander[1]; [1]Univ North Carolina Pembroke

Bilirubin (BR) is produced by catabolism of the heme component of red blood cells and is a common clinical marker for diagnosis of numerous pathological conditions. Several clinical methods are available for the determination of BR in body fluids, many of which entail measurement of visible spectral changes accompanying its reaction with chemical or enzymatic reagents. We have followed a similar approach to develop a serum BR assay in which spectral changes accompanying controlled potential electrolysis are monitored, a measurement strategy known as spectroelectrochemistry (SEC). Potential advantages of such an SEC assay include lower cost, reduced chemical waste, and the possibility of increased selectivity compared to some existing assays. Results obtained to date indicate the assay compares favorably to the most common clinical methods with respect to sensitivity, precision, and analysis time, and that it will be effective in discriminating against commonly encountered interferants such as ascorbic acid, uric acid, and hemoglobin. A summary of these results and a description of on-going efforts to further characterize the SEC assay will be presented.

(548) **Fibre Spectroscopy with Clean in Place Probes for PAT**

Viacheslav Artyushenko[1], Joachim Mannhardt[1], Gary Colquhoun[1]; [1]Fibre Photonics Ltd.

All the fibre spectroscopy methods available in broad spectral range from 180nm to 18µm (55.500 to 550cm-1 – see Fig.1) can be used in laboratory and industrial applications, including transmission, reflection, fluorescence, Raman and evanescent absorption spectroscopy. Clean in Place (CIP) demand is of absolute necessity in any applications of optical methods for PAT, and industrial applications requires CIP in automated mode. All above listed fibre spectroscopy methods are compared in their applications for PAT when used with different process interfaces for CIP, including "Light House Probe" (LHP) based on silica fibres for 0.18-2.4µm range and two process-interfaces made by Company KNICK, Berlin: "Ceramat" & "Sensogate". These two last process-interfaces have been developed for CIP of electrochemical probes (to control pH, Oxygen, etc.), but have been already tested for different fibre probes used in a broad range from 180nm to 18µm. These tests were done with silica fibre probes for transmission spectroscopy and with two ATR-probes for Mid-IR range: 1.5-18µm. One ATR-probe was assembled for 1.5-6µm range (6500-1500cm-1) with Chalcogenide IR-glass (CIR)-fibres

and ATR-cone from cubic ZrO, while the other probe was made for 3.3-18µm (3000-550cm-1) with Polycrystalline PIR-fibres from Silver Halides assembled with Diamond or ZnSe ATR-cones. These ATR-probes for Mid IR evanescent absorption spectroscopy cover the whole "finger-print" part of spectrum which is the most informative on absorption bands of specific molecular vibrations. Remote spectroscopy of chemical reaction, real time monitoring of components concentration in liquid or gaseous stream in pipe, fermentation process control, etc. can be realised by immersion of sensing heads of flexible fiber probes coupled with different type of spectrometers – based on diffraction grating and detector arrays, FTIR-interferometers, IR-LED, optical filters or using Tunable Diode or Quantum Cascade lasers. Thus almost universal fibre spectroscopy solutions can be provided for industrial PAT in single or multispectral versions with a fully automated process-interface. This synergy will enable PAT solutions with CIP to be programmed for specific customer application.

(549) **Proteomic Analysis of the Rice Blast Fungus Magnaporthe oryzae**

Emine Gokce[1], Timothy Collier[1], Yeon Yee Oh[2], William Franck[2], Ralph Dean[2], David Muddiman[1]; [1]W.M. Keck FT-ICR Mass Spectrometry Laboratory; [2]Center for Integrated Fungal Research

Magnaporthe Oryzae is the fungus responsible for rice blast and destroys millions of hectares of rice each year. It has been reported in over 85 countries including China, Japan, Korea and United States. Our objective is to identify the whole proteome of this fungus, to understand its mechanisms during plant infection to achieve permanent resistance against it. In preliminary experiments M. oryzae spores were lysed via bead beating and protein extraction was accomplished by utilizing a 1xPBS, 0.1%SDS and 2M Urea buffer. The total protein concentration was assessed by BCA assay. 50µg Protein was loaded onto a 10-20% Tris-HCl Criterion Gel and an in-gel Trypsin digestion was performed. The samples were run through a nanoLC-FT-ICR mass spectrometer and we identified 589 proteins (6.9% of the whole genome) at 1% false discovery rate using the MASCOT database. In order to increase this number we compared the effect of different enzymes, such as Lys-C, for the digestion. The performance of these methods was evaluated based on number of proteins identified, the confidence of their identifications, and the diversity of biological functions to which the proteins are ascribed. Preliminary data obtained from the MASCOT search of GeLC-MS/MS analysis revealed proteins belonging to a variety of cellular compartments including the cytoplasm, ribosomes, mitochondrial matrix and plasma membrane. The biological functions represented by this initial data range from carbohydrate metabolism and cell development to protein translation and oxidative stress response. Further we investigated different pathways for the bioinformatics to analyze the raw data. Algorithms including MASCOT and SEQUEST were compared. The overlap between the two at 1% false discovery rates was determined. These comparisons are presented within and across tissue types (e.g. spores vs. germinated spores). We will also present the precision and dynamic range of spectral counting within data sets as a precursor to performing label-free quantification between tissue types and in response to external stimuli.

(550) **Chemical and Surface Structure Correlations Using Tip Enhanced Raman Spectroscopy**

James Marr[1], Zachary Schultz[1]; [1]University of Notre Dame

In recent years Raman spectroscopy has become an ever-increasing player in the field of chemistry. Traditionally, Raman spectroscopy suffers from weak signal and diffraction- limited resolution. The use of electromagnetic field enhancements associated with metal nanostructures, such as surface enhanced Raman scattering (SERS), have emerged to address these issues. Tip enhanced Raman spectroscopy (TERS) couples the chemical sensitivity of Raman spectroscopy with surface characterization of atomic force microscopy (AFM) to correlate chemical and surface structure information at a level of detail that has never been seen before. To increase the effectiveness of TERS in a back-scattering configuration, radially polarized light is being coupled with this system to enhance signal along the Z-axis, between the tip and the surface molecules. This methodology enables the ability to perform Raman imaging at a spatial resolution of about 100 nm. We have been successful thus far in imaging graphene sheets on sub diffraction-limited scales. With this ability we are investing edge effects on the graphene in relationship to the number of layers of graphene present at the edge. We have also shown the ability to, scan in air, a SAM layer with the Raman signal being amplified by both SERS and TERS. We are also working to perform *in situ* TERS of self-assembled monolayers (SAM) and lipid bilayers. With the end goal being to deposit lipid bilayers on a surface and be able to image the supported bilayers obtaining both structural and chemical information simultaneously.

(551) **Quantitative Determination of Polymorphic Purity of Crystalline API in Tablets Using Raman Spectroscopy and Multivariate Analysis**

Yong Xie[1], Rick Chiu[1], Nina Cauchon[1]; [1]Analytical Research and Development, Amgen Inc

The polymorphic purity of the drug substance can impact the quality, safety and efficacy of drug products. Often, the crystal forms of active pharmaceutical ingredients (APIs) are preferred due to better processabiity and chemical stability. In these cases, monitoring amorphous content and other crystalline forms of API in drug substance and drug product is critical in assisting the development towards rational final drug formulation and storage conditions. Raman spectroscopy has been widely used in recent years in polymorphic characterization of pharmaceuticals. Less water interference, flexible sample preparation requirement, and better signal sensitivity of aromatic API compared with non-aromatic excipients make Raman scattering technology especially helpful for the analysis of pharmaceutical tablet samples. In conjunction with multivariate data analysis, Raman technology has been used to develop accurate and sensitive quantification methods for polymorphic purity determination of API in drug substance and tablet formulation. The case studies presented in this work also include the design of experiments to understand the stability of selected polymorphic form with different storage or stress conditions including heat, humidity, and compression forces. Chemometrics data analysis using multivariate curve resolution (MCR), and partial least squares (PLS) algorithms are compared in terms of method accuracy and sample preparation requirements. Raman imaging technology is also used to demonstrate the distribution of polymorphic purity over tablet surfaces. Data collected using orthogonal technologies including differential scanning calorimetry (DSC), X-ray powder diffraction (XRPD) and solid-state nuclear magnetic resonance (ssNMR) are used to confirm the results obtained by Raman spectroscopy. Among these commonly used technologies for pharmaceutical solids, Raman spectroscopy was demonstrated to the technology that can be used for trace amorphous detection in both drug substance and pharmaceutical dosage forms, which is critical to aid development of stable dosage forms.

(552) **Design Considerations and Best Practices, for the Implementation of a Fluorescence Spectrophotometer in Laboratory and Plant Pharma PAT**

Susan Bragg[1]; [1]Expo Technologies

Monitoring content uniformity of powder blends by NIR is fast becoming an established technique. The limitations, challenges and benefits of this technique are known, the mechanics of plant installation are understood. However, drug development pushes forward with higher potency and lower dosage formulations that offer a new set of challenges for the PAT Scientist or Engineer. Fluorescence spectroscopy is emerging as a technique for both off line and real time content uniformity monitoring, to compliment NIR and offer some improvement in areas where NIR may not be ideal. Light Induced Fluorescence can offer very fast measurement speeds, sensitivity in the order of 100X that of NIR and a much simpler data handling route. This presentation considers the potential of light induced fluorescence as a PAT application together with the design criteria that allow it to move from a laboratory bench to the process environment. Measurement protocols and the approach to validation for routine use are also proposed. Where permissions allow, accounts of process studies and early feasibility work will be shared.

(553) **Spectroscopic Analyses for Quantification of Key Biological Components in Microalgae as Indicators of Environmental Change**

Rebecca Horton[1], Edward Duranty[1], Morgan McConico[1], Frank Vogt[1]; [1]University of Tennessee, Department of Chemistry

Microalgae biodiversity has been considered a fast and promising indicator of environmental change. In preliminary studies, FTIR in combination with chemometric algorithms is a proven technique for identification of species as well as for detecting spectroscopic changes due to environmental conditions. However, quantification of these chemical changes in microalgae would open novel pathways in understanding and assessing changes in marine ecosystems due to human impacts. For this purpose, several different microalgae species were cultured using different nitrogen and carbon nutrient sources to mimic environmental changes of these two key parameters. The effects of different nitrogen sources were studied spectroscopically after changing the nitrogen-containing nutrient, ammonium or nitrate ions, in the growing medium at different concentrations. Similarly, the impacts of the carbon source on the spectroscopic signatures were studied by varying the concentration of bicarbonate ions in the culture medium. After sufficient quantities of biomass were grown, algae samples were fixed, dried, mixed with KBr powder and pressed into pellets of known algae weight percentages. These pellets were then analyzed by means of FTIR spectroscopy regarding concentrations of selected lipids, amino acids, proteins, carboxylic acids, mono- and polysaccharides.

However, such solid samples of biological materials impose several challenges regarding data evaluation. Challenges are imposed by light scattering causing baseline shifts, variations in sample thickness (=absorbance pathlength) and highly overlapping analyte spectra which require innovative data pre-processing methods for chemometric calibration and evaluation. Novel methods for baseline correction and pathlength normalization will be presented. For quantification of selected target analytes, standard Principal Component Regression (PCR) has been applied. For calibration purposes, a database comprising concentration series of 30+ analytes (approx.1,500 samples) has been constructed and is continuously augmented. Results concerning concentration precision in multicomponent mixtures will be discussed. Current investigations are determining the feasibility to utilize such an artificial calibration for quantification of key compounds in real-world biological materials. Impacts from analytes not contained in the calibration will also be investigated along with methods to minimize them.

This methodology will provide a better understanding of the chemical composition of microalgae invoked by environmental change.

(554) **Bayesian Classification of Microalgae FTIR Spectra as Innovative Method to Detect Chemical Changes in Ecosystems**

Morgan McConico[1], Edward Duranty[1], Rebecca Horton[1], Frank Vogt[1]; [1]University of Tennessee, Department of Chemistry

Human activities have profound impacts on aqueous ecosystems which are numerous, complex and interrelated. Consequently, a simple measurement of one chemical parameter does not yield a comprehensive assessment of biological changes in ecosystems. Microalgal biodiversity, however, has been reported to respond quickly and sensitively to changes in marine ecosystems while adapting via homeostasis. For detecting and monitoring these chemical changes, novel analytical tools are required for facilitating microalgae as *in-situ* probes. Novel sensors will be based on classification of microalgae species and their chemical composition along with measuring their relative abundance. Biomaterials contain many characteristic, infrared-active components including amino acids, carbohydrates, and lipids. Relative composition of these analyte groups is hypothesized to reflect changes in their overall cell composition imposed by their growing conditions. Thus, FTIR spectroscopy is a promising technique but must cope with a large number of microalgae species and environmental conditions. In order to ensure high species classification rates, algorithms were developed based on Bayesian statistics. This method tests the hypothesis whether a certain algae sample belongs to a specific class which is determined by species and environmental conditions. Along with evaluating spectroscopic information, Bayes' classification gains from incorporating additional physical, chemical and geometric background information about the samples. In order to connect environmental conditions and the microalgae' chemical signatures, samples were cultured under different growth parameters. In the first step, these conditions focused on concentrations of different nutrient sources, specifically carbon (CO3-) and nitrogen (NO3- and NH4+). After a sufficient culturing process, algae samples were fixed, dried, mixed with KBr powder and pressed into pellets of known algae weight percentages. Secondly, FTIR spectra were acquired from these samples and compiled into a calibration database for Bayesian classification of future unknown algae samples by identifying the most probable class. These Bayesian classification methods along with the calibration databases have been incorporated into the remotely-accessible, custom-made Virtual Chemometrics Lab for straightforward utilization of these tools. The expected significance of this research is to utilize microalgal biodiversity as a novel probe for assessing ecosystems or chemical changes of the marine ecosystems.

(555) **Fast Methods for Simultaneous Wavelength Selection and Grouping**

Erik Andries[2], John Kalivas[1]; [1]Central New Mexico Community College; [2]Center for Advanced Research Computing; [3]Idaho State University

In spectroscopy, spectra are ordered in a meaningful way---by wavelength. Other data sets do not have this a priori or continuous ordering. For example, in microarray data sets, genes are discrete features. When selecting the most discriminating features for a regression or classification task, permuting the gene order in a data set does not affect which genes are picked. However, most feature selection algorithms treat all features as discrete, in spite of the fact that the data may have continuous attributes (e.g. wavelengths, time or dosage). Spectroscopically, it makes sense to find intervals or

bands of discriminating wavelengths. Algorithmically, it also makes sense to explicitly enforce this interval grouping into the wavelength selection procedure--provided regression/classification performance does not suffer or the computational burden is not prohibitive. Recently, algorithms have emerged that encourage both wavelength sparsity (many zero coefficients in the regression vector) and wavelength grouping. However, many of these algorithms are computationally slow. Adopting the stance that speed matters, we examine the faster variants that preserve wavelength sparsity and grouping, implement speed-up modifications to the slower variants and propose our own fast alternative.

(556) **Calmagite Assay for Quickly Screening Potential Magnesium Chelators**

Joshua Kimball[1], Leonard Moothart[1], Laurent Bernad[1], Thomas Kirkland[1]; [1]Promega Biosciences LLC

Using a formulation similar to commercially available magnesium detection kits that utilize 3-Hydroxy-4-[(2-hydroxy-5-methylphenyl)azo]-1-naphthalenesulfonic acid (calmagite), an assay has been developed to quickly screen potential chelators of magnesium. This assay is linear from 0.5-2mM Mg++, stable over time, consistent day-to-day, and can be run in water or common buffers. Measurements are made on a UV-vis spectrophotmeter, which is readily available in most laboratories. The assay was benchmarked with ethylenediaminetetraacetic acid (EDTA, a strong chelator) and ethylenediaminediacetic acid (EDDA, a weak chelator). Our initial screen consists of varying the concentration of the putative chelator in the presence of a fixed concentration of magnesium to get a qualitative assessment of binding strength. Compounds identified as strong chelators in the initial screen are then tested at a static concentration against variable magnesium. Following this second analysis, a dissociation constant (Kd) can be estimated using a Scatchard plot. A similar assay was developed simultaneously using a fluorescent magnesium indicator, and was found to be useful in determining the properties of chelators weaker than EDDA. Because magnesium chelators generally contain many acidic groups, adjustment of the pH is often required for solubility in water or buffer. Although KOH is effective in adjusting the pH, even a slight excess of hydroxide ion can have a detrimental effect on the assay. Alternatives to hydroxide ion for pH adjustment that do not interfere with the assay will be discussed.

(557) **Spatial and Time Resolved Measurements by a New Acousto-Optical Imaging Spectrometer in Combination with Glow Discharge Sources**

Maxim Voronov[1], Volker Hoffmann[1], Till Wallendorf[2], Swen Marke[2], Gary Hieftje[3], Steven Ray[3], Carsten Engelhard[3], Wolfgang Buscher[4]; [1]IFW Dresden, Institute for Complex Materials; [2]IfU Diagnostic Systems GmbH; [3]Indiana University; [4]University of Münster

First results of the 2D elemental analysis by a new imaging spectrometer in combination with glow discharge sources are presented. Pulsed RF discharges as well as pulsed DC discharges were used as sources of the light emission. The atoms of the sample are sputtered by the pulsed discharge and then diffuse to the discharge chamber walls or back to the cathode. To achieve good spatial resolution, the radiation of sample atoms must be detected immediately after the beginning of the discharge pulse, before atoms have had time to migrate far. For these measurements of the plasma radiation an optical spectrometer with a good spectral, time and spatial resolution is needed. Space- and time-resolved measurements of the pulsed glow discharge radiation are accomplished by means of a newly developed acousto-optical imaging spectrometer, fitted with an ICCD camera. A compact UV / VIS / NIR chromatically compensated optical design which incorporates a wide wavelength range high resolution acousto optical tunable filter, is combined with a wide wavelength range image amplifier based on a microchannel plate. The time resolution of the image amplifier is down to 1 ns. The spatial resolution of the system is about 0.1 mm at a distance of 150 mm from the light input window. In the wavelength range 250-800 nm the spectral resolution changes from 0.1 – 0.6 nm. Due to electronic tuning the access time to any wavelength is about 200 µs. These characteristics allow the measurement of discharge images for a wide range of different tasks including RF and DC micro- and millisecond pulsed discharge evolution and phase-correlated investigations of light emission during the RF cycle.

(558) **Single Modified Starch Granules Rapidly Imaged with Focal Plane Array FT-IR Microspectrometer Illuminated by Multiple Combined Synchrotron Beams**

David Wetzel[1,2], Michael Nasse [3,4]; [1]Microbeam Molecular Spectroscopy Laboratory; [2]Kansas State University; [3]Synchrotron Research Center; [4]University of Wisconsin-Milwaukee

This is the first report of chemically imaging a single starch granule in minutes with the newly commissioned, custom-built synchrotron illumination infrared environmental imaging (IRENI) system designed for environmental investigation by members of the Physics Department of the University of Wisconsin-Milwaukee. In the carbohydrate chemistry community, the utility and importance of octenyl succinic anhydride (OSA) modified starch is well known. Esterification with OSA produces emulsifying properties, improves emulsion stabilizing properties, gives water resistance to films and improves the flow properties of dry starch. The intergranular modification level differences are expected to affect functionality of the bulk product. The intergranular uniformity of modification has been a goal on the industrial scale and FT-IR microspectroscopy has recently been employed to obtain a carbonyl group census, one granule at a time. On a microscopic sale, because all dipole-dipole interactions are highly localized, the intragranular homogeneity or lack thereof is also of concern. Imaging a single 15 micron diameter modified waxy maize granules has presented an analytical challenge requiring a multihour mapping procedure by the usual procedure. The object of this study is to meet the challenge with the sophisticated optical system which allows rapid, direct chemical imaging. With the IRENI system, customization of a bending magnet in the synchrotron storage ring was required to extract multiple beams that are redirected by an intricate mirror system, and recombined and focused to provide 320 x 25 mrad2 synchrotron swath to fully illuminate the target on the microscope stage. The result is a diffraction limited spatially resolved image that is wavelength dependant. With a 64 x 64 element focal plane array and a 74x Schwarzschild mirror lens condenser. A 32 x 32 micron2 image is illuminated on the stage. Imaging data is acquired in 0.5 microns increments.

(559) **Remote Hyperspectral Imaging of Human Skin**

Kerri Moloughney[1], Kiersten Schiliro[2], Diane Williams[3]; [1]Oak Ridge Institute of Science and Education; [2]FBI, Operational Technology Division; [3]FBI, Laboratory Division

The ability to remotely detect human skin, human remains, and items of possible evidentiary value would be invaluable to search and rescue operations and criminal investigations. Preliminary statistical work using a hand-held short-wave infrared (SWIR) spectrometer confirmed that there are unique spectral signatures that can be used to distinguish skin from environmental objects commonly found in search scenes. The next phase of testing involved the use of remote hyperspectral imaging (HSI) to determine if an airborne sensor could spectrally discriminate human skin from background environmental factors. This capability would allow search areas to be processed quickly and

easily, enabling faster searches of larger areas and targeted ground search teams. Aerial tests were completed using a hyperspectral imaging camera that is sensitive in the 400-2350 nm range. The airborne system was mounted onto a Black Hawk helicopter and images were captured from test samples at altitudes ranging from 200-1500 feet. The surrounding area and environment included a paved road, heavy vegetation, mud, and water. Multiple atmospheric correction methods were compared, and the images were classified against known spectra. Results show the successful classification of human skin and various other samples of interest, to the exclusion of background variables.

(560) **Cellular Crystallography: Generation and Detection of 2D Membrane Protein Crystals in Living Cells**

Ellen Gualtieri[1], Fei Guo[1], David Kissick[1], Joyce Jose[1], Richard Kuhn[1], Wen Jiang[1], Garth Simpson[1]; [1]Purdue University

The elucidation of integral membrane protein structures is crucial for an understanding of the molecular basis of human diseases and the development of drugs to combat them. A major reason for the relatively slow pace of membrane protein structure discovery is the difficulty in detergent extraction and stabilization of the protein from the lipid environment. The observation by electron microscopy of spontaneous 2D crystal formation of membrane proteins in live cells suggests that cells themselves might prove to be viable media for protein crystallization. The central challenge of detection is shown to be overcome with the use of second order nonlinear optical imaging of chiral crystals (SONICC) to detect unlabeled 2-D membrane protein crystals in living cells. SONICC relies on the unique symmetry requirements of second harmonic generation (SHG) for selective detection of protein crystal array formation. The expression levels of bacteriorhodopsin (bR) in living cells as a function of culture growth time and the identification of specific cells expressing bR were determined by SONICC. SONICC was also used to detection the expression of proteins associated with virus infection in mammalian cells.

(561) **Characterization and Differentiation of Phthalates Using FTIR Fingerprinting, NMR Spectroscopy, and GC/MS**

Martin Best[1], Melanie Silinski[1], Joseph Licause[1], James Blake[1], Stephen Cooper[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

Phthalates were developed in the 1920s to substitute for camphor as a softening agent in the production of plastics. In recent years, phthalates have been extensively investigated for their potential hazards to human health and the environment. While some phthalates, such as dimethyl phthalate (DMP) and diethyl phthalate (DEP), are generally considered as non-toxic, some of the more structurally complex members like di(2-ethylhexyl) phthalate (DEHP) and di-n-butyl phthalate (DBP) are suspected of being carcinogenic and endocrine system disruptors in both humans and laboratory animals. The purpose of this study is to explore analytical techniques that can be used to differentiate and unequivocally identify different members of the phthalate family. A number of different analytical techniques including, FTIR, GC/MS, proton NMR, and carbon NMR were employed to differentiate six different di-substituted phthalates [DMP, DEP, DBP, DEHP, di-n-octyl phthalate (DnOP), and benzyl butyl phthalate (BBP)]. The IR spectra were compared and contrasted to determine how the signature of the various functional groups (aryl, ester and aliphatic) within these compounds are expressed, and what effects the chain length and the substitutions have on the fingerprint pattern. The DMP, DEP, and DBP resulted in discernible patterns in their individual IR spectra to account for the increasing lengths of the aliphatic chains. The NMR spectra for these three compounds were also distinct enough to allow definitive confirmation of identity for each compound. The IR spectra for DnOP, DEHP, and BBP exhibited similarities in their patterns and a lesser degree of differentiation than their short-chain members. There is also notable similarity in the NMR spectra for DnOP and DEHP, whereas the NMR spectrum for BBP has some unique characteristics. The GC/MS spectra provided the most conclusive results; all six phthalates exhibited differentiable fragmentation patterns. The results of this study show that FTIR, GC/MS, and NMR spectra can be used in conjunction for unequivocal identification of these different phthalates.

(562) **Highly Sensitive Imaging of Protein Crystals at Cryogenic Temperatures**

David Kissick[1], Ellen Gualtieri[1], Kevin Battaile[3], Michael Becker[3], Robert Fischetti[3], Steve Ginell[3], Lisa Keefe[3], Anne Mulichak[3], Vadim Cherezov[2], Garth Simpson[1]; [1]Purdue University; [2]Scripps Research Institute; [3]Advanced Photon Source

Second order non-linear optical imaging of chiral crystals (SONICC) was applied to protein crystal detection and characterization at cryogenic temperatures. The need for automated crystal centering has motivated the development of many techniques that determine the position of protein crystals frozen in loops (e.g. brightfield image analysis, birefringence, intrinsic UV fluorescence and X-ray diffraction based centering). These techniques have been limited by a lack of contrast, minimum detectable crystal size, and sample damage respectively. SONICC provides the necessary contrast by effectively eliminating the background from centrosymmetric materials (e.g. amorphous water and cryoprotectants). SONICC images have been taken at multiple angles, of both soluble and membrane protein crystals, ranging in size from several hundred microns down to 2 microns squared by a few molecular layers thick. In addition, preliminary diffraction studies showed no evidence of structural damage to crystals that were exposed for fifteen minutes with ~500mW laser power, where a typical image requires three minutes exposure at ~100mW. Further experiments are planned to assess the suitability of SONICC for automated crystal centering, especially with respect to crystal damage.

(563) **Second Harmonic Generation in NaCl**

Scott Toth[1], Garth Simpson[1]; [1]Purdue University

Sodium chloride, an achiral crystal, which would be unexpected to generate second harmonic generation (SHG), has been observed to give strong SHG from localized points. The following research probes the subject of the possibility of induction (by application of pressure) of SHG in achiral NaCl. Our hypothesis is that point defects, surface effects, or non-centrosymmetric polymorphs could be the origin/source of SHG. An unground sample of NaCl was compared with ground/crushed samples, and the unground samples showed no significant SHG generated, while the ground and crushed samples exhibited significant SHG. The possibility of surface effects was ruled out, meaning that there is a possibility of a new and undiscovered NaCl polymorph. Future research would include imaging at greater magnification, and proposing NaCl models that have the potential for NaCl production.

(564) **NIR Assessment of Water Correlates to Mechanical Properties in Articular Cartilage**

Alireza Hosseini[1], Somaieh Moghadam[1], Roza Mahmoodian[2], Sorin Siegler[2], Nancy Pleshko[1]; [1]Temple University; [2]Drexel University

Articular cartilage (AC) is a translucent tissue that covers the end of articulating bones and serves as the load bearing surface within synovial joints. Water accounts for 60 - 85 % of cartilage wet-weight and the interaction between the fluid and solid phase of the cartilage influences the mechanical properties of the tissue. Joint degeneration in the early stages of osteoarthritis (OA) may be

reflected in changes in water content and in mechanical properties in articular cartilage. Currently, water content is assessed by destructive methods. In the current study we sought to assess whether NIR spectroscopy could be used as a non-invasive technique to evaluate water content. This study provides preliminary data to support the use of NIR clinically. We determined the stress relaxation behavior of cartilage plugs obtained from young and old bovine knee in unconfined and confined compression geometries using the biphasic mixture theory. The elastic modulus (Es) and aggregate modulus (HA) were determined by fitting a line to the equilibrium stress-strain curves of unconfined and confined compression tests, respectively, and the axial permeability parameter was obtained by finding the best fit to the stress-time curve from the confined compression tests. Results show that permeability increases and the equilibrium modulus decreases with age, while the Poisson's ratio *(ν)* was similar for both ages. HA Young = 0.25 ± 0.07 MPa, while HA Old= 1.54 ± 0.22 MPa, $^{\nu}$Young=0.3±0.01 and $^{\nu}$Old=0.3±0.04, Es Old = 2.125 ± 0.03 MPa, Es Young = 7.86 ± 0.07 MPa ,and permeability coefficients k Young = 1.25 ± 0.35 ×10-16 m4 N-1 s-1 and k old = 2.78 ± 0.42 ×10-16 m4 N-1 s-1. NIR water data were measured from the integrated area of the absorbance between 4748-5376. This area decreased significantly from 233.7±1.8 to 208.2 ± 7.0 from young samples to old samples respectively. Water content was therefore positively correlated with the elastic modulus (Es), aggregate modulus (HA), and axial permeability and could potentially be used as a non-invasive surrogate for mechanical parameters.

(565) **A Novel Matrix Modifier for the Analysis of Arsenic and Antimony in High-Sulfate Acid Mine Drainage**

Ronald Smith; [1]Indiana University, Indiana Geological Survey

A mixture of palladium and magnesium nitrate has been proposed as a universal matrix modifier for the determination of several elements using Zeeman Corrected Graphite Furnace/AA. However, it has been known since the 1970's that this modifier is unable to eliminate the interferences caused by sulfate concentrations of 10 g/L or greater. Acidic mine drainage often contains sulfate at levels greater than this, preventing the detection of trace metals in concentrated mine drainage. Moreover, digestion solutions of other difficult matrices also may contain high concentrations of sulfuric acid that may interfere with metals analysis. Data will be presented demonstrating the use of a new modifier, barium acetate, on such samples. The comparison of signal peak size, shape, and coherence in these environmental samples using the traditional Pd/NO3 modifier with results obtained using the new modifier show significant improvements in signal characteristics. Using palladium, sulfate concentrations greater than 10 g/L completely suppressed the analyte signal generated by > 20 ug/L As. A failure to detect this concentration of As can have major environmental and regulatory consequences. On the other hand, barium acetate permitted the detection of as little as 1 ug/L As. Similar results were demonstrated for the element Sb. An optimized furnace temperature program is presented. The chemical reaction pathways whereby Barium acetate achieves this effect are explored and evaluated. The approach demonstrates the importance of applying methods development techniques to analytical challenges that defy the prevailing cookbook approach to environmental testing.

(566) **Development and Validation of an Analytical Method for the Determination of Zinc Carbonate Basic in Zinc-Deficient Rodent Feed**

Randy Price[1], Glenn Ross[1], Jason Perlmutter[1], Keith Levine[1], Donna Browning[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

Zinc carbonate basic is commonly employed in water-based drilling fluids to scavenge hydrogen sulfide and is the test chemical in an ongoing toxicological investigation. A critical component of this investigation was to provide confirmation that the correct test chemical was being administered at specified dose concentrations in a homogeneous manner. To this end, the present work describes the development and validation of a dose formulation analytical method for the determination of zinc carbonate basic (as zinc) in AIN-93M zinc-deficient rodent feed. The concentration of the test chemical was determined by inductively coupled plasma optical emission spectrometry (ICP-OES) following a rapid, atmospheric pressure microwave digestion procedure. Diet dose concentrations ranged from 2.80 to 550 mg Zn/kg diet. The preparation of homogeneous formulations within this concentration range was challenging because zinc is a ubiquitous element and was present in the zinc-deficient feed at a concentration approaching the lowest dose. In addition, zinc carbonate basic is insoluble in solvents appropriate for the preparation of animal feed formulations. As a result, it was necessary to develop and employ specialized premix and blending procedures to ensure the homogenous distribution of the test chemical within the diet formulations. Analytical method validation parameters included linearity, range, accuracy, precision, homogeneity, stability, and detection and quantitation limits, and dilution verification for doses up to 1000 mg Zn/kg feed. Validation was conducted using a 10-g feed sample size, which was pre-digested before removal of a 0.5-g homogenous aliquot from the slurry. Test chemical recoveries (n=9 at each dose formulation concentration) ranged from 89.2 to 109%, with %RSD values less than 15%. Linearity across the validation range was demonstrated with a correlation coefficient of 0.9998, dose formulation stability was demonstrated for a period of 42 days, and the mean extraction recovery across the entire validation range was 105%.

(567) **Co(II) Determination by Photoacoustic Spectroscopy with 3-(2-Pyridyl)-5,6-bis(4-sulfophenyl)-1,2,4-triazine as Ligand**

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This work introduces 3-(2-Pyridyl)-5,6-bis(4-sulfophenyl)-1,2,4-triazine (FST) as ligand for the Co(II) determination under Photo-Acoustic Chemical Sensor Analysis. The sensor was composed by ligand retained in anionic resin DEAE-Sephadex-A-25 and the formation of Co(II)-FST occurs on the coated solid phase. Photoacoustic spectroscopy (PAS) is based on measurement of acoustic waves produced by modulated light absorption. A conventional PA cell embedded with an electret microphone was used. Solid samples were irradiated using a 250W QTH lamp source after passing through a monochromator. Detection of PA signal was made by microphone-preamplifier circuitry and a lock-in amplifier yields the PA absorption spectra of the samples. The sample was prepared as following : mass of FST-DEAE-Sephadex A-25 50 mg, final dilution volume 100 mL, pH 5 (buffer acetic acid/sodium acetate) and shaking time 20 min. Solutions of Co(II) were prepared from a aliquot of a standard solution 25 mg/L. Raw PAS amplitude signals of Co(II)-FST-resin samples were recorded by averaging 100 measurements at each wavelength, at room temperature and 10 Hz modulation frequency .To compensate source intensity effects, the raw PAS amplitude signals were normalized to PAS amplitude signals obtained from carbon black sample. PA spectra shows an absorption band (480-520 nm) for the presence of Co (II), in agreement with the UV-VIS spectroscopy. The PAS signal of the band of the Co(II)-FST-DEAE-Sephadex-A-25 is produced in different wavelength absorption bands of the sensor, therefore there is no spectral interference. The metal complex spectra quantification is analyzed by means calibration curve generated with synthetically prepared Co(II) samples

between 50 and 300 ng/L, with analytical parameters : sensitivity of 0.00107 [PAS AU L/µg], LOQ 45 [µg/L], LOD 14 [µg/L] and Lineal Range 14-250[µg/L]. Each sample's PAS signals were measured in duplicate (rsd <5%). The method is been test in spiked samples of tap water. Acknowledgments to Projects Milenio ICM-P06-067-F and FONDECYT Nº1100103.

(568) **Optical Optimization of Phytoplankton Classification Instrument**

Joe Swanstrom[1], Laura Hill[1], Tammi Richardson[1], Timothy Shaw[1], Michael Myrick[1]; [1]University of South Carolina

Our laboratory has designed a real time instrument for the classification of individual plankton into one of three group target species (Emiliania huxleyi, Thalassiorira pseudonana and Synechococcus sp. (pink)) using multivariate optical elements. An optical ray tracing program, OSLO EDU, by Lamda Research was used to optimize the instruments optics to make a flat field projection of the modulated excitation source onto the sample flow cell.

(569) **The Dual-Sample CapNMR Probe: Application Diversity and Performance Enhancement through Miniaturization and Automation**

Timothy Peck[1], James Norcross[1], Craig Milling[1], Robert Albrecht[1], Dean Olson[1], Steve Xu[1]; [1]Protasis Corporation

Microflow NMR systems are specifically designed for analysis of low-mass, µg-quantity samples. The range of 1-100 µg is representative of sample amounts available in pharmaceutical compound libraries, low-abundance analytes of interest in liquid chromatography (impurity and degradation studies), and often represents the total analyte quantity isolated in natural product and discovery inquiries. The CapNMR measurement platform is uniquely configured for small samples in wellplates and microvials, deliberately excluding the incongruent NMR tube in favor of more standard laboratory sample preparation and handling equipment, including web-based sample management, liquid handlers, and system automation. The Dual-Sample Probe (DSP) represents a new entrant to NMR detection. Simultaneous introduction of two separate samples is accomplished using a single autosampler with dual injection ports and two completely independent, parallel flow paths each leading to an individual flowcell in the DSP. Each NMR flowcell in the DSP delivers the same spectral resolution and S/N as a single-sample CapNMR probe, but with a doubling of overall system capacity. The electrical isolation between the two flowcells enables the DSP probe to function in a variety of acquisition modes. For instance, both radiofrequency microcoils can be shimmed to high resolution with a single shim set for simultaneous acquisition. Alternatively, individual shim settings are readily called via spectrometer subroutines to enable sequential operation. In addition, a number of significantly novel strategies emerge where the probe can be configured to accommodate individual applications for each flow cell. For example, some users employ one flowcell for monitoring reaction studies and the second for metabolomic applications. Others maintain flow paths for two different solvents, or dedicate one flow cell for open-access use. This poster provides an overview of the Dual-Sample Probe in multiplexed NMR detection. Example data sets from a recent DSP deployment in a pharmaceutical lab for automated structure verification are presented and discussed.

(570) **Microscopic FTIR/DSC System Used to Investigate the Thermal-induced Intramolecular Cyclization of Eudragit E Film or PVA Copolymer Film**

Shan-Yang Lin[1], Wen-Ting Cheng[1], Yen-Shan Wei[1], Yu-Ting Huang[1]; [1]Yuanpei University

Microscopic FTIR/DSC System Used to Investigate the Thermal-induced Intramolecular Cyclization of Eudragit E Film or PVA Copolymer Film Shan-Yang Lin*, Wen-Ting Cheng, Yen-Shan Wei and Yu-Ting Huang Department of Biotechnology, Yuanpei University, Hsin-Chu, Taiwan, Republic of China Transmission FTIR/DSC microspectroscopy was applied to investigate the thermal stability of Eudragit E (terpolymer of butyl methacrylate, 2-dimethylaminoethyl methacrylate and methyl methacrylate) film or PVA copolymer (terpolymer of polyvinyl alcohol, acrylic acid and methyl methacrylate) film used in pharmaceutical industry. Both copolymers were evidenced to form six-membered cyclic anhydrides via ester condensation in the heating process. The anhydride-related IR peaks at 1801, 1763 and 1010 cm-1 for Eudragit E appeared from 180oC and increased their peak intensities with the heating temperature, but the appearance of IR peaks at 1799, 1759 and 1012 cm-1 was found from 167oC for the anhydride formation of PVA copolymer. Since the peak intensity ratio of 1763 cm-1/1801 cm-1 for Eudragit E or 1759 cm-1/1799 cm-1 for PVA copolymer was about 2.47 or 2.02, respectively, suggesting the predominantly intramolecular condensation of anhydride formation in the film of Eudragit E or PVA copolymer. A unique peak at 1140 cm-1 related to crystalline C–O stretching vibration of PVA copolymer disappeared from 167oC, also implying that the thermal-induced ester condensation altered or destroyed the crystallinity phenomenon in the film of PVA copolymer. The result indicates that FTIR/DSC microscopic system was a unique and rapid analytical technique to simultaneously determine the thermal stability of samples. Acknowledgement: This work was supported by National Science Council, Taipei, Taiwan, Republic of China (97-2628-B-264-001-MY3).

(571) **Formation and Characterization of Nanosized Organic Molecular Crystals on Engineered Surfaces**

Andrea Centrone[1], Kitae Kim[2], T. Alan Hatton[1], Allan S. Myerson[2]; [1]Massachusetts Institute of Technology; [2]Illinois Institute of Technology

The formation of organic molecular crystals with sizes below 500 nm is of great interest to the pharmaceutical industry since an enhanced solubility and dissolution rate can potentially increase drug bioavailability. In this work, patterned engineered surfaces were used for crystallizing glycine with a lateral dimension below 200 nm in a confined volume while controlling supersaturation. Individual crystals were characterized with AFM and Raman spectroscopy and determined to be the metastable beta form. The solubility of crystals obtained was measured as a function of crystal size and employed along with the Ostwald-Freundlich equation to determine the surface tension (1929 +/- 85 erg/cm2) and predict solubility enhancement vs crystal size. From this calculation the estimated solubility of 100 nm sized (radius) crystals is 0.589 mg/mL which is approximately twice the glycine equilibrium solubility (0.290 mg/mL).

(572) **NIST SRM for the Relative Intensity Correction of Raman Spectrometers Utilizing 830 nm Excitation**

Aaron Urbas[1], Steven Choquette[1]; [1]National Institute of Standards and Technology

Raman spectroscopy is an analytical method that has many applications in the chemical, forensic, pharmaceutical, and biotechnology industries. However, because Raman is an emission process, the spectra obtained are significantly influenced by the instrument response function. Consequently, no single atlas of Raman data exists for chemical compounds that can cover the many choices of lasers and instrumentation used in this technique. This is a significant stumbling block for industries that are subject to quality standards or vendors that are currently producing instrumentation specific Raman libraries. Spectra corrected for the,

instrument response function, much like absorbance spectra, would be transferable between instruments. NIST has undertaken the development of simple to use emission standards (SRM 224X series) to allow users to correct the sample spectra for instrument response. This work introduces preliminary data for the relative intensity correction of systems utilizing 830 nm excitation using an existing glass calibration artifact, NIST SRM 2244. NIST SRM 2244 is a chromium oxide doped borosilicate glass currently available for intensity correction of Raman systems utilizing 1064 nm excitation. The process of transferring a NIST radiometric white light correction to Raman instruments via the use of a secondary glass calibration artifact will be presented.

(573) **Imaging Nonlinear Optical Stokes Ellipsometry (iNOSE)**

Garth Simpson; [1]Purdue University

The polarization-dependence of nonlinear optical measurements contains rich information related to local structure and orientation. Nonlinear optical ellipsometry (NOE) performed with rapid polarization modulation and a static Stokes ellipsometry detection configuration was designed to reduce acquisition times for NOE by ~8 orders of magnitude with no loss in measurement precision laying the foundation for precise polarization analysis in imaging applications. Nonlinear optical ellipsometry retains additional phase information lost in more conventional polarization-dependent measurements, increasing the number of observables that can be used for analysis. A systematic framework for designing NOE experiments will be presented. Fundamentals and applications of fast, precise NOE in microscopy, thin film, and microcrystal analysis will be described, along with a discussion of practical challenges.

(574) **Re-Investigation of Excited State Proton Transfer Reaction in 2-naphthol in the Presence of Sodium Acetate**

K Singh, G.C. Joshi; [1] HNBGarhwal University, Srinagar

The excited state proton transfer reaction of 2-napthol has been a subject of extensive study and has been considered as a model for such reaction mechanisms [1-4]. There is an enhancement in the proton transfer rate on addition of Sodium acetate. In the present work we have re-investigated the 2-naphthol excited state proton transfer reaction in the presence of different concentrations of sodium acetate at pH=5. Both the steady state and time resolved techniques have been used in the present study. It is observed that there is a deviation from the single exponential for the decay curve of 2-naphthol emission in the absence of sodium acetate measured at 350 nm which becomes single exponential on addition of a small amount of Sodium acetate (0.005M). On adding higher quantities of Sodium acetate the decay curves again become double exponential. The investigations of the steady state spectra also reveal absence of the isosbestic point between naphthol and naphtholate ion emissions. Both these observations suggest that the excited state proton transfer reactions of 2-naphthol are not as simple and require thorough investigation. Some possible suggestions have been made.

(575) **New Directions in AFM-Raman BioImaging in Liquids**

David Lewis[1], Anatoly Komisar[1], Andrey Ignatov[1], Rimma Dekhter[1], Hesham Taha[1], Aaron Lewis[2]; [1]Nanonics Imaging Ltd.; [2]Hebrew University of Jerusalem

A new feedback mechanism for AFM imaging in liquids will be introduced. This new approach is based on tuning fork technology which has been shown to be the most effective method of AFM feedback. Thanks to their high Q factors, tuning forks allow for improving feedback sensitivity and imaging quality. It will be shown how these unique characteristics can now be extended to liquid environments for bioimaging. Very small working distances allow for working with many high NA objectives, such as water immersion objectives. A completely open liquid cell from above provides no Raman signal loss, which is very important for biological samples. Such advanced techniques allow for using recently reported frequency modulation techniques for ultra high resolution biomolecular pulling. Moreover, it permits full integration with other optical techniques such as fluorescence, TERS, confocal, NSOM, TIRF, FRET and DIC with no interference that usually occurs with standard laser based optical feedback.

(576) **Proposal of a New Calibration Strategy for LA-ICP-MS Based on Dried Residues of Individual Picoliter Droplets**

Jan H. Petersen[1], Jan Massmann[1], Niklas Schaper[1], Nicolas H. Bings[1]; [1]University of Mainz, Analytical Chemistry

Laser ablation (LA) in combination with inductively coupled plasma mass spectrometry (ICP-MS) has evolved to a mature tool in the field of spatially-resolved elemental analysis of solid samples. However, accurate calibration is still difficult, since it is hampered by elemental fractionation and the lack of available standard reference materials. The presented novel calibration strategy is based on the ablation of dried residues from small droplets of known volume of standard solutions. Therefore, a novel droplet generator using thermal inkjet technology was designed for the reproducible transfer of minute amounts of sample mass onto various sample targets through individual droplets. The so called “drop-on-demand generator (DOD)” is micro controlled, uses a modified “stand alone” printer cartridge for droplet generation and is capable of dosing sample volumes in the picoliter-range. It is possible to create droplets with adjustable diameter in both individual and continuous modes, allowing a variable droplet generation frequency. Furthermore the DOD generator contains an electronic, labview driven interface allowing to fully control the dosing process and repetitive and reproducible sample transfer. In this poster we present a fundamental characterization of the novel DOD system regarding the transferred mass. Data on the achievable precision concerning the droplets’ size (distributions) and the achievable droplet volume determined by TXRF, AFM and SEM investigations will be presented. The systems potential for both precise transfer of known picoliter volumes of standard solutions onto solid samples and for a new calibration strategy in LA-ICP-MS will be outlined.

(577) **Multiphoton-Excited Intrinsic Fluorescence of Protein Crystals**

Jeremy Madden[1], Ellen Gualtieri[1], David Kissick[1], Garth Simpson[1]; [1]Purdue University

Reliable detection and characterization of protein crystals is a critical step in protein structure determination. Two complementary methods are described based on multi-photon excitation of intrinsic fluorescence and second-order nonlinear optical imaging of chiral crystals (SONICC). SONICC imaging has been shown to compare favorably to conventional detection methods in membrane protein studies, particularly with crystal sizes under 5 microns and/or for crystals grown in turbid media. One example is membrane protein crystals that are grown in lipidic cubic phase (LCP), which mimics the conditions of the natural protein environment. Voids and defects, including LCP crystals, can arise in these conditions, making it difficult to discern the protein crystals from those of the LCP. As a complement to SONICC, two-photon excited fluorescent (TPEF) imaging allows access to the intrinsic UV fluorescence of membrane proteins. By using multi-photon visible excitation, the high concentrations of aromatic amino acids, specifically tryptophan, can be differentiated from small-molecule crystals or any surrounding matrix. In these studies, the use of an ultrafast fiber laser provides incident light at 1030 nm and 515 nm, permits SHG and TPEF measurements of the protein crystals to be

conducted on the same platform without the need for UV compatible optics.

(578) **ETD and CID Characterization for the Automated Top-Down Analysis of Intact Large Proteins via High Resolution FTMS**

Aaron Behr[1], Jeremy Wolff[2], Christopher Thompson[2]; [1]The Rivers School; [2]Bruker Daltonics, Inc.

Top-Down analysis is quickly becoming a routine method for the identification and characterization of intact proteins that are believed to be modified, or whose sequence is incorrect or unknown. In the Top-Down approach, MS/MS is performed on the intact protein, producing a very complex product ion mass spectrum. Fourier Transform Mass Spectrometry (FTMS) is ideally suited for analysis of these complex spectra due to its high resolving power (>100k in broad band mode) and high mass accuracies (< 1 ppm). This precision allows for the unambiguous assignment of product ions and ultimately in protein identification and determining any potential modifications. For this technique to become a standard analytical tool in Proteomics laboratories, this process needs to be automated through LC-MS/MS. Electron Transfer Dissociation (ETD), Electron Capture Dissociation (ECD), and Collisionally Induced Dissociation (CID) are the commonly used ion activation methods for Top-Down mass spectrometry. While these ion activation methods produce abundant product ions, ETD and CID are preferred as they can be used within the time requirements of LCMS. ETD and CID methods have been shown to produce complementary fragment ions, increasing the ability to identify unknown proteins. While there have been studies that examine fragmentation efficiency as a result of ion activation energy for small molecules and peptides, relatively little work has involved larger molecules such as proteins. This work will systematically examine ETD and CID fragmentation energies on large proteins and is an extension of our work on the optimization of CID energies for automated LCMSMS analysis. It is known that there is a linear relationship between fragmentation energies and m/z, and this relationship has been optimized for peptide ions with low charge states (2+, 3+). These results are now being extended for intact protein ions with high charge states (~8+ through 15+). Preliminary results show a difference in optimized fragmentation efficiencies versus sequence coverage, which is charge and molecular weight dependant. The results of this study will be incorporated in the overall optimization of fragmentation energies of proteins.

(579) **Surface-Enhanced Raman Scattering from Au and Ag-coated Magnetic Microspheres**

Gulay Ertas[1], Haci Osman Guvenc[1]; [1]Bilkent University, Chemistry Department

A novel surface-enhanced Raman scattering (SERS) substrate was prepared by coating magnetic microspheres with gold and silver without the requirement of a linker or capping agent. The micron-sized magnetic microspheres were prepared in two steps: In the first step, an inorganic core that consisted of oleic acid-coated magnetite nanoparticles was prepared by the co-precipitation method. The second step was the encapsulation of oleic acid-coated magnetite nanoparticles by a modified suspension polymerization method. The magnetic microspheres were coated by gold and silver directly by hydroxylamine or sodium borohydride reduction methods for gold coating and only sodium borohydride for silver coating. From their SEM images, the average size of magnetic microspheres is measured to be 22 µm. EDX analysis demonstrated that the microspheres were indeed coated with gold and silver. The gold- and silver-coated magnetic microspheres were highly efficient surface-enhanced Raman scattering (SERS) substrates, with an enhancement factor estimated using Rhodamine 6G as a model adsorbate to be larger than 10 7. Our results indicated that these gold-and silver-coated magnetic microspheres, with their low manufacture cost, high stability (for at least nine months), reasonable reproducibility and reusable SERS-active substrates, have a rather promising prospect for future surface-enhanced molecular detection.

(580) **Toward High-Speed, Near-Field Raman Acquisition Utilizing Ag Nano Junctions**

Steven Asiala[1], Zachary Schultz[1]; [1]University of Notre Dame

To facilitate high-speed acquisition in near-field Raman microscopy experiments, we are studying enhancements obtained from junctions created between an AFM tip with a nanoparticle and an array of nanostructures. These junctions allow for the coupling of Surface-Enhanced Raman Spectroscopy (SERS) and Tip-Enhanced Raman Spectroscopy (TERS) effects, and are facilitated by the use of radially polarized light. Current SERS results obtained from a substrate comprised of Ag nanopillars prepared by vapor deposition indicate Raman spectra can be obtained in a few milliseconds with good signal to noise levels. Combining the nanostructured arrays with TERS measurements in our laboratory is a promising new approach to high spatial resolution Raman imaging. A driving force in this work is the potential to study local heterogeneity in model lipid membranes.

(581) **Trace Element Analysis of Seminal Fluid by ICP-MS**

Todor Todorov[1], Gustavo Guandalini[2], Dennis Hoover[3], Larry Anderson[3], Jose Centeno[2], Katherine Squibb[4], Melissa McDiarmid[4]; [1]US Geological Survey; [2]Armed Forces Institute of Pathology; [3]University of Maryland; [4]Veterans Administration - Baltimore

Seminal fluid has the important function of protecting and transporting spermatozoa. Previous studies have indicated that the major changes in trace elemental composition might be related to proper spermatozoal function and reproductive problems. In the present study, the concentrations of 22 trace elements were measured in seminal fluid from 33 patients using inductively coupled plasma mass spectrometry. The samples were decomposed in nitric acid at 110oC . From all elements measured Zn levels were highest followed by Fe, Sr, Cu, Al and Ni. Other toxicologically relevant elements such as Cd, Pb, Tl and As were found in the ng/L ranges for all subjects. For Zn, Fe, Sr, Al, Cu and Mn our values compare well with previous investigations. However, for the rest of trace elements studied, our values are much lower (in some cases by 2-3 orders of magnitude). The main reason for this discrepancy is the use of various additives to seminal fluid such as liquefiers, preservatives, etc by other authors. In the present study, no additives were used and thus we believe that the obtained trace element concentrations represent normal population background seminal fluid levels.

(582) **Development of a High-speed and High-sensitivity Near-Infrared Spectrometer and Short -Time Transmission Measurement of Tablets by Using It**

Koudai Murayama[1], Makoto Komiyama[1], Takuma Genkawa[2], Mikiko Konta[2], Yukihiro Ozaki[2]; [1]Yokogawa Electric Corp.; [2]Kwansei Gakuin Univ.

A high density photo-diode array near infrared (NIR) sensor with 640 elements was developed, and by using it a prototype of a high-speed and high-sensitivity NIR spectrometer of a polychrometer type (P-NIRs) has been build. We have tried a short time tablet transmission measurement by using P-NIRs, and have succeeded in the quantitative analysis of tablets in several seconds. Recently, pharmaceutical industry has keen interest in on-line process monitoring, and NIR spectroscopy is one of the most promising monitoring techniques. The content test and inspection of entire

tablets are thought to be an end goal. A diffusion reflection method is often attempted in the content measurement of the tablets. The diffusion reflection method acquires only the surface information on the tablets. Therefore, there is a possibility that this method doesn't realize the central content of the tablet. A predominant technique to avoid this inconvenience is a transmission measurement. However, the short time transmission measurement of tablets has not been easy because transmission of NIR light through a tablet is extremely low. We have developed a NIR spectrometer of a polychrometer type with the in-house 640 elements photo-diode array sensor. Then, we have attempted transmission measurement of tablets, and have succeeded in the transmission measurement in the short time and its quantitative analysis. The tablets for this test were made from a powder consisting of magnesium stearate, lactose, and cornstarch, etc. by a press machine. The concentration of the magnesium stearate was in the range of 0-5 wt%. We attempted the transmission measurement of these tablet samples by using P-NIRs, and acquired the spectral data in several seconds. Then, we transformed the spectral data and constructed a calibration model with partial least squares regression. Correlation coefficient R2 of the calibration model showed a high correlation. It was confirmed that we can perform the transmission measurement of tablet in short-time with P-NIRs.

(583) **Identification of Fish Species by Protein Profiling Using MALDI-TOF Mass Spectrometry**

Alexander Post[1], Sergei Dikler[1]; [1]Bruker Daltonics Inc.

Previously, MALDI-TOF protein profiling techniques have been used to successfully identify microorganisms in environmental and clinical applications. This work is focused on identification of fish species based on protein profile in the mass range from 3000 to 15000 Da. A low-cost, effective fish verification tool is needed in order to thwart the fraudulent substitution of high quality seafood with low quality fish, which is becoming increasingly common. MALDI-TOF analysis can accurately, quickly, and inexpensively identify the species of unknown fish samples. MALDI-TOF spectra collected from fish samples are exceedingly consistent and species-unique, making it easy for the software to easily ascertain the correct taxonomy of the sample. Samples from seventeen species of fish were processed into a novel database in Bruker Daltonics MALDI Biotyper software. Each database entry was created from 16-36 MALDI-TOF spectra acquired from 4-8 protein extracts, which were generated from 2 fish specimens. No such database has ever been created for seafood identification with MALDI-TOF. The selection of fish used to create the database was diverse, but did have some very closely related species including 2 species from Thunnus genus (Thunnus thynnus and Thunnus albaceres) and 3 species from Salmonidae family. When the software processed one of the closely related species, the related fish did receive higher scores than non-related fish; however, in the trials performed, the correct fish was always unambiguously identified with a satisfactory statistical margin. As this method relies only upon the proteome of the fish, any species could be added to the database for identification or verification. Current efforts are focused on identification or *de novo* sequencing of selected peptides and small proteins from fish protein profiles.

(584) **Chromatographic Detection Using a Micro-Fluidic Attenuated Total Internal Reflection (ATR) Cell Coupled to a Planar Array Infrared Spectrograph**

Willie Tran[1,2], Andre Sommer[1,2]; [1]Molecular Microspectroscopy Laboratory; [2]Miami University

Patterson et al. recently demonstrated the detection of analytes separated by liquid chromatography and capillary electrophoresis using a single bounce attenuated total internal reflection (ATR) cell coupled to a conventional infrared microscope. The results of those studies demonstrated that the method possessed excellent volumetric resolution (540 femto-liters) and a detection limit of 20 ppm for moderate infrared absorbers. In that study the detection efficiency was improved by closely matching the evanescent volume to the cell volume thereby eliminating the detrimental dead volume. The limiting drawback to the method was the speed at which (~4seconds) spectra could be collected due in part to the modulation efficiency of the detector. Lanzarotta et al. retained the volumetric resolution advantage, added a multichannel detection advantage and addressed the temporal resolution limitation demonstrated by Patterson et al. by coupling a microchannel/cylindrical hemisphere internal reflection element (IRE) sample cell with a planar array infrared (PA-IR) spectrograph. Although this preliminary study demonstrated feasibility for employing ATR-PA-IR spectroscopy as a real-time detection technique for liquid chromatography, the method was limited by a large dead volume and the flow rate was not optimized to accommodate the frame rate of the detector In this investigation we describe an optimized ATR cell based on a zinc sulfide hemi-cylinder internal reflection element and micro-fluidic channel. The cell is then coupled to a planar array infrared spectrograph (PAIRS) which permits 256 spectra to be collected in ~ 15 milli-seconds. The speed at which spectra can be collected is limited by the frame rate of the array. Once in the cell, the analyte is detected 256 times as it traverses through the cell. Design considerations for optimizing the integration of the chromatographic column to the ATR cell will be presented in addition to experimental results.

(585) **Comparison of Atomic Absorption and Molecular Spectrophotometry for the Indirect Determination of Phosphate Compounds**

Neil Danielson[1], Matthew Collins[1]; [1]Miami University

The indirect determination using the suppression of the Ca atomic absorption (AA) signal by not only ortho-phosphate but also other phosphate species such as polyphosphates and polyphosphonates has been investigated analytically. In particular, tripolyphosphate and hexametaphosphate are of interest since they are two major phosphorus containing components in Calgon and fluorophosphate is found in some toothpaste brands. In addition, polyphosphonates such as ethylenediaminetetra(methylene phosphonic acid) found in Dequest products are promising candidates. Using a 1.2 mM calcium solution, linearity in AA response from 0.02 to 0.1 mM for ortho-phosphate is observed but the change in absorbance is only about 0.1 absorbance unit (AU) with a correlation coefficient of about 0.965. The sensitivity as measured by the slope is about the same for hypophosphate and fluorophosphate but higher for both tri- and hexa-polyphosphate. The indirect determination of similar phosphate compounds as indicated above has been done by measuring the loss of absorbance at 525 nm of the Fe(III)-salicylate complex due to preferential phosphate interaction with ferric ion. A change in AU of about 0.7 from 0.05 - 0.6 mM ortho-phosphate with a higher correlation coefficient of 0.997 is found. By using 0.05 M nitric acid (pH = 1.3) as the solvent, selective determination of fluorophosphate (pKa = 0.55) in the presence of ortho-phosphate (pKa =1.97) seems feasible. Both the AA and molecular spectrophotometric methods are simple, rapid, and have the potential for automation.

(586) **Identification of Unknown Pharmaceuticals in Hospitals with a Small Coded Aperture Raman Spectrometer**

Prasant Potuluri[1], Brett Guenther[1], Yuting Qi[1]; [1]Centice Corporation

Identification of unknown pharmaceutical pills is crucial in the emergency room where patients are often admitted for adverse drug reactions. Proper identification of an ingested drug ensures effective treatment, however in some cases this information is not

available or is misleading, and in many cases, the care giver has only an unlabeled pill to identify the drug in question. Visual identification of an unlabeled pill is possible using the Physician's Desk Reference (PDR) or an online alternative; however these methods may be time consuming and assume that the pill is in good condition. Here we present a compact, easy-to-use device that makes pill identification faster and more effective. The device, known as "Rx ID," uses Raman spectroscopy to identify a pill from a library of more than 2500 unique pharmaceuticals in seconds. The speed of the device arises from our unique coded aperture that enables efficient light collection over a wide field of view without sacrificing resolution and uses lower laser intensities. The algorithm for analyzing the Raman spectra was built using linear discriminant analysis (LDA) and trained on more than 2500 unique drugs from our onsite pharmacy. The algorithm matches the unknown pill's Raman spectrum with the spectral library and returns the most likely matches. Stock pictures are displayed with the results so that the physician can easily narrow down the possibilities and make the final identification. Testing has shown that the "Rx ID" device can accurately display the top 10 results 98% of the time. In this talk we will introduce the "Rx ID" product We will present hardware and algorithm design details, and discuss how they were chosen to meet or balance design requirements. Practical results from testing on actual pharmaceuticals will be shown and will illustrate the effectiveness of the device.

(587) **Development of Halide Sensor Based on 4-(2-Pyridylazo)resorcinol (PAR)**

Michal Sidlo[1], Premysl Lubal[1], Pavel Anzenbacher Jr.[2]; [1]Masaryk University Brno, Czech Republic; [2]Bowling Green State University

Halide (Cl-, Br-, I-) salts widely present in the Nature are used as raw materials in numerous industrial processes and they are also present in natural mineral waters. Due to the wide impact of those anions on health and environment there is a need for fast, inexpensive and practical methods for halide anion determination. The azodyes (e.g. PAR) are well known as widely utilized chromogenic reagents for determination of a number of metal ions. Unfortunately, the reliable determination is in many cases precluded due to the presence of interfering anions. In this contribution, we will discuss the optimization of experimental conditions and the results of the Artificial Neural Network (ANN) analysis of selected Tl(III)-PAR system which was employed for determination of iodide, bromide, and chloride. Since the color change in aqueous solution caused by a ligand substitution reaction is sufficiently large, the Tl(III)-PAR system can be potentially used as inexpensive halide chemosensor available for general practice. Acknowledgement: The research was supported by Ministry of Education of the Czech Republic (project ME09065 and BIO-ANAL-MED (LC06035)).

(588) **Improvement of Analytical Method for Methylmercury in Seafood by High Performance Liquid Chromatography-Inductively Coupled Plasma Mass Spectrometry**

Kyung-Yoal Yoo[1], Kyeong-Nyeo Bahn[1], Yang-Sun Kim[1], Eun-Jung Kim[1], Seong-Chul Shin[1], Ji-Hyeon Seok[1], Hye-Young Park[1], Mi-Sun Lee[1], Yeo-Won Sohn[1], Hae-Seong Yoon[1]; [1]Gyeongin Regional KFDA

The main source of human exposure to mercury is dietary intake of fish and fish products. The Korea have established guideline of 1.0 mg/kg for methylmercury in tuna species, billfish species and abyssal fish. Most of mercury measurement in fish has been quantified by mercury direct analyzer and gas chromatography with electron capture detection. Recently, methylmercury is also analyzed by HPLC-ICP/MS because of the simplicity for sample preparation steps and the interface to the detector. However, most of pretreatment methods for methylmercury need a further pH adjustment of the extracted solution for HPLC and removal of organic matter. Also, the techniques for sample preparation are time-consuming and prone to unpredictable analyte losses and contamination. The aim of this study is to improve a pretreatment and quantitative method for determination of methylmercury in seafood by HPLC-ICP/MS. We extracted the analytes with adding 20 mL aqueous 1% L-cysteine¡¤HCl¡¤H2O and heated for 20 min at 60¡É in microwave(ETHOS-1, Milestone). This method was effective in removal of organic matter for methylmercury in seafood. The determination of methylmercury was carried out with HPLC-ICP/MS. Clear peak of methylmercury was obtained by using a C18 (Synergi Hydro-RP, 4 ¥ìm, 150¡¿4.6 mm) column and aqueous 0.1% L-cysteine¤ýHCl¤ýH2O + 0.1% L-cysteine mobile phase at 25¡É. The presence of cysteine in mobile phase and sample solution was essential to eliminate adsorption, peak tailing and memory problems. The methylmercury eluted from HPLC system was detected by ICP-MS at mass-to-charge ratio 202. The accuracy was evaluated using certified reference materials and tuna samples spiked with methylmercury. Recovery of the methylmercury for the samples were 93.14~98.81%. The limit of quantification for methylmercury in bluefin tuna was 1.04 ¥ìg/kg. The calibration curve obtained using these conditions was linear with good correlation coefficient(r2) of 0.9996 in the rang of 1.0~100.0 ¥ìg/kg. The relative standard deviation of the peak area and repeatability for methylmercury were below 1.50% and 1.68% respectively. The advantage of microwave-assisted extraction method is that the extracted solution can be directly injected into the HPLC column without pH adjustment. We expect that the established method is successfully applied to the determination of methylmercury in seafood.

(589) **Validation of Low-e Slides for FT-IRIS Polarization Measurements**

Arash Hanifi[1], Nancy Pleshko[1]; [1]Temple University

Collagen is the primary protein of connective tissues such as bone, skin and cartilage. The orientation of collagen contributes to its mechanical properties and friction coefficient. Changes in the collagen network can result in cartilage diseases such as osteoarthritis (OA). Fourier transform infrared imaging spectroscopy (FT-IRIS) has been used to characterize the structure, distribution and orientation of molecules in histological sections of connective tissues. In recent studies polarized FT-IR images have been calibrated to assess the alignment of collagen fibers in cartilage tissue sectioned on BaF2 windows. The use of low-e slides (Kevley Technologies, Chesterland, OH) in FT-IR is of great interest due to their significantly lower price, and the ability to stain histological sections on these slides. Since the mechanism of data collection from low-e and BaF2 slides are different (transflectance and transmittance respectively), we assessed whether the FT-IRIS-derived collagen orientation values were equivalent for both methods. In current study, polarized FT-IRIS amide I/II area ratios, previously shown to correlate to collagen orientation, were obtained from highly oriented bovine tendon and decalcified bovine cartilage to assess the effect of type of slide on the FT-IRIS derived values for collagen fibers orientation. Bovine tendon and cartilage were sectioned on both low-e slides and BaF2 windows. Polarized FT-IR spectra were collected in different polarization angles (0-180°). There was no significant difference of the changing amide I/II area ratios for the tissues sectioned on low-e slides and BaF2 windows. Low-e slide results were in the same range as the BaF2 slide, where the amide I/II area ratio was > 2.7 for collagen fibers oriented parallel to the surface of the sample for data collected at the angle of polarization equal to 0°. A sine function was fit to the amide I/II area ratios with an R squared > 95%. We conclude that the FT-IRIS derived properties for collagen

fiber orientation can be utilized equivalently for both low-e slides and BaF2 windows.

(590) **Calibration of NIR Water Assessment in Articular Cartilage Using a Model System of Gelatin and Chondroitin Sulfate**

Mugdha Padalkar[1], Karl Lewis[1], Nancy Pleshko[1], Richard Spencer[2]; [1]Temple University Philadelphia; [2]National Institute of Aging Baltimore

Calibration of NIR water assessment in articular cartilage using a model system of gelatin and chondroitin sulfate Mugdha Padalkar1, Karl Lewis1, Richard Spencer2, Nancy Pleshko1 1 Temple University, Philadelphia 2 National Institute of Aging, Baltimore Articular cartilage is a hyaline cartilage which covers the subchondral bone in a diarthrodial joint. In diseased conditions such as osteoarthritis, there is an increase in water content from the normal of 60-85% to greater than 90%. As cartilage is avascular, and thus has very little capability for self repair, methods of early detection of degeneration are urgently required. One potential useful diagnostic method is the assessment of water. The techniques of FT-IR microscopy and imaging have been used for analyses of water in food, pharmaceuticals and skin. Here, we hypothesize that NIR spectra can be utilized to assess water content in cartilage. We developed a model system of gelatin and chondroitin sulfate gels, representing the primary matrix components of cartilage, collagen and proteoglycan, in water with 60 to 80% water and 40-20% of gelatin and/or chondroitin sulfate to mimic the water content in cartilage.We assessed whether the NIR spectral absorbance bands that arise from water correlate to the water content in these mixtures. The gels were sliced to 1mm thickness and the spectra were obtained from the gels using PerkinElmer Spotlight 400 Spectrometer at 8cm -1 spectral resolution, 50µ spatial resolution and 16 scans per pixel. For the spectral analysis, the water content was initially determined by finding the integrated areas under the absorbance bands centered at 5190 cm-1 and 6890 cm-1. Our preliminary results suggest that there is a correlation between integrated area under the water peaks and percentage of water content Gelatin + Water model; area under 5190 cm-1 , R2=0.7056 and area under 6890 cm-1 R2=0.646. In Gelatin +chondroitin sulfate, area under 5190 cm-1 R2=0.6246 and area under 6890 cm-1 R2=0.5801). These calibration curves will then be utilized to to assess the water content in normal cartilage.

(591) **Enhanced Ion Mobility Shift Reagents for Peptide Labeling**

Thomas J. Kerr[1], Randi L. Gant-Branum[1], John A. McLean[1]; [1]Vanderbilt University

Post-translational modifications (PTMs) account for the vast diversity found within the proteome. Novel techniques to study these protein modifications are required in order to fully characterize and understand the importance of these modified peptides and their effect on the proteome. Ion mobility-mass spectrometry, IM-MS, offers a post ionization separation step that reduces sample complexity prior to mass spectrometry. PTM specific shift reagents offer a practical method to easily isolate signals that contain a specific PTM within an IM-MS spectrum. An ideal high density shift reagent should have the properties of high mass and relatively small surface area, require minimal sample treatment, and be robust in application. Lanthanide-based shift reagents provide these properties (especially the high mass and low surface area), allowing for a significant signal shift from the peptide correlation band (i.e. > 10%). Lanthanide-based shift reagents consist of a metal chelation moiety, a short linker region that may contain a charge enhancing moiety, and a reactive moiety specific for a desired functionality (e.g. maleimide or NHS ester). Since the lanthanides have similar ionic radii, many different metals can be chelated to the metal chelation moiety, allowing for relative quantitation experiments similar in nature to isotope coded affinity tags. Multiplex experiments are also possible using lanthanide-based shift reagents. For example, a peptide containing both a cysteine residue and a primary amine (e.g. N-terminus or Lys residue) can be differentially labeled using two different lanthanide metals. That is, the primary amines may be labeled with a terbium based shift reagents while the cysteine residues on the same peptide are labeled with a holmium based shift reagent. Since these shift reagents remain intact through tandem MS experiments, site identification of the thiol or primary amines is possible. Multiplexed experiments open the possibility of readily determining how many functionalities a peptide has. That is, neither a primary amine nor a cysteine, either a primary amine or a cysteine, or both a primary amine and a cysteine. Recently synthesized charge enhancing shift reagents will be presented as a method to increase MS ionization of peptides that ionize poorly, e.g. phosphopeptides.

(592) **Elucidation of the Binding of Alkanes to Transition Metals Using Quantum Cascade Lasers and Time-resolved Infrared and NMR Spectroscopies**

James A. Calladine[1], Olga Torres[2], Khuong Q. Vuong[1], Steven L. Matthews[2], Simon B. Duckett[2], Robin N. Perutz[2], Michael W. George[1]; [1]The University of Nottingham; [2]The University of York

Organometallic alkane complexes are key intermediates in the C-H activation process.[1] These complexes, however, are usually short-lived at room temperature (100 mW) and ease of operation. Preliminary experiments have demonstrated a capability to observe intermediates on the deltaOD = 10-6 scale. In the second part, the characterisation of two new organometallic alkane complexes of manganese and rhenium using a combination of fast TRIR and low temperature FTIR and NMR spectroscopies is presented. References: [1] C. Hall, R. N. Perutz, Chem. Rev.,1996, 96, 3125 [2] X.-Z. Sun, M. Poliakoff, M. W. George, et al., J. Am. Chem. Soc., 1997, 119, 7521 [3] G.E. Ball, A.J. Cowan, M.W. George, et al., Proc. Natl. Acad. Sci. U.S.A., 2007, 104, 6927 [4] J. A. Calladine and M. W. George, in Spectroscopy Europe, 2009, 21, pp. 6-9

(593) **SPR Signal Amplification with Nanoparticles and *in situ* Polymer Growth**

Q Cheng[1], Ying Liu[1]; [1]Univ of California Riverside

There are a number of biomimic structures for cell membrane and one of the best known systems is the supported bilayer membrane (SBM) on a solid substrate. However, protein detection on supported membranes with SPR has limitations in sensitivity, since common amplification methods are typically not applicable to fragile SBMs. To solve this problem, we have developed a novel signal amplification method for SPR that is performed in an all-aqueous environment. Ultra-sensitive protein detection was achieved via gold nanoparticles (AuNP) coupled with *in-situ* ATRP (atom transfer radical polymerization) reaction. Phosphatidylcholine (PC) vesicles were fused on a calcinated gold chip to form SBM and bacterial cholera toxin (CT) was used as the model protein. CT was captured onto the surface through the membrane receptor (GM1) and further recognized by biotinylated anti-CT, which brings ATRP initiator-functionalized AuNP to the binding site through a neutravidin bridge. The signal amplification was realized by triggering the localized growth of polymer brushes of poly(hydroxyl-ethyl methacrylate) (PHEMA). The resulting polymer film has been characterized by optical and atomic force microscopy. A calibration curve for CT detection has been obtained that displays a dynamic range from 6.3 x 10-16 to 6.3 x 10-8 M with a detection limit of 0.158 fM. This approach is simple and

effective, offering a significant enhancement of the detection sensitivity under mild conditions for SPR methods.

(594) **Localized Surface Plasmon Resonance Response of Surface-Attached Gold Nanoplates to Protein Binding: Effect of Binding Location and Distance**

Francis Zamborini[1], Srinivas Beeram[1]; [1]University of Louisville

In this talk, we will describe the synthesis and purification of gold nanoplates grown directly on surfaces and their localized surface plasmon resonance (LSPR) response to protein binding, focusing on the effect of binding location and distance between the protein and gold nanoplate surface. We synthesized gold nanoplates by seed-mediated growth and purified them by removing spherical gold nanoparticles from the surface by taping or sonicating the sample. Functionalization of the gold nanoplates with carboxylic acid terminated alkanethiol self-assembled monolayers (SAMs) allows attachment of anti-IgG to the gold nanoplate surfaces. With thiol place-exchange reactions, we controlled the attachment of anti-IgG to gold nanoplate edge and vertex sites. The use of different length SAMs leads to control over the distance between anti-IgG and the nanoplates. We correlated the LSPR response of the nanoplates with the binding location, as determined by atomic force microscopy (AFM), and binding distance. Anti-IgG binding to edge and vertex sites led to increased LSPR response relative to those bound to terraces, even with a much lower coverage of anti-IgG on the surface. This is expected based on the fact that the electric-field enhancement is greatest at edge and vertex sites, but has not been measured experimentally before. In addition, binding of proteins to C3 monolayers led to larger LSPR responses compared to C11 and C16, consistent with an exponential decrease in the field with distance from the nanoplates. Gold nanoplates with edge-attached anti-IgG at short distances detected

(595) **Surface Optical Sensing: SPR, Interferometry and Grating Reflection**

Roger Terrill; [1]San Jose State University

The primary and oft-cited advantage of SPR, namely label-free detection, is by no means exclusive to this optical phenomenon. A quantitative comparision of the sensitivity of SPR and comparable label-free sensing methods can shed light on the field of surface optical sensing. The analysis herein attempts to make a coherent comparision between the sensitivity of SPR, Fabry-Perot interferometry and Grating Reflection methods that may be helpful in guiding further development of this type of sensor.

(596) **Development of a Surface Plasmon Resonance Sensor for Monitoring Cytochrome P450 Activity**

Brent Cameron[1], Rui Zheng[1]; [1]University of Toledo

A significant group of enzymes, known as cytochrome P450s (CYPs), are responsible for drug metabolism and bioactivation in the body. Specifically, CYPs play a key role in influencing drug clearance rates, interactions between drugs, and potential toxicity effects. Toxicity effects are of key interest in individuals that may be taking various therapeutic-type pharmaceuticals. Therefore, methods to monitor CYP activity could be used in developing therapeutic drug monitoring technology (e.g. determining optimal individualized dosing protocols), as well as, playing an essential role in determining how new pharmaceutical drugs will be metabolized by certain P450 enzymes. We will be reporting on a novel method which has the potential to provide rapid, specific, and sensitive measurements to allow for immediate assessment of *in vivo* CYP activity. The developed biosensor(s) and corresponding analysis methods allow for the detection of key probe analytes and their metabolites which can be correlated to CYP activity. The technology utilized in our investigation is based on Surface Plasmon Resonance (SPR) which is an optical based sensing approach. Gold (Au) SPR sensing surfaces are functionalized to target key CYP probes and metabolites with custom designed molecularly imprinted polymer (MIPs) thin films. The use of such films significantly improves the sensitivity and specificity of the SPR technique. The developed sensor has the potential to provide a simple, accurate, and low-cost alternative for monitoring CYP activity compared to conventional technologies.

(597) **Direct Detection of Biomarkers in Whole Biological Matrixes Using Ultralow Fouling Peptide SAM and SPR**

Olivier R. Bolduc[1], Joelle N. Pelletier [1, 4], Jean-François Masson [1, 2, 3]; [1]Département de Chimie, Université de Montréal; [2]Center for Self-Assembled Chem. Struct.; [3]Centre for Biorecognition and Biosensors; [4]PROTEO Network for Protein Structure

Low nanomolar concentrations of cancer biomarker were quantified directly in crude bovine serum without any pretreatment or signal amplification techniques using near-zero fouling monolayers based on binary patterned peptides. Proteins contained in whole biological matrixes induce a nonspecific response masking the one due to biomarkers of clinical interest. Development of anti-biofouling surfaces is of primary importance to reach direct detection capability for biosensors reducing the time and human resources needed to obtain front line diagnosis leading to better medical treatments. Polar and ionic amino acids attached to a short thiol (3-MPA, 3-mercaptopropionic acid) forming self-assembled monolayers (SAM) have shown a great potential in reducing nonspecific interactions due to bovine serum proteins. A solid phase synthesis approach allowed the building of short thiolated homo-, binary-patterned and more complex peptides leading to nonspecific interaction level as low as 10 ng/cm² while the most commonly used surfaces PEG and CM-Dextran respectively lead to 100 ng/cm² and 800 ng/cm². Peptide based surfaces were characterized using SPR, FT-IR, contact angle measurement, circular dichroism to determine their secondary structures and hydrophilicity. Quantifications of beta-lactamase (LOD: 10nM) and matrix metalloproteinase-3 (MMP-3, LOD: 0.14nM) were accomplished in crude cell lysate (total protein: 20 mg/mL) and whole bovine serum (total proteins: 80 mg/mL) respectively.

(598) **New Tools for Surface Science: Robust Multifunctional Chemically Patterned Amorphous Carbon Substrates**

Stephen Weibel; [1]GWC Technologies, Inc.

Biosensor and chemical and biological arrays employing planar substrates typically use glass surfaces with silane linkages and gold surfaces with thiol bond linkage. These surfaces (both commercial sourced and laboratory fabricated) generally are functionalized with a single chemical species. We will demonstrate new strategies for creating low-cost multi-functional patterned surfaces using amorphous carbon thin films and UV-mediated chemical attachment. The carbon layer presents a new interface for chemically patterned surfaces and provides flexibility for attaching different chemistries in varied spatial geometries using flexible optical patterning masks. Multi-functional patterned surfaces with C=C bond attachment to the surface will be shown for carboxyl, hydroxyl, ethylene glycol, fluorous, and amine terminated surfaces. These surfaces are compatible with both surface plasmon resonance and infrared external reflection techniques. PM-IRRAS and SPR are utilized to characterize the surface chemistry and demonstrate desired functionality.

(599) **Indicator for Flagging Matrix Effects in Axial-Viewing Mode Inductively Coupled Plasma–Atomic Emission Spectrometry**

George Chan[1], Gary Hieftje[1]; [1]Indiana University

It is well established that the inductively coupled plasma (ICP) operated in axial-viewing mode offers better atomic emission (AES) sensitivities and detection limits than in the conventional radial-viewing mode, but that it is also more prone to matrix interferences. Clearly, reducing interferences would be a desirable advancement. To date, this goal of interference-free analysis is not achievable; it is therefore crucial to have indicators that can successfully flag the presence of a matrix effect so immediate remedial work can be undertaken. Previously, we have developed a simple all-in-one indicator that is effective to flag the presence of matrix interferences originating from any one of the three matrix-effect categories (spectral interferences, sample-introduction-related and plasma-related) in radial-viewing ICP–AES. The applicability of this indicator for axial-viewing ICP–AES is investigated in the present study. In this presentation, the spatial characteristics of matrix interferences in axial-viewing mode will be described, the theoretical basis of this matrix-effect indicator will be discussed, and its effectiveness will be evaluated.

(600) **Tungsten Coil Atomic Emission Spectrometry**

Brad Jones[1]; [1]Wake Forest University

This talk describes a novel technique for the detection of trace amounts of metals in environmental samples. The portable atomic emission spectrometer uses a simple tungsten coil atomizer extracted from an inexpensive light bulb. A twenty microliter sample aliquot is deposited onto the coil with a micropipette. The sample is dried by passing a small current through the resistively heated tungsten filament. The constant current supply is solid state and can be powered by a 12 V battery. Once the solvent has been removed, the sample residue is vaporized at high temperature by applying full power to the filament (150 W). Simultaneous multi-element analyses are possible by detecting the light emitted by the sample using a portable CCD spectrometer. Typical detections are at the part per billion level. The device is small, rugged and portable. There are no moving parts, and the components are mounted on an aluminum plate measuring less than one foot across. Instrument design, analytical figures of merit, and potential applications will be discussed.

(601) **ICP Signals: Model and Experimental Results**

Josh Dettman[1], John Olesik[1]; [1]The Ohio State University

The goal of this research is to use one line from one element in a single standard to semi-quantitatively determine the concentrations of all 70 elements measurable by ICP-OES. Unlike ICPMS, ICP-OES sensitivities may vary over tens of thousands of times for analytically useful emission lines dependent on element, line, and temperature. We will test a partial local thermodynamic equilibrium (pLTE) model for determining relative sensitivities in a variety of matrices. A practical model can only require inputs measurable on a commercial ICP-OES instrument. Temperatures estimated from different ion/atom emission intensity ratios using the pLTE model are 8500±300 K. Concentrations based on the pLTE model and one experimentally measured line in one standard differ by a factor of ~3 from the correct value. Matrix effects that would induce a 6x error in concentrations can be corrected to within 3x using the pLTE model. Accuracy of traditional calibration can be improved using the model to estimate approximate concentrations of inorganic matrix elements for preparing matrix matched standards and by adjusting experimental intensities for differences between standard and sample plasma temperatures. In this presentation temperatures and semi-quantitative concentrations using the pLTE model will be assessed. Limitations of the model, including assumptions made and the availability of fundamental data will be considered. The accuracy of concentration results from the pLTE model will be compared with using sensitivities measured from standards once over months without accounting for possible variations in plasma conditions or matrix effects.

(602) **Optical and Mass Spectrometric Studies of a Helium Dielectric Barrier Discharge used as an Ambient Ionization Source**

Matthew Heywood[1], Jonathan Wright[1], Paul Farnsworth[1]; [1]Brigham Young University

Plasma sources have been shown to be fast and effective ionization sources in the quickly developing field of ambient ionization-mass spectrometry. Of these sources, the low temperature plasma (LTP), direct analysis in real time (DART), and the flowing atmospheric pressure afterglow (FAPA) have received considerable attention. In our lab, we have conducted experiments with a dielectric barrier discharge that shares some traits with these three sources, but also exhibits some significant differences. We are conducting experiments to characterize our source with optical, mass spectrometric, and physical measurements in an effort to distinguish fundamental differences among the different sources from superficial differences that reflect changes in operating conditions and physical orientation of the source. We will present a detailed characterization of the dielectric barrier discharge source, including microscopic imaging to track sample removal from a test surface, and simultaneous emission spectrometric and mass spectrometric measurement to correlate changes in optical properties of the plasma with changes in its performance as an ion source. Similar measurements with a FAPA source will serve as benchmarks.

(603) **Atmospheric Desorption and Detection of Organic Compounds by Atmospheric Pressure Glow Discharge Mass Spectrometry (APGD-MS)**

Tim M. Brewer[1], Marcela Najarro[1], Jennifer Verkouteren[1], Greg Gillen[1]; [1]NIST

The detection of illicit drugs and trace explosives represent one the most significant challenges for law enforcement and forensic communities. Particular interest to the forensic analyst is the ability to rapidly identify suspected illicit drug materials and explosives residues in their native state (powder, tablet or liquid form), under atmospheric conditions and with a high level of specificity and sensitivity. Atmospheric pressure glow discharge mass spectrometry (APGD-MS) can remove and ionize materials from any sample surface, including human skin, liquid and gas matrices under atmospheric conditions. Here, APGD-MS is used to analyze two classes of organic compounds, 1) a series of over-the-counter (OTC) drug formulations (Tylenol AllergyTM, Alka-Seltzer NighttimeTM, SudafedTM, AleveTM and Mucinex MDTM), illicit drugs (crack cocaine, methamphetamine, MDMA and hydrocodone), as well as the agents used for cutting (i.e. filler and diluents) the illicit drugs, and 2) explosive residues in all three states of matter; liquid, solid and gas. For the drugs, the active ingredients in the OTC medications are readily distinguished from tablets containing controlled substances. Bulk materials, including tablets, are sampled simply by allowing the plasma to come in contact with the solid substance. Characteristic molecular ion mass spectra were identified for the active ingredients in OTC and illicit drugs. All drugs studied produced protonated ion signals as well as fragmentation patterns that are EI-NIST database searchable. For explosives analyses, a 100 ug/mL aqueous solution of TNT, solid bulk C4 samples and a flowing gas stream of 100 ng/mL of TNT, RDX and PETN are analyzed by allowing the plasma to come in direct contact with each analyte in their respective states of matter.

As with the drugs, all cases demonstrated characteristic molecular ion spectra for the explosives. In the case of the C4 solid samples the binders and plasticizers were also readily identified, producing characteristic ion signals as well as fragmentation patterns that are EI-NIST database searchable. These results highlight the techniques ability to detect multiple species at once in atmospheric pressure without a chromatographic separation in any state of matter.

(604) **Examinations and Improvements in Low-Temperature Plasma (LTP) Ambient Desorption/Ionization Mass Spectrometry**

Joshua Wiley[1], Jacob Shelley[2], Ayanna Jackson[1], Gary Hieftje[2], R. Graham Cooks[1]; [1]Dept. of Chemistry - Purdue University; [2]Dept. of Chemistry - Indiana University

Low-temperature plasma (LTP) probe, based on a dielectric barrier discharge, was recently introduced as an ambient desorption/ionization source for mass spectrometry (MS). In conventional operation, interaction of the plasma plume with a sample surface results in direct desorption and ionization of analytes for mass spectrometric detection. The non-destructive nature and mild operating conditions of the LTP probe enable analysis of delicate samples, such as a human finger. However, it has been found that some analytes, such as those with extremely low vapor pressures, can often be difficult to desorb, which ultimately limits the versatility of LTP-MS. The studies within have focused on fundamental investigations with the use of optical and mass spectrometric techniques to enhance performance. With the help of these fundamental examinations, improved unique discharge-cell geometries and optimized plasma conditions have emerged. For example, miniaturization of the plasma probe has demonstrated improved desorption capabilities for some analytes while minimizing power and gas consumption, potentially leading to implementation on field-portable mass spectrometers. LTP miniaturization has also yielded a more practical approach to ambient MS imaging due to enhanced spatial confinement of the excited species emanating from the plasma plume when compared with conventional LTP. All efforts of increasing the overall analytical performance of LTP-MS have been made with hopes of maintaining the inherent non-destructive nature of the technique.

(605) **Enhanced Chemical Synthesis and Scale-Up in Micro Reactors**

Paul Watts; [1]University of Hull

Throughout the numerous stages of product development there are many associated risks, potentially none as costly as the failure to scale a synthetic process to achieve the required production capacity. Micro reactor technology (MRT) is an emerging technique that enables those working in research and development to rapidly screen reactions utilising continuous flow, leading to the identification of reaction conditions that are suitable for use at a production level. Furthermore, in addition to using conventional reaction methodology, the inherent safety associated with the use of small reactor volumes enables users to employ reaction conditions previously thought to be too hazardous for use within a production environment; such as extreme reaction conditions or the use/generation of 'hazardous' compounds. Consequently, the types of reactions available to the R&D chemist increases through the use of this technology. Compared to stirred reactor methodology, the benefits of MRT are acknowledged to be: Increased reaction control, Efficient mixing, Accurate control of reaction time, temperature and pressure, Increased run-to-run and reactor-to-reactor reproducibility , Improved atom efficiency, product selectivity, yield and purity, Increased catalyst turnover and lifetimes Increased process safety. Due to rapid dissipation of heat of reaction, Low reactant hold-up, Real-time *in-situ* analytical evaluation of reactions Lower cost and shorter development cycles, Higher chemical selectivity leading to higher yield, Reducing the amount of reagents and catalyst, Reducing the size of the plant, Faster scale-up from lab- to plant scale It is this system flexibility that has the potential to reduce both the time taken and risk associated with transferring reaction methodology from research to production. In this talk, we will present an overview of chemical reactions conducted in these reactors. Furthermore an outlook into integrated analysis will be given.

(606) **Designing Sustainable Chemical Systems in a Process Intensified Environment**

Michael Gonzalez[1]; [1]United States Environmental Protection Agency

As the importance of increasing the sustainability of a chemical synthesis or processes becomes increasingly evident, it is necessary to ensure that research undertaken as well as applied in practice is of increased sustainability with respect to its predecessor. As demonstrated in the literature and in application, there is a growing awareness of the necessity to merge disciplines and knowledge when tackling a new chemical synthesis or making improvements to an existing process. In order to achieve this, chemists and chemical engineers are now working in tandem to apply their respective principles and theories when designing and implementing a new research project. The research to be presented will highlight the use and application of Green Chemistry principles and philosophy, the use of process intensification to achieve the goal of a minimized environmental and physical footprint, faster and more accurate process optimization using *in-situ* real-time and within process analytical analysis and the development and application of metrics to measure the improvements to the process in terms of sustainability.

(607) **Benefits of On-line Sensors for Advanced Flow Reactor Analysis, Optimization and Control**

Brian Marquardt[1], Wesley Thompson[1], Thomas Dearing[1]; [1]University of Washington

Process Analytical Technology (PAT) has been used by various industries for gathering data to develop and monitor processes. It has proven a valuable tool to optimize productivity and quality for decades, and recent advances in analytical sensors and sampling technology have continued to improve measurement capabilities. New manufacturing and materials developments have allowed for advances in the miniaturization of instrumentation and sensors. These advances are apparent in the new continuous flow reaction equipment and analytical tools available today. Many micro-analytical developments have been focused on the need for measurement tools to support the growing use of micro-systems. These systems have been used predominantly for high throughput experimentation, a widely used technology for new material discovery. This approach to working with a large number of reactions and achieving fast results has been recently broadened to include process development, process optimization, and product development activities. Recently a program was initiated to develop and optimize new chemistries using advanced flow reactors, analytical measurement tools and NeSSI (New Sampling and Sensor Initiative) sampling components. The program is a collaboration between the Food and Drug Administration (FDA), Corning, Kaiser Optical Systems, Parker and CPAC (Center for Process Analytical Chemistry) to demonstrate the value of the advanced flow platform in conjunction with micro-analytical tools and modular sampling systems components to improve process understanding and control. Results from recent experiments will be presented. Results from initial experiments show significant progress towards the ultimate goal of Process Understanding and a future path towards control.

(608) **Ultrasonically Levitated Microreactors: Continuously Stirred or Shaken?**

Rachel Behrens[1], Alexander Scheeline[1]; [1]University of Illinois at CU

Microreactors are desirable for initial exploration of chemical processes, as reactant consumption is minimal and safety most easily managed. Scale-up may be challenging, as the surface area to volume ratio drops as volume increases (typically ~ cube root of volume). Passivation of reactor walls may limit the extent of such complications, but if free radical reactions occur such passivation may be impossible. For example, bovine serum albumin, a common biochemical passivator, is reactive to radicals. Thus, we have been developing ultrasonically-levitated drops as microreactors either free of solid surfaces, or with limited solid/liquid interfaces where capillaries feed or drain the drop or where electrochemical sensors sense drop contents. Convective mixing accelerates drop homogenization. We discuss fluidics and sensing in these systems.

(609) **Microreactors for the Large Scale Manufacture of Life Science Compounds**

David Ager[1]; [1]DSM

DSM employs microreactor technology and flow chemistry for the preparation of a number of Life Science intermediates and products. The emphasis is on process intensification especially where reactions would be hazardous to perform in batch mode. Case studies will be presented: The first is for a very exothermic reaction used in the manufacture of an agrochemical intermediate. A laboratory scale reactor was used to determine the process parameters. Scale up involved numbering up the microreactor channels. A smaller scale method that uses in line analysis for the preparation of amines will also be illustrated. The final example involves cGMP manufacture of a drug candidate where formation of a nitrate ester is the key step. This process involves continuous separation, which has proven useful in other flow reactions even when microreactors are not used. IR is used to monitor the reaction.

(610) **Synthesis in Microreactors: From Materials to Small Molecules**

D. Tyler McQuade[1]; [1]Florida State University

The McQuade Group's mission is to develop new synthetic methods and techniques with the goal of creating both faster and more efficient routes to molecules of commercial interest using catalysts, microreactors or a combination of both. The FACSS seminar will present an overview of the McQuade Group's work in the field of microreactors, beginning with a brief examination of the unique features offered by microreactors and the positive impact these features can have on the preparation of a variety of important molecules. We will demonstrate how to create highly effective microreactors from simple tubing and syringe pumps and then illustrate how this simple set-up can be applied to a wide spectrum of chemical reactions. This seminar will highlight the many benefits of using these simple microreactors to prepare polymeric materials, run reactions that produce slurries, create packed-bed microreactors, and develop continuous syntheses.

(611) **Mapping Contact Surfaces in Protein Complexes by Solution-Phase H/D Exchange Monitored by Ultrahigh Resolution FT-ICR Mass Spectrometry**

Alan Marshall[1], Greg Blakney[2], George Bou-Assaf[1], Mark Emmett[2], Christopher Hendrickson[2], Santosh Valeja[1], Hui-Min Zhang[2], Qian Zhang[1], Alan Marshall, Alan Marshall; [1]Florida State University; [2]Nat'l High Magnetic Field Laboratory

The rates of hydrogen/deuterium exchange of protein backbone amide hydrogens reflect solvent accessibility, and thus afford a probe of the site(s) and structural changes induced by binding of enzyme inhibitors and drugs. Compared to NMR monitoring of H/D exchange, mass spectrometric detection has lower primary amino acid sequence resolution, but offers much higher concentration sensitivity, and can be applied to proteins too large or too insoluble for NMR. Representative applications include: (a) validation of whether or not a truncated "domain" of a large enzyme accurately represents the behavior of the intact enzyme (wild-type and drug-induced mutants of tyrosine kinase, and its complexes with imatinib and sunitinib anti-cancer drugs); (b) validation of the use of bacterially expressed protein (i.e., no post-translational modifications) to represent a highly glycosylated mammalian enzyme, the receptor for advanced glycation endproducts; and (c) exposition of the allosteric mechanism of L-leucine feedback inhibition of á-isopropylmalate synthase (the rate-limiting step in leucine synthesis by Mycobacterium tuberculosis, and thus a drug target for TB therapy). Work supported by NIH GM-78359 and NSF Division of Materials Research through DMR-06-54118 and the State of Florida.

(612) **Mass-Spectrometry-Based Hydrogen/Deuterium Exchange, PLIMSTEX, and Protein Digestion for Elucidating Metal Binding in Proteins**

Michael L Gross[1], Richard Huang[1]; [1]Washington Univ in St Louis

Troponin C (TnC) is a muscle protein that acts as a Ca(II)-activated trigger initiating myocyte contraction. The binding of Ca(II) to TnC initiates a cascade of conformational changes involving the constituent proteins of the muscle thin filament. The binding of TnC to Ca(II) has significant regulatory influence on muscle contraction. Although we know that troponin binds four Ca(II) ions and can identify the binding sites, we don't know which of the four binding sites binds the first Ca, which binds the second Ca, etc. Here we report the implementation of PLIMSTEX (Protein Ligand Interaction by Mass Spectrometry, Titration and H/D Exchange) to elucidate the binding affinity of TnC with Ca(II), to determine the conformational changes upon binding of Ca(II), and to elucidate the binding order. We are able to confirm the stoichiometry for the calcium interaction of the protein at high concentration (4-10 micromolar) to be four Ca(II) per mole of protein. A PLIMSTEX Ca(II) titration at low TnC concentration (~ 0.10 micromolar) affords the four binding constants of TnC with Ca(II). From the equilibrium constants, we can calculate fractional species as a function of the analytical concentration of Ca(II). These calculations are the basis for a new set of experiments with HDX whereby we digest with pepsin, separate by UPLC, and analyze by MS the relevant four peptides, each of which contains one binding site. Examining the deuterium distributions of the peptides at a given [Ca(II)] and knowing the fractional species, we can determine how each binding site's deuterium distribution changes as the fractional species changes. The outcome provides an answer to the question of which site binds the first Ca, which binds the second, etc. Overall, we are able to determine in a set of HDX and MS experiments the stoichiometry of binding, the binding constants, and the order of binding with respect to the various binding sites. The outcome offers hope that the order of binding and the stoichiometry of metal binding to other proteins can be rapidly and sensitively determined by an application of PLIMSTEX, HDX, and mass spectrometry.

(613) **SUPREX: An H/D Exchange and Mass Spectrometry-Based Protein-Ligand Binding Assay with a High Throughput Capability**

Michael C. Fitzgerald[1]; [1]Duke University

SUPREX, (stability of unpurified proteins from rates of H/D exchange), is an H/D exchange- and mass spectroscopy-based method for evaluating the free energy and cooperativity of protein folding reactions. In SUPREX, the denaturant dependence of a protein's amide H/D exchange reaction is determined and used to -

quantify the cooperativity and free energy associated a protein's global unfolding/refolding reaction. The evaluation of a protein's folding free energy in the absence and in the presence of a ligand is ultimately used to generate thermodynamic information about the strength the protein-ligand binding interaction. Two application of SUPREX will be discussed. One application of SUPREX to be discussed will be our work using the technique to study the binding interaction between the molecular chaperone Hsp33 and four different unfolded protein substrates including citrate synthase, lactate dehydrogenase, malate dehydrogenase, and aldolase. In this work an increase in the cooperativity of the Hsp33 folding/unfolding reaction upon binding with the denatured protein substrates was detected in the SUPREX experiment. This is consistent with the burial of significant hydrophobic surface area in Hsp33 when it interacts with its substrate proteins. Dissociated constants for Hsp33 complexed with each of the four unfolded substrates were evaluated and all found to be within a range of 3-300 nM, a range similar to that found for other protein chaperone substrate interactions. One advantage of SUPREX over other H/D exchange and mass spectrometry-based protocols for characterizing protein-ligand binding interactions is the speed at which analyses can be performed. This makes the technique especially well-suited for use in high throughput screening projects were the goal is to identify novel ligands to a target protein. The second application to be discussed in this presentation will be our work using SUPREX to screen the compounds in several chemical libraries for binding to cyclophilin A (CypA). CypA is an overexpressed protein in lung cancer tumors and as a result is a potential therapeutic and diagnostic target. The throughput and efficiency of the screens that we have conducted will be discussed, along with the novel ligands we have discovered.

(614) **Mass Shift Perturbation Methods for Structural Proteomics**

David Schriemer[1], Andrew Percy[1]; [1]University of Calgary

The proteome is a dynamic and responsive entity at virtually every level of its definition, which suggests that its analysis will never truly be complete. Increasingly, our attention must shift to the characterization of its perturbation on a level beyond composition, as a means of gaining greater insights on complex cellular functions. This characterization includes high resolution analysis of structure and dynamics. While such activities are not typically associated with the tools of proteomics, there is a strong role for the mass spectrometer in this area. In a fashion conceptually related to NMR spectroscopy, restraints can be derived using mass spectrometry to support structure/function mapping of proteins, especially their complexes. In this talk, I will present new concepts in measuring restraint data by hydrogen/deuterium exchange mass spectrometry, which we broadly define as mass shift perturbation (MSP) methods. I will describe how MSP measurements can be made using triple-quadrupole instruments operated in scheduled-MRM mode, enabling exchange measurements of unprecedented speed and sensitivity. Using this "MSP by MRM" approach, several applications in structure/function analysis will be presented, including drug lead evaluation, mapping of protein mechanisms, and protein structure modeling. Overall, it will be demonstrated that MSP methods provide complementary data to conventional structural tools such as NMR and x-ray crystallography.

(615) **The Utility of H/D-Exchange for Epitope Screening to Assist with Biopharmaceutical Candidate mAb Selection**

Jennifer F. Nemeth[1], Seng-Jiun Wu[1], Steve Tuske[2], Yoshi Hamuro[2]; [1]Centocor Research and Development; [2]ExSAR Corporation

Hydrogen/deuterium-exchange (H/D-Ex) coupled with mass spectrometry is having an impact in the biopharmaceutical arena in the area of epitope mapping and lead candidate selection. This technology, which is orthogonal to other accepted epitope mapping techniques, provides an assessment of a molecule's solvent accessibility under different experimental conditions. An example of the utility of this technology is highlighted during lead selection in a monoclonal antibody (mAb) drug program. A number of factors are initially evaluated as a means for identifying candidates for further development including biological activity, ease of development, structural attributes, protein heterogeneity, and intellectual property. Most of the results showed that the mAbs were similar, so additional data was needed to provide more specificity. An assessment of the binding epitopes was proposed as a means for choosing a mAb with a unique epitope to the antigen for improved exclusivity in the biopharmaceutical drug space. As the timelines and sample amounts were limited, hydrogen/deuterium-exchange (H/D-Ex) was chosen for mapping out the epitopes on the target antigen, as opposed to more traditional techniques. Hydrogen/deuterium-exchange proved to be a fast and efficient means for screening the leads, as well as a small panel of commercially available mAbs. Based on the results, lead and back-up mAbs were selected that had desirable epitopes for further product development.

(616) **Ligand Screening with Hydrogen Deuterium Exchange Mass Spectrometry**

Michael Chalmers[1], Jun Zhang[2], Rachelle Landgraf[1], Graham West[1], Janelle Lauer[1], Scott Novick[1], Scooter Willis[1], Bruce Pascal[1], Scott Busby[1], Patrick Griffin[1]; [1]TSRI

In this work we describe the use of HDX MS to characterize protein-ligand and protein-protein interactions to aid the understanding of the mechanism of action of protein kinases and nuclear receptors. In each case HDX was able to provide new insights into the determinants that regulate these signaling proteins. Differential HDX experiments were conducted with an automated HDX system utilizing a LEAP HTS twin PAL robot interfaced with an ESI Orbitrap mass spectrometer. Data analysis was performed with in-house developed software (HD Desktop). Here we describe and validate the required data analysis workflow to facilitate the use of HDX as a robust approach for ligand screening. Following acquisition of HDX data at a single on-exchange time point ($n > 3$), one way analysis of variance in conjunction with the Tukey multiple comparison procedure is used to establish the significance of any measured difference. Analysis results are graphed with respect to a single peptide, ligand or group of ligands, or displayed as an overview within a heat map. Hierarchical clustering is used to bin compounds with highly similar HDX signatures. The workflow is evaluated with a data set showing the ligand binding domain (LDB) of the nuclear receptor peroxisome proliferator-activated receptor gamma (PPARg) screened against 10 functionally selective ligands. A significantly larger dataset comprised of 127 differential HDX experiments was then used to determine the precision of our automated system over a period of eight months. From a total of 4191 HDX measurements, over 96% of the data points were within 10% of their mean values (n=127). The workflow has been applied to further our understanding of the mechanism of action of the retinoid related receptor (ROR), PPARg and protein kinases.

(617) **Expanding Versatility of SERS with Construction of Various Nanostructures**

Zhong-Qun Tian[1], Jiang-Feng Li[1], Yi-Fan Huang[1], Zheng Liu[1], Zhi-Lin Yang[2], Bin Ren[1]; [1]Chemistry Department, Xiamen University; [2]Physics Department, Xiamen University

Surface-enhanced Raman scattering (SERS) stems from surface plasmon resonance (SPR) which takes place on various nanostructures with suitable dielectric constant, shape and scale [1].

It had been very difficult to apply conventional SERS to study probed molecules adsorbed at atomically-flat single-crystal surfaces and surface components of diverse materials because they cannot effectively support SPR. Based on the borrowing SERS activity strategy, we have utilized some nanostructures to expand SERS studies to Pt and Si single crystal surfaces and various molecules adsorbed on surfaces as diverse as those of platinum, yeast cells or citrus fruits [2]. The latest progress made in our group is a new method named as Shelled-Isolated Nanoparticle-Enhanced Raman Spectroscopy (SHINERS) [2]. We chemically synthesized Au nanoparticles coated with ultra-thin shells (ca. two to four nanometers) of chemically inert silica and alumina, respectively. About monolayer of such nanoparticles is spread over the surface that is to be probed. The ultrathin coating keeps the nanoparticles from agglomerating, separates them from direct contact with the probed substance and allows the nanoparticles to conform to different contours of substrates. High-quality Raman spectra were obtained on various molecules adsorbed at Pt and Au single-crystal surfaces and from Si surfaces with hydrogen monolayers. These measurements and our studies on yeast cells and citrus fruits with pesticide residues illustrate that our method significantly expands the flexibility of SERS for wide applications in surface, materials and life sciences, as well as for the inspection of food safety. Finally, further developments of SERS will be briefly discussed with emphasis on the structural study on a single molecule junction for correlating binary quantum conductance of molecular electronics. References: 1. M. Moskovits, J. Chem. Phys., 1978, 69, 4159; Rev. Mod. Phys., 1985, 57, 783; J. Raman Spectrosc. 2005, 36, 485. 2. J.F. Li, et. al., Nature 2010, 464, 392.

(618) **Pathogen Detection with Plasmonic and Superparamagnetic Nanoparticles**

Li-Lin Tay[1], Peilin Chen[2], John Hulse[1], Jamshid Tanha[1]; [1]National Research Council Canada; [2]Academia Sinica, Taipei, Taiwan

The plasmon resonance of gold nanoparticles (NP) manifests itself in a variety of extraordinary optical properties. In particular, resonant excitation of the conduction electrons by incident radiation generates a localized surface plasmon resonance (LSPR) that is responsible for a variety of surfaced enhanced optical phenomena. This unique optical property coupled with the well established surface chemistry of gold allows one to utilize Au NPs as target specific optical labels to probe and monitor microorganisms. In this study, we demonstrate a rapid and ultrasensitive assay that employees the antibody-labeled plasmonic and superparamagnetic nanoparticles in a core-shell structure for the detection of Staphylococcus aureus and Salmonella typhimurium. While the silver shell provides the plasmonic transduction element, the superparamagnetic nature of the core enables a rapid magnetic pre-concentration and purification of the bacterial samples which ultimately leads to an increase in the detection sensitivity. The core-shell plasmonic magnetic nanoparticles are co-labeled with a benzenethiol Raman reporter molecule and a target specific antibody. The particles are exposed to the pathogen and samples isolated by magnetic field. Detection of the concentrated bacterial species is achieved with the surface enhanced Raman spectroscopy (SERS) and light scattering techniques. In this presentation, we will outline the different chemistries involved in the nanoparticle-antibody conjugation and discuss the optical detection limit. In a pre-concentrated and isolated bacterial sample, SERS detection of a single bacterium can be easily achieved.

(619) **Biomolecule Sensing with Adaptive Plasmonic Nanostructures**

Vladimir Shalaev[1], Vladimir Drachev[1]; [1]Birck Nanotechnology Center, Purdue University

One of the challenges of biomolecule sensing with surface-enhanced Raman scattering (SERS) is to preserve all of the advantages of Raman spectroscopy applications for structural biology. We demonstrate the appropriateness of nanostructured adaptive silver films (ASFs) to enhance the strength of Raman spectroscopy in biological applications. The restructuring under biomolecule deposition allows one to match the metal-particle aggregate geometry with large molecules such that the conformation and functionality are preserved. This produces excellent conditions for SERS enhancement. Note here that there is a promising way to improve further the sensitivity of SERS-based biosensors by including a bulk metal layer as a sublayer between the adaptive silver film and a glass substrate. The interaction of the silver film with the biomolecule solution acts to stabilize the system, making it applicable for all possible protocols for bioarray treatment and detection. The most straightforward application of SERS in immunoassays appears to be the active signaling reporter for binding reactions. However, we believe the most attractive benefits would be in the direct detection of Raman features of protein–ligand interaction, including a follow-up comprehensive analysis of the vibrational fingerprints.

(620) **Surface-Enhanced Resonance Raman Scattering Imaging with Langmuir-Blodgett Monolayers**

Ricardo Aroca[1], Golam Moula[1], Nicholas Pieczonka[1]; [1]University of Windsor

The potential and advantages of the 2D structures to explore the spectral properties in the transition from the average SERRS spectra to the single molecule regime are demonstrated with a large amount of data collected for a single Langmuir-Blodgett (LB) on silver island nanostructures (SIF). The chemical images created with the SERS/SERRS data provide a mapping of the enhancement factor distribution on the surface coated with an LB containing the probe molecule. The collection of large SERRS maps permits to discuss the strongly interconnected nature of the inherent Raman cross section, the enhancement factors and the occurrence of SM events on silver island films covered with a monomolecular layer of the target material. In addition, the micrometer resolution of Raman-microscopy and the spectral acquisition via fast mapping techniques, permit the development of “plasmonic writing”, where the laser induced inscription formed by surface reactions in the LB coated silver island film can be revealed as chemical images constructed using the SERRS mapping. In this lecture several examples of the high resolution inscription produced by induced photoreactions in a single LB monolayer are discussed.

(621) **Surface-Enhanced Spectroscopies of Biomolecules**

Naomi Halas; [1]Rice University

One of the major challenges and goals of surface enhanced spectroscopic sensing is the identification of biomolecules and biomolecular complexes and their properties. A facilitating aspect is substrate design: by controlling the properties of the SERS substrate in a reproducible and reliable manner, one can focus instead on complex molecular properties and conformations. Nanoshell-based SERS substrates have provided such a platform, and have enabled us to examine the SERS of peptides, DNA, DNA aptamers, and lipid-based molecular complexes in detail. The identification of endogenous SERS markers in biomolecules provides useful strategies for label-free molecular detection, and extending SERS to infrared wavelengths and other spectroscopic modalities holds promise for the identification of molecular unknowns.

(622) **New Panel for SERS-Based Screening of Influenza Viral Nucleoproteins Using Anti-Influenza Aptamer**

Pierre Negri[1], Richard A. Dluhy[1]; [1]University of Georgia - Department of Chemistry

We have developed a highly sensitive aptamer-modified nanobiosensor consisting of a disulfide-terminated anti-influenza aptamer immobilized on the surface of an aligned silver nanorod array that facilitated binding of the aptamer to the nucleoproteins of specific Influenza vaccine strains. The binding affinity of the viral target- aptamer complex was probed using surface-enhanced Raman spectroscopy (SERS) and the spectral signature due to their highly selective interaction was differentiated from that of the immobilized aptamer alone based on their intrinsic SERS spectra. Partial least squares-discriminate analysis (PLS-DA) was used to establish statistically significant differences between SERS spectra and was further used to depict and identify specific binding of the aptamer to its viral target. This highly adaptable bioassay platform shows potential application for biomedical sensing as a diagnostic biosensor as well as in the development of antiviral agents and their bio-recognition with pathogen targets. Additionally, this novel aptamer-modified array provides a new panel for SERS-based screening of Influenza virus allowing specific, selective and sensitive detection and is to this day the first direct evidence of the use of aptamer-modified SERS substrates as diagnostic tools for virus detection in complex biological media.

(623) ***In situ* Investigation of the Structure of the RPLC Stationary Phase with Sum Frequency Spectroscopy**

Arthur Quast[1], Alexander Curtis[1], Anthony Peterson[1], Steven Goates[1], James Patterson[1]; [1]Brigham Young University

Reversed-phase liquid chromatography (RPLC) is widely used as a separation technique, however the molecular level interactions that control chromatographic retention are not completely understood. Previous researchers have studied the effects of various mobile phase compositions on the structure of the stationary phase at ambient pressure. Others investigated changes in the structure of the stationary phase following, but not during, exposure to typical mobile phase mixtures at elevated pressures. We have used vibrationally resonant sum-frequency generation (VR-SFG) spectroscopy to study the effects of sample storage conditions, pressure, and mobile phase composition on the structure of polymeric octadecyltrichlorosilane (OTS) model stationary phases. Samples were prepared by coating fused silica windows with OTS by liquid phase deposition. Following storage in a solvent of choice, the samples were placed into a high-pressure sample chamber filled with a common mobile phase solvent to observe the effects of pressure on stationary phase structure *in situ*. Following storage in methanol, a stationary phase that was exposed to water at ambient pressure exhibited structural changes over several hours. At 900 psi, however, the applied pressure inhibited the structural changes. At no time did immersion in water lead to a complete collapse of the stationary phase, either at ambient or elevated pressure. We have also probed the response of the stationary phase to methanol/water mixtures at ambient and 900 psi hydrostatic pressure. To our knowledge, this represents the first work of this kind at the elevated pressures typically encountered on an RPLC column. This research effort will help us to establish links between changes in stationary phase structure and chromatographic retention.

(624) **Effect of Mobile Phase Modifiers on Chromatography and Negative Ion Electrospray Response: A Case Study with Flavonoids and Phenolic Acids**

Christine Hughey[1], Bruce Wilcox[1], Carina Minardi[2], Crisand Anderson[2]; [1]James Madison University; [2]Chapman University

Negative ion electrospray (ESI) is widely used to ionize acidic compounds for mass spectrometric detection, yet the mechanisms involved in ionization are not well understood due to the many variables that affect molecule-to-ion conversion. Here we focus on the effect of solution composition and analyte chemistry in hopes of gaining insight into how negative ions are most effectively formed in the ESI source under commonly used LC gradient elution conditions. The ESI response of five flavonoids and three phenolic acids, which ranged in acidity and polarity, was measured over four orders of concentration with an Agilent 6460 triple quadrupole mass spectrometer operated in dynamic MRM mode. The eluent of one of two binary LC pumps was directed through an Eclipse Plus C18 column using a ten port valve. Pump 1, with mobile phases of water with 26.5mM (0.1% v/v) formic acid and acetonitrile with 26.5 mM formic acid, was used as the control. Changes in peak area, height, width, symmetry and retention time for each compound upon addition of a new modifier were calculated relative to the control. We found that formic acid and acetic acid produced similar chromatographic results (e.g., similar retention times and peak shapes). The ammonium salts, however, decreased the retention of early-eluting compounds and caused significant tailing of late-eluting compounds. The effect of the modifiers on the negative ion ESI response was class-dependent. The acids exhibited the highest response with acetic acid. At 2x10-6M the response of vanillic acid in acetic acid was ~10-15X higher than with the other modifiers. The phenolic compounds (both early and last eluters) exhibited a greater response with the ammonium salts. In general, the response of all analytes increased with a decrease in modifier concentration (e.g., 8 mM and 13.25 mM were higher than 26.5 mM). In fact, unbuffered solutions produced the highest ESI response but poor chromatography. In future work, we will conduct flow injection experiments to see if the more basic acetate ion is responsible for the increased ESI response of the acids.

(625) **The Development of an Interpolation-Based Approach to the Alignment of Fast LCxLC-DAD Chromatograms**

Robert Allen[1], Sarah Rutan[1]; [1]Virginia Commonwealth University

The use of parallel factor (PARAFAC) analysis to analyze local regions of comprehensive two dimensional liquid chromatography (LCxLC) diode array detector (DAD) chromatograms allows for the direct quantification of analytes present across multiple samples. However, retention time shifts in both the first and second chromatographic dimensions results in PARAFAC producing poor results due to the lack of multilinearity present in the data set. In order to align LCxLC-DAD chromatograms (to produce multilinear data), the undersampled first chromatographic dimension must be interpolated. The interpolation techniques examined in this study consisted of Hermite polynomials, cubic splines, Fourier zero-filling, wavelets, and Gaussian fitting. A thirty five injection run, consisting of fifteen calibration samples and twent validation samples, was simulated using a Gaussian function for the second chromatographic dimension peaks and undersampled Gaussian peaks for the first chromatographic dimension. The simulated peak retention time shifts in both the first and second chromatographic dimensions were comparable to those found within experimental chromatograms. The peak widths for both the first and second chromatographic dimensions were assumed to remain constant for the entire run. Singular value decomposition (SVD), iterative key set factor analysis (IKSFA) and multivariate curve resolution-alternating least squares (MCR-ALS) were used to determine the number of components and to generate appropriate initial guesses for the PARAFAC algorithm. The accuracy and precision of the concentration results for the validation samples will be discussed in this presentation.

(626) **Biotransformation of Aflatoxin B1 in Soil**

Mustafa Selim[1]; [1]East Carolina University

Aflatoxin B1 (AFB1) is a highly toxic and carcinogenic secondary metabolites produced by Aspergillus flavus and A. parasiticus fungi which are commonly found soil. The fate of AFB1 in soil is important for determining its potential migration and contamination of ground and /or surface water. Previous studies have identified aflatoxin B2 and or G2 as primary transformation product of AFB1 in soil. However, these identifications were based on the use of non-specific techniques (e.g. TLC) and did not provide viable transformation mechanism. This study will describe the use of combined high pressure liquid chromatography, ultraviolet (UV) spectrometry, and mass spectrometry (HPLC/UV-ES/MS) to identify the transformation products of AFB1 in soil. The transformation mechanism will be presented and explained in view of the UV and MS data of all identified aflatoxin-transformation products.

(627) **The Metabolism, Excretion, and Pharmacokinetics of Resveratrol in Pregnant and Lactating Rats**

Brenda Fletcher[1], Franz Thomas[1], Teruyo Uenoyama[1], Norman Gaudette[1], Timothy Fennell[1], Stephen Cooper[1], Melanie Silinski[1], James Blake[1], Reshan Fernando[1], Bradley Collins[2]; [1]RTI International; [2]NIEHS/NTP

Resveratrol (3,5,4'-trihydroxystilbene) is a naturally-occurring phenolic phytoalexin found in grapes and red wine that is believed to play a role in the prevention of cancer, cardiovascular disease, and neurodegenerative diseases. Resveratrol undergoes extensive first pass metabolism in the liver and gut. Little is known about the kinetics and distribution of the resveratrol metabolites, however, and information regarding the effects of resveratrol during pregnancy and lactation is especially limited. The objective of this work was to develop, validate, and apply analytical methods for simultaneous determination of resveratrol and its major metabolites in rat plasma, urine, fetus, and pup tissues in order to assess the toxicity, perinatal exposure, and metabolic fate of resveratrol. Resveratrol was administered orally to pregnant and lactating female Wistar-Han rats. Dam plasma, fetuses, and urine were collected from study animals at various timepoints. Plasma samples were spiked with internal standard (IS, 2',4'-dihydroxypropiophenone) and extracted with acetonitrile. Pups were frozen in liquid nitrogen and shattered. Pups and fetuses were homogenized and spiked with IS; proteins were precipitated with methanol, and lipids were removed by freezing the extracts at -80 °C followed by centrifugation. Urine samples were diluted 1:100 in methanol and filtered using Whatman Mini-UniPrep syringless filters. UPLC-MS/MS analysis was conducted on an Acquity UPLC with a 4000 QTRAP mass spectrometer using electrospray ionization in negative ion mode and multiple reaction monitoring. Resveratrol (RES) and two of its major metabolites 3-O-B-D-glucuronide (GLUC) and 3-sulfate (SULF) were quantitated in rat plasma, urine, pups, and fetuses. Preliminary results show that PND 21 pups from dams dosed at 1250 mg/kg exhibited plasma levels as high as ~ 2000 ng/mL resveratrol, ~ 40 µg/mL 3-sulfate, and ~ 500 µg/mL 3-O-â-D-glucuronide, indicating significant perinatal exposure and extensive metabolism. The tissue concentration of resveratrol and the metabolites in plasma, urine, pup, and fetus will be presented and the metabolism, excretion and degree of perinatal exposure of resveratrol will be discussed.

(629) **Hyperspectral Imaging of Post-Blast Explosives Residues**

Daniel Mabel[1, 3], Kerri Moloughney[1], Diane Williams[2]; [1]Oak Ridge Institute of Science and Education; [2]FBI, Laboratory Division; [3]Virginia Commonwealth University

Previous study has shown that pure high explosives such as pentaerythritol tetranitrate (PETN) and cyclotrimethylene trinitramine (RDX) have characteristic spectral signatures in the short-wave infrared (SWIR) region of the electromagnetic spectrum. Using lab-based hyperspectral imaging systems that are sensitive in the SWIR region, data were collected to identify unique spectral features of the post-blast by-products of PETN and RDX. Identification of the spectral signatures of post-blast explosives residues could allow for the determination of the type of explosive used in a detonation. Initial method development involved the creation of a device for collecting "clean" post-blast explosives residues, using pure explosives with known compositions and detonating them on several known substrates. The results will be presented in the context of the type of explosive used in the detonations, the specific substrates used for the device, and the spectral data that were obtained from the post-blast residues that were collected.

(630) **Experimental Considerations for Quantitation of Solid Mixtures for Focal Plane Array Near-IR Imaging Data**

Mark Boatwright[1, 2], David Wetzel[1,2]; [1]Microbeam Molecular Spectroscopy Laboratory; [2]Kansas State University

Mixtures of granular commodities present an analytical challenge. However, focal plane array near infrared imaging offers a potential convenient approach. By advanced multivariate statistical characterization of individual single components with approximately 240,000 spectra or by selective wavelength contrast, individual pixels are identified as predominantly representative of one component. Pixel counting subsequently readily enables calculation of the relative number of pixels containing the analyte commodity. The reliability of this process depends upon a number of factors such as the pixel size relative to the particle size, the precision of replicate fields drawn from the same mixture lot, the optical range of linearity, and potential spectral changes resulting from continuous flooding of the specimens with light. Data is presented to illustrate the effect that these factors have on image pixel counting results for solid mixtures.

(631) **Effect of Scale of Scrutiny in the NIR Spectroscopic Evaluation of Blend Homogeneity in Continuous and Batch Pharmaceutical Blending Processes**

Rodolfo Romañach[1], Yleana Colon[1], Jackeline Jerez-Rozo[1], Luis Obregon[2], Rafael Mendez[2], Carlos Velázquez-Figueroa[2]; [1]Dept of Chemistry, Univ. Puerto Rico-Mayaguez; [2]Chemical Eng., Univ.Puerto Rico-Mayaguez

Most pharmaceutical processes for tablet production include batch blending. Batch blending requires fixed amounts of materials and is not adaptable to changes in product demands from customers. Furthermore, batch blending processes often lack the capability for breaking down agglomerates of highly cohesive active pharmaceutical ingredients, leading to variation in the final concentration of the drug product. These disadvantages have lead to significant industrial interest in continuous mixing processes. This presentation will discuss the use of near infrared and near infrared chemical imaging in understanding a pharmaceutical batch blending process, and then evaluating the performance of a new continuous mixing process. The evaluation of homogeneity depends on the scale of scrutiny used to evaluate a blend. Near infrared (NIR) spectra were obtained with a small 5 mm diameter beam and a wider 25 mm beam. Partial least squares regression (PLS) was used to develop calibration models for drug concentration in a number of blends produced through both batch and continuous blending processes. The PLS models were used to evaluate drug homogeneity in both scales of scrutiny (smaller and narrower NIR beams). At the same time, NIR chemical imaging has been used to further evaluate the blends produced through both processes. These preliminary evaluations will be used a basis for real time process measurements. Finally, the drug concentration of

the final product (tablets) will be evaluated through both near infrared and destructive whole sample (ultraviolet spectroscopy) methods. The study will provide an understanding of the effect of the scale of scrutiny on the evaluation of pharmaceutical blending processes. The study will provide a greater understanding of the sources of error associated with near infrared spectroscopic measurements of blend homogeneity. In this manner, near infrared spectroscopy contributes to the design of a new pharmaceutical continuous blending process.

(632) **Combining Physical Morphology and Spectroscopic Classification on Multi-Component Samples**

Justin Pritchard[1], Martin Warman[1]; [1]Vertex Pharmaceuticals

In pharmaceutical development, understanding how particle properties contribute to the variability of a drug product is important to the processability of powders and often the efficacy of the product. The particle measurement capabilities typically employed in the pharmaceutical industry are expanding to meet the demands of designing more elegant and robust commercial formulations. Here we present data on the physical and chemical identities of particles in mixed populations by vision-based image analysis and Raman spectroscopy, incorporated into a single prototype instrument platform. Vision-based image analysis is gaining acceptance as an alternative and/or complementary technique to laser light scattering for particle characterization. Image analysis allows for more specific interrogation on two dimensional morphological attributes that effect drug performance and, unlike light scattering, is not biased towards large particles. Vision-based systems are particularly useful in analyzing and classifying mixed populations. By using a relatively small laser spot (when compared with NIR) to acquire Raman spectra, morphological attributes defined by the vision-based component are related to physical and chemical properties of individual particles. Inter- and intra-particle homogeneity is assessed by Raman spectroscopy. The cumulative power of coordinating Raman spectra with morphological distributions of particles is presented with data on mixed physical and chemical populations, generated as a result of process steps such as crystallization and wet and dry granulation.

(633) **The Development of Infrared Spectroscopic Imaging-Based Histochemical Methods for the Prediction of Kidney Ischemia**

Scott Huffman[1], Caitlin Williams[1], Nicole Crane[2], Ira Levin[3], Eric Elster[2]; [1]Western Carolina University; [2]Naval Medical Research Center; [3]National Institutes of Health

Ischemia is a restriction of blood supply and thus, oxygen, which can lead to tissue damage. The quantification of ischemia in tissue biopsies and sections is difficult using histopathological methods, where the traditional morphological changes (such as swelling and extravesation of red blood cells) are highlighted with chemically nonspecific hematoxylin and eosin (H&E) staining. Other immunohistochemical stains are available, but are neither quantitative nor sensitive to early or mild tissue damage. The lack of high quality, specific diagnostic tools is exacerbated by a lack of knowledge of the chemical changes that occur in tissue as a result of ischemia and subsequent reperfusion (re-establishing the blood flow). Ischemia and reperfusion are necessary during transplant surgeries, and a generally accepted critical ischemia time has not yet been established in humans. Ischemia/reperfusion injury may greatly affect organ function after transplantation. The numbers of kidney transplants is increasing every year, and therefore, the need for understanding of the chemical nature of ischemia induced tissue damage is increasing. Infrared spectroscopic imaging has the ability to spatially resolve materials with chemical heterogeneity such as kidney tissue. We have chosen to use this imaging technique, coupled with chemometrics, to discern ischemia/reperfusion injury changes at the molecular level. Herein, we present the results and findings from a study of a canine ischemia/reperfusion model probed with spectroscopic imaging.

(634) **Mid-IR Imaging for Identification of Cells and Mucin Subtype in the Gastrointestinal Tract**

Michael Walsh[1], Jason Ip[1], Caroline Caroline Cvetkovic[1], Rohit Bhargava[1]; [1]University of Illinois at Urbana-Champaign

Mid-Infrared (IR) imaging represents a fast and potentially automated approach to cell and mucin identification in the gastrointestinal (GI) tract. IR imaging of colon tissue microarrays allowed for an automated classification of the main components of colon tissue to be created. A six class model of epithelial cells, loose connective tissue, goblet cells, muscle, blood and general mucin showed a high degree of accuracy in validation tissue arrays. There are primarily two types of mucin within the GI tract; sulfated and sialylated mucins which can be identified using the high iron diamine-alcian blue (HID/AB) staining method. Sialomucin is primarily expressed in the ileum and sulphated mucin in the right colon. The left colon and rectum have a mixed expression of the two mucins. Abnormal expression of mucins in the GI tract may be observed in various GI diseases such as Inflammatory Bowel Disease and certain adenocarcinomas. FTIR imaging of surgical resections from the ileum, left colon, right colon and rectum were acquired. Average IR spectra of the two mucin types were found to be distinct and a subsequent classification program demonstrated a high degree of sensitivity for the identification of mucin type. Further work is ongoing to determine the use of Mid-IR imaging in the identification of cell types and mucins in a range of GI diseases.

(636) **From Handheld to Huge to Handheld: Raul Curbelo and the Continuing Evolution of Commercial FT-IR Spectrometers**

Richard Crocombe[1]; [1]Thermo Fisher Scientific

The rapid-scanning interferometer was invented and developed by Larry Mertz at Block Engineering for astronomical studies. Myron Block applied it to government/military applications, for instance observation of rocket plumes during the boost phase, and had the vision of applying it to analytical chemistry. In one of the (in)famous JOSA advertisements, Block Engineering featured a hand-held FT-IR (mounted on a gunstock) in the 1960s. Raul Curbelo took Mertz's technology and almost singlehandedly turned it into the first commercial rapid-scanning instrument, the FTS-14. The FTS-14 and its immediate successors (FTS-15, FTS-20), along with its direct competitors (Nicolet 7199) were huge, free-standing instruments. With improvements in electronics, notably microprocessors, tabletop instruments were developed in the late 1970s and early 1980s (Nicolet MX-1, Digilab Qualimatic). Raul was the driving force behind the increasing sophistication of Digilab's instruments, through to the late 1990s, including dynamic alignment, step-scan and digital signal processing. Now a new generation of small and portable FT-IRs is emerging, and although developed independently, they show some familial resemblance to the early Block designs. This talk will trace that history, Raul's contributions, and compare very recent to early designs.

(637) **Spectroscopy through the Engineering Lens: Lessons from Raul Curbelo**

Norman Wright[1]; [1]Applied Instrument Tech, Hamilton Sundstrand

This paper will discuss product innovation and the development of scientific instrumentation, with some historical context. Many organizations working on advancing technology bear the stamp of a single influential individual; the Digilab organization was no exception, carrying the stamp of Raul Curbelo. The Digilab product family (of past years) will provide examples, in particular the hyphenated techniques as sampling devices attached to FT-IR

spectrometers, as a vehicle to ask those not quite simple questions about what constitutes a successful measurement and by extension a spectrometer system. The experience of working in an engineering environment as a spectroscopist can be viewed as having the best of both worlds while polishing the tools necessary to successfully guide instrument development projects, relevant today even with a much different marketplace and business environment. The constant is the ability to be creative in challenging conditions. Although design tools may be better and the knowledge base better, there are more corporate and government restrictions, smaller budgets, more compliance, and seemingly more obstacles in getting to the end product. What does innovation today look like today? Different, but it still starts with fundamental questions and a necessary balance between science and engineering.

(638) **Development of Digital Signal Processing (DSP) Software for Step-Scan Modulation Measurements**

Dave Drapcho[1]; [1]Thermo Fisher Scientific

Raul Curbelo applied his knowledge of radio telecommunication theory to the problem of demodulating the sample signal intensity and phase from the complex mixture of signals that are generated in step-scan FTIR modulation measurements. His work led to a series of patents covering these techniques and a series of software tools that were provided in the software package of the Bio-Rad step-scan FTIR spectrometers, first applied to step-scan Photoacoustic Spectroscopy, then to Sample Modulation Spectroscopy (polymer stretching), and finally to Polarization Modulation experiments. This paper will describe the algorithms Raul developed for these experiments, the subsequent software packages that followed, and a few examples from samples demonstrating the techniques.

(639) **On the Shoulders of Giants: An FT-IR Legacy**

Andrew Hind[1, 2]; [1]Agilent Technologies; [2]Varian Inc

In leveraging the understanding afforded by noted thinkers who have gone before us, we stand on the shoulders of giants. In the arena of Fourier transform infrared spectrometry, Raul Curbelo was one such giant. With almost 20 FT-IR related patents to his name, his contribution to the field of FT-IR spectrometry over many years cannot be questioned. In 1969, as the engineer in charge of the development of Digilab's FTS-14, he helped bring us the worlds first commercially available FT-IR spectrometer: a rapid scanning interferometer system featuring frictionless air bearing for the moving mirror, laser reference system for wavelength calibration, and interchangeable beamsplitters, sources and detectors for customizable wavelength range. Ahead of its time, the FTS-14 became the industry standard upon which all commercially available FT-IR spectrometers are based today. There is no better evidence of this legacy than the Varian 600 series FT-IR spectrometer platform. Released some 40 years after the FTS-14, the Varian 600 series incorporates technologies which leverage eleven patents bearing the Curbelo name. In addition, the development of the 600 series has resulted in a new patent application which stems from the work of Curbelo and Johnson in US Patent 5166749: 'Step scanning technique for interferometer'. This paper will describe how this and other technologies remain at the heart of a modern day FT-IR spectrometer, focusing on the development role Raul Curbelo played in absentia. The tradition continues …

(641) **Building a Community Resource of Open Spectral Data**

Antony Williams[1]; [1]Royal Society of Chemistry

ChemSpider is an online database of almost 25 million chemical compounds sourced from over 300 different sources including government laboratories, chemical vendors, public resources and publications. Developed with the intention of building community for chemists ChemSpider allows its users to deposit data including structures, properties, links to external resources and various forms of spectral data. Over the past three years ChemSpider has aggregated almost 3000 spectra including Infrared and Raman Data and continues to expand as the community deposits additional data. The majority of spectral data is licensed as Open Data allowing it to be downloaded and reused in presentations, lesson plans and for teaching purposes. This presentation will provide an overview of our efforts to build a structure-indexed online database of spectral data, initiate a call to action to the community to participate in improving this resource for the community at large and discuss how such a resource could be used as the basis of a spectral game to teach students spectral interpretation.

(642) **Establishing Raman Spectroscopy as a First Choice Method in Forensic Casework**

Steven Bell[1], Samantha Stewart[1], W. James Armstrong[2], George Kee[2], S. James Speers[2]; [1]Queen's University; [2]Forensic Science (N.I.)

Many of the features of Raman spectroscopy, particularly the ability to carry out non-contact analysis on small samples, suggest that it should be the first choice in examination of many types of forensic casework samples. However, there is a significant difference between demonstrating that the Raman spectra of a few samples recorded by expert spectroscopists show statistically significant differences and having a method that is fit for purpose in a working forensic laboratory. New methods will not be accepted unless it is unambiguously demonstrated that they give a significant improvement in the evidential value of an exhibit or have a speed /cost advantage over existing techniques, many of which have been refined and optimized over years or even decades. This presentation will centre on our current work on integrating Raman spectroscopy into examination of seized drugs, paint transfer evidence and document examination. With the drug seizures the objective is not simple identification of illicit compounds within the samples but instead is the rapid chemical profiling of the samples for the purposes of drugs intelligence work. Here the main issues are associated with establishing representative sampling protocols and data mining/ reporting. It is essential to have a method to reduce large amounts of raw data (typically 100s or 1000s of spectra) into information which is useful for law enforcement purposes. For the household paints our focus has been on the use of Raman spectroscopy for analysis of multi-layer white paint samples which are commonly encountered in casework as transfer evidence and have little evidential value under existing methods due to their poor discrimination of the various layers. Here the challenge is not merely to establish that Raman methods give superior performance but also how to integrate them into well-established paint examination protocols. Finally, SERS can be useful for document examination but again, it is important to demonstrate real improvements over existing methods.

(643) **Raman Spectroscopy in Forensic Geoscience and Contraband Materials**

Howell GM Edwards; [1]University of Bradford

The forensic analysis of materials from the geoscientific record and contraband seizures of illegal substances at ports of national entry may seem unrelated at first sight but the application of Raman spectroscopic interrogation is a unifying theme. The ability of Raman spectroscopic techniques to provide molecular analytical information nondestructively , obviating the necessity to chemically or mechanically pretreat a sample or to resort to desiccation , are very attractive attributes for a first-pass interrogation and screening to identify potential specimens for the adoption of further analytical procedures. Here , the advantages of Raman spectroscopy for the identification of biomaterials and

associated pigments in a depositional environment and a survey of our experiments carried out in the detection of drugs of abuse , contraband ivories and explosives precursors in a busy airport environment will be demonstrated . In this respect the ability of the Raman spectra to provide information about both the organic and inorganic components of a heterogeneous specimen is invaluable , especially where it is believed that an interaction between them could have taken place .An important example is the use of portable Raman spectroscopic instrumentation for the interrogation of suspect illegal substances *in situ* and rapidly (ca. 10 seconds) , as powders and concealed in liquids inside glass bottles and as deposits on clothing . The demonstration of portable Raman spectroscopic equipment in a forensic archaeological field is provided by the analysis of suspect human brain tissue in the cranium of decapitated skeletal remains and the preservation of skin in mummified bodies ; molecular information from these studies informs conservators about degradation processes . Another example addresses the forensic analysis of badly damaged fresco paintings resulting from gunfire and conflagration and the identification of the original pigments used for restoration purposes.

(644) **Raman and Near-Infrared Spectroscopy for the Forensic Analysis of Intact Tablets**

Tony Moffat[1], Sulaf Assi[1], Robert Watt[1]; [1]The School of Pharmacy, University of London

Forensic scientists often need to identify tablets and capsules for a variety of reasons. It may be that some tablets are found near the body of a deceased person and the coroner wants their identity confirmed, or it may be that a product is suspected of being counterfeit. In the latter case the questions most often asked are: was the product manufactured by the license holder and does the product contain the correct active pharmaceutical ingredient (API) and potency? Both near-infrared (NIR) spectroscopy and Raman spectroscopy are used because they are fast, non-destructive, require no sample preparation, can be used through blister packaging and hand-held instruments are available that can be taken to the site of inspection (eg a hospital or customs shed) rather than taking the samples to a laboratory. Spectral libraries of samples of products are set up for identification purposes. For each product, it is best to incorporate samples from many different batches and from as many different manufacturing sites as possible to encompass all expected manufacturing variations. Calibrations for quantification of the active can be prepared using powdered dilutions and standard additions of authentic tablets. Raman spectroscopy has the advantages that it often detects APIs in a matrix of excipients which are usually not very Raman active, water is transparent so that solutions may be analysed, and powdered and intact tablets give the same spectra. However, it suffers from fluorescence effects and sometimes low Raman activity of some APIs which makes extracting a usable Raman spectrum for a product difficult. NIR spectroscopy has the advantages that it is cheaper and can detect nearly all the components in a tablet so that it gives a fingerprint for each individual product. A counterfeiter would have to get the all the components in a tablet correct in terms of identity, percentage and particle size to pass a test. Raman and NIR imaging techniques have the added advantages that each component may be identified, their relative amounts estimated, their particle sizes measured and the homogeneity of the components measured, but require more extensive laboratory sample preparation and measurement.

(645) **Raman Mapping of Chocolate and Cells**

Duncan Graham[1], Karen Faulds[1], Iain Larmour[1], Ross Stevenson[1], Joanna Loose[1]; [1]University of Strathclyde

Advances in modern instrumentation for Raman spectroscopy have enabled the rapid mapping of surfaces with high resolution and the ability to identify multiple species based on their vibrational signature. In this presentation two examples will be given where Raman mapping has been used to provide chemically specific information to produce maps of composition of chocolate and in a more biological application, to show the pH values across a cell. In the example of the chocolate, a piece of white chocolate was mapped for composition in terms of fats and sugars and false colour images were produced showing how the fats were distributed over the chocolate and also in relation to the sugars. This is highly significant as the composition of chocolate affects the way that it tastes and a number of samples were obviously examined to verify, confirm and test this hypothesis. In the second application, nanoparticles functionalised with a pH sensitive molecule were introduced to cells. The cells were HeLa cells and the spectra from the pH sensitive molecule recorded across the area of the cell using confocal Raman mapping. By taking the ratio of the intensity of two marker bands, pH values were identified over the area of the cell and reproduced in a false colour contour map which allowed nanoscale resolution of pH values from within the cell. The typical lateral resolution for this experiment was around 500 nm although it could be pushed down to 100 nm when high resolution was required. These two examples show the power and flexibility of modern Raman mapping as applied to two very different applications and really the main hurdle in exploiting Raman mapping now is the handling to the large amounts of data which result from the experiments.

(646) **Sampling for Success: Quantitative *in situ* Raman Spectroscopy**

Ian Lewis[1], Kevin Davis[1], Sean Gilliam[1], Maryann Cuellar[1], David Strachan[1], Carsten Uerpmann[2], Herve Lucas[2], Pat Wiegand[1], Joe Slater[1]; [1]Kaiser Optical Systems, Inc.; [2]Kaiser Optical Systems, SARL

Sampling remains an important if not vital component of any industrial application where the results are to be used for process monitoring and/or control. When the sample size is too small, the bulk material is typically under-sampled. If the sample size is too large, spatial and temporal information could be reduced or even lost. Further, if the analysis is destructive, then the impact to overall product yield may be significant. Sampling has historically presented a challenge to industrially inclined Raman spectroscopists due to the need to precisely focus a laser onto a sample to maximize the Raman signal. In this presentation, parameters important to both improving and standardizing Raman sampling will be identified and discussed. These discussions will include relevant applications across a wide range of industries as well as across several phases of materials. Gases, solids, and liquids each present unique sampling challenges that will be addressed. Several application examples of proper and improper sampling, including those from the pharmaceutical and petrochemical areas, will be discussed.

(647) **Transmission Raman Spectroscopy for Quantitative Analysis**

Hanna Matic[1], Magnus Fransson[1], Jonas Johansson[1], Anders Sparén[1], Olof Svensson[1]; [1]AstraZeneca PAR&D, Sweden

Raman spectroscopy can be an alternative to near-infrared spectroscopy (NIR) for non-destructive quantitative analysis of solid pharmaceutical formulations. Compared with NIR spectra, Raman spectra have much better selectivity, which opens new opportunities for a more rapid development of simple calibrations,

compared with traditional, often extensive NIR calibrations. Raman spectroscopy has been widely used for solid-state determination of pharmaceutical materials. However, for quantitative measurements very little has been reported. The main drawback with Raman spectroscopy for quantitative assessment has been sub-sampling, which leads to higher than necessary prediction errors. In backscatter mode, this problem has been addressed in several ways, e.g. by spinning the sample and using wide area illumination and sampling probes. Transmission Raman spectroscopy virtually samples the entire sample, which further reduces the problem with sub-sampling. In this presentation, transmission and backscatter Raman spectroscopy will be compared for quantitative assessment of pharmaceutical tablet formulations. The experimental set-up for backscatter and transmission modes consisted of a Raman spectrometer, using a 785 nm laser for excitation. The effect of sample heterogeneity on the Raman signal in transmission mode will be shown. The accuracy, precision and robustness of transmission and backscatter Raman spectroscopy for quantitative assessment of pharmaceutical tablet formulations will be evaluated and compared. In addition, different methods for quantitative evaluation of transmission Raman spectra will be discussed.

(648) **Signal Intensity Dependence on Depth in Transmission Raman Spectroscopy of Pharmaceutical Tablets**

Pavel Matousek[1,2], Neil Everall[3], Alison Nordon[4], David Littlejohn[4], Matthew Bloomfield[2]; [1]Rutherford Appleton Laboratory; [2]Cobalt Light Systems; [3]Intertek MSG; [4]University of Strathclyde

Transmission Raman spectroscopy is an emerging analytical tool with yet unexplored potential in pharmaceutical analysis. Technique's benefits include volumetric probing capability with largely reduced sub-sampling compared with conventional Raman methods and the ability to suppress surface capsule and tablet coating fluorescence and Raman interference permitting accurate quantification of intact pharmaceutical capsules and tablets non-invasively [1]. In comparison with conventional Raman spectroscopy the subsampling issue is largely removed although some residual dependence of the signal intensity on the depth of a layer within sample still remains present. This effect has been reported by several groups both theoretically and experimentally [2,3,4,5]. This presentation will focus on studying this dependence both theoretically using Monte Carlo simulations as well as experimentally on test samples consisting of 0.5 mm cellulose discs with a thin test layer of PET sheet doped with Ti:O2 inserted at different depths. The presentation will also discuss potential means of reducing this residual depth dependence. The understanding of these effects is important for the effective deployment of the technique in practical pharmaceutical settings. References 1. N. A. Macleod, P. Matousek, Appl. Spectrosc. 62 (2008) 291A-304A. 2. P. Matousek, A.W. Parker, Appl. Spectrosc. 60 (2006) 1353-1357. 3. N. Townshend, D. Littlejohn, A. Nordon, M. Myrick, J. Andrews, P. Dallin, PhAT Raman analysis of pharmaceutical tablets, 2009, unpublished results. 4. J. Johansson, O. Svensson, S. Folestad, A. Sparen, and M. Claybourn, Transmission Raman Spectroscopy for Robust Tablet Assessment, FACSS Conference Proceedings, Abstract 300 (Louisville, Kentucky, 2009), p. 131. 5. N. Everall, P. Matousek, N. Macleod, K.L. Ronayne, I.P. Clark, Appl. Spectrosc. 64 (2010) 52-60.

(649) **Raman Reaction Monitoring in Opaque Vessels when No Ports are Available for a Raman Probe**

Michael Pelletier[1]; [1]Pfizer Global Research and Development

Real-time analysis of chemical reactions using Raman spectroscopy has proven valuable in the pharmaceutical industry for determining reaction endpoint, intermediate formation, unexpected reaction pathways, crystal polymorph transformation, and reaction kinetics. A Raman probe is typically inserted directly into the reaction mixture, avoiding the complications and artifacts caused by withdrawing material from the reaction vessel, or imaging through transparent reaction vessel walls. Sometimes, though, no ports are available for a Raman probe. Even when ports are available it may be challenging to place the Raman probe window in a high-shear, well mixed region of the reactor in order to avoid probe fouling or measurement of non-representative volumes in the reactor. It is possible to incorporate the Raman probe optics into the overhead stirrer. This approach eliminates the need for a separate port for the Raman probe. It eliminates the possibility of damage that can occur when a separate Raman probe is accidentally placed in the path of the impeller. Side-looking optics in the overhead stirrer place the Raman detection volume at a high-shear location, reducing the chances of probe fouling. Since the point in the reactor being sampled by the Raman probe changes as the stirrer rotates, a greater reactor volume is sampled by the Raman probe, possibly leading to more representative sampling. Finally, specially shaped impellers can produce unique and beneficial flow patterns in the Raman probe volume when the rotational velocity is temporarily increased above that normally used for mixing. This talk will illustrate some approaches for incorporating Raman probe optics into overhead stirrers. It will also include some examples of small-scale (few ml to a few hundred ml) reaction monitoring with a Raman-enabled overhead stirrer.

(650) **Application of *in-situ* Raman Spectroscopy in Pharmaceutical Chemical Development**

Susan Barnes[1], Gregory Gervasio[1], James Rydzak[1]; [1]GSK

The application of *in-situ* Raman spectroscopy to gain increased understanding during pharmaceutical process development is becoming common practice. *In-situ* Raman spectroscopy is a valuable real-time technology which is well integrated into quality by design (QbD) of active pharmaceutical ingrediants (API). This presentation will detail the application of Raman spectroscopy for *in-situ* reaction monitoring and determination of polymorphic form on laboratory and manufacturing scale.

(651) **Application of Raman Spectroscopy in Pharmaceutical Process Development**

Ming Huang[1], Robert Wethman[1], John Wasylyk[1]; [1]Bristol-Myers Squibb Co.

The utilization of spectroscopic techniques is increasing due in part to their ability to monitor the progress of chemical reactions. Information and knowledge gained from research and development studies using spectroscopic tools, as well as manufacturing experience, have yielded scientific understanding for the determination of the design space, enabling Quality by Design (QbD) while enhancing overall productivity. Raman spectroscopy is one tool which has provided valuable information when both off-line and in-line studies are completed. Recent applications of off-line Raman spectroscopy have allowed the Research and Development chemist to improve experimental design. When appropriate, these at-line methods, including chemometric models, are transferred to an in-line system to allow better reaction control. Several examples of spectroscopy applied to process screening, improvement, and finally, in-line productivity enhancement will be presented. Issues that arise during development and transfer of methods will also be covered.

(652) **Evaluation of Granules Made by High Shear Granulation using Raman Mapping and Imaging**

Tatsuo Koide[1], Toru Kawanishi[1], Yukio Hiyama[1]; [1]National Institute of Health Sciences

Granulation is often used and one of the most critical steps in pharmaceutical manufacturing process. In the case of Japanese

pharmaceutical industries, the share of granulation was more than 80%. Therefore it is necessary to understand granulation mechanisms, to identify the critical factors determining quality attribute and then to establish robust process. In order to understand the granulation mechanism well enough, highly capable evaluation methods are desired. Chemical imaging technique is one of the methods which provide characterization of heterogeneous solid dosage forms at a micron scale with spatial and chemical information. Previously we studied high shear granulation process using NIR chemical imaging to understand granulation process and we detected significant segregation of components in the granules made by high shear granulation using NIR(Near Infrared) chemical imaging technique. However we were not able to observe the segregation in detail because NIR chemical imaging system did not have enough spatial resolution to detect small components. In this study, to observe the segregation granules in detail, we measured the granules by Raman chemical mapping and imaging that have higher spatial resolution than that of NIR chemical imaging. The formulation we used in this study contained 4 compounds (ethenzamide, cornstarch, lactose and methylcellulose). These ingredients were mixed, added water and granulated with different granulation time and impeller speed by high shear granulation method. Raman chemical mapping and imaging of those granules were measured and chemical images on granules were generated by univariate or multivariate analysis methods. Segregated ethenzamide and lactose was observed in the granules by NIR chemical imaging. But cornstarch was not detected clearly because its size was too small. In the result of Raman mapping analysis, distribution of cornstarch domain was observed clearly. These results suggested that Raman mapping technique could provide detailed information on smaller particle, which could not be obtained by the NIR imaging.

(653) **Multiparametric Surface Plasmon Resonance Imaging Systems and Nano- Micro- Milli- Structured Biochip Substrates**

Michael Canva[1, 2], Anuj Dhawan[2], Aurélien Duval[1], Mohamed Nakkach[1], Buntha Ea-Kim[1], Alain Bellemain[1], Julien Moreau[1], Tuan Vo-Dinh[2]; [1]Institut d'Optique; [2]Duke University

Surface Plasmon Resonance (SPR) sensing is a label free technique that allows monitoring of biomolecular surface interactions. The plasmonic effect requires the interaction to be localized in the vicinity of a thin metallic film. In conjunction with imaging capabilities (in Surface Plasmon Resonance Imaging, i.e. SPRI) as well as surface functionalization and structuration, SPR sensing is the basis of powerful real-time biochip systems, which physically monitor the evolution of reflectivity R as a function of biochip space coordinate, x and y, and time t, i.e. R(x, y, t). Furthermore, many parameters of these systems can be tuned, in particular the incident wavelength, λ, and coupling angle, θ, as well as the plasmon propagation direction, P. The measurement of the reflectivity in the multidimensional space R(x, y, t, θ, λ, P) not only allows access to precise quantitative information about how much of material has been captured by the sensor chip but also provides access to spectral and organization information of the biomolecular material binding to the metallic film. Examples include genotyping of single Nucleotide Polymorphism (SNP) from ssDNA or PCR amplified DNA material from patients. In parallel, for many applications, end users require more sensitive systems that would detect very small concentrations of analytes and also consume less biological materials. The current SPR systems are now being operated near their theoretical limits based on homogeneous substrate with propagating plasmons. To overcome such limitations nano- and micro- structured substrates are being investigated. Theoretical simulations are being carried on using Rigorous Coupled Wave Analysis (RCWA) codes. Substrate fabrications have been done using deep UV lithography techniques. We studied both one dimension (line gratings) and two dimensions structuration (with different base motive shapes) of the thin metallic film, varying periodicity as well as filling factors, in a wide spatial range from nanometers to micrometers, allowing us to study the transition regime from the classical propagating plasmon to localized plasmon, via quasi-propagating plasmon as well as coupled localized plasmon. The materials characterization allows quantification of the real structural parameters. The SPRI systems allow quantification of the initial plasmonic reflectivity as well as its dependence to change in optical index and/or binding of biomolecular thin film.

(654) **Characterization of Diazonium-Salt Monolayers as a Linker for Surface Plasmon Resonance Spectroscopy Analyses**

Karl Booksh[1], Nicola Menegazzo[1], Qiongjing Zou[1], Laurel Kegel[1]; [1]University of Delaware

Diazonium salts offer an intriguing alternative to thiol chemistry for forming reactive monolayers on SPR active surfaces. Diazonium salts offer the possibility to prefunctionalize the antibody with the diazonium salt and electrograft the adduct under mild conditions in less than 30 seconds. This presentation will detail the construction and characterization of the functionalized diazonium salt monolayer. A microfluidic device with electrochemical contacts has been constructed to functionalize the SPR sensing pad *in-situ* with the diazonium salt adduct and perform quantitative determination of target proteins in solution.

(655) **Grating-Assisted Optical Fiber SPR Sensor with Self-Referencing Capability**

Jacques Albert[1]; [1]Carleton University

The use of a tilted fiber Bragg grating photo-induced in the core of a conventional single mode fiber by intense ultraviolet irradiation from an excimer laser allows the excitation of well-defined stable plasmon resonances in a thin metal film deposited along the fiber surface. By monitoring changes in the narrowband spectral resonances of the transmission spectrum of these 1 cm-long gratings, we obtain SPR wavelength shift sensitivities of the order of 1000 nm/RIU (refractive index units), as well as demonstrate the detection of individual 1 nm-thick polyelectrolyte monolayers and of sub-monolayers of aptamers for bio-chemical sensing. An additional advantage of this configuration is that the multiple optical resonances of the grating structure provide a self-referencing capability that is used to eliminate power and spectral fluctuations of the light source and to reduce the temperature cross-sensitivity to negligible levels. While our current sensors use gold coatings and operate at wavelengths near 1550 nm (where these low cost telecommunication fibers have their lowest loss, thereby allowing remote operation of the sensors over km length scales), the sensor design is amenable to other conducting films and wavelength ranges, visible light in particular, as long as the underlying fiber is single mode for the chosen wavelengths. This is potentially the lowest cost, simplest, and most practical fiber optic SPR sensor ever developed : the fiber itself costs nearly nothing, the grating fabrication process is identical to that used for the mass production of a mature commercial product, and the interrogation uses standard optical instrumentation that is widely used for telecommunication applications.

(656) **Extending SPR to Novel Substrates**

Josh Guske[1], Stefan Franzen[1]; [1]NCSU

Surface plasmon resonance (SPR) has been extended to conducting metal oxides and hybrid materials. In this talk we show the general considerations for novel plasmonic effects in a range of materials including gold, silver, indium tin oxide (ITO), aluminum-doped zinc oxide and titanium nitride. The spectral and material properties of these materials can be correlated and understood quantitatively

using 3- and 4-layer Fresnel equations, which leads to the ability to predict new plasmon effects. The charge carrier density, mobility and thickness of the film can be controlled experimentally. The combination of these process parameters with material properties leads to a new field of plasmon design. Free electron conductors provide the greatest flexibility in the area of plasmon design since they have no band-to-band transitions. We show that free electron conductors provide novel information spectral bandwidth of the plasmon that can be used to understand other plasmonic phenomena such as surface-enhanced Raman scattering. As an example of a free electron conductor, ITO is one of the more versatile materials. We close by showing that ordered monolayers or self-assembled monolayers can be formed on ITO. The demonstration of order is novel and provides a strong motivation for further study of ITO and related conductive metal oxides.

(657) **Detection of Influenza Virus Using Evanescent Fields of Waveguide Modes**

Makoto Fujimaki[1], Subash Gopinath[1], Koichi Awazu[1]; [1]AIST

Influenza is an infectious disease caused by RNA viruses of the family Orthomyxoviridae that affects birds and mammals. Influenza A viruses are spherical or filamentous and enveloped; they range in diameter from 80 to 120 nm. Influenza viruses have two major proteins, hemagglutinin (HA) and neuraminidase (NA), on their surfaces, which are the principal determinants of variation among the major types of influenza viruses. The influenza A virus subtypes are classified according to their H number (for HA) or N number (for NA). Many methods have been developed for the detection of influenza viruses. However, a sensor that is able to detect and classify an influenza virus quickly and simultaneously is still highly aspired. Sensors that detect molecules by observing changes in the dielectric environment due to specific adsorption of molecules using near-field optics, such as sensors using surface plasmon resonance, optical waveguide modes, or ring resonators, are well known. Recently, we developed a sensor designed for detecting changes in optical absorption, or 'change in color', which was developed using concept of a monolithic sensing plate of evanescent-field-coupled waveguide-mode sensors. In the present research, we applied the color-sensitive sensor for the detection of influenza viruses. In the detection, we used the anti-HA antibody as the capturing molecule. To facilitate the detection of viruses at lower concentrations, the strategy of complexing viruses with dye was used to amplify the signal. The dye used was Coomassie Brilliant Blue. By applying this method, we succeeded in the detection of HA proteins from an avian influenza virus (H5N1) and a human influenza virus (A/Panama//2007/1999). We also succeeded in the detection of an intact human influenza virus (A/Brisbane/59/2007). The developed method is suitable for the detection of virulent viruses other than influenza that may emerge at various stages of evolution. This study was partly supported by Industrial Technology Research Grant Program in 2009 from New Energy and Industrial Technology Development Organization (NEDO) in Japan.

(659) **Surface Plasmon Resonance Imaging for Applications of Binding Kinetics in Multiplex Throughput**

Karen Gall[1], Philippe Kerouredan[1]; [1]Horiba Scientific

Surface plasmon resonance (SPR) measurements have long been used to measure the binding kinetics of molecules coupled to gold or silver surfaces. The surface plasmon created at a gold or silver surface can be used to detect small changes in reflectivity due to a molecular binding event and this change is plotted versus time for one or two samples. The method of surface plasmon resonance imaging (SPRi) uses a CCD to image multiple binding phenomena in one experiment. SPRi multiplexing and the use of an array spotter enables the measurement of up to 400 interactions in one experiment using SPRi on a gold biochip surface. The technical aspects and effects of how molecules are coupled to an SPRi biochip surface with different spotting techniques are discussed. Applications of SPRi are presented such as oligosaccharide-protein binding, pathogen detection, DNA interactions, antibody screening, and patient immunology. Another application involves the use of SPRi for the detection of binding on a surface spotted with a specific antibody. The same biochip is then directly inserted into a MS/MS MALDI-TOF spectrometer for the identification and confirmation of the molecules that bound to the target. SPR imaging enhances the method of SPR to monitor many binding phenomena in both a graphical and visual manner in multiplex throughput.

(659) **Laser Ablation ICP-MS: A Progress Report of Problems, Pitfalls and Potential for Geological and Biological Applications**

Alan Koenig[1]; [1]US Geological Survey

New developments in reference materials, novel integration of multiple methods of analysis and an honest outline of what we know about laser ablation ICP-MS and what we still do not really know or understand fully will be presented. A summary of new reference materials produced by the US Geological Survey along with the testing of additional reference materials for a variety of applications will be presented. Successful integration of complimentary elemental and molecular or structural information from electron microprobe, micro-XRF, SEM, cathodoluminescence and raman with chemical mapping via LA-ICP-MS will be outlined. A new method for rapid analysis of bulk powders by large spot LA-ICP-MS will also be presented. As those are some of the new (and review) methods that are fairly well characterized, understood and validated some additional discussion of the trials, tribulations and some challenges of things we do not fully understand regarding LA-ICP-MS will be also presented.

(660) **From Sub-Micron Crater Size to High Repetition Rate Femtosecond Laser Ablation Inductively Coupled Plasma Time-of-Flight Mass Spectrometry**

Jhanis Gonzalez[1], Vassilia Zorba[1], Dayana Oropeza[1], Travis Owens[1], Xianglei Mao[1], Richard Russo[1]; [1]L. Berkeley National Laboratory

Laser ablation-based chemical analysis is a straightforward process in which a high-energy laser pulse is used to transform a portion of a solid sample into aerosol for subsequent chemical analysis. Despite its simplicity, the efficiency of the process depends strongly on the combination of experimental parameters (energy, spot size, repetition rate, wavelength, pulse length, etc.). Nevertheless, laser ablation has been extensively used for chemical analysis over a wide range of materials, in a number of diverse applications. Among the main challenges of this technology, is maximization of the spatial resolution, as well as process throughput. Improving spatial resolution to the nanoscale becomes increasingly important as the fields of nanoscience and nanotechnology develop. The implementation of femtosecond laser pulses is a basic strategy for increasing resolution due to the spatially localized material damage. Another factor that has hindered laser ablation from wide implementation in production lines, or any other industry that requires analysis of a large number of samples, is the slow throughput. One aspect limiting the speed of laser ablation-based chemical analysis is the laser repetition rate; most commonly, lasers with repetition rates 100Hz for chemical analysis of solid samples. Our goal is to determine the prospect of much faster throughput by ablating a larger sample area in a shorter period of time.

(661) **Combined LA-ICP-MS and Raman Microspectroscopy for Pharmaceutical and Nutraceutical Imaging Analysis**

Todor Todorov[1], Alan Koenig[1]; [1]US Geological Survey

Composition, homogeneity and quantification of impurities in pharmaceuticals have gained interest over the past decade. Variety of techniques could be used for characterization of these materials, including (1) molecular analysis by Raman, IR, UV-Vis, mass spectrometry, (2) elemental composition by atomic spectroscopy, inductively coupled plasma, (3) particle analysis and other. In this paper we present a combined Raman spectroscopy/laser ablation ICP-MS study to investigate the composition of the active ingredients and binding materials of several tablets, including cold relief medicines, painkillers, multivitamins and dietary supplements. We used Raman spectroscopy followed by multivariate curve resolution analysis to generate rapid hyperspectral images. Raman spectroscopy provided invaluable information about the homogeneity of tablets identifying all active ingredients, binder components and coating materials in the pharmaceuticals as well as many of the components of multivitamins. Pharmaceuticals showed higher homogeneity when compared to nutraceuticals. Elemental analysis of medical tablets can be used for identification of toxic elements and their dose quantification, and for provenance studies to establish whether the pharmaceutical has been manufactured by an illegal drug manufacturer. LA-ICP-MS was used to generate distribution maps for over 30 elements. These elemental images were overlaid with the hyperspectral Raman data and show where the trace elements are located within the tablets: active ingredient, binding materials and/or coating.

(662) **Liquid Nitrogen Cooled Stage for Laser Ablation Inductively Coupled Plasma Mass Spectrometry**

William Hoffmann[1], Aaron Hart[1], Dr. Guido Verbeck[1]; [1]University of North Texas

Recent developments have greatly enhanced the spatial resolution available in LA-ICP-MS. These developments have made it desirable to utilize this technique for surface sampling of specimens that would not normally suit laser ablation techniques. Our group has designed and implemented a cold stage capable of cooling samples to sub-ambient temperatures prior to analyses. Our primary interest lies in probing oil based and biological samples. Oil based samples are of particular interest because of the inherent suitability of ICP-MS methodologies to determine metals concentrations very accurately. Biological samples are also of significant interest because of the spatial resolution afforded by laser ablation. This allows the analyst to spatially determine the bioaccumulation of metals by organs of fish. The cold stage is comprised of two orthogonal copper slugs in thermal contact. Liquid nitrogen is flowed through one of the slugs to generate the desired temperature. These slugs are housed in Rexolite inserts, which are then encased in an aluminum housing. Temperature is measured by a Lakeshore platinum resistance temperature detector. Testing has shown our system to sustain temperatures as low as 83.6 K with a constant liquid nitrogen flow. Typical analysis proceeds as focusing on the specimen in question, cooling the stage to the desired temperature, and initiating the ablation/detection event. Bringing the samples down to liquid nitrogen temperatures decreases the degrees of freedom available for the samples to dissipate the 213 nm radiation. Cooling the oil based samples has significant advantages because of the consistency of the ablation event. Additionally, lower laser powers can be used to generate similar ablation efficiencies and may have the added benefit of decreasing the extent of preferential fractionation. Biological samples are a relatively new arena for laser ablation, and this technique shows significant promise. Our lab has completed laser ablation studies of sectioned fish livers in paraffin. Utilizing the cold stage eliminates the cumbersome step of impregnating the livers with paraffin and may reduce matrix effects that interfere with analytes.

(663) **Characterization of Genesis Solar Wind Sample Surface Contamination by Total Reflection X-ray Fluorescence Spectrometry**

Martina Schmeling[1], Munir Humayun[2], Donald Burnett[3]; [1]Loyola University Chicago; [2]Florida State University; [3]California Institute of Technology

The Genesis mission was the first mission returning solar material to Earth since the Apollo program. Solar wind was collected during a period of 854 days on specially designed collectors made of various ultra clean materials such as silicon and sapphire. Unfortunately the collector material acquired surface contamination and fractured into small pieces upon return of the capsule to Earth. This presents new challenges for analysis of the solar wind material embedded into the collectors. Different cleaning procedures were developed and are still in the process of development for removal of larger and smaller particles as well as film contamination. An ultrapure water and ozone UV radiation treatment is routinely applied to the samples by the curatorial team at NASA's Johnson Space Center. Other efforts mostly involving various forms of acid treatment are currently tested. Initial acid cleaning with concentrated hydrochloric acid decreased inorganic contaminants in most samples analyzed, but additional more aggressive cleaning steps are needed for removal of persistent contaminants and are currently tested. After each cleaning step the samples are checked for remaining contamination. The analysis method employed for this has to be surface sensitive, non-destructive, capable of handling small size samples with reasonable analysis times and should be readily accessible to accommodate a larger number of samples. Laboratory based total reflection X-ray fluorescence spectrometry fulfils all these requirements and a number of samples were analyzed before and after acid treatment. Parameters such as surface roughness, remaining contamination and quantification of contaminants are explored to identify the most successful cleaning procedure.

(664) **Multi-element RIMS Analysis of Genesis Solar Wind Collectors**

Igor Veryovkin[1], Emil Tripa[1], Alexander Zinovev[1], Bruce King[1,2], Michael Pellin[1], Donald Burnett[3]; [1]Argonne National Laboratory; [2]University of Newcastle; [3]California Institute of Technology

Fragments of Solar Wind (SW) collectors of the NASA Genesis spacecraft present significant challenges for analytical techniques, in part due to severe terrestrial contamination during the crash landing of the spacecraft, in part due to low concentrations of SW implants (ppb to ppt) that peak at about 10-15 nm under the surface. We will describe our efforts aiming at accurate and precise determination by Resonance Ionization Mass Spectrometry (RIMS) of the fluences of SW Mg, Ca and Cr in the Genesis collectors. To this end, concentration versus depth profiles were obtained by a sequence of alternating sessions of sputtering and TOF MS analysis. We developed new RIMS photoionization schemes that allowed us to simultaneously detect Mg, Ca and Cr, using only three tunable lasers. In order to improve accuracy of our quantitative RIMS analyses, we used specially prepared reference samples of composition closely resembling the one expected for the SW samples. These "standards" are 1 keV / a.m.u. ion implants (25Mg, 44 Ca, 54Cr) in the same materials as Genesis SW collectors (Si and diamond film on Si, DOS) and fluence 3×1013 at cm-2 each. RIMS analyses of a Genesis sample and the "standard" were performed on the same day, in order to assure very similar photo-ionization conditions. We have compared three approaches to depth profiling: (1) the pulsed analysis beam probed only the center of the rectangular sputtering crater, (2) the analysis beam

was raster scanned over the entire area of this crater, with gating signal assuring data acquisition only from 50% of this area, (3) prior to depth profiling, four 5µm deep "trenches" were ion milled around this crater by line scans of the DC primary beam, thus forming a so called "mesa" structure, and then analyses were performed in the same fashion as mode (2) but with 80% signal gating. The "mesa" approach, with primary ion beam energy set to 5 keV, yielded the best depth resolution and dynamic range of analysis, with profiles of both reference and SW ion implants for Mg closely resembling SRIM simulations. We will present and discuss these results at the Meeting. This work is supported by UChicago Argonne, LLC, under contract No. DE-AC02-06CH11357 and NASA through grants NNH08AH761 and NNH09AM48I.

(665) **Blood, Sweat and Tears: What can DCDRS Tell Us about Bodily Fluids?**

Nicholas Stone[1, 2], Catherine Kendall[1,2], Jacob Filik[1]; [1]Gloucestershir Hospitals NHS Foundation Trust, UK; [2]Cranfield University, UK

Clinical diagnostics regularly utilizes changing analyte concentrations in bodily fluids associated with abnormal physiological conditions and disease. Raman spectroscopy is able to measure many of the key constituents with minimal sample preparation, leading to the potential for rapid testing for numerous conditions. However, bodily fluids cover a broad range of molecular and cellular concentrations, many of which are challenging to Raman using traditional approaches. Sweat and tears are dilute fluids, but contain particularly useful diagnostic molecules. Blood is a highly complex medium with a high density of constituents. Here the use of drop coating deposition Raman spectroscopy (DCDRS), a combination of evaporative preconcentration and Raman spectroscopic analysis is explored on a selection of fluids. Sample preparation, consistency of results and the potential of DCDRS for clinical diagnostics will be discussed.

(666) **Drops on Superhydrophobic Surfaces Provide New Analytical Tools**

Noah Weiss[1], Antonio Garcia[2], Mark Hayes[1]; [1]Arizona State University, Chemistry; [2]Arizona State University, Bioengineering

Analytical chemistry relies on spatially confining fluids for sample preparation, separations, and measurement. Here we accomplish this task by utilizing superhydrophobic surfaces to precisely confine drops and minimize solid/liquid contact area. This simplistic approach provides several advantages (e.g., reduced surface adsorption) and unique opportunities for analytical chemistry. For instance, we have developed superhydrophobic targets for MALDI mass spectrometry sample preparation to pre-concentrate sample deposition. Additionally, we present a new paradigm of separations by carrying out a molecular separation in an open drop without the use of channels. These methods demonstrate the significance of drops and superhydrophobic surfaces in providing solutions to the challenge of spatially confining fluids.

(667) **Morphological and Chemical Analysis of Dried Biofluid Drops**

Karen Esmonde-White[1], Francis Esmonde-White[2], Blake Roessler[1], Michael Morris[2]; [1]University of Michigan Medical School; [2]University of Michigan

Drop deposition is a widely studied analytical technique for the preparation of low abundance biofluids. The features of drop deposition, such as low volume requirements, minimal sample preparation are amenable to current clinical practices of biofluid collection. Drop deposition also provides a course separation of fluorescing impurities from the biofluid and the fluid of interest is localized at the drop edge. The preconcentration effect improves detection limits. Microscopy and Raman spectroscopy of dried biofluid drops can provide information on the drying patterns and chemical composition. Morphological image analysis was applied to microscopy images of dried biofluid drops to quantify visual features such as crystallization patterns, cracking and accumulation of thin films. Raman spectra collected from dried drops at high and low magnification will be compared. Other measurements, such as contact angle and fluid viscosity, provided insight into the dynamics of drop formation.

(668) **Towards the Use of Levitated Drops as Microreactors to Study Enzyme Kinetics**

Alexander Scheeline[1], Zakiah Pierre[1], Oluwafemi Masha[1], Edward Chainani[1]; [1]University of Illinois at CU

Free radicals react and recombine at surfaces. There is thus a tension between the desire to study biomolecular reactions in microfluidic systems and the separation of homogeneous from heterogeneous reaction dynamics when free radicals play an essential role in reaction because, for small characteristic dimensions, surface area to volume ratio is high. Ultrasonically-levitated drops show promise as reactors free (or largely free) of solid-liquid interfaces for the study of biochemical reaction kinetics, especially kinetics of enzymes producing or consuming free radicals. We present examples of kinetics successfully observed in levitated drops. We also show some of the challenges which, to date, have prevented routine use of such drops for study of, e.g., mechanisms of peroxidase reactions and other aspects of immune response. Developments in reactant delivery and mixing, optical diagnostics, and electrochemical diagnostics are reported. Reactant mixing, both spontaneous and driven, is examined. Strategies for improving drop stability and, consequently, signal stability are reviewed.

(669) **Quantitative Protein Characterization Using Drop Coating Deposition Raman Spectroscopy**

Dongmao Zhang[1]; [1]Mississippi State University

Drop coating deposition Raman spectroscopy (DCDR) is a simple, sensitive and reliable normal Raman sampling technique that has become increasingly popular in bioanalytical applications. Most of the DCDR applications to date have been qualitative in nature. In this talk we will demonstrate two quantitative DCDR applications. First, using protein oxidation as an example, we developed a novel DCDR based ratiometric Raman method for quantification of protein carbonyl level. Current detection sensitivity is ~1 nmol carbonyl/mg protein in terms of protein carbonyl level and sub picomole in terms of total protein consumption per measurement. In addition to the general strategy we envisioned for DCDR quantification of protein post-translational modifications, obstacles and possible solutions will also be discussed for further enhancing the sensitivity of this ratiometric DCDR method. The second quantitative DCDR application to be presented is the Raman predication of protein compositions. Taking advantage of the high reproducibility of the DCDR technique, a small library of the protein Raman spectra were acquired. Using the Raman feature of phenylalanine as internal Raman references, we demonstrate that numbers of tryptophan, tyrosine residues can be accurately estimated together with other important protein skeletal and side chain features such as the number of peptide bonds, etc. Implications of this DCDR result for an all-optical protein characterization tool will be discussed in this talk.

(670) **Analysis of Rapidly Solidified TiO2 Droplets by Micro-Raman Spectroscopy**

Christopher Young[1], Jose Colmenares-Angulo[1], Giovanni Bolelli[2], Valeria Cannillo[2], Luca Lusvarghi[2], Clive Clayton[1]; [1]Stony Brook University; [2]University of Modena and Reggio Emilia

Phase distribution of titanium dioxide coatings is a key characteristic in improving photocatalytic activity of the material under UV light. The phase content of titanium dioxide coatings processed by the thermal spray technique is analyzed from an individually solidified droplets (or splats) perspective. Both high velocity oxygen fuel and atmospheric plasma spray methods are used to spray individual splats on a stainless steel substrate. Splats are found to mainly consist of the rutile phase, but with certain areas containing higher anatase peak intensities, and smaller splats having higher antase content. Micro-Raman spectroscopic mapping has been performed on splats of varying sizes sprayed by the two above methods. The relative content of anatase and rutile phase TiO2 in each has been characterized. A relationship between splat size and anatase phase content has been established. Possible processing reasons for the difference in phase content are explored.

(671) **Mass Spectrometry for Analyzing the Proteome of a Small Number Of Cells**

Liang Li[1]; [1]University of Alberta

There are a number of analytical platforms available in dealing with the proteome analysis of a large number of cells. However, analyzing the proteome from a limited amount of sample is a very challenging task at present. In this presentation, several analytical methods based on the shotgun proteome analysis platform will be described. Issues related to cell lysis, protein extraction, protein digestion, peptide quantification, and peptide identification by LC/MS will be discussed. It will be demonstrated that hundreds of proteins can be identified from as few as 500 cancer cells. Limitations of the current methods for sample handling, mass spectrometric detection and data analysis along with the possible approaches for overcoming these limitations will be discussed.

(672) **Microchip Separations with Integrated Electrospray Ionization**

J. Scott Mellors[1], Andrew Chambers[1], J. Michael Ramsey[1]; [1]University of North Carolina at Chapel Hill

Separations performed in microfluidic devices have potential advantages in speed, solvent consumption, automation, and ease of use over conventional separation methods. Microfluidic devices also enable integration of sample preparation functional elements and/or multiple separation dimensions, offering advantages in analysis time and sample consumption. Over the past 2 decades these advantages have been demonstrated by many researchers for a wide range of separation methods. However, most of the work reported has utilized optical detection methods, which couple easily to microfluidic separations, but provide very little information about the chemical structure of the analytes. The development over the last few years of a sensitive, stable, and efficient microchip electrospray ionization (ESI) interface has enabled the integration of mass spectrometry (MS) detection with several different microchip separation methods. The interface uses an electroosmotic pump to drive fluid out of the chip and ESI is performed directly off of a corner of the glass device, introducing virtually no dead volume. The first application that was demonstrated was microchip capillary electrophoresis (CE)-MS of peptides and proteins. Theoretical plate counts of over 200,000 were obtained in under 3 minutes. Additionally, microchip capillary electrochromatography (CEC)-MS, liquid chromatography (LC)-MS, and LC-CE-MS have been demonstrated. Recent improvements including higher run voltages, new microchip designs, and the use of a higher acquisition rate mass spectrometer have allowed for even faster and more efficient separations. Current work is focused on making further improvements to this technology, applying it to a range of different samples, and testing the limits of high speed microchip CE-MS. This presentation will focus on the most recent results obtained for microchip CE-MS and LC-CE-MS of biologically relevant samples.

(673) **A Proteomic Perspective of the Dynamic Interplay between Viruses and Hosts**

Ileana Cristea; [1]Princeton University

Viruses have evolved finely tuned interactions with their hosts to manipulate and adapt complex cellular processes for their own use. The study of virus-host interactions has therefore emerged as a key driving force in the research of infectious disease during the post-genomic era. Despite these efforts, our understanding of the protein interactome remains, in large part, unknown. The development and incorporation of new approaches that can reveal the dynamics of virus-host protein interactions is a necessity. Modern proteomic techniques have the ability to provide access to such interactions, and the ever increasing sensitivity of mass spectrometry allows the identification and quantification of relatively low levels of proteins. This presentation will describe targeted proteomic approaches for studying virus-host macromolecular assemblies. We have approached the dynamic interface between viruses and hosts by implementing genetic-proteomic approaches from both the virus and host perspectives. Strategies for isolating protein complexes, quantifying changes in interactions during the progression of viral infections, and assessing the specificity of protein interactions will be presented. Highlights will be shown from our studies on infections with human immunodeficiency virus (HIV) and human cytomegalovirus (HCMV). The results discussed will include the discovery of parallel processes occurring at distinct cellular sites during the assembly of virions. Additionally, the presentation will cover aspects of our work on chromatin remodeling complexes, such as histone deacetylases, which are modulated by viruses, possibly in part to gain control over host gene expression and the outcome of an infection.

(674) **Isotopic Labeling Strategies for Quantitative Glycomics**

Ron Orlando[1]; [1]CCRC, University of Georgia; [2]BioInquire; [3]Walter Reed Army Med Cntr; [4]Windber Research Institute

Glycosylation is one of the most common post-translational modifications encountered in eukaryotic systems. One of the analytical challenges facing scientists in the characterization of glycoproteins involves the ability to identify and quantify changes in the attached glycans. This topic is of importance to a variety of researchers ranging from those involved in the batch to batch analysis of recombinant glycoproteins to those involved in glycomics. One of the most accurate methods currently available for quantification, regardless of the analyte, involves the use of isotopic labeling followed by mass spectrometric detection. To this end, we have developed a range of *in vivo* and *in vitro* isotopic labeling strategies for glycan quantification. Four different isotopic labeling strategies have been developed, and have been shown to provide significantly more accurate and reproducible results than label free approaches. Two of these use isotopic variants of methyl-iodide (12CH3I/13CH3I and 13CH3I/12CH2DI) to label the glycans during permethylation. The advantages are that this is simple, and does not alter the normal glycomic workflow. Unfortunately, reproducibility can be problematic since small changes in permethylation efficiency cause large changes in the detected relative abundances because labeling occurs at many sites on each glycan. The third *in vitro* procedure incorporates 18O during PNGaseF deglycoslation. Because this only labels one site per glycan, the reproducibility is significantly improved compared to the permethylation approaches. A second advantage is that 18O

labeling allows glycans from two samples to be combined prior to clean-up and permethylation, therefore eliminating errors resulting from parallel sample processing. The *in vivo* approach relies on the hexosamine biosynthetic pathway that uses the side-chain of glutamine as its sole donor source of nitrogen for aminosugars in the production of sugar nucleotides, and offers all of the benefits associated with SILAC, including the investigation of glycan turnover rates. The focus of this presentation will be to highlight these techniques and to demonstrate their utility on a variety of biological systems.

(675) **Mass Spectrometry-Based Proteogenomic Approaches Reveal Insight into the Activities and Functions of Microbial Isolates and Communities**

Robert Hettich, Alison Russell[1], Brian Erickson[1], Chongle Pan[1], Nathan VerBerkmoes[1], Jillian Banfield[2]; [1]Oak Ridge National Lab; [2]University of California - Berkeley

With the availability of complete microbial genome sequences for cultured organisms as well as more complex environmental samples, systems biology in both isolates as well as microbial communities is becoming feasible by combining genomic, transcriptomic, proteomic and metabolic information. Our current work seeks to develop and demonstrate advanced "shotgun" mass spectrometry techniques for the comprehensive characterization of microbial proteomes. The goal of this research is to enable a detailed glimpse into the functional state and metabolic activities of microbial systems. The approach is based on multidimensional liquid chromatography interfaced on-line with tandem mass spectrometry. A variety of mass analyzers are employed, ranging from quadrupole ion traps (high throughput, rapid tandem mass spectrometry) to Orbitraps and Fourier transform ion cyclotron resonance instruments (high performance, accurate mass measurements). We have focused on experimental approaches for both qualitative and quantitative proteome determinations. While the qualitative work focuses on high-throughput measurements for "deep and wide" proteome characterization, the quantitation is conducted with either label-free or stable isotope-labeled samples. In particular, we have demonstrated how metabolic labeling in either microbial isolates or more complex microbial communities (in lab bioreactors) can yield detailed protein abundance information at the proteome level. More recently, we have incorporated selected reaction monitoring (SRM) on a triple quadrupole MS to pursue absolute quantification determinations. As might be expected, a combination of these approaches is often used to unravel the molecular level details of proteome machinery. We have used these MS-based techniques to characterize a variety of microbial species that are involved in bioremediation, bioenergy, human microbiome health, and environmental communities (such as acid mine drainage). * Research support was provided by the U.S. DOE, Office of Biological and Environmental Research. Oak Ridge National Laboratory is managed and operated by the University of Tennessee-Battelle, L.L.C., under contract DE-AC05-00OR22725 with the U.S. Department of Energy.

(676) **Label-Free Mass Spectrometry-Based Proteomics Study of Ovarian Adenocarcinoma in the Chicken**

Adam Hawkridge[1], Rebecca Wysocky[1], James Petitte[1], Kenneth Anderson[1], Paul Mozdziak[1], Oscar Fletcher[1], Jonathan Horowitz[1], David Muddiman[1]; [1]NC State University

Epithelial ovarian cancer (EOC) remains the most lethal gynecological cancer in the Western world due to a combined lack of effective therapeutics and screening strategies. The inability to identify effective EOC screening strategies has been attributed to the heterogeneous pathogenesis of EOC, difficulty in obtaining significant numbers of human EOC tumors at the initial stages of development, and the lack of *in vitro* and *in vivo* experimental models that recapitulate the onset of EOC. The chicken has emerged as a promising animal model for studying spontaneous EOC due to high disease prevalence (5-35%) and molecular level similarities to human EOC including CA-125 expression, the mutational frequency of the p53, and the up-regulation of HER-2/neu. Specific to our interests and efforts is the ability to collect longitudinal blood samples from chickens over a window of time that spans health, EOC onset, and EOC metastasis and then retrospectively analyzing these samples with advanced proteomics technology. The central hypothesis is that by measuring the within- and between-individual biological variability of the plasma proteomes of chickens as a function of health and EOC, we will be able to evaluate the predictive value of protein biomarkers in the context of both population-based versus personalized-based screening potential. In this study, blood was drawn from 148 age-matched chickens starting at 2.5 years of age every three months for one-year followed by tissue resection. Pathological assessment of the tissues confirmed that 6.8% of birds developed EOC at various degrees of severity. A mass spectrometry-based proteomics workflow including in-gel digestion, nanoLC coupled to high performance mass spectrometry, and label-free (spectral counting) quantification was used to measure the biological intra-individual variability (CVW) of the chicken plasma proteome of two birds; one bird was considered "healthy" and the second bird developed late-stage EOC. Furthermore, the within- and between-run analytical variability (CVA) of the label-free proteomics workflow was also measured using a single plasma sample analyzed multiple times. The short-term goal of this study is to assess the feasibility of using label-free proteomics to measure the biological variability of plasma protein concentrations such that candidate biomarkers can be identified.

(677) **Standoff Detection Using Raman Spectroscopy: Current Status and New Directions**

S. Michael Angel[1], J. Chance Carter[1]; [1]University of South Carolina

Recently there is renewed interest in standoff Raman spectroscopy, in part because of the potential of technique to be used for detection of hazardous materials at a distance, including high explosive (HE) materials. High explosive (HE) detection is becoming more important, with increased suicide bomber and vehicle born improvised explosive device (VBIED) activity. The availability of conventional military explosives means that more comprehensive and sensitive methods of HE detection are needed, especially remote detection of HE and related material residues and vapors near hidden HE. Residue is important because the bulk of HE will normally be hidden and contained in the improvised explosive device (IED). But small amounts of HE and HE-related residue is expected to be found on exposed surfaces and on vehicles in and around areas where IEDs are manufactured or transported from IED handling and manufacture. Current HE detection methods span from simple colorimetric screening to more sophisticated speciation with advanced spectroscopic measurements. These techniques generally require contact or proximal sampling, which can be extremely hazardous if the device detonates. Thus there is a need for reliable, remote and/or standoff detection methods, in some cases covert, to reduce the danger of exposure of first responders and to provide early warning to prevent US military and civilian casualties, and to provide tracking for potential attribution. There are a few spectroscopic techniques that look promising for standoff HE detection including laser-induced fluorescence (LIF), laser ionization (LI), terahertz techniques, passive infrared, laser-induced photo-fragmentation/LIF, laser-induced breakdown spectroscopy (LIBS), and Raman scattering. This talk will be an overview of the current state of the art in standoff Raman detection, optimization considerations and a discussion of new directions.

(678) **Integrated Optical Sensing: Microresonators, Metamaterials, and Chip Scale Integrated Optical Systems**

Nan Jokerst[1]; [1]Duke University

Leveraging nano and micro fabrication technologies developed for integrated circuits, new optical sensing technologies are emerging. Resonant structures at the microscale, such as microresonators, are interesting biochemical sensors that can be integrated in large arrays at the chip scale with integrated optical components such as lasers and photodetectors toward portable sensing systems. Nanofabrication technologies have enabled metamaterial structures for transformational optics – structures that can control the path of light through engineered optical properties. Candidate structures, and the potential impact that resonant and nonresonant optical metamaterial structures may have on sensing will be explored.

(679) **Planar Waveguide ATR Spectroscopy in the Time- and Frequency-Domains: Development and Application to Photochemical and Electrochemical Reactions in Molecular Films**

S. Scott Saavedra[1], Zeynep Ozkan Araci[1], Anne Simon[1], Anne Runge[1], Walter Doherty[1], Clayton R. Shallcross[1], Neal R. Armstrong[1]; [1]University of Arizona

This talk will describe the development and application of waveguide-based tools to measure reaction kinetics in thin molecular films. a) Potential-modulated, attenuated total reflection spectroscopy (PM-ATR) is a frequency-domain technique in which the intensity of visible light propagating in a planar waveguide coated with indium-tin oxide (ITO) is monitored while an ac potential modulation is simultaneously applied to the ITO layer. A thin film of redox-active chromophores is deposited on the ITO layer, which functions as the working electrode. Changes in the absorbance of the film as a function of the light polarization, modulation frequency, and amplitude provide information about electron transfer rates, electro-optical switching rates, and molecular orientation. We have used PM-ATR to study the relationship between electrochemical kinetics and molecular orientation in cytochrome c monolayers, the influence of ITO surface chemistry on electro-optical switching in conducting polymer films, and reversible charge injection into semiconductor (CdSe) nanoparticles tethered to ITO. b) Transient or time-resolved absorbance spectroscopy, also known as flash photolysis, is used widely to study photochemical kinetics in bulk samples. Measurements on substrate-supported films are usually performed in a transmission geometry, which requires that the film thickness be sufficient to generate a path length large enough for adequate sensitivity. Measurements on monolayer films are therefore problematic. We have constructed a transient absorbance instrument based on a multiple-reflection planar waveguide ATR geometry. This capability of this approach for characterization of photo-induced reaction kinetics and heterogeneous charge transfer in thin molecular films will be described.

(680) **RIfS: A Versatile Biosensor Technology for Label-Free Detection**

Guenther Proll[1]; [1]University of Tuebingen

Direct optical detection principles for label-free investigation of bio-molecular interaction processes are widely used in many different fields of research. Currently the improved performance of biosensors capable for array read out and other smart applications stimulates the interest of industry and scientists. Information gained with label-free technologies can be extremely valuable during basic research as well as during drug screening, particularly in combination with other complementing techniques. Label-free optical biosensor detection is performed either by methods based on micro-refractometry (e.g. surface plasmon resonance, grating couplers, Mach-Zehnder interferometry, etc.) or micro-reflectometry (e.g. ellipsometry or reflectometric interference spectroscopy). After an overview of both principles demonstrating benefits and drawbacks of various approaches the focus will be on reflectometric interference spectroscopy (RIfS) and its latest improvements towards imaging-RIfS. This versatile tool can be used for screening of small molecules up to cell based assays. Major advantages are the robustness against temperature changes and the ability to use standard disposables such as microtiter plates and microscopy slides or any other kind of transparent material. The newly developed imaging-RIfS setup allows bio-molecular interaction analysis on free scalable arrays with up to 1000 spots with unmatched flexibility and high performance. This will be demonstrated by means of assays developed for small molecules and nuclear receptors, as well as for cell based assays. Furthermore, it could be demonstrated to combine the advantageous of infrared absorption with the RIfS principle to open up new possibilities for label-free detection with added value, reaching even in a wavelength range up to now not in focus. [1] Pröll F, Fechner P, Proll G, Direct optical detection in fragment-based screening, Anal Bioanal Chem 393(6-7), 1557-1562, 2009 [2] Penttinen P., Jaehrling J., Damdimopoulos A.E., Inzunza J., Lemmen J.G., van der Saag P., Pettersson K., Gauglitz G., Makela S., Pongratz I., Diet-derived polyphenol metabolite enterolactone is a tissue-specific estrogen receptor activator, Endocrinology, 148 (10), 4875-4886, 2007 [3] Fechner P, Pröll F, Carlquist M, An advanced biosensor for the prediction of estrogenic effects of endocrine-disrupting chemicals on the estrogen receptor alpha, Anal Bioanal Chem 393(6-7), 1579-1585, 2009 [4] Möhrle B., Köhler K., Jaehrling J., Brock R., Gauglitz G., Label-free characterization of cell adhesion using reflectometric interference spectroscopy (RIfS), Anal Bioanal Chem 384 (2), 407-413, 2006 [5] IR absorption and reflectometric interference spectroscopy (RIfS) combined to a new sensing approach for gas analytes absorbed into thin polymer films, Leopold N, Busche S, Gauglitz G, Spectrochimica Acta Part A Molecular and Biomolecular Spectroscopy, 72(5), 994-999, 2009

(681) **Raman Spectroscopy for Environmental Analysis**

Karl Booksh[1]; [1]University of Delaware

Four applications of Raman spectroscopy to determine inorganic species in the environment will be presented and discussed. First the use of a field portable Raman spectrometer with a capillary waveguide to determine sulfates, nitrates, and carbonates in geothermal wells will be presented. Second, the application of SERS with silver nanoparticles to quantify ammonium in ground water will be demonstrated. Third, the construction and deployment of a Raman spectrometer to monitor the chemistry of deep sea hydrothermal vents will be dissected. Finally, the use of a Raman microscope to characterize the heterogeneity of sulfur nanoparticles produced and consumed by extremophile microbes will be presented.

(682) **Recent Advances in Surface-Enhanced Infrared Spectroscopy**

Peter Griffiths[1], Ayuba Fasasi[1]; [1]University of Idaho

Two different ways of producing substrates for surface-enhanced infrared absorption (SEIRA) spectroscopy will be described in this talk. In the first, very small gold nanoparticles (AuNPs) are prepared inside micelles of the surfactant, sodium bis (2-ethylhexyl) sulfosuccinate (AOT). AuNPs are formed by reduction of metal cations inside the water-core of a reverse micelle where the micelle acts as a template for controlling the final particle size. A few microliters of an aqueous solution of HAuCl4 are added so that the AuCl4- ions enter the micelles. Gold NPs are then formed by reducing the AuCl4- ions with a saturated solution of NaBH4. The size of the nanoparticles is controlled by the ratio of the concentration of water to AOT in hexane. The alternative approach

involves the deposition of reducible metals (silver, gold, copper and platinum) on germanium and silicon substrates by electroless deposition (ED). Metals formed in this way have been found to have some unusuall properties. For example, copper only forms an oxide layer when in contact with air over a period of a few days. When a monolayer of p-nitrothiophenol (PNTP) is bonded to silver nanoparticles that have been prepared on a germanium substrate (AgGe NPs) by ED, the PNTP is reduced to p-aminothiophenol simply by reaction with water. The reaction rate is increased by the addition of bromide or iodide ions, which facilitate the removal of the oxide layer from the germanium substrate. The same reaction does not take place with AuGe, AgSi or AuSi NPs for which there is apparently a Schottky barrier that prevents electrons from flowing from the germanium substrate to the nitro group of the PNTP.

(683) **Integrated Probing of Protein Purity and Conformation by Capillary Electrophoresis with Wavelength-Resolved Fluorescence Detection**

Bregje J. de Kort[1], Gerhardus J. de Jong[1], Govert W. Somsen[1]; [1]Utrecht University

In the pharmaceutical and biomedical fields there is a growing demand for integrated analytical approaches that allow the assessment of intact proteins. However, the complex and adsorptive nature of proteins makes their characterization quite challenging. Capillary electrophoresis (CE) is a very attractive technique for purity and stability analysis of proteins, as it offers fast and efficient separations and requires only small sample volumes. Protein emission spectroscopy, based on the native fluorescence of aromatic amino acid residues, is a selective and sensitive detection technique which can provide information on protein conformation. This presentation outlines the on-line coupling of CE with wavelength-resolved fluorescence detection (WRFlu) for protein analysis. WRFlu is accomplished using a dedicated fluorescence cell that employs wave-guiding principles to lead protein emission light to a spectrograph with CCD detector. The cell is installed in a commercial CE instrument enabling recording of full protein spectra in a routine fashion. The new CE-WRFlu set-up allows fast separation and detection of proteins down to 10 nM, permitting detection of minor protein impurities and degradation products. Moreover, it will be shown that changes in protein conformation can be probed by the recorded emission characteristics as well as the measured electrophoretic mobility. The ability to monitor protein folding/ unfolding kinetics will be demonstrated for several test proteins, such as carbonic anhydrase II and â-lactoglobulin B. The presented results will indicate that CE-WRFlu provides a highly promising tool for the simultaneous monitoring of protein purity and conformation.

(684) **Characterization of an Automated Capillary Electrophoresis System for Single Cell Analysis**

Alexandra J. Dickinson[1], Dechen Jiang[1], Christopher E. Sims[1], Nancy L. Allbritton[1, 2]; [1]University of North Carolina; [2]North Carolina State University

Capillary electrophoresis (CE) has been demonstrated to be a valuable tool for single cell analysis, but is limited by low throughput (5-35 cells per day.) To increase throughput, a CE system for serial single-cell analysis was designed. This system was comprised of a physiological and electrophoretic buffer flowing perpendicularly to each other. The capillary traveled in an L shape between these buffers. This system was capable of analyzing 1.8 cells per minute. Since the physiological and electrophoretic buffer fluid flows intersected, the stability of this system was dependent on the relative flow rates of the two buffers. If the physiologic buffer flow is interrupted, the electrophoretic buffer can access and lyse the cells. In addition, the capillary requires three seconds to transit between buffers due to the L-shaped path, which decreases the cell analysis rate. To enhance the throughput as well as to minimize cross contamination between the buffers, an air gap was utilized to separate the two buffer systems. In this H design the buffer channels were parallel to each other with the capillary transitioning the trough between the two buffers. The capillary traveled through the air-filled connection channel in less than one second. The buffer streams do not intersect, thus the system is flow rate independent. Cross contamination of buffers, the health and well-being of the cells of cells prior to analysis, and the speed of single cell analysis were characterized for this system.

(685) **Investigation of Electroosmotic Flow Dynamics in Response to Biological Sample Introduction for Capillary Electrophoresis**

Funda Kizilkaya[1], S. Douglass Gilman[1]; [1]Louisiana State University, Department of Chemistry

Capillary electrophoresis (CE) has been used to directly analyze biological samples such as cells, body fluids and other tissues. Molecules in biological samples often adsorb to the inner wall of the capillary, altering the surface chemistry and changing the electroosmotic flow (EOF). Such adsorption affects the migration times of analytes, leading to irreproducible results. There is a limited amount of data describing how biological samples affect EOF dynamics. The continuous monitoring of EOF provides precise information regarding EOF changes throughout the entire separation procedure. This is achieved by measurements of migration rate of photobleached zones of dilute, neutral flurophore at ~1 Hz during a CE run. In order to understand changes in EOF dynamics due to biological samples, model compounds representing biological cell constituents (proteins, carbohydrates, lipids and DNA) were injected into the CE system, and the EOF was monitored as a function of time. Proteins had the largest effect on the EOF by adsorbing to the wall, changing the surface chemistry and zeta potential. The peak widths of the photobleached zones broadened as a result of protein injections. Possible causes of this peak broadening were investigated, and it was found that a parabolic flow profile was the major contributor to the peak variance. The other model molecules did not change the EOF dynamics significantly. Cell lysate and serum samples were also introduced into the system, and these caused the EOF to decrease and peak widths to increase, possibly due to the proteins in the cells.

(686) **Single Cell Analysis of EGFR Activity by Capillary Electrophoresis**

Ryan Phillips[1], Nancy Allbritton[1,2,3], David Lawrence[1, 2]; [1]Department of Pharmacology, UNC Chapel Hill; [2]Department of Chemistry, UNC Chapel Hill; [3]Joint BME Program, UNC Chapel Hill/ NCSU

Cardiovascular disease is the leading cause of death in the US for both men and women. Diesel fuel combustion generates fine and ultrafine particulate matter (PM) that, when inhaled, deposits in the airways and is correlated with hypertension, arrhythmias, and significantly increased risk of heart attack and stroke. While the mechanism underlying these health effects has not been fully elucidated, mounting *in vitro* evidence implicates increased Epidermal Growth Factor Receptor (EGFR) activity as a major contributor to the inflammatory response to PM exposure and the deleterious health effects that result. Analysis of *ex vivo* airway samples taken from human subjects exposed to PM would provide valuable information on the mechanism of cardiovascular disease resulting from PM inhalation, but these samples are difficult to analyze due to very small sample size, cell heterogeneity, and low viability. To address this problem, we will apply a capillary electrophoresis (CE)-based approach to single cell analysis of

kinase activity. Briefly, cells are loaded with a fluorescently labeled peptide substrate, phosphorylated in the intact cell, a single cell is lysed with a nanosecond laser pulse and cell contents are aspirated into a thin glass capillary. Analytes in the cell contents are then separated by CE and detected by laser-induced fluorescence. The high separation efficiency of CE allows separation of phosphorylated and nonphosphorylated species, while the high sensitivity of LIF affords detection limits approaching 10-21 mol, meaning very low concentrations of peptide can be loaded into cells to avoid competitive inhibition of natural signaling events. In the present work, we have successfully synthesized the EGFR peptide reporter, and have demonstrated phosphorylation by EGFR in the test tube and intact A431 cells, including single cells. Furthermore, resistance to degradation by peptidases has been observed in cell lysates and intact cells. Finally, specificity of the EGFR reporter has also been investigated using EGFR kinase-dead B82L cells as well as small-molecule inhibitors of EGFR. Future directions include structural modification of the reporter to improve affinity and specificity for EGFR, as well as analysis of primary airway epithelia in the presence and absence of PM exposure.

(687) **Particle Isolation by Insulating Gradient Dielectrophoresis (iDC-GDEP)**

Sarah Staton[1], Kang Ping Chen[2], Tom Taylor[3], Jose Rafael Pacheco[2, 4], Mark Hayes[1]; [1]Arizona State University Dept. of Chemistry; [2]Mechanical and Aerospace Engineering; [3]Mathematics and Statistical Sciences; [4]Center for Environmental Fluid Dynamics

Separation of bioanalytes is frequently complex and difficult. Bioanalyte samples contain a multitude of components that range in size over several orders of magnitude. For example, bacteria (~1 μm – 10 μm) and viruses (several tens of nanometers to slightly over 100 nm) are found greatly outnumbered in complex matrixes like blood. Current technology to separate and isolate bioanalytes is generally time consuming and focused on a single analyte. In response to these limitation we present a scheme to separate particles according to their characteristic physical properties, including size, charge, polarizability, deformability, surface charge mobility, dielectric features, and local capacitance. Separation is accomplished using an insulating DC gradient dielectrophoresis (iDC-GDEP) based microdevice that can isolate and concentrate multiple analytes simultaneously at different positions. The device is dependent upon dielectrophoretic and electrokinetic forces in a DC field using shaped insulating features within the channel acting in opposition. Dielectrophoretic and electrokinetic forces each have a rich set of properties by which they separate, however by placing these forcing in opposition a fuller set of their combined vectors of separation can be exploited for sample refinement. Sulfate- capped polystyrene nano and microparticles (20nm, 200nm, and 1 μm) were used as probes to mimic the bioanalyte sizes of interest in preliminary tests to demonstrate the influence of channel geometry and separation behavior of iDC-GDEP within that size range. These results are consistent with models using similar channel geometry and indicate that species can be isolated within a distinct portion of the device. Controllable and selective isolation of the three particle types were achieved, as well as the co-capture of 200 nm and 1 μm particles simultaneously in different sections of the device. Particle enrichment found during the isolation of 200 nm particles was concentrated by a factor of 103 experimentally, and theoretically could be as high as a 106 concentrating factor. iDC-GDEP shows promise for future bioanalytical separation and isolation in the size range of nanometer and micron bioanalytes, a size range encompassing several interesting targets.

(688) **Magnetic Bead Microreactors for Studies of On-Column Two-Enzyme Reactions**

Rachel Henken[1], S. Douglass Gilman[1]; [1]Louisiana State University

We report here the impact of microreactor configuration and composition for studies of on-column two-enzyme reactions using capillary electrophoresis with laser induced fluorescence detection. The enzymes, choline oxidase and horseradish peroxidase (HRP), were covalently bound to two batches of magnetic beads. The two enzyme microreactor relies on a product of choline oxidase as a substrate for HRP, which produces the fluorescent species resorufin. The magnetic beads are immobilized within the capillary by rare earth magnets external to the capillary. Substrates were introduced to the microreactors by electrophoretic flow both through injection and continuous flow. The effect of order, placement, composition, and distance between the enzyme coated bead microreactors on reaction kinetics are presented.

(689) **Developing a New Toolbox for Analysis of Warrior Wound Biopsies: Vibrational Spectroscopy**

Nicole Crane[1], Eric Elster [1, 2, 3]; [1]Naval Medical Research Center; [2]National Naval Medical Center; [3]USUHS

The management of modern traumatic war wounds remains a significant challenge for clinicians. This is a reflection of the extensive osseous and soft-tissue damage caused by blasts and high-energy projectiles. The ensuing inflammatory response ultimately dictates the pace of wound healing and tissue regeneration. Consequently, the eventual timing of wound closure or definitive coverage is often subjectively based. Some wounds fail to close, or dehisce, despite the use and application of novel wound-specific treatment modalities. Additional wound complications include wound infection and biofilm formation and heterotopic ossification (the pathological mineralization of soft tissues). An understanding of the molecular environment of acute wounds throughout the debridement process can provide valuable insight into the mechanisms associated with the eventual wound outcome. Currently, we are examining wound biopsies and wound effluent (a proteinaceous mixture that oozes from the wound) from combat-wounded warfighters, and exploring the use of vibrational spectroscopy to answer three clinical questions: 1) Are there bacteria present in the wound (i.e. infection)? 2) Is the wound developing heterotopic ossification? 3) Will the wound heal normally? The analysis of Raman spectroscopic maps of wound biopsy tissue sections revealed a decreased 1665 cm-1/1445 cm-1 band area ratio in impaired healing wounds, indicative of an impaired remodeling process. The examination of debrided tissue reveals mineralization during the early development of heterotopic ossification. Finally, preliminary results suggest that FT-IR images of wound effluent may be able to provide early microbiological information about the wound. Vibrational spectroscopy, specifically Raman and FT-IR spectroscopies, have the potential to offer improved objective assessment of combat wounds, resulting in faster healing times, decreased infection rates, and decreased local and systemic complications of injury. This, in turn, will produce improved clinical outcomes, decreased patient morbidity, and reduced medical costs.

(690) **Raman Spectra of Scars**

Adrian Eugenio VIllanueva Luna[1], Jorge Castro Ramos[1], Sergio Vazquez- Montiel[1], Jose Alberto Delgado Atencio[1]; [1]Instituto Nacional de Astro., Op y Elec.(INAOE)

This paper presents a study on different types of scars caused by cuts, burns and scrapes, which have long been done. This study helps us see the changes suffered by the skin along the wound healing. Therefore the Raman spectra obtained show the change between the new skin of the scar and skin that was before having the wound. The results are derived from taking 10 people with

different scars. These Raman spectra will be linked in the future with the changes that generate malignant melanomas that cause skin cancer.

(691) **Raman Spectroscopy of Neonatal Mouse Skull Soft Spot Reveals Cyclic Mineral Deposition Dynamics**

John-David McElderry[1], Guisheng Zhao[2], Renny Franceschi[2], Michael Morris[1]; [1]Department of Chemistry, U of Michigan; [2]School of Dentistry, U of Michigan

Bone formation is affected by physiologic pathways which are known to fluctuate in diurnal cycles. Systemic markers for bone formation, such as osteocalcin, display cyclic fluctuations consistent with diurnal cycles. However, direct detection of cyclical, local changes in bone formation has not been previously reported. Using a custom 830 nm Raman inverted microscope and stage incubation chamber, we tracked the kinetics of first mineral formation in the soft spot of *ex vivo* tissue cultures of neonatal mouse skulls. We achieved a time resolution of 20-30 minutes to observe mineral dynamics occurring on the hour time scale. Mineral phosphate levels in the tissue increased in short (1-4 hour) intervals several times during the first 6 days after birth. Time between mineralization bursts varied from 8 to 12 hours. We simultaneously probed a series of points 25 μm apart in a line perpendicular to the edge of the interparietal skull plate within the soft spot. Mineral was detected sequentially along the line. The mineral nearest the interparietal plate displayed a linear propagation rate similar to values previously reported for osteoblast cell culture mineral formation [1]. Earliest measurements of the mineral contain a phosphate ν_1 Raman band centered at 958 cm $^{-1}$ which matches values for apatitic mineral in mature murine long bones. Time resolved gene expression assays were performed on osteoblast and clock gene mRNAs to compare physiologic rhythms to bone formation rhythms. Based on these studies, we conclude that mineralization in developing bone tissue undergoes periodic step-wise increases in mineral content driven by local factors in the bone. This is the first reported evidence for periodic biomineralization in bone development. Our findings suggest a more complex mechanism for bone mineralization than previously thought. 1. Dallas et al. Cells Tissues Organs 189:6, 2009.

(692) **Non Invasive Pathology the 'Raman Style': Detecting Breast Cancer by Probing Microcalcifications**

Marleen Kerssens[1,2], Keith Rogers[2], Pavel Matousek[3], Nick Stone[1,2]; [1]Gloucestershire Hospitals NHS Foundation Trust; [2]Cranfield University; [3]Central Laser Facility, RAL

Microcalcifications in breast are an important indicator for breast cancer, and often the only sign of its presence. Several studies have suggested that the type of calcification formed may act as a marker for malignancy and its presence may be of biological significance. Microcalcifications can be found in both benign and malignant breast lesions and their composition can indicate the possible disease state. The accurate and safe diagnosis of breast cancer is a significant issue, with annual incidence of 44,000 women and around 300 men in the UK. Early diagnosis of the disease allows more conservative treatments and better patient outcomes. Type I microcalcifications are composed of calcium oxalate dihydrate (COD) and are associated mainly with benign lesions, whereas calcium hydroxyapatite (HAP, type II) can be present in both benign and malignant breast lesions. It has been shown that small chemical variations in HAP are related to the pathology of the surrounding tissue. The standard screening procedure mammography cannot reliably distinguish between COD and HAP, let alone see more detail of HAP. So although calcifications are an indicator, breast biopsies always have to be done in order to make a correct diagnosis. In contrast, Raman spectroscopy has the inherent capability to distinguish the two types of calcifications and also, for example different amounts of carbonate substitution levels in HAP. However, due to low signal intensity it is difficult to distinguish this non-invasively through several millimetres of soft tissue. Here we report results of a preliminary study evaluationg the chemical differences of calcifications and investigating whether our technology meets clinical requirements. The new results pave the way towards a new generation of non-invasive methods for cancer screening based around transmission Raman and Spatially Offset Raman spectroscopy.

(693) **Assessing Bone Fragility in Chemically-Aged Bone with Raman Spectroscopy**

Jessica Lopez[1], Gurjit S. Mandair[1], Michael D. Morris[1]; [1]University of Michigan, Department of Chemistry

The accurate assessment of bone fragility depends on a greater understanding of bone compositional contributors to bone quality. Raman spectroscopy has widely been used to examine the mineral and matrix composition of bone compromised by ageing, disease, or injury. Assessment of bone mineral heterogeneity may also be used as a measure of bone quality, whereby tissues with different degrees of mineralization, age, or turnover are taken into consideration. In this study, Raman spectroscopy is used to examine the heterogeneity of bone mineral distribution in chemically-aged cortical and cancellous bone specimens. Canine tibial bone sections were progressively demineralized in 0.5 M EDTA solutions in order to simulate the conditions of age-related bone mineral losses and changes in bone mineral heterogeneity. Changes in compositional measures of bone quality, such as mineral/matrix and carbonate/mineral ratios were monitored using Raman mapping techniques. For each Raman map, pixel histograms were generated and fitted with a Gaussian distribution curve to calculate the mean mineral/matrix and carbonate/mineral distributions. Full width at half-maximum (FWHM) of the Gaussian curves were then used to determine the heterogeneity of the distributions as a function of increased demineralization time. Preliminary results show that increased demineralization of cortical bone resulted in reduced mineral/matrix ratios and altered bone tissue heterogeneity. In conclusion, we show that measures of bone mineral heterogeneity taken together with bone compositional measures provides greater insights into bone quality, which may be used as potential predictors of fracture risk in prospective clinical studies.

(694) **Deep Ultraviolet Resonance Raman Spectroscopy of Formulated Insulin**

Sergey Arzhantsev[1], Connie Gryniewicz-Ruzicka[1], John Kauffman[1]; [1]US Food and Drug Administration

A formulated therapeutic protein contains chemicals that stabilize the protein secondary structure and protect it from degradation. To ensure the safety and quality of protein drugs it is beneficial to find methods that can be used to determine the degradation stage of the protein inside a formulated product. Raman spectroscopy is a powerful analytical method that can be used to evaluate degradation of isolated proteins in solution. Unfortunately, Raman scattering from stabilizers can mask the protein signal and as a result Raman spectroscopy is often ill-suited for the analysis of formulated products without separation. Using deep UV excitation can greatly enhance Raman scattering from the protein due to resonance enhancement of the amide bonds in the protein backbone. Insulin is one of the oldest formulated protein products on the US market, and the degradation processes of insulin are well studied. This presentation will evaluate the ability of deep UV resonance Raman spectroscopy to extract insulin's Raman spectrum from a mixture of materials, such as a formulated insulin product. The emphasis of this work is to develop a simple and rapid method for the detection of insulin degradation in formulated

products using deep UV resonant Raman spectroscopy. Chemometric algorithms to improve the isolation of the protein signal from the total solution spectrum will be applied. The results for model systems as well as formulated product will be presented and discussed.

(695) **Application of Chemometric Techniques to Open-Path FT-IR Spectra Measured under Conditions of Severe Interference**

Peter Griffiths[1], Limin Shao[2], April Leytem[3]; [1]University of Idaho; [2]University of Science and Technology of; [3]U. S. Department of Agriculture

The measurement of open-path Fourier transform infrared (OP/FT-IR) spectra is always subject to strong interference by the rotational bands in the vibrational spectra of water and carbon dioxide. Water vapor presents the largest interference as the absolute intensity of each line is directly proportional to the absolute humidity and the relative intensity of all lines is strongly dependent on temperature, so that water lines cannot be removed from OP/FTIR spectra by simple subtraction and classical least squares regression (CLS) cannot be used to compensate for their absorption. The situation is exacerbated by the fact that most OP/FT-IR spectra are measured at a resolution that is considerably broader then the true width of the lines, making the spectra susceptible to resolution errors. We have been able to improve upon the performance of CLS by a procedure that involves partioal least squares regression (PLS). A calibration set is acquired by measuring a large number of single-beam OP/FT-IR spectra through pristine atmospheres with a wide range of temperatures, humidities and path-length and ratioing each against a short-length background. The spectra were converted to absorbance and to each was added the reference spectra of the analytes of interest. The performance of this approach was far better than the more commonly used CLS procedure used for OP/FT-IR spectroscopy. Examples of measurements made at a dairy farm and under simulated battlefield conditions where fireworks were exploded in the infrared beam will be shown.

(696) **Airborne Monitoring of Volatile Organic Compounds by Passive Infrared Spectroscopy**

Gary Small[1]; [1]University of Iowa

The detection of airborne chemicals is a key capability in a variety of environmental monitoring scenarios. For these applications, passive infrared remote sensors collect infrared emissions from natural and manmade sources such as the radiant emission from the earth or emissions from the stacks of a chemical plant. Chemical compounds absorb or emit infrared energy at characteristic wavelengths, and the profile of these absorption or emission signatures can be used to identify a chemical and to estimate the amount present. Passive infrared remote sensors can be implemented in either imaging or non-imaging configurations and can be constructed to acquire infrared emission data in either multispectral or hyperspectral modes. Implementing these measurements successfully requires the construction of rugged and portable instruments capable of being mounted on platforms such as moving aircraft or ground vehicles. In addition, sophisticated computer processing techniques must be designed to allow the automated analysis of the large quantities of data acquired by these sensors. The research presented here describes the development of novel signal processing and pattern recognition methodology for application to multispectral imaging data and to non-imaging data acquired with a hyperspectral instrument. Remote sensing data were collected with these instruments mounted on an aircraft platform as part of the U.S. Environmental Protection Agency's ASPECT emergency response program. Remote measurements collected during several field responses will be used to evaluate the data analysis methodology and to assess the strengths and weaknesses of the imaging and non-imaging remote sensing approaches.

(697) **Predicting Wastewater Treatment Efficacy**

Lisa Morkowchuk[1], Michael Krein[1], Alison Kennicutt[2], Curt Breneman[1], James Kilduff[2]; [1]Rensselaer Polytechnic Inst, Chemistry & Chem Bio; [2]Rensselaer Polytechnic Inst. Civil & Env

The removal of endocrine disrupting compounds (EDCs) and pharmaceuticals and personal care products (PPCPs) from wastewater is important for the preservation of the health of ecosystems and to prevent contamination of drinking water sources. Common techniques used by treatment facilities to remove these compounds include filtration, oxidation, and activated carbon adsorption. In this talk, we present work to date on building robust, cross-validated quantitative structure-activity relationship (QSAR) models capable of predicting the efficacy of these common techniques on previously unseen compounds. We will describe our modeling process, including molecular structure encoding and descriptor generation, with an emphasis on validation techniques. Challenges include the responsible merging of data sets, accounting for matrix properties, such as pH, that may vary across source waters and process parameters, such as contact time, that may vary across treatment protocols. We will also discuss the creation of two publicly available online tools. The first is a QSAR model-building tool capable of partial least squares, kernel partial least squares, and support vector machine regression, as well as principle component analysis, cross-validation, sensitivity analysis, and y-scrambling. The second is a reverse-QSAR prediction tool that will allow water treatment facilities to access and use our models to inform removal procedures.

(698) **Multiway Analysis of EEMs to Analyze Microbial Carbon Cycling in Estuarine Waters**

Gregory Hall[1], Jennifer Edmonds[2]; [1]U.S. Coast Guard Academy; [2]University of Alabama

Evaluating the chemical composition of dissolved organic carbon (DOC) in stream ecosystems can elucidate both the source of the material and its bioavailability to the heterotrophic microorganisms (Bacteria and Archaea) that utilize it. DOC contributions to the surface flow of a stream from wetlands present in the catchment may alter carbon cycling through increased C supply and higher lability, stimulating mineralization and changing the organic chemistry of water exported from the stream. Coupling measures of microbial community composition to patterns in the timing and percent contribution of wetland DOC sources to stream chemistry could identify which microbial groups are able to utilize this material. Excitation-Emission Matrices (EEMs) analyzed by PARAFAC and NPLS can be used to evaluate DOC chemical composition of surface water samples and elucidate these patterns. Our work focuses on two stream ecosystem types where wetland contributions could be significantly influencing DOC pools. One dataset includes Alaskan streams positioned along a gradient in wetland contribution, and the second dataset comes from a coastal plain stream in Alabama where beaver wetlands are embedded in the stream network.

(699) **Using Lignin-Derived Phenols to Measure Terrestrial Organic Matter Inputs to Western Long Island Sound**

Eric Miller[1], Annelie Skoog[1], Greg Hall[2]; [1]University of Connecticut, Dept. of Mar. Science; [2]U.S. Coast Guard Academy, Science Dept.

Measuring lignin-derived phenols produced from marine and estuarine sediment samples provides useful information about terrigenous organic matter inputs to these environments. When lignin is heated under alkaline conditions in the presence of CuO, an oxidative hydrolysis reaction generates acid, aldehyde, and

ketone products that reflect the biopolymer's four basic structural families. Specifically, eleven phenols are derived: nine aldehyde, ketone, and acid analogues for the syringyl, vanillyl, and p-hydroxyphenyl families, and two acid analogues for the cinnamyl family. Once isolated and quantified, lignin-derived phenols are used in different ratios and summations to provide clues about the geographic origin, degree of terrestrial influence, and extent of biodegradation in a sample. Western Long Island Sound (WLIS) experiences frequent episodes of hypoxia that may be linked to high shellfish mortality rates and other water quality problems in the region. Analyses of the lignin-derived phenols and other organic parameters of WLIS may contribute to a better understanding of the organic carbon and nutrient biogeochemical cycles within these commercially important but troubled waters. Sediment cores were collected from three different stations located within a 12-km transect in WLIS where seasonal hypoxia occurs. Lignin-derived phenol, stable isotope (ä13C), and total organic carbon measurements were made on six different horizons cut from each core. Scores from PCA models of these chemical parameters indicate significant variance between the three stations and PCA Loadings help to explain the differences observed between the locations. The most hypoxic and western station (#4) is closely associated with the highest phenolic acid to aldehyde ratios of the three stations, thereby suggesting that terrestrial material deposited at this site either arrives in a degraded form or undergoes increased degradation *in-situ*. The eastern-most station (#2) was most closely associated with the highest cinnamyl to vanillyl ratio and total lignin-derived phenol measurement, thereby suggesting that terrestrial material is being deposited and/or maintained in a less-degraded condition at the least hypoxic station. These results suggest that there may be an interplay between the degree of hypoxia and the quality and quantity of terrestrial organic matter in WLIS.

(700) **Computational Aspect of Two-Dimensional Correlation Spectroscopy**

Isao Noda ,; [1]The Procter & Gamble Company

Two-dimensional (2D) correlation analysis has become a popular and versatile tool in the last two decades for the analysis of various spectral data. Spectral intensity variations, which are induced by a chosen perturbation applied to the sample, are systematically examined by a cross correlation method. The result is displayed in the form of a set of graphical 2D maps, defined by two independent spectral axes, to provide improved spectral resolution, as well as valuable information about the relative directions and sequential order among the spectral intensity variations. Theoretical foundation of 2D correlation, originally developed for the examination of simple sinusoidal signals, is based on the classical time-series analysis. The extension of the technique to the analysis of signals arising from a perturbation with an arbitrary waveform, combined with the introduction of fast discrete Hilbert transform algorithm, made 2D correlation analysis a generally applicable tool of choice for many practical problems in analytical science. In this talk, basic topics often ignored even by experienced practitioners, such as why 2D correlation approach really works and how the current computational method used in generating 2D correlation spectra are derived, will be discussed to provide the intuitive understanding and insight into the inner working of 2D correlation spectroscopy.

(701) **Methodologies for Extracting Useful Insights from Spectral Images**

Curtis Marcott[1]; [1]Light Light Solutions

The combination of spectroscopy and imaging, known as Spectral (or Hyperspectral) Imaging, is a rapidly emerging area of analytical science. It is now possible to perform spectral imaging measurements from the ultra-violet region all the way into the far-infrared (terahertz) region of the electromagnetic spectrum. An important concept is the representation of spectral imaging data as a cube: x and y directions being spatial coordinates, with the wavelength of light absorbed being represented along the z direction of the cube. Current array detectors used in spectral imaging applications can contain up to 80,000 pixels, or more, presenting a tremendous data analysis challenge. A number of approaches for extracting useful chemical information from these massive data sets will be discussed, including chemometric techniques.

(702) **Transfer and Implementation of Chemometric Models**

John Wasylyk[1], Ming Huang[1], Robert Wethman[1]; [1]Bristol-Myers Squibb Co.

Chemometric models have become the backbone for the analysis of complex spectroscopic data and have been applied to solve both descriptive and predictive problems. The resulting predictions are highly dependent on the quality of the model and reliable models are typically created with numerous replicas. Replicate data includes data from experiments designed to build robustness into the model, extend the design space and ultimately enhance its transferability. Considerations such as establishing a cohesive off-line and in-line environment will aid in preparation of a transferable chemometric model. Likewise, choosing the best predictive model, avoiding pitfalls such as over- or under-fitting, and establishing consistent work practices improves transfer success. Once a chemometric model has been optimized and tested 'in-house' the chance for a successful transfer to a manufacturing environment is greatly improved. We will present case studies on developing and transferring chemometric models from lab, to pilot plant, and finally to manufacturing in a regulated and compliant environment.

(703) **Calculation of Vibrational Spectra for the Assignment of Chemical Structure**

Don Pivonka[1], Steven Wesolowski[1]; [1]AstraZeneca

Vibrational spectroscopy can provide an extremely selective compound signature for the investigation of unknown compounds. While collection of an actual spectrum is often not an issue, interpretation of unknown spectra (especially when the compounds are novel and or undocumented) can be difficult if not impossible. The task is particularly formidable in the absence of spectral libraries containing the specific compounds or at least similar analogs to the compound in question. With recent advances in computational technologies, it is now often possible to accurately calculate infrared (IR), Raman and vibrational circular dichroism (VCD) spectra for use as primary references in the interpretation of unknowns. This technology is especially important for the interpretation of unknown isomers for which a discrete set of structural possibilities exists and is known. Isomer classes including ring substitution isomers, cis/trans isomers, and chiral compounds often lend themselves to informative computational spectral analysis. This presentation will serve as an introduction to the fundamentals of calculating vibrational spectra. Conformational searching, energy minimization, and Boltzmann weighting of conformational contributions in the generation of calculated spectra will be discussed.

(704) **Analysis of Data Arrays from Hyphenated Instrumentation - Multi-Way Methods Such As Parallel Factor Analysis (PARAFAC)**

Karl Booksh[1]; [1]University of Delaware

Multiway calibration methods offer the possibility of accurate and reliable quantitative analysis in the presence of unknown unclaibrataed spectral interferents. This presentation will

demonstrate the power and limitations of multiway methods for application with hyphenate methods such as excitation-emission matrix fluorescence and HPLC-UV/Vis spectroscopies. The advantages and disadvantages of different multiway methods will be discussed and demonstrated the data. The use of the multiway methods for exploratory analysis, quantitative analysis, and classification will be covered.

(705) **Mid-Infrared Imaging as a Label-Free Alternative to Immunohistochemistry for Breast Cancer Pathology**

Michael Walsh[1], Andre Kajdacsy-Balla[2], Rohit Bhargava[1]; [1]University of Illinois at Urbana-Champaign; [2]University of Illinois at Chicago

Histologic diagnosis is the gold standard for evaluating the presence and severity of most cancers. Unfortunately, the manual nature of histopathologic recognition leads to low throughput analysis, delays in decision-making and errors. Here, we report on the evaluation of an automated means for accurate histologic recognition using mid-infrared (IR) spectroscopic imaging. This method does not require dyes or probes and dispenses with human input but relies on the inherent biochemistry of unstained tissue coupled with computational approaches to provide histologic information. We present here examples of this approach using normal and cancerous breast tissue microarrays. Comprehensive diagnosis of breast tissues require serial tissue sections being collected which are subsequently stained with a panel of immunohistochemical (IHC) stains to identify key cell types and cancer subtypes. The identification of cell types, such as myoepithelial cells, is critical for accurate cancer diagnosis and staging. Subtyping of breast cancer using IHC, in particular testing for estrogen, progesterone and Her2/neu receptor status is critical for effective treatment. A panel of twelve IHC stains that are routinely used in clinical practice for cell type and cancer subtype identification were compared to IR imaging. Results demonstrate that IR imaging is capable of accurate segmentation of cell types and disease subtypes that can potentially become competitive with that attained by conventional IHC analyses. Progress in high resolution IR imaging has led to the ability to resolve cell types and tissue structures that are not readily identifiable using conventional transmission FTIR. The identification and chemical characterization of these features may also be critical for accurate staging and diagnosis of breast cancers.

(706) **Standardization of Raman Spectral Library Methods for Rapid Screening of Pharmaceutical Raw Materials**

Jason Rodriguez[1], Lucinda Buhse[1], Benjamin Westenberger[1], John Kauffman[1]; [1]FDA Div. of Pharmaceutical Analysis

Recent studies by the FDA Division of Pharmaceutical Analysis have demonstrated that portable Raman spectrometers are capable of achieving sub percent-level surveillance of contaminants in pharmaceutical materials using chemometric methods. Although chemometric methods are highly sensitive, they also require substantial sample preparation and model development. One alternative is to utilize Raman spectral library-based methods for materials verification. Library-based material verification methods do not require extensive model development and are capable of making qualitative identification of an unknown sample through a measure of similarity such as a hit quality index (HQI). While library-based methods have not been traditionally employed to make quantitative assessments, they can be used to screen material needing more in-depth analysis. The biggest drawback of using library based methods is the lack of transferability of spectral libraries between different Raman instruments. We report our efforts to establish a transferrable, standardized library of pharmaceutical raw materials that will be used to evaluate the usefulness of library-based methods for rapid pharmaceutical screening. Particular emphasis is given to the procedures involved in determining the instrument-specific spectral corrections for each Raman spectrometer included in the study and the standardization of the library spectra. Our findings indicate that there are several crucial aspects in establishing a standardized Raman library that include calibration of each spectrometer for both Raman shift and relative intensity.

(707) **Raman Analysis of Pharmaceutical Powders and Tablets**

David Littlejohn[1], Pamela Allan[1], Luke Bellamy[1], Alison Nordon[1], Nichola Townshend[1], John Andrews[2], Paul Dallin[2]; [1]University of Strathclyde; [2]Clairet Scientific

Recent advances in wide area illumination methods to increase the sampling volume, and thus improve the representativeness of the sample measured, have resulted in new opportunities for Raman spectrometry in the analysis of solids. In this work, a Kaiser PhAT probe system was used to acquire non-invasive reflectance Raman spectra of a powder blending process. Initially, experiments were carried out on static materials to establish the information depth of the technique. The mixing of aspirin and microcrystalline cellulose in a small-scale convective blender was then monitored. The ability to study the mixing dynamics and to make quantitative measurements will be discussed, and comparisons made with results obtained using near infrared spectrometry. In addition, a Kaiser workstation was used to acquire both reflectance and transmission Raman spectra of tablets with an active pharmaceutical ingredient (API) content of 3 % w/w. Calibration tablets were prepared and used to predict the API concentration. The relative merits of reflectance and transmission measurements for the quantitative determination of API in tablets will be discussed.

(708) **Using PCA to Understand Raman-DSC Data**

Richard Spragg, Kevin Menard; [1]PerkinElmer LAS

Raman spectroscopy and differential scanning calorimetry (DSC) are complementary techniques with the spectra providing qualitative information not available from DSC alone. Simultaneous measurements are important because material properties are often affected by the thermal history of the sample. The resulting data sets can consist of several hundred spectra. The spectra are often quite noisy because the ideal sample for DSC is a thin layer and acquisition times are short. PCA is a versatile tool for investigating these data. We will illustrate a number of applications using data for pharmaceutical compounds and polymers. Where necessary the data quality can generally be improved by reconstructing the spectra from a limited number of principal components so reducing the noise. PC scores plots can confirm that changes seen in the Raman spectra correspond to the peaks in a DSC curve and can help separate overlapping events that are seen as a complex envelope by DSC. They are valuable in distinguishing forms with very similar spectra as is seen in the much studied case of acetaminophen where it is apparent that the three well known crystalline polymorphs are not sufficient to account for all the spectra that can occur. They reveal slow changes that are not evident by DSC where the signal depends on the rate of a change. Scores plots also provide quantitative information, for example about the degree of crystallinity in a polymer. Loadings plots identify subtle spectral changes of band position and width, appearing as derivative-shaped features.

(709) **Assessing the Sample Heterogeneity and Determining the Minimum Sampling Ratio for the Healthy Statistical Representation Using Raman Spectroscopy**

Eunah Lee[1], Sergey Mamedov[1], Fran Adar[1], Andrew Whitley[1]; [1]HORIBA Scientific

The demand and pressure on pharmaceutical development is constantly increasing, and as a consequence the requirement put on analytical technologies for improved instrumentation and methodologies is constant. There are needs for increased accuracy, reproducibility, robustness, easy-of-use, and automation for faster and better understanding between processing and performance. One critical aspect in reducing both measurement and development time is determining the level of statistical sampling. Whether it is measuring a portion of a sample or a subset of a batch, both are critical in the data reliability and speed of measurement. On one hand, a certain level of sampling ratio is required for the measured part to be the healthy statistical representation of the whole. On the other hand, a high sampling ratio calls for a high number of measurements, and thus causes a high time penalty. It is imperative for the effective process analysis to determine the minimum sampling ratio that can provide reliable statistical representation of the entire sample or sample set. However, the required sampling ratio changes as the degree of heterogeneity of the sample or sample set with theoretical extremes. On one end, a completely homogeneous sample or completely uniform set of samples would require an infinitesimal sampling ratio. On the other end, a completely heterogeneous sample or completely random set of samples would require 100 % sampling ratio. Therefore, determining the required sampling ratio is a two step process – first assess the heterogeneity of the sample or sample set, and second determine the required sampling in correlation to the degree of sample heterogeneity. A previous study[1] demonstrated a method of determining the degree of heterogeneity using NIR chemical imaging. This paper will expand and extend the method to Raman microscopy. Raman imaging will be used to determine the degree of sample heterogeneity within individual sample, and results from high dose and low dose samples will be compared. [1] 'Assessing Homogeneity Using Chemical Imaging', Andrew Brookes, IFPAC 2008

(710) **Accuracy of Quantification of Pharmaceutical Formulations Using Transmission Raman Spectroscopy**

Matthew Bloomfield[1], Darren Andrews[1], Craig Tombling[1], Paul Loeffen[1], Pavel Matousek[1,2]; [1]Cobalt Light Systems; [2]Central Laser Facility, RAL

Transmission Raman spectroscopy is an emerging analytical tool with wide unexplored potential in pharmaceutical analysis, in particular process and quality control. The technique's benefits include volumetric probing capability with largely absent sub-sampling, which otherwise plagues conventional Raman spectroscopy, and the ability to suppress surface capsule or tablet coating interfering fluorescence and Raman components permitting accurate quantification of intact pharmaceutical capsules and tables non-invasively with no sample preparation(1). This presentation will discuss the dependence of the accuracy of quantification of pharmaceutical formulations as a function of various experimental parameters including spectral signal-to-noise ratio, spectral resolution of the detection system, the choice of spectral region selected for the analysis and the choice of data analysis method. The understanding of these parameters is crucial for the effective deployment of the technique in practical applications. Reference 1. N. A. Macleod and P. Matousek, Appl. Spectrosc. 62, 291A-304A (2008).

(711) **Calibration Transfer Across Multiple Portable Raman Spectrometric Instruments Used for Pharmaceutical Surveillance**

Connie Gryniewicz-Ruzicka[1], Sergey Arzhantsev[1], Benjamin Westenberger[1], Lucinda Buhse[1], John Kauffman[1]; [1]US Food and Drug Administration

The past decade has witnessed a dramatic increase in the number of pharmaceutical products and materials imported into the US. A small number of imported materials have been found to be adulterated with contaminants that pose a serious health risk to consumers. To ensure the safety of the American drug supply, rapid and reliable screening methods are being developed by the FDA to assess the quality of drug products and excipients. Portable Raman spectrometers are particularly attractive for this application because they require little or no sample preparation and can be used for on-site testing at manufacturing and import facilities. Raman spectroscopic methods using multivariate calibration models were developed to screen for the presence of contaminants in common pharmaceutical excipients. Partial least squares (PLS) calibration models for the quantitative determination of a contaminant in an excipient were developed using training sets measured on a single "master" instrument. The Transfer-of-Calibration (ToC) repeat samples used to standardize the slave instruments were selected from the training sets using the Kennard-Stone algorithm. Calibration models were distributed to four "slave" instruments using piecewise direct standardization as the calibration transfer procedure. ToC samples and a set of test samples were analyzed on each of the slave instruments, and the calibration model developed on the master instrument were used to predict the composition of the test samples. The performance of the calibration models applied on the slave instruments were evaluated based on the root mean square error of prediction (RMSEP). Several issues regarding model development, selection of ToC samples, and field implementation of portable Raman spectrometers with chemometric calibration models will be discussed.

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