



**2010 Winter Conference on
Plasma Spectrochemistry
Fort Myers, Florida
January 3 - 9, 2010
Sanibel Harbour Resort & Spa**

2010 Winter Conference on Plasma Spectrochemistry
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Dr. Ramon M. Barnes, Conference Chairman

2010 Winter Conference on Plasma Spectrochemistry

Short Courses

***Saturday – Monday,
January 2 – 4, 2010***

Course Descriptions

Course Hours:

8 am - 12 noon

1 pm - 5 pm

7 pm - 11 pm

***Fort Myers,
Florida***

January 4 – 9, 2010



2010 Winter Conference Short Courses

Saturday, January 2 – Monday, January 4, 2010

Summary

Analysis by Plasma Spectrochemistry

- SA-01 Elemental Testing in Toxicology**, Sunday, January 3, 8 am, Ela Bakowska
- SA-02 Analysis of Foods and Food Products**, Sunday, January 3, 1 pm, Deborah Bradshaw
- SA-03 Mass Spectrometry in Protein Quantification**, Sunday, January 3, 7 pm, Jörg Bettmer, Maria Montes-Bayón
- SA-04 Arsenic and Mercury Speciation in Biological Samples**, Monday, January 4, 7 pm, Jörg Feldmann, Eva Krupp
- SA-05 Clinical ICP-MS I**, Sunday, January 3, 1 pm, Robert Jones, Kathy Caldwell, Jeff Jarrett, Carl Verdon
- SA-06 Clinical ICP-MS II**, Sunday, January 3, 7 pm, Kathleen Caldwell, Robert Jones
- SA-07 Analysis of Petroleum Products and Industrial Materials by Plasma Spectrochemistry**, Saturday, January 2, 7 pm, Robert Botto, Kelly Mason
- SA-08 Environmental Sampling Techniques and Practices**, Saturday, January 2, 7 pm, Brian Buckley
- SA-09 Pharmaceutical Applications of Atomic Spectrometry**, Saturday, January 2, 8 am, Nancy Lewen
- SA-10 Quantitative Imaging LA-ICP-MS of Tissues**, Sunday, January 3, 7 pm, Sabine Becker
- SA-11 Sample Decomposition and Analysis of Refractory and Advanced High Technology Materials**, Saturday, January 2, 8 am, Joe Brenner
- SA-12 Environmental Monitoring and Fingerprinting: The Use of ICPMS-Based Isotopic Methods**, Saturday, January 2, 8 am, Michael Ketterer

Spectrochemical Instrumentation

- SI-01 Calibration and Correction for Interferences in Spectrochemical Analysis**, Sunday, January 3, 8 am, José A.C. Broekaert
- SI-02 Isotopic Analysis for Beginners**, Monday, January 4, 7 pm, Frank Vanhaecke, Michael Ketterer
- SI-03 High-Resolution ICP-MS**, Sunday, January 3, 1 pm, Norbert Jakubowski
- SI-04 Plasma Spectroscopy of Solids, Surfaces, and Interfaces**, Sunday, January 3, 7 pm, Norbert Jakubowski, Volker Hoffmann
- SI-05 Time-of-Flight Mass Spectrometry for Elemental Analysis**, Saturday, January 2, 7 pm, Steve Ray, Gary Hieftje
- SI-06 ICP-MS I: Introduction**, Saturday, January 2, 8 am, Sam Houk
- SI-07 ICP-MS II: Advanced Topics**, Saturday, January 2, 1 pm, Sam Houk
- SI-08 Ambient Desorption/Ionization Mass Spectrometry**, Sunday, January 3, 8 am, Jacob Shelley, Carsten Engelhard, Zoltan Mester, Ralph Sturgeon
- SI-09 Reaction Cells and Collision Cells for ICP-MS**, Sunday, January 3, 1 pm, John Olesik
- SI-10, Matrix Effects in ICP Techniques**, Sunday, January

3, 7 pm, José Todoli

- SI-11 Identification and Correction of Interferences in Practical ICP-OES and ICP-MS**, Sunday, January 3, 8 am, Deborah Bradshaw

Sample Introduction Approaches

- SS-01 Sample Introduction for ICP Spectrochemistry**, Monday, January 4, 7 pm, James A. Holcomb, Vassili Karanassios
- SS-02 Flow Injection Analysis Techniques and Applications**, Monday, January 4, 7 pm, Fernanda Giné
- SS-03 Nebulizer Diagnostics and Characteristics**, Saturday, January 2, 8 am, Akbar Montaser
- SS-04 Laser Ablation Atomic and Mass Spectrometry I**, Saturday, January 2, 1 pm, Henry Longerich, Detlef Günther
- SS-05 Laser Ablation Atomic and Mass Spectrometry II**, Saturday, January 2, 7 pm, Detlef Günther, Henry Longerich

Plasma Spectrochemical Techniques

- ST-01 Pharmaceutical Applications of Laser-Induced Breakdown Spectroscopy (LIBS)**, Saturday, January 2, 1 pm, Lydia Breckenridge
- ST-02 Fundamentals and Recent Developments in Isotope Dilution ICP-MS**, Sunday, January 3, 1 pm, Klaus G. Heumann
- ST-03 Extraction Chromatography for Enhanced Elemental and Isotopic Analysis by ICPMS**, Saturday, January 2, 1 pm, Michael Ketterer
- ST-04 Antimony and Its Species in the Environment**, Sunday, January 3, 7 pm, Patricia Smichowski, Michael Krachler
- ST-05 Microwave Sample Preparation for Bioanalytical, Speciation, and Accurate Analysis**, Saturday, January 2, 7 pm, Skip Kingston, Mizamur Rahman
- ST-06 Contamination Control for Trace Element Analysis**, Sunday, January 3, 1 pm, Brad McKelvey
- ST-07 Trace Element Speciation**, Monday, January 4, 7 pm, Olivier Donard
- ST-08 Plasma Diagnostics: Fundamentals, Measurements, Applications**, Saturday, January 2, 1 pm, Paul Farnsworth
- ST-09 Preparing Your Laboratory for Trace Analyses**, Sunday, January 3, 7 pm, Ela Bakowska
- ST-10 Microwave-Assisted Sample Preparation for Spectrochemistry**, Sunday, January 3, 8 am, Joaquim A. Nóbrega
- ST-11 Metallomics: Basic Concepts, Analytical Methodologies and Emerging Applications**, Saturday, January 2, 7 pm, Katarzyna Wrobel, Kazimiere Wrobel
- ST-12 Isotopic Analysis of Natural Inorganic Samples by Laser Ablation ICP-MS**, Sunday, January 3, 8 am, Jan Kosler, Mike Tubrett, Rebecca Lam
- ST-13 Speciation Isotope Dilution Mass Spectrometry**, Saturday, January 2, 1 pm, Skip Kingston, Mizamur Rahman
- ST-14 Bioinorganic Speciation Analysis and Metall-**

omics, Sunday, January 3, 8 am, Ryszard Lobinski, Joanna Szpunar

ST-15 Experiences of an Expert Witness, Monday, January 4, 7 pm, Ela Bakowska

ST-16 Clean Microwave Digestions for Ultra-Trace Analysis, Saturday, January 2, 8 am, Skip Kingston

ST-17 Trace Element and Isotope Analysis of Forensic Evidence, Sunday, January 3, 1 pm, Christopher Latkoczy

ST-18 Reference Materials and Standards for Quality Control of Speciation Analysis, Monday, January 4, 7 pm, Heidi Goenaga Infante

ST-19 Proficiency Testing Issues Relating to Clinical ICP-MS, Saturday, January 2, 1 pm, Christopher Palmer, Patrick Parsons

Arranged by Date and Time

(SC1) Saturday, January 2, 8 am - Noon

SA-09 Pharmaceutical Applications of Atomic Spectrometry, Nancy Lewen

SA-11 Sample Decomposition and Analysis of Refractory and Advanced High Technology Materials, Joe Brenner

SA-12 Environmental Monitoring and Fingerprinting: The Use of ICPMS-Based Isotopic Methods, Michael Ketterer

SI-06 ICP-MS I: Introduction, Sam Houk

SS-03 Nebulizer Diagnostics and Characteristics, Akbar Montaser

ST-16 Clean Microwave Digestions for Ultra-Trace Analysis, Skip Kingston

(SC2) Saturday, January 2, 1 - 5 pm

SI-07 ICP-MS II: Advanced Topics, Sam Houk

SS-04 Laser Ablation Atomic and Mass Spectrometry I, Henry Longerich, Detlef Günther

ST-01 Pharmaceutical Applications of Laser-Induced Breakdown Spectroscopy (LIBS), Lydia Breckenridge

ST-03 Extraction Chromatography for Enhanced Elemental and Isotopic Analysis by ICPMS, Michael Ketterer

ST-08 Plasma Diagnostics: Fundamentals, Measurements, Applications, Paul Farnsworth

ST-13 Speciation Isotope Dilution Mass Spectrometry, Skip Kingston, Mizamur Rahman

ST-19 Proficiency Testing Issues Relating to Clinical ICP-MS, Christopher Palmer, Patrick Parsons

(SC3) January 2, 7 - 11 pm

SA-07 Analysis of Petroleum Products and Industrial Materials by Plasma Spectrochemistry, Robert Botto, Kelly Mason

SA-08 Environmental Sampling Techniques and Practices, Brian Buckley

SI-05 Time-of-Flight Mass Spectrometry for Elemental Analysis, Steven Ray, Gary Hieftje

SS-05 Laser Ablation Atomic and Mass Spectrometry II, Detlef Günther, Henry Longerich

ST-05 Microwave Sample Preparation for Bioanalytical, Speciation, and Accurate Analysis, Skip Kingston, Mizamur Rahman

ST-11 Metallomics: Basic Concepts, Analytical Methodologies and Emerging Applications, Katarzyna Wrobel, Kazimiere Wrobel

(SC4) Sunday, January 3, 8 am - Noon

SA-01 Elemental Testing in Toxicology, Ela Bakowska

SI-01 Calibration and Correction for Interferences in Spectrochemical Analysis, José A.C. Broekaert

SI-08 Ambient Desorption/Ionization Mass Spectrometry, Jacob Shelley, Carsten Engelhard, Zoltan Mester, Ralph Sturgeon

SI-11 Identification and Correction of Interferences in Practical ICP-OES and ICP-MS, Deborah Bradshaw

ST-10 Microwave-Assisted Sample Preparation for Spectrochemistry, Joaquim A. Nóbrega

ST-12 Isotopic Analysis of Natural Inorganic Samples by Laser Ablation ICP-MS, Jan Kosler, Mike Tubrett, Rebecca Lam

ST-14 Bioinorganic Speciation Analysis and Metallomics, Ryszard Lobinski, Joanna Szpunar

(SC5) Sunday, January 3, 1 - 5 pm

SA-02 Analysis of Foods and Food Products, Deborah Bradshaw

SA-05 Clinical ICP-MS I, Robert Jones, Kathy Caldwell

SI-03 High-Resolution ICP-MS, Norbert Jakubowski

SI-09 Reaction Cells and Collision Cells for ICP-MS, John Olesik

ST-02 Fundamental and Recent Developments in Isotope Dilution ICP-MS, Klaus G. Heumann

ST-06 Contamination Control for Trace Element Analysis, Brad McKelvey

ST-17 Trace Element and Isotope Analysis of Forensic Evidence, Christopher Latkoczy

(SC6) Sunday, January 3, 7 - 11 pm

SA-03 Mass Spectrometry in Protein Quantification, Jörg Bettmer, Maria Montes-Bayón

SA-06 Clinical ICP-MS II, Kathleen Caldwell, Robert Jones

SA-10 Quantitative Imaging LA-ICP-MS of Tissues, Sabine Becker

SI-04 Plasma Spectroscopy of Solids, Surfaces, and Interfaces, Norbert Jakubowski, Volker Hoffmann

SI-10 Matrix Effects in ICP Techniques, José Todoli

ST-04 Antimony and Its Species in the Environment, Patricia Smichowski, Michael Krachler

ST-09 Preparing Your Laboratory for Trace Analyses, Ela Bakowska

(SC7) Monday, January 4, 7 - 11 pm

SA-04 Arsenic and Mercury Speciation in Biological Samples, Jörg Feldmann, Eva Krupp

SI-02 Isotopic Analysis for Beginners, Frank Vanhaecke, Michael Ketterer

SS-01 Sample Introduction for ICP Spectrochemistry, James A. Holcomb, Vassili Karanassios

SS-02 Flow Injection Analysis Techniques and Applications, Fernanda Giné

ST-07 Trace Element Speciation, Olivier Donard

ST-15 Experiences of an Expert Witness, Ela Bakowska

ST-18 Reference Materials and Standards for Quality Control of Speciation Measurements, Heidi Goenaga Infante

*Manufacturers' Seminars
Roundtable Discussions
Workshop*

Workshop on Plasma Instrumentation, January 5 - 7,

2010 Winter Conference Short Courses

Saturday, January 2 - Monday, January 4, 2010

Descriptive Abstracts

Analysis by Plasma Spectrochemistry

SA-01 Elemental Testing in Toxicology. Ela Bakowska, NMS Labs, 3701 Welsh Rd, Willow Grove, PA 19090, ela_bakowska@yahoo.com

The field of elemental testing in toxicology is underutilized, despite many famous cases of arsenic poisoning. Despite its notoriety, even arsenic is not always on a "short list" of tests performed upon sudden illness or death. This course will cover the topics of toxicity of several elements. Some high profile poisoning cases will be presented: arsenic poisoning in the church in New Sweden, Maine; Cynthia Sommer's story; murder of Robert Curley; poisoning of Alexander Litvinenko. An exposure to harmful elements not always is a result of a crime. Examples of everyday risks of heavy metals poisoning will be presented.

Keywords: Arsenic, thallium, polonium, heavy metals, poisons, toxicity

Elzbieta (Ela) Bakowska has 30 years of experience as an analytical chemist. She received her master's degree from the University of Warsaw, Poland and her Ph.D. from the University of Massachusetts/Amherst. She worked at the Institute of Technology of Semiconductor Materials (Warsaw, Poland), University of Massachusetts, Dow Chemical, Hewlett-Packard/Agilent and since 2002 at NMS Labs, a private clinical and forensic laboratory. Ela is an author of 14 publications and over 100 abstracts and presentations at national and international conferences. In the past she taught courses and workshops at Winter Conference on Plasma Spectrochemistry, EAS, FACSS and AAFS conferences. Ela is a member of American Academy of Forensic Sciences and a guest lecturer at Arcadia University Forensic Program.

SA-02 Analysis of Foods and Food Products. Deborah K. Bradshaw, Atomic Spectroscopy Consulting, P.O. Box 536307, Orlando, FL 32853-6307, bradshawdk@cs.com

The analysis of foods and food products for metals and minerals content can be of great importance for the determination of nutritional and contamination issues. To accomplish these determinations, a variety of atomic spectroscopy techniques can be used: flame and graphite furnace atomic absorption, inductively coupled plasma atomic emission and inductively coupled plasma mass spectrometry. During this course, all of these techniques will be compared, including advantages, disadvantages and potential interferences. Extended possibilities such as solid sampling and elemental speciation will also be discussed.



Sample preparation techniques, always an integral part of any method, will also be presented with examples from the literature. The course will conclude with a presentation and discussion of data validation protocols.

Keywords: Foods, food products, atomic absorption, ICP-AES, ICP-MS, sample preparation

SA-03 Mass Spectrometry for Protein Quantification. Jörg Bettmer and Maria Montes-Bayón, Universidad de Oviedo, Departamento de Química Física y Analítica, C/ Julián Clavería 8, E-33013 Oviedo, Spain, bettmerjorg@uniovi.es



Quantitative protein analysis has become an important task in many fields of biochemical research and clinical application. Besides the qualitative characterization of the proteome or subproteome in a specific tissue or body fluid, quantitative aspects have been identified to be relevant for a better understanding of biological processes on the protein and/or peptide level. In this regard, different mass spectrometric techniques could be used as analytical tools for quantitative protein studies, especially since isotopically labeled compounds have been discovered for this purpose. This short course intends to provide an overview on the strategies used in conjunction with elemental and structural mass spectrometric techniques for quantitative protein analyses.

The course is thought to be divided into two main parts: i) strategies based on mass spectrometric techniques with soft-ionization sources like ESI and MALDI, and ii) strategies based on the application of ICP-MS. Here, mainly two different quantification strategies based on isotope dilution analysis (IDA) will be discussed for its current and future potential in quantitative protein analysis, but also other purposes will be presented related to this field.

Keywords: protein quantification, isotope dilution analysis, protein labeling, heteroatom-tagged proteomics

SA-04 Arsenic and Mercury Speciation in Biological Samples Using HPLC Coupled Simultaneously to ICP-MS and ES-MS. Jörg Feldmann and Eva Krupp, University of Aberdeen, College of Physical Sciences Department of Chemistry, Aberdeen AB24 3UE Scotland, United Kingdom, j.feldmann@abdn.ac.uk, e.krupp@abdn.ac.uk

This course is divided in three parts. Part 1 shows how speciation analysis is done when only an element-selective detector is available. It will be elaborated in particular on identification strategies of known and unknown species using HPLC-ICP-MS. Part 2 introduces electrospray mass

spectrometry and discusses the advantages and limitation of this technique for complex sample matrices. Part 3 focuses on quantitative aspects in element speciation analysis when chromatographic separations are used.

Keywords: Electrospray MS, ICP-MS, food analysis, arsenic, arsenosugars, mercury, phytochelatins, plant physiology

Jörg Feldmann received his PhD at University of Essen (Germany) in 1995; he studied volatile metal and metalloids in the environment by using GC-ICP-MS. He was Feodor Lynen Postdoc (Alexander von Humboldt) at University of



British Columbia, Canada in 1995-1997 when he investigated complimentary use of GC-MS and GC-ICP-MS for volatile tin, antimony and bismuth compounds. Since 1997 he was a Lecturer at University of Aberdeen, Scotland and became full Professor in 2003. He has published more than 100 papers in peer-reviewed journals mainly about arsenic speciation. His focus is on the determination of the arsenosugar metabolism by seaweed-

eating sheep and the transport and biotransformation of arsenic in plants and the pro and cons of ES-MS and ICP-MS and the online combination of both MS techniques

SA-05 Clinical ICP-MS I: The Devil's in the Details.

Robert Jones, 4770 Buford Highway, Mailstop F-50, Atlanta, GA 30341-3724, rljones@cdc.gov; Kathleen L. Caldwell, Jeff Jarrett, Carl Verdon

"The Devil's in the Details", is your lab prepared to generate data for multimillion dollar public health decisions? This course will take a look at ICP-MS requirements for long term QC; how QC pools are prepared and validated; whether the matrix matters, and a focused look at how LODs are calculated: just how low can you go? These very small details are ultimately important components to the larger task of clinical ICP-MS at CDC. It pays to avoid pitfalls which are preventable by remembering that small laps of attention can translate into useless results.

Keywords: Clinical ICP-MS, QC\QA, LOD, biomonitoring, public health



SA-06 Clinical ICP-MS II: Responding to Inorganic Chemical Exposure Evaluations and Emergency Response.

Kathleen L. Caldwell and Robert Jones, 4770 Buford Highway, Mailstop F-18, Atlanta, GA 30341-3724, klc7@cdc.gov

This course will compare and contrast ICP-MS requirements for biomonitoring vs. exposure investigations or emergency responses. We will discuss CDC's use of ICP-MS as related to clinical analysis. We will highlight several clinical methods to include mercury and arsenic speciation methods. We will discuss how ICP-MS has been, and can be, a useful tool in making public health



decisions. We will walk thru a CDC inorganic response to an emergency response scenario, evaluating the use of ICP-MS during emergency responses requiring short turn around times on a large number of samples.

Keywords: Clinical ICP-MS, emergency response, biomonitoring, and speciation

Kathleen L. Caldwell is Chief, Inorganic Toxicology Laboratories and assistant Chief of the Inorganic and Radiation Analytical Toxicology Laboratory. Her responsibilities include the planning, implementation, oversight, and completion of programs related to public health that involve non-radioactive and radioactive elements or their isotopes. These programs involve research and development of a wide variety of analytical methods to enable the Centers for Disease Control and Prevention to assay and monitor the exposure of populations to toxic or essential elemental exposures. Her other responsibilities include the implementation and laboratory aspects of multiple local, state, regional, national and international studies or investigations. In addition, the laboratory group responds to EPI Aids, "emergency responses" and is involved with terrorism preparedness.

SA-07 Analysis of Petroleum Products and Industrial Materials by Plasma Spectrochemistry.

Robert Botto, Analytical Services Laboratory, Baytown Complex Laboratory Department, Exxonmobil, 5000 Bayway Dr., Baytown, TX 77520, bob.i.botto@exxonmobil.com, and Kelly Mason, ExxonMobil Research and Engineering, Analytical Sciences 1545 Route 22 East, Annandale NJ 08801-3059, kelly.j.mason@exxonmobil.com

This course will provide an overview of petroleum and petrochemical analysis using current ICP-AES and ICP-MS techniques and in depth information on specific applications and challenges. Sample types will include crude oil, distillate fractions, volatile hydrocarbons and solvents, used oils and other materials encountered in petrochemical processing. Methods for obtaining quality results for the wide variety of sample matrix types will be discussed. There will be time for class discussion on topics of individual interest.

Keywords: ICP, petroleum, plasma, metals, crude, oil, hydrocarbons, emission, spectrometry, mass spectrometry

Bob (Robert I.) Botto was born in Buffalo, NY. His dad was a chemist, and Bob developed an interest in chemistry in the fourth grade. With the help of a chemistry set and encouraged by his parents and teachers, Bob learned much about the world of chemistry before entering college at the State University of New York at Buffalo where he became a chemistry major. In 1975 as Bob completed his doctorate in geochemistry at Cornell University. He moved to Baytown, Texas the same year and began working for Exxon Research and Engineering Company (ER&E). Bob's first assignment at ER&E was to commission the new ICP atomic emission spectrometer. Thus he was able to pioneer many plasma spectrochemical techniques for petroleum and petrochemicals. Bob joined the ExxonMobil Baytown Chemical Plant in 1995 where he works today. His laboratory specializes in analytical prob-



lem solving for ExxonMobil refineries and chemical plants. Bob has published 30+ papers in the area of analytical atomic spectroscopy. Bob continues to be active as a promoter of chemistry for kids and has led the American Chemical Society Greater Houston Section's Community in Chemistry Committee. Each year he presents his traveling "Magic in Chemistry" show to thousands of kids. Bob is an ultra marathon runner and a veteran of 30 races longer than 25 miles including three 100-mile finishes. Bob is the team captain of the ExxonMobil Corporate Track & Field Team.

Kelly J. Mason received a doctorate in Analytical Chemistry from University of Vermont in 1987 following which he became a research associate for ExxonMobil Research & Engineering (EMRE). His research has focused on the analysis of petroleum products, deposits and catalysts using various atomic spectroscopy techniques (ICP-AES, ICP-MS, AAS, WDXRF) and sample decomposition approaches. Much of his research has been used to develop industry standard test methods published by ASTM. Currently, he holds the ASTM subcommittee vice-chairman position for spectroscopic methods for D02.03, committee on Petroleum Products and Lubricants, subcommittee on Elemental Analysis. For almost 10 years, Kelly was the Team Leader for EMRE's Inorganic Analysis Group where he manages research programs and method development covering atomic spectroscopy, precious metals assay, micro-chemical analysis and other related techniques for two research centers, ExxonMobil's Research Center at Annandale, NJ and Technical Center at Paulsboro, NJ. These two laboratories provide critical analytical data in support of product, process, and catalyst development, as well as addressing environmental issues. He is now responsible for the Analytical Sciences Section at the two EMRE sites. This section has approximately 45 employees with a third being PhD professionals covering a broad range of analytical disciplines to support research and affiliate problem solving characterization needs.

SA-08 Environmental Sampling Techniques and Practices. Brian Buckley, Rutgers University, EOHSI 681 Frelinghuysen Road, Piscataway NJ 08855-1179, bbuckley@eohsi.rutgers.edu

This course will cover the fundamentals of environmental sampling for metals and organics with a focus on how to obtain a clean sample. It is designed for the individual who may occasionally go into the field to do sample collection as well as for those individuals who have to prepare samples collected from the field. It is designed to provide a basic understanding of what is required to collect and transport a field sample, so that useful analytical information can be obtained from that sample. Field techniques for maintaining a clean sampling environment as well as for sample collection will be presented. Sample matrices discussed will include; soil, sediment, dust, air, and others. Field collection problems and how to overcome them will be a primary discussion topic. How to sample in both indoor and outdoor environments will be an important topic for the course.



Preparation for sampling trips and sample transport techniques will also be discussed.

Keywords: Sampling, environmental media, sample handling

Brian Buckley is Executive Director at the Environmental and Occupational Health Sciences Institute and a member of the graduate faculty at Rutgers University. His research involves trace metal quantification and speciation by chromatography coupled ICP-MS as well as microwave assisted solvent extraction.

SA-09 Pharmaceutical Applications of Atomic Spectroscopy. Nancy Lewen, BMS, 1 Squibb Dr., New Brunswick, NJ 08903, nancy.lewen@bms.com

The short course will cover the field of atomic spectroscopy as it relates to the pharmaceutical industry. It will cover topics including: sample preparation, regulatory considerations, safety issues, emerging techniques, instrumental techniques used, real-life examples, process trouble-shooting, contamination issues.

Keywords: Atomic spectroscopy, pharmaceuticals, drugs, API's, ICP, ICP-OES, ICP-MS

Nancy Lewen is a Senior Research Scientist with the Bristol-Myers Squibb Pharmaceutical Research Institute.



She has been supervisor of the atomic spectroscopy laboratory for 15 years. The laboratory provides support for all phases of the drug development process, including method development, technology transfer and process trouble-shooting. The laboratory is equipped with flame and graphite furnace AAS systems, as well as ICP-AES, ICP-MS and LA-ICP-MS equipment.

SA-10 Quantitative Imaging LA-ICP-MS of Tissues. J. Sabine Becker, Research Centre Jülich GmbH, Central Department for Chemical Analysis, Postfach 1913, D-52425 Jülich, Germany, s.becker@fz-juelich.de

The quantitative bioimaging of elements (e.g., Cu, Fe, Zn, Se and others) in biological tissues is of growing interest in life science especially in brain research, (e.g., for studying neurodegenerative diseases such as Alzheimer's or Parkinson's disease). Imaging mass spectrometry using LA-ICP-QMS can be applied to study the uptake and the transport of nutrient and toxic elements in plant tissues. In the short course powerful analytical imaging techniques using LA-ICP-MS to study element distribution in biological tissues in the routine mode and the combination to metallomics will be discussed.

Keywords: Animal tissues, human brain tissues, bioimaging, gels, LA-ICP-MS, metallomics, plant tissues, quantification



J. Sabine Becker (Head of Trace and Ultratrace Analysis, Research Centre Juelich, Germany) is author of the comprehensive handbook: *Inorganic Mass Spectrometry: Principles and Applications*, Wiley 2007 as the result of 35 years extensive experience in all fields of inorganic mass spectrometry (ICP-MS,

SSMS, TIMS, LIMS, rf-GDMS, SIMS, SNMS, LA-ICP-MS). She pioneered the quantitative bioimaging of metals and non-metals in thin section of biological tissues (e.g., human, mouse and rat brain tissues by LA-ICP-MS), combined Bioimaging of Metals in Brain and Metallomics (BrainMet) to study neurodegenerative diseases (Alzheimer's and Parkinson's diseases, cancer growing, aging and stroke), and created the Nano-LA-ICP-MS for future metal imaging in single cells.

SA-11 Sample Decomposition and Analysis of Refractory and Advanced High Technological Materials. Isaac (Joe) B. Brenner, Brenner Scientific, 9 Dishon Street, Malkha, Jerusalem, Israel 96956, brenner@cc.huji.ac.il

The development of high technological materials such as refractory ceramics, high temperature-resistant metals and complex alloys in the aerospace, electronic, and nuclear industries and exploration and beneficiation of geological deposits, necessitates their quantitative analysis for major and trace element compositions. In this short course we will review the current status of sample preparation strategies for the analysis of these chemically and physically complex materials. Protocols for decomposition of ferro-chrome and W alloys and metal powders, Zr-oxides, Cr-slugs, carbides, borides and nitrides, alumina and Al-silicate rocks and minerals, coals, REE-bearing silicate rocks, and concentrates will be evaluated. This evaluation will be based on conventional figures such as recovery, effect on LODs, spectroscopic and nonspectroscopic interference effects. Procedures using ICP-AES and MS will be detailed. Alternative direct solids analysis such as DC arc and glow discharge-OES and MS, direct sample insertion, ETV, slurry nebulization, and spark and laser ablation will be considered.



Keywords: Refractory materials, sample preparation, ICP-AES/ MS, DC arc, glow discharge-OES/ MS, ETV, slurry nebulization, spark and laser ablation

SA-12 Environmental Monitoring and Fingerprinting: The Use of ICPMS-Based Isotopic Measurements. Michael E. Ketterer, Department of Chemistry and Biochemistry, Northern Arizona University, Box 5698, Flagstaff, AZ 86011-5698, michael.kettere@nua.edu

The how and why of using quadrupole and sector ICP-MS for environmental monitoring and fingerprinting determinations will be presented. This course will describe types of samples, sample treatment methods, separation/preconcentration procedures, and ICP-MS strategies. "Fingerprinting" has two main objectives: a) developing a qualitative but definite yes/no answer about the presence of a specific environmental source, and b) where possible, providing quantitative apportionment information. This course will cover the reasons for isotopic variations, and will describe how these systems can be used for either tracing of the element of varying isotopic composition, or as a pathfinder for source elucidation of another element or pollutant. The course will cover how to select isotopic systems, design

monitoring approaches, and collect the requisite data by Q-ICPMS, SF-ICPMS, and MC-ICPMS. The course will examine case studies from several important examples based upon published literature and/or the instructor's own work.

Keywords: Environmental monitoring, ICP-MS, isotope dilution, isotope ratios, case studies, fingerprinting

Michael E. Ketterer received his undergraduate education at the University of Notre Dame (BS, Chemistry, 1980) and earned a PhD in Analytical Chemistry in 1985 from the University of Colorado. His PhD research was in the area of electroanalytical chemistry, but he now considers himself a "recovering electrochemist". He was employed in industry for two years, then he worked from 1987-1993 at the USEPA's National Enforcement Investigations Center. In 1988, while employed at EPA-NEIC, he began working with quadrupole ICPMS. From 1993-1998 he was Assistant Professor of Chemistry at John Carroll University and in 1998 he moved to Northern Arizona University, where he is currently Professor of Chemistry and Biochemistry. He now manages a laboratory equipped with a VG Axiom sector ICPMS current research interests are in isotopic measurements, and studies of naturally occurring and artificial radionuclides in the environment.



Spectrochemical Instrumentation

SI-01 Calibration and Correction for Interferences in Spectrochemical Analysis. José A.C. Broekaert, Institute for Inorganic and Applied Chemistry, University of Hamburg, Martin-Luther-King-Platz 6, D-20146 Hamburg, Germany, jose.broekaert@chemie.uni-hamburg.de

The course will treat procedures for calibration in plasma spectrochemical analysis including statistical evaluation of the data; calibration by standard addition, use of internal and external standards; importance of and procedures for the acquisition of the spectral background and spectral interferences in atomic spectrometry (additive interferences) and of matrix induced signal enhancements and depressions (multiplicative interferences). Other topics include definition and determination of detection and determination limits in analytical spectrometry; noise and signal-to-noise considerations; methods for optimization (trial-and-error, Simplex) and chemometrics (data display, clustering and multivariate analysis), and the concept of traceability. Examples from ICP-AES/MS with solutions, slurry nebulization ICP-AES, ETV-ICP-AES, MIP-AES, glow discharge atomic spectrometry, extraction, and HPLC-based separations and speciation work will be given.

Keywords: calibration, statistical evaluation, figures of merit, detection limits, interferences, optimization, data treatment

José A.C. Broekaert received his Ph.D. in chemistry at University of Gent (Belgium) in 1976, an Alexander-von-Humboldt Research Fellow Germany (1977), and was a scientist at ISAS-Institute for Analytical Sciences, Dortmund, Germany from 1978 to 1991. He



was “Geaggregeerde voor het hoger onderwijs”, University of Antwerp (Belgium) (1985), Associate Professor of Analytical Chemistry at University of Dortmund (1991-1998), Professor of Analytical Chemistry at University of Leipzig (1998-2002) and subsequently at University of Hamburg (since 2002). He was a Visiting Fulbright Research Fellow at Indiana University, Bloomington (1998), and is an Adjunct Professor of Chemistry at Indiana University (since 2004). His Research interests include analytical chemistry especially with atomic spectrometric methods.

SI-02 Isotopic Analysis for Beginners. Frank Vanhaecke, Ghent University, Department of Analytical Chemistry, Krijgslaan 281 – S12, BE9000 Ghent, Belgium, frank.vanhaecke@UGent.be, and Michael Ketterer, Department of Chemistry and Biochemistry, Northern Arizona University, Box 5698, Flagstaff, AZ 86011-5698, michael.kettere@nu.edu

The course starts with a general introduction and reviews the various origins of natural variations in the isotopic composition of elements. The capabilities and limitations of various types of ICP-MS instrumentation for isotope ratio measurements are discussed. Attention is also devoted to instrumental bias caused by detector dead time and mass discrimination and approaches for correction for these biases are covered. A next section focuses on applications relying on the quantification of induced changes in isotope ratios and includes elemental assay using isotope dilution and tracer experiments with stable isotopes to obtain a more profound insight into (bio)chemical reactions or physical processes. A separate sub-section is devoted to nuclear applications. Next, attention will be devoted to applications based on the natural variation in the isotopic composition of metallic and metalloid elements as a result of (a) the presence of radiogenic isotopes and (b) isotope fractionation effects. Examples coming from the field of geochemistry, nuclear chemistry, environmental research and biomedicine will be discussed.

Keywords: isotope ratio, accuracy, precision, single-collector ICP-MS, multi-collector ICP-MS, isotope dilution, tracer experiments, provenance determination, isotope fractionation, mass discrimination

SI-03 ICP Sector ICP-MS. Norbert Jakubowski, Institute for Analytical Sciences (ISAS), Bunsen-Kirchhoff-Str. 11, P.O. Box 10 13 52, D-44013 Dortmund, Germany, jakubowski@-ansci.de

This course is an introduction to ICP-MS with a double focusing magnetic sector mass analyzer. It offers fundamental background, a thorough discussion of analytical features, and state of the art information on applications. Different types of double focusing instruments also are considered. Specific topics include fundamental aspects of ICP-MS (physical properties of a double focusing instrument, operational characteristics in comparison with quadrupole



instruments); analytical characteristics (spectral and non-spectral interferences, figures of merit in low and high resolution modes, blanks and memory effects, HPLC and GC interfaces), and applications (industrial including ultra-pure reagents and alloys, environmental, geological, and biomedical materials).

Keywords: High-resolution ICP-MS, figures of merit, interfaces, applications, ultra-trace analysis

SI-04 Plasma Spectroscopy of Solids, Surfaces, and Interfaces. Volker Hoffmann, Leibniz Institute for Solid State and Materials, Research Dresden PO Box 27 00 16, D-01171 Dresden, Germany, v.hoffmann@ifw-dresden.de, and Norbert Jakubowski, Institute for Analytical Sciences (ISAS), Bunsen-Kirchhoff-Str. 11, PO Box 10 13 52, D-44013 Dortmund, Germany, jakubowski@-ansci.de

This course is designed to serve as an introduction to the fundamental operating principles of glow discharge devices and their applications in atomic emission (GD-AES) and mass spectrometries (GD-MS) and will be given by international experts. Hard- and software (quantification) will be explained and applications will be compared with other methods of direct solids elemental analysis. Analytical figures of merit for the two spectrometric methods are discussed. Direct current, radio frequency and pulsed glow discharges will be described with respect to the plasma and electrical discharge parameters. Finally, the session will be concluded with a discussion of future trends in instrumentation and applications.

Keywords: Glow discharge, atomic emission, mass spectrometry, elemental analysis

SI-05 Time-of-Flight Mass Spectrometry for Elemental Analysis. Steven J. Ray and Gary Hieftje, Indiana University, Department of Chemistry 800 E. Kirkwood Ave., Bloomington IN 47405-7102, sjray@indiana, hieftje@indiana.edu

Since its introduction time-of-flight mass spectrometry (TOFMS) has demonstrated extensive analytical utility and potential for elemental analysis. The acceptance of the technique can be attributed to both the characteristics of TOFMS and the introduction of commercial ICP-TOFMS instrumentation. This acceptance is illustrated by the increased presence of ICP-TOFMS systems in laboratories throughout the world. Among the simplest of mass analyzers, TOFMS offers exceptionally high spectral generation rates (typically 20,000 complete mass spectra per second), adequate mass spectral resolving power (1500), high abundance sensitivity, the promise of high ion transmission efficiency, and relatively low cost. Additionally, since each packet of ions is sampled from the plasma source at virtually the same instant, ion signals can be ratioed to improve precision for all isotopes and elements simultaneously. These features combine to eliminate spec-



tral skew and thus, TOFMS is an ideal mass spectrometer for transient analyses. In this short course, the fundamentals of TOFMS will be reviewed, and instrumental and experimental considerations will be discussed. Applications of TOFMS with various plasma sources will be examined, and particular attention will be given to methods that exploit the characteristics of TOFMS to overcome typical experimental difficulties. Moreover, approaches for improving figures of merit will be described and future generations of TOFMS systems will be suggested.

Keywords: Time-of-flight, mass spectrometry, elemental analysis, ICP-MS, GD-MS, instrumentation, plasma sources

Gary M. Hieftje is Distinguished Professor and Mann Chair of Chemistry at Indiana University in Bloomington, Indiana. His research interests include the investigation of basic mechanisms in atomic emission, absorption, fluorescence and mass spectrometric analysis, and the development of atomic methods of analysis. He is interested also in the on-line computer control of chemical instrumentation and experiments, the use of time-resolved luminescence processes



for analysis, the application of information theory to analytical chemistry, analytical mass spectrometry, near-infrared reflectance analysis, and the use of stochastic processes to extract basic and kinetic chemical information. He currently serves on the editorial boards of various journals including *Analytica Chimica Acta*, *Journal of Analytical Atomic Spectroscopy*, *Journal of Mass Spectrometry*, *Laboratory Microcomputer*, *Spectrochimica Acta, Part B*, *Advances in Inorganic Mass Spectrometry*, *Talanta*, and *Spectroscopy and Spectral Analysis*. He has received numerous awards, and he was chairman (1985-86) of the Analytical Division of the American Chemical Society. In 1991 he served as President of the Society for Applied Spectroscopy. From 1999-2000 he served as Director of the newly established Linda and Jack Gill Center for Instrumentation and Measurement Science at Indiana University and was the first holder of an endowed Gill Chair. In 2000 he was appointed to the Robert and Marjorie Mann Chair of Chemistry. Most recently, he was the 2000-2001 Indiana Academy of Science Speaker of the Year, and the recipient of the 2001 Pittsburgh Spectroscopy Award. Hieftje is the author of over 400 scientific publications, 10 books, and holds 13 patents. Over 50 students have received doctorates under his direction.

SI-06 ICP-MS I: Introduction. Sam Houk, Iowa State University, Ames Laboratory USDOE Department of Chemistry, Ames IA 50011, rshouk@iastate.edu

This course is intended mainly for the newcomer to ICP-MS. ICP characteristics as an ion source, ion extraction, operating principles of ion optics, ion focusing, quadrupole and sector mass analysis, and detectors will be described. The general analytical capabilities, applications survey, and an introduction to matrix effects will be presented.

Keywords: ICP-MS characteristics, instrumentation, interferences, applications

SI-07 ICP-MS II: Advanced Topics. Sam Houk, Iowa State University, Ames Laboratory USDOE Department of Chemistry, Ames IA 50011, rshouk@iastate.edu

This course is intended for those who complete Part I and for the experienced ICP-MS user. Detailed consideration will be given to basic cases of matrix effects, removal of polyatomic ion interferences (solvent removal, collisional dissociation, high resolution, cool plasma), alternate mass analyzers, solid sampling, analysis of limited solution volumes, and combining ICP-MS with chromatography for speciation and removal of interferences.



Keywords: ICP-MS operation and measurement effects, interference corrections, sampling, chromatography

SI-08 Ambient Desorption/Ionization-Mass Spectrometry: From Fundamentals to Applications. Jacob T. Shelley and Carsten Engelhard, 800 E. Kirkwood Ave., Bloomington, IN, 47405, jtshelle@indiana.edu; Zoltan Mester and Ralph Sturgeon, National Research Council of Canada, Institute for National Measurement Standards, 1500 Montreal Road, Ottawa ON K1A 0R6, Canada, zoltan.mester@nrc.ca, ralph.sturgeon@nrc.ca

Recently a number of atmospheric-pressure ionization sources for mass spectrometry have emerged in the literature to yield a field that is collectively referred to as Ambient Desorption/Ionization-Mass Spectrometry (ADI-MS). These sources include Desorption ElectroSpray Ionization (DESI), Direct Analysis in Real Time (DART), and Flowing Atmospheric-Pressure Afterglow (FAPA). Collectively, these ADI-MS sources have numerous advantages over conventional ionization sources, including high ionization efficiency, soft ionization, and direct analysis of solid, liquid, and gaseous samples. This course will cover fundamental principles of desorption and ionization processes, as well as real-world applications of ADI-MS sources. A particular emphasis will be placed on plasma-based systems.

Keywords: Direct Analysis, soft ionization, mass spectrometry, DART, DESI, glow discharge, ambient ionization sources, pharmaceutical analysis, homeland security

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 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. Par-

ticular focus has been on the Flowing Atmospheric-Pressure Afterglow (FAPA) source.

Carsten Engelhard was Born and raised in Siegen, Germany. Carsten attended the University of Münster (WWU) from 1997-2003 before receiving his Diploma in Chemistry. At WWU he performed undergraduate research in the research group of Karl Cammann and Wolfgang Buscher on developing instrumentation for electrothermal vaporization microwave induced plasma optical emission spectrometry. He received his Dr. rer. nat. degree majoring in analytical chemistry in 2007. His graduate studies were mainly focused on developing alternative plasma sources for ICPs. The so-called SHIP torch reduced the total argon consumption of conventional ICPs by 95% and was applied for example, to the analysis of doped luminescent nanoparticles. Carsten started his postdoctoral research with Gary M. Hieftje in 2008. His current research interest lies in imaging and quantitation of proteins and biopolymers with novel glow discharges (GDs), in addition to fundamental studies of GDs. He received the 2009 Young Scientist Award of the Analytical Division of the German Chemical Society.



Zoltan Mester is a Research Officer of the Institute for



National Measurement Standards, National Research Council of Canada (NRC), where since 1999 he has been leading speciation-related research. He received his PhD in 1998 in Budapest, Hungary, and he worked with Professor J. Pawliszyn, the inventor of SPME, at the University of Waterloo, developing inorganic analytical applications for SPME.

He has published 30 papers on SPME.

Ralph Sturgeon received his PhD in analytical chemistry in 1977 under the guidance of Professor C.L. Chakrabarti. For the past 25 years he has been employed in Ottawa at the



National Research Council of Canada, where he is currently a Principal Research Officer and Group Leader for Chemical Metrology within the Institute for National Measurement Standards. His research interests include trace element analysis and organometallic speciation with emphasis on atomic and mass spectrometric detection. He has published approximately 190 papers in these areas.

He serves on the advisory board of a number of analytical journals and is an editor for *Spectrochimica Acta Reviews*.

SI-09 Theory and Operation of Reaction Cells for ICP-MS. John Olesik, Ohio State University, Department of Geological Sciences 125 S. Oval Mall, 026 Mendenhall Labs, Columbus OH 43210-1002, jolesik@-geology.ohio-state.edu

The design and operation of reaction cells and collision cells used for isobaric interference removal in ICP-MS will

be discussed. The course begins with an introduction to the principals and kinetics of ion-molecule reactions and the operation of the rf devices (quadrupoles, hexapoles, octapoles, etc.). The various efficiencies of the ion chemistry (primarily ion reactivity, reactivity, production of the ions within the cell) will be evaluated, concluding that where high efficiency of the primary chemistry is obtained, reaction of the analyte ion with impurities and the formation of new interferences within the cell becomes important. Various means of suppressing these effects, including kinetic energy discrimination and band pass operation of the cell will be contrasted and compared, and effects related to the order of the multipole device will be considered. Examples of the application of reaction cell and collisions cell methods in the semiconductor, environmental, clinical, geochemical and isotopic analysis will be presented.



Keywords: Collision cell, reaction cell, spectral interference, chemical resolution, pressurized multipole, chemistry, ion dynamics, ion-molecule chemistry, in-cell produced interferences, multipole operating point, energy discrimination

SI-10 Matrix Effects in ICP Techniques. José Luis Todolí, Department of Analytical Chemistry, Nutrition and Food Sciences, University of Alicante, P.O. Box 99, 03080 Alicante, Spain, jose.todoli@ua.es

In many situations matrix effects are one of the current drawbacks of ICP techniques. If a given method is able to provide excellent sensitivities and limits of detection but it affords bad recoveries, then this technique is not appropriate to be applied for routine analysis. Non spectral interferences can arise from almost every component of the spectrometer. In the present course the different sources of matrix effects will be addressed. Attention will be paid to the effect of the liquid sample introduction system on these unwanted phenomena. Advises to try to solve interferences will also be given.

Keywords: Matrix effects, liquid sample introduction system, inorganic acids, organic solvents, easily ionized elements, ICP-AES, ICP-MS

SI-11 The Identification and Correction of Interferences in Practical ICP-OES and ICP-MS. Deborah K. Bradshaw, Atomic Spectroscopy Consulting, P.O. Box 536307, Orlando, FL 32853-6307, bradshawdk@cs.com

The identification and correction of interferences for ICP-OES and ICP-MS can be critical to obtaining accurate data in the analytical laboratory. The interferences and their corrective techniques which will be addressed include sample transport, sample matrix, and spectral. Procedures used to correct for transport and matrix interferences include the use of appropriate internal standards, matrix matching, optimizing plasma conditions, using buffer solutions, and the correct choice of sample introduction systems. Spectral interference correction approaches include optimizing the method parameters as well as choosing the appropriate

numerical correction technique. Some of these may be limited to the specific instrumental hardware and software available to the analyst. The merits and disadvantages of the various correction approaches that are used will be examined, with practical examples of the use of these corrections to obtain accurate data.

Keywords: Transport interferences, matrix interferences, spectral interferences, inductively coupled plasma atomic emission, inductively coupled plasma mass spectrometry

Sample Introduction Techniques

SS-01 Sample Introduction Systems for ICP Spectrometry. James Holcombe, University of Texas at Austin, Department of Chemistry and Biochemistry, 1 University Station A5300, Austin TX 78712-0165, holcombe@mail-utexas.edu, and Vassili Karanassios, University of Waterloo, Department of Chemistry, Waterloo ON N2L 3G1, Canada, vkaranassios@uwaterloo.ca

In this course, the fundamentals of sample introduction into ICP's will be reviewed, and the capabilities and limitations of conventional, pneumatic nebulization sample introduction will be described, compared and contrasted with those offered by micro- and nano-sample introduction approaches. The application of electrothermal vaporization techniques (ETV) for the ultra-trace analysis of micro samples will be covered. Furthermore, the reduction (and often elimination) of spectral interference effects in ICP-MS obtained using discrete, "dry" sample introduction systems will be highlighted, and capabilities for speciation in the field and analysis in the lab will be demonstrated. The determination of isotope-ratios in enzymes by ICP-MS; and of trace elements in bio-molecules, and the capability to obtain micro-homogeneity information from powdered solids on a per-particle basis will be shown. Many cases exist in which only a limited amount of sample is available for analysis. Examples abound in clinical and biological analysis and in nanoscience and nanotechnology. For sample-limited solids, acid-digestion of minute amounts leads to dilution, thus low concentration analytes in solids often fall below the detection limit of ICP-AES. As a consequence, relatively more expensive ICP-MS systems must be used. Would it not be ideal if solids were analyzed without acid-digestion, thus also reducing sample preparation and eliminating waste disposal concerns arising from unused metal-loaded acid-digested samples?



Indirect, molecular detection by atomic spectrometry approaches will be outlined using the determination of prostate-specific antigen in microliter volume samples by ICP-MS as an example. Overall, it will be shown that in some cases it is preferable to analyze liquid or solid samples as micro- or nano-samples even if there is plenty of sample available for analysis. ETV offers several distinct advantages besides the well known use for microsamples. Complex matrices (including slurries and solids) can be easily handled with ETV. This includes biological samples, and some time will be dedicated to the analytical utility of the ETV's thermal program in simplifying the analysis of such

samples. The course will discuss simple use of the heating programs to optimize analytical performance. Explanations also will be given of how to adapt a modified graphite furnace (for AA) easily into an ICP system for automated use as an ETV. Time will be allotted for discussions of samples and analytical situations that are of particular interest of the attendees. Emphasis will be placed on the nano particle sample introduction (PSI) and on the electrothermal, in-torch vaporization (ITV) systems.

Keywords: ICP-AES, ICP-MS, electrothermal vaporization, sample introduction, micro- and nano-samples, trace analysis, isotope ratio determination

Vassili Karanassios is a Professor of Chemistry (<http://orchard.uwaterloo.ca>) and a founding member of the Nano-technology Engineering degree-program at the University of Waterloo (www.nanotech.uwaterloo.ca). His research interests include (among others) sample introduction for ICP's and microplasma devices (MPD's), and in general, micro-/nano-analytical chemistry and miniaturization of analytical instrumentation. VK has published more than 100 papers and has given more than 300 presentations at national and international conferences.



SS-02 Flow Injection for Elemental and Speciation Analysis by ICP OES or ICP-MS: Fundamentals and Applications. Maria Fernanda Giné, Av. Centenário 303, CEP 13416-000, Piracicaba SP, Brazil, mfgine@cena.usp.br

This course will introduce the main figures of flow injection analysis for on-line elemental or species quantification by inductively coupled plasma optical emission (ICP OES) or mass spectrometry (ICP-MS). Analyte dilution, pre-concentration or transference to the vapor phase either by hydride generation, electrothermal vaporization or laser ablation will be described. Flow facilities to overcome matrix effects by applying calibration procedures such as standard additions or isotope dilution and the instrumentation involved for data acquisition in transient signals to attain precise measurements will be discussed. Advantages of sample pre-treatments by sequential injection to the ICP will be evaluated.



Keywords: Flow injection analysis, on-line calibration procedures, speciation analysis, pre-concentration, ICP OES, ICP-MS, on-line chemical processes.

SS-03 Nebulizer Diagnostics and Analytical and Fundamental Characteristics. Akbar Montaser, George Washington University, Department of Chemistry, Washington DC 20052, amestp@aol.com, montaser@gwu.edu

This short course deals with the introduction of micro- and nanoliter quantity of samples into plasmas and relevant aerosol diagnostic techniques essential for gaining improved fundamental understanding of aerosol to achieve ultimate analytical performance indices in plasma spectrochemical measurements. A fundamental knowledge of the

properties and limitations of the sample introduction system and the plasma is important in conducting viable spectrochemical analyses. Recent research on several micro-nebulizers is reviewed, along with the most common micronebulizers and desolvation approaches that are used in plasma spectrometry. However, direct injection nebulizers are stressed as well since the design of these devices not only conserves samples, but also reduces memory effect and band broadening in chromatographic separations. Further, current benefits and limitations of micronebulizers and direct injection devices and aerosol diagnostic technique are also summarized.

Keywords: Low-sample consumption, nebulizers, spray chambers, desolvation, aerosol diagnostics, droplet size, modeling, transport efficiency

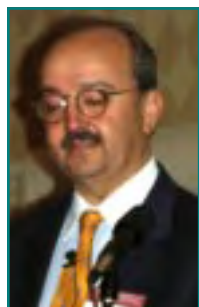
SS-04 Laser Ablation Atomic and Mass Spectrometry I.

Henry Longerich, Earth Sciences, Memorial University of Newfoundland, St. John's, NL A1B 3X5, Canada, HenryL@mun.ca, and Detlef Günther, Laboratory of Inorganic Chemistry, ETH Hönggerberg, HCI, G113CH-8093, Zürich, Switzerland, Guenther@inorg.chem.ethz.ch

The course is designed to give participants an introduction to the analytical capabilities of laser ablation - inductively coupled plasma spectrometry and should be of particular benefit to spectroscopists interested in developing a laser ablation facility. LA-ICP-MS has become one of the most common solid sampling technique for major, minor and trace element analysis. The course will explain how LA-ICP-MS works. Details about basics in lasers and ICP-MS instrumentation and their combination will be explained. Examples for transient data acquisition, method development, and a large number of applications will be discussed to give a feel for the quantification capabilities of this analytical technique. Topics also will include terms and terminology, laser selection, ablation cell design and interfacing, ablation processes, transport phenomena, and measurement systems. Participants do not need previous knowledge about this technique.

Keywords: Lasers, ablation, aerosol transport systems, carrier gas, calibration, atomization and ionization, ICP-MS, interface, applications

Henry Longerich graduated from Indiana University with a PhD in Analytical Chemistry in 1967. He then joined the chemistry faculty at the University of Alaska in Fairbanks, following which he did post doctoral research at Dalhousie University in Halifax, Nova Scotia, Canada. Henry then moved to Memorial University of Newfoundland, where he is now holds the title of Professor Emeritus in the Department of Earth Sciences. He was responsible for the electron probe X-ray microanalysis facility, X-ray fluorescence instrumentation, and development of computerization in the department. Henry became involved with ICP-MS in 1984 when he installed an ELAN 250 instrument, which was the tenth ICP-MS instrument sold



and installed by Sciex. After several years of developing solution nebulization methods for geosciences and environmental applications, the Memorial University research group obtained funding in 1988 to build a micro sampling laser ablation (LA) system to use with the ICP-MS. The landmark 1992 LA paper (Jackson, Longerich, *et. al*, *Canadian Mineralogist*, **30**, 1049-1064), which received the Hawley Medal for the best paper of the year in the *Canadian Mineralogist*, contributed significantly to the establishment of LA micro sampling in the earth sciences. It was clearly shown in this paper that the 1064 nm Nd:YAG laser used was not optimum, especially for samples, which were transparent at this wavelength. This paper suggested, "Use of different wavelengths of the laser can be expected to allow further improvement ...". This led to the development of a frequency quadrupled 266 nm system, and later in 1998 to the publication of the first report using a quintupled 213 nm system (Jeffries, Jackson, and Longerich, *JAAS*, **13**, 935-940). In 1996 Henry took voluntary early retirement from Memorial University, where he continues to have students and carry on funded research. While not in residence in Newfoundland, Henry has become an "itinerant" professor, doing research and teaching at the University of Alberta, Chemex Laboratories, St. Francis Xavier University, Trent University, and Clarion University of Pennsylvania.

SS-05 Laser Ablation Atomic and Mass Spectrometry II.

Detlef Günther, Laboratory of Inorganic Chemistry, ETH Hönggerberg, HCI, G113CH-8093, Zürich, Switzerland, Guenther@inorg.chem.ethz.ch, and Henry Longerich, Earth Sciences, Memorial University of Newfoundland, St. John's, NL A1B 3X5, Canada, HenryL@mun.ca

The course will provide detailed knowledge about recent instrumentation and method developments in LA-ICP-MS. Various problems in laser sample interaction, aerosol transport, and atomization and ionization will be discussed. Elemental fractionation, its source, and various strategies to overcome this problem for different laser and ICP-MS systems will be extensively discussed. Furthermore, different quantification strategies and non-matrix matched calibration examples will be given. A few non-routine applications will also be discussed. Participants should have some knowledge or practical experience with LA-ICP-MS or should have taken the basic LA-ICP-MS course (SS-04).

Keywords: Laser-sample interaction, elemental fractionation, aerosol size and aerosol transport, atomization and excitation, ICP-MS, interface and ICP-optimization



Plasma Spectrochemical Techniques

ST-01 Pharmaceutical Applications of Laser-Induced Breakdown Spectroscopy (LIBS).

Lydia Breckenridge, 1 Squibb Drive, Mail Stop 107-1-1250, New Brunswick, NJ, 08903, lydia.breckenridge@bms.com

Laser-induced breakdown spectroscopy (LIBS) has emerged as a unique analytical technique for the qualitative and quantitative analysis of pharmaceutical materials. The minimal sample preparation requirements, high spatial reso-

lution capabilities, rapid analysis time, simple instrumentation and applicability to all media make LIBS especially appealing to the pharmaceutical industry. This course will introduce the fundamentals and instrumentation of LIBS and provide a brief review of current applications. The utility of LIBS in the pharmaceutical field will be covered; including its application to coating analysis, batch homogeneity determination, contaminant identification and elemental quantitative analysis.

Keywords: Pharmaceuticals, LIBS, atomic spectroscopy and solid analysis

Lydia Breckenridge is a Research Investigator in the Atomic Spectroscopy group at Bristol-Myers Squibb in New Brunswick, New Jersey. Lydia's research concentrates on pharmaceutical applications of plasma-based analytical techniques, such as ICP-AES, ICP-MS, LA-ICP-MS and LIBS. Specifically, she has worked on applying LIBS to a broad range of challenges encountered in pharmaceutical formulations, including batch homogeneity determination, tablet coating analysis, particle size investigations, and contaminant identification. Lydia attended graduate school at the University of Florida, where she earned her Ph.D.



under the direction of Dr. James Winefordner. Her doctoral research focused on the application of LIBS to the determination of carbon in soil. Lydia also earned an M.S. degree in Forensic Drug Chemistry under Dr. Ian Tebbett, also at the University of Florida.

ST-02 Fundamentals and Recent Developments in Isotope Dilution ICP-MS. Klaus G. Heumann, Institute of Inorganic and Analytical Chemistry, Johannes Gutenberg-University Mainz, Duesbergweg 10-14, 55099 Mainz, Germany, heumann@uni-mainz.de

Isotope dilution mass spectrometry (IDMS) is well known for a long time as a relatively accurate analytical method for trace element determinations. The fundamental principles of ICP-IDMS and its applications as an accurate validation and/or routine method will be discussed. Elemental speciation is a strongly increasing field with growing importance in analytical chemistry. ICP-IDMS coupled online with separation techniques such as GC, HPLC, CE and GE therefore also became an important tool for the accurate determination of elemental species at very low concentration levels. In this connection the difference of the species-specific and species-unspecific spiking mode will be explained, and representative examples will be presented to demonstrate the power but also the limitations of ICP-IDMS analyses for elemental trace and elemental species determinations.

Keywords: isotope dilution mass spectrometry, ICP-MS, trace element analysis, elemental speciation

Klaus Gustav Heumann is Emeritus Professor of Analytical Chemistry at the Johannes Gutenberg-University Mainz, Germany. He received his Diploma degree in Chemistry at the Technical University Darmstadt in 1966. After his



Habilitation in Analytical Chemistry, he was appointed in 1974 Professor for Inorganic and Analytical Chemistry at the University of Regensburg/Bavaria, where he taught from 1974 to 1996. In 1996 he became Professor of Analytical Chemistry at the University of Mainz. His research interests lie in the development and application of analytical methods for the determination of trace elements and trace amounts of elemental species, using ICP-MS, TIMS, different types of optical atomic spectrometry, and electroanalysis as detection methods, and HPLC, CE or capillary GC as separation methods. His research group has considerable experience in isotope dilution mass spectrometry (IDMS). They were the first to use IDMS in connection with the negative thermal ionization technique (NTI-MS) for trace element analysis and to introduce on-line IDMS into hyphenated techniques for elemental speciation. He has received several awards that include the Clemens-Winkler-Medal of the German Society for Analytical Chemistry in 1994 for his scientific lifework and for continuous support of Analytical Chemistry in Germany. In 2007 he received the European Award for Plasma Spectrochemistry. He is member of several national and international scientific societies and he was a member of the IUPAC Commission on Atomic Weights and Isotopic Abundances for twelve years and chairman from 1991 to 1995. Since 2002 he is Editor of *Analytical and Bioanalytical Chemistry* (ABC).

ST-03 Extraction Chromatography for Enhanced Elemental and Isotopic Analysis by ICPMS. Michael E. Ketterer, Department of Chemistry and Biochemistry, Northern Arizona University, Box 5698, Flagstaff, AZ 86011-5698, michael.ketterer@nau.edu

Many aspects of ICPMS require some type of preparative separation prior to determination of component(s) of interest. Separations are useful for removing high concentrations of matrix elements, which produce undesirable space charge effects; polyatomic ion interferences can be eliminated by appropriately designed separations, and detection limits can be improved by pre-concentration of the analytes. "Extraction chromatography", involving a hydrophobic, polymer supported resin, is useful in the separation of many elements; analytes are retained from aqueous solution by complexation with ligands and/or ion-pairing mechanisms. Many types of resins are commercially available, and others can be prepared from readily available starting materials. This course will describe the principles of solid-phase extraction using selective resins, with the intent of demonstrating how extraction chromatography is highly useful and versatile as a pre-treatment step prior to elemental and isotopic analysis by ICPMS. The course will cover how to design a separation, based upon the known k' behavior of the resins in different aqueous media. Column design, batch vs. column mode, rinsing of non-retained components, and elution of desired components will be discussed. The advantages and disadvantages of off-line vs. on-line separations, and the regeneration and re-use of resins will be evaluated. Specific analyti-



cal examples, such as Pb, Sr, rare earth elements, Th, U, and transuranics will be described for applications in environmental chemistry, geochemistry, and radiochemistry.

Keywords: Extraction chromatography, resins, separation, pre-concentration, elemental and isotopic analysis, ICPMS

ST-04 Antimony and Its Species in the Environment.

Patricia Smichowski, Comisión Nacional de Energía Atómica, Centro Atómico Constituyentes A. Gral. Paz 1499, San Martín B1650KNA, Argentina, smichows@-cnea.gov.ar, and Michael Krachler, University of Heidelberg, Institute of Environmental Geochemistry Im Neuenheimer Feld 236, D-69120 Heidelberg, Germany, krachler@ugc.uni-heidelberg.de

Antimony occurs fairly widely in the natural environment with over 100 Sb-bearing minerals known, and antimony is



released into the environment from natural and anthropogenic sources. Most humans are now chronically exposed to low levels of antimony in their food, water and air. Antimony and its compounds are regarded as pollutants of priority interest. Knowledge of the concentrations of potentially toxic elements in atmospheric aerosols is fundamental for assessing pollution levels and provides valuable information for evaluating the long-term exposure of the population. In spite of regulatory requirements, available data for estimating the environmental exposure and assessing the risks of antimony in the environment on human health remains very limited. In general, arsenic and antimony share some chemical and toxicological properties, and their biological half-lives are relatively short in mammals, although there is evidence that certain parts are retained in the body for longer periods. The toxicological and physiological behavior of Sb depends on its oxidation state, the presence of binding partners and potential ligands, and on the solubility of the Sb compound. In parallel to its periodic companion arsenic, studies of the biogeochemistry of antimony have focused on methods for determining its chemical speciation, biotically mediated transformations, and the kinetic stability of thermodynamically unstable species. Several surprises have arisen from these studies. The conventional method for determining arsenic and antimony speciation is selective hydride generation followed by element specific detection via atomic absorption or fluorescence, ICP/MS, or gas chromatography with photoionization detection. Antimony is a fascinating and enigmatic element in terms of its environmental chemistry and effects



on human health. Environmental aspects of Sb, measuring techniques, and recent applications will be described in this course.

Keywords: Antimony, environment, hydride generation, atomic fluorescence, ICP-MS, speciation

Patricia N. Smichowski is a research scientist, and head of the Analytical Methodologies Division, Atomic Energy Commission (CNEA), Buenos Aires, Argentin

na. She completed her doctoral thesis at Complutense University of Madrid (Spain) under the direction of Carmen Cámara. She has published numerous papers on analytical chemistry, atomic absorption spectrometry, and inductively coupled plasma spectrometry.

ST-05 Microwave-Enhanced Sample Preparation Chemistry for Bioanalytical, Speciation, and Accurate Analysis.

H.M. (Skip) Kingston, Duquesne University, Department of Chemistry and Biochemistry, Pittsburgh PA 15282, kingston@duq.edu

This course presents both selected applications and the theoretical non-intuitive relationships in microwave sample preparation featuring solvent and acid dissolution and extraction for elemental, ultra-trace elemental analysis and species analysis. Specific sample preparation for ICP-MS and ICP-AES and ESI-TOF-MS including fundamental relationships of analyte recovery, power, reagent temperature, pressure, matrices, chemical compatibility, and practical standard methods will also be discussed. Basic features of microwave decomposition methods, equipment, vessel design, and accessories will be covered. Methods for transfer of procedures between equipment are highlighted, and applied methods such as the new 3052, 3200, 3051A, 3015a, 3546, 3030K, and others developed for EPA SW-846 and other standards organizations using microwave sample preparation for analysis in soils, sediments, water, tissues, foods, blood, urine and combination of these samples are reviewed. Safety considerations in laboratory microwave-enhanced sample preparation and analysis are stressed.

Keywords: Microwave heating, acid dissolution, organic and inorganic sample preparation, extraction, speciated extraction, elemental analysis, environmental analysis, microwave equipment, microwave theory, safety

H.M. (Skip) Kingston is Professor of Analytical Chemistry and Environmental Chemistry at Duquesne University

in Pittsburgh, PA. Kingston is the author of the EPA method 6800 and participated in the validation of many speciated methods for environmental health. From 1976 to 1991 he was a Supervisory Research Chemist and Program Director in the Analytical Research Division of the National Institute of Standards and Technology (NIST). In the early 1990s he conceived and headed the Consortium on Automated Analytical Laboratory Systems (CAALS), which was dedicated to developing automated analytical standards. For the past decade, Kingston has been actively involved in advancing the areas of speciation, analytical automated analysis and microwave enhanced chemistry analysis and applied analysis in autism and other environmental health area through basic research and methods development. He has invented and authored over a dozen standard methods for EPA, ASTM, NCCLS, SEMI and other organizations including EPA Methods 6800, 3200, 7473, 3052, and others. He co-authored two landmark Professional Reference books for the American Chemical Society



(ACS) entitled *Microwave-Enhanced Chemistry: Fundamentals, Sample Preparation and Applications*, and *Introduction to Microwave Sample Preparation: Theory and Practice* (in 1997 and 1988). Since 1987, he has received numerous awards for these innovative inventions, including the 1996 R&D 100 Award for development of Speciated Isotope Dilution Mass Spectrometry (SIDMS) and the 1987 IR 100 Award for development of the microwave-enhanced chemistry and in 1989 for the invention of chelation ion chromatography. He holds a dozen patents in the field of (SIDMS), mass spectrometry automation, microwave-enhanced chemistry, chelation chromatography, and has others pending and remains interested in the development and automation of analytical analysis methods.

ST-06 Contamination Control for Trace Element Analysis. Brad McKelvey, Seastar Chemicals Inc., 10005 McDonald Park Rd., Sidney, BC V8L5Y2, Canada, bmckelvey@seastarchemicals.com

Current instrumentation has the ability to determine many elements down to the ppt and ppq level. However, many analytical techniques for trace element determinations are limited by the blank. The focus of this course will be contamination control and strategies to determine and eliminate contamination sources. This course will discuss contamination sources from the environment, labware, reagents, sample handling and sample introduction systems. Participants will be encouraged to discuss their contamination problems and experiences.

Keywords: Contamination Control, Trace Element Analysis, Blanks



Brad McKelvey is the Quality Assurance Manager at Seastar Chemicals Inc. Seastar Chemicals is a global supplier of high purity reagents for trace element analysis. Dr. McKelvey has over 20 years experience in ICP-MS analysis, sample preparation, and contamination control for ultra-trace elemental analysis.

ST-07 Trace Element Speciation. Olivier F.X. Donard, Laboratoire de Chimie Analytique Bio-Inorganique et Environnement, UMR CNRS 5034, Université Pau et des Pays de l'Adour, F-64000 Pau, France, olivier.donard@univ-pau.fr

The chemical forms of metals in natural, aqueous, and solid environments are of considerable interest. The approaches and techniques used to sample, preserve, and measure the chemical forms of trace elements in environmental samples will be the focus of this course. Regulatory procedures, development of methodology, and collaborative exercises also will be examined. Chromatographic and other separation techniques, generation of species-specific gases and vapors, and other approaches will be discussed. The role of plasma and furnace spectroscopic detection systems will be examined.



Keywords: Arsenic, chromium, mercury, tin, chemical forms, separation, speciation

ST-08 Plasma Diagnostics: Fundamentals, Measurements, Applications. Paul B. Farnsworth, Department of Chemistry and Biochemistry, Brigham Young University, Provo, UT 84602, paul_farnsworth@byu.edu

This course will provide an introduction to the fundamental characteristics of plasmas that affect their analytical performance. The introduction will be followed by discussions of how to measure those plasma characteristics and manipulate them to improve the performance sources used for spectrochemical analysis. Among topics to be covered are temperatures, thermal equilibrium, electron densities, excitation processes, line profiles, temporal and spatial resolution.

Keywords: Thermal equilibrium, excitation processes, electron densities, line profiles, time resolution, imaging.

Paul Farnsworth is currently chair of the Department of Chemistry and Biochemistry at Brigham Young University.



He earned his B.S. degree in 1977 from BYU, and then went on for doctoral work at the University of Wisconsin in Madison under the direction of John Walters. He did postdoctoral work with Gary Hieftje at Indiana University, and then returned to BYU as an assistant professor in 1983. He has had two appointments as a visiting scientist at the Joint Research Center

of the European Commission in Ispra, Italy, the first in 1989 and the second in 1998, working in the laboratories of Nicolò Omenetto. Professor Farnsworth's primary research interests are laser and atomic spectroscopy. He began his research career studying energy transport and excitation mechanisms in inductively coupled plasmas used as emission sources. His emphasis has slowly shifted to elemental mass spectrometry. In recent years he has been using laser-excited fluorescence as a tool to study ion transport through the vacuum interface of an inductively coupled plasma mass spectrometer. His ion transport has been twice recognized with the *Spectrochimica Acta* atomic spectroscopy award, first in 1998, and then in 2006. He received the 2006 Lester W. Strock Award from the Society for Applied Spectroscopy and the 2006 Utah Award from the American Chemical Society.

ST-09 Preparing Your Laboratory for Trace Analyses. Ela Bakowska, NMS Labs, 3701 Welsh Rd, Willow Grove, PA 19090, ela_bakowska@yahoo.com

This course is designed to assist participants in installation of their first ICP-MS, renovating of a laboratory, remodeling of a laboratory, improving trace level capabilities by eliminating and reducing contamination and optimization of the laboratory layout. ICP-MS offers trace and ultra-trace level measurement capabilities. Those levels require careful planning of the laboratory. The choices of water supply, cabinets, benches, hoods, reagents, standards, reference materi-



als, consumables and other items will be presented. Cost-savings alternatives (without compromising of the quality) will be presented. Different applications require different levels of the purity. Specific examples will be provided for semiconductor, environmental and other laboratories.

Keywords: Lab design, trace analysis, contamination, optimization, standards, water, consumables

ST-10 Microwave-Assisted Sample Preparation for Spectrochemistry. Joaquim A. Nóbrega, Federal University of São Carlos, Department of Chemistry, São Carlos, SP, Brazil, djan@terra.com.br

This course presents both selected applications and the theory for sample preparation featuring microwave-assisted acid digestion for elemental and ultra-trace elemental analysis. Specific sample preparation approaches for AAS, ICP-MS and ICP OES including microwave power, reagent temperature, pressure, matrices, chemical compatibility, and practical standard methods will be discussed. Basic features of microwave decomposition methods, equipment, vessel design, and accessories will be covered. Methods for transfer of procedures between equipment are highlighted, and applied methods such as the new 3052 developed for EPA SW-846 for total microwave digestion of soils, sediments, ash, tissues, foods, and combination of these samples are reviewed. Safety considerations in laboratory microwave sample preparation and analysis are stressed. Special applications based on digestions with diluted acids or bases in closed vessels, vapor-phase digestion, and extractions will be discussed.

Keywords: Microwave heating, acid digestion, organic and inorganic sample preparation, elemental analysis, environmental analysis, microwave equipment, microwave theory, safety



ST-11 Metallomics Approaches for Speciation in Biological Samples. Katarzyna Wrobel and Kazimierz Wrobel, University of Guanajuato, Chemistry Department, L. de Retana 5, 36000 Guanajuato, Mexico, katarzyn@quijote.ugto.mx

This course will describe sample treatment, separation and detection approaches to obtain biologically relevant analytical information on heteroelements within a given system. Basic principles and real-life applications of different leaching, extraction, volatilization and enzymatic treatments will be discussed. Furthermore, the preparation of



biological samples in terms of cell disruption, separation of sub-cellular fractions and isolation of a specific biomolecules will be addressed. The course will focus on hyphenated techniques that couple chromatographic separation with ICP-MS detection, yet the mass spectrometry tools providing structural information will be also included. Epigenetics as an emerging field in metallomics will be introduced, highlighting the potential of ICP-MS in this area. Some examples from

our laboratory will be presented, demonstrating the feasibility of internal and external elemental tags for the evaluation of DNA and RNA methylation.

Keywords: Analytical speciation, metallomics, epigenetics, sample treatment, HPLC, ICP-MS

ST-12 Isotopic Analysis of Natural Inorganic Samples by Laser Ablation ICP Mass Spectrometry. Jan Kosler, Department of Earth Science, University of Bergen, N-5007 Bergen, Norway, jan.kosler@geo.uib.no; Mike Tubrett, Inco innovation Centre, MAFIIC, Memorial University of Newfoundland, mtubrett@mun.ca, and Rebecca Lam, Inco Innovation Centre, MAFIIC, Memorial University of Newfoundland, rlam@mun.ca

This course will describe the techniques used to measure ratios of stable and radiogenic isotopes in natural inorganic materials, such as geological samples, using laser ablation attached to single- and multiple-collector ICP-MS. The topics covered will include optimization of the laser and ICP mass spectrometer for precise measurement of isotopic ratios, effects of laser beam parameters on elemental and isotopic fractionation and signal stability, correction procedures for laser-induced fractionation of elements and isotopes, instrument mass bias and isobaric interferences. The techniques will be demonstrated on real data sets and examples will include applications of Fe, S, Sr, Hf, Nd and Pb isotopic ratio measurements and U-Th-Pb dating methods.



Keywords: Laser ablation ICP-MS, laser-induced fractionation, instrument mass bias, isotopic ratios, stable and radiogenic isotopes

ST-13 Speciated Isotope Dilution Analyses by Mass Spectrometry: New Standards Focusing on Environmental Health, Forensics, and Homeland Security. H.M. (Skip) Kingston, Matt Pamuku, and Mizanur Rahman, Duquesne University, Department of Chemistry and Biochemistry, Pittsburgh PA 15282, kingston@duq.edu

This course will focus on the use of the newly promulgated Federal government speciation methods adopted by EPA and other government agencies known as EPA Method 6800, 3200 and on related methods for speciation analysis. Species that transform during sample handling and speciated analysis, such as those of chromium, mercury, arsenic, cyanide, azide, toxic adducts of glutathione and other species of interest in environmental health, environmental forensics and homeland defense will have their qualitative and quantitative protocols described. Fundamentals of sample preparation and handling, including extraction, stable enriched isotopic spiking and species stabilization integrated with multiple mass spectrometry detection platforms will be covered. The course will describe and compare isotope dilution mass spectrometry (IDMS) and Speciated Isotope

Dilution Mass Spectrometry (SIDMS) as qualitative, quantitative and diagnostic tools for molecular and ionic species analyses by mass spectrometry. LC-ICP-MS, GC-MS, ESI-TOF-MS, and MALDI-TOF-MS methods will be covered, compared and enabled. The course provides support methods and materials for current available instrumentation, reagents and standards used in carrying out these analyses. Example software for solving simultaneous equations and implementation tools is provided on CD as part of the course. New and additional techniques and methods to accomplish these recently approved methods will be developed in addition to the method documentation provided by US government agencies to enable the analyst to perform these challenging speciated analyses.

Keywords: Environmental forensics, environmental health, speciation, ICP-MS, isotope dilution, speciated isotope dilution, arsenic, chromium, mercury, selenium, cyanide, species, EPA methods.

ST-14 Bioinorganic Speciation Analysis and Metallomics. Ryszard Lobinski and Joanna Szpunar, LCABIE, UMR5254, CNRS, Hélioparc, 2, av. Pr. Angot, F-64053 Pau, France, ryszard.lobinski@univ-pau.fr



The short course discusses analytical techniques and methods for the probing of interactions between metal ions and the cellular genome and the derived -omes: proteome and metabolome. Particular attention is given to the *in-vivo* screening for the native metal-protein and metal-metabolite complexes by hyphenated techniques, which combine a high-resolution separation technique (gel electrophoresis, chromatography or capillary electrophoresis) with sensitive elemental (ICP MS) or molecular (electrospray or MALDI MS) detection. The role and contribution of bioinformatics to the prediction of metal-binding sequences in proteins and of molecular biology approaches for detecting metal-dependent genes, proteins and metabolites are highlighted.

Keywords: bioinorganic speciation analysis, metalloproteomics, metallometabolomics, metallomics, chromatography, electrophoresis, hyphenated techniques

Ryszard Lobinski obtained his Ph.D. (1989) and D.Sc. (habilitation) (1994) degrees from the Warsaw University of



Technology. He held postdoctoral positions at the Institute of Spectroscopy and Applied Spectrometry (ISAS) in Dortmund and at the University of Antwerp (U.I.A.). Since 1994 he has been working for the French National Research Council (CNRS). Ryszard Lobinski is research director at the CNRS, professor of chemistry at the Warsaw University of Technology and the co-director of UltraTrace Analyses Aquitaine (UT2A). He is the author of 3 books and ca. 200 research papers in the areas of atomic spectroscopy, trace element speciation and molecular description of metal interactions with bioligands.

ST-15 Experiences of an Expert Witness. Ela Bakowska, NMS Labs, 3701 Welsh Rd, Willow Grove, PA 19090, ela_bakowska@yahoo.com

This short course is based upon my personal experience as an expert witness. I testified in the fields of elemental testing, arsenic speciation and gunshot residue (GSR) testing in several murder cases in six states: California, Delaware, New Jersey, New York, North Dakota and Pennsylvania. I was called as an expert witness both by the prosecution and by the defense. This course will describe how to be an effective expert witness and what the major challenges associated with this role are. Also discussed will be the differences between testimonies in regular and high profile cases, especially televised trials.

Keywords: Expert testimony, expert witness, murder, Arsenic, gunshot residue, trial

ST-16 Clean Microwave Digestions for Ultra-Trace Analysis. H.M. (Skip) Kingston and Mizanur Rahman, Duquesne University, Department of Chemistry and Biochemistry, Pittsburgh PA 15282, kingston@duq.edu

This course presents both selected applications and the theoretical non-intuitive relationships in microwave sample preparation featuring solvent and acid dissolution and extraction for elemental, ultra-trace elemental analysis and species analysis. Specific sample preparation for ICP-MS and ICP-AES and ESI-TOF-MS including fundamental relationships of analyte recovery, power, reagent temperature, pressure, matrices, chemical compatibility, and practical standard methods will also be discussed. Basic features of microwave decomposition methods, equipment, vessel design, and accessories will be covered. Methods for transfer of procedures between equipment are highlighted, and applied methods such as the new 3052, 3200, 3051A, 3015a, 3546, 3030K, and others developed for EPA SW-846 and other standards organizations using microwave sample preparation for analysis in soils, sediments, water, tissues, foods, blood, urine and combination of these samples are reviewed. Safety considerations in laboratory microwave-enhanced sample preparation and analysis are stressed.

Keywords: HEPA filters, clean rooms, clean laboratories, microwave digestions, sample preparation, extraction, trace element analysis, clean laboratory chemistry, applications

ST-17 Trace Element and Isotopic Analysis of Forensic Evidence. Christopher Latkoczy, ETH Zurich (D-CHAB), Lab of Inorg Chem, HCI G111, Wolfgang-Pauli-Strasse 10, 8093 Zurich, Switzerland, Latkoczy@inorg.chem.ethz.ch

The short course covers background material on the theory and application of trace elemental and isotopic analyses of materials and includes examples on the origin of trace elements and isotopes in commonly encountered materials in forensic laboratories, the pro and cons of analytical systems and data presentation and interpretation. The aim of this short course is to teach participants how to use trace elements and isotopes to analyze forensic samples and to interpret and present the data produced. Educational objectives of this short course are to understand the basic

principles of trace elemental and isotopic analysis, become aware of how these analytical methods can be applied to forensic sciences and learn how to access these methods for their own work.

Keywords: forensic sciences, analytical techniques, laser-based techniques, trace elements

Christopher Latkoczy has been a research assistant at the Eawag (Siedlungswasserwirtschaft, particle lab), Switzerland studying the field of LIBD for the analysis of colloidal nanomaterials in aquatic systems since April 2007. He conducted post-doctoral studies (Habilitation) at the ETH Zurich (D-CHAB), Switzerland, Laboratory of Inorganic Chemistry, (Prof. Dr. Detlef Günther) in the field of laser ablation, ICP-MS processes, and confocal microscopy imaging techniques (Habilitation May 2007) from January 2001. He was Director of the Laboratory for Isotope and Trace Element Research, Old Dominion University, Department of Chemistry and Biochemistry, Norfolk, Virginia, USA - in the field of elemental and isotopic analysis of oceanographic samples. Earlier he was a research assistant at the University of Agricultural Sciences - BOKU Vienna, Institute of Chemistry in the field of strontium isotopic ratio analysis in Prehistoric human bone samples by means of ICP-SF-MS, where he did his Ph.D. studies in the field of determination of heavy metals in soil and water samples and isotopic ratio analysis.



ST-18 Reference Materials and Standards for Quality Control of Speciation Measurements. Heidi Goenaga Infante, LGC, Queens Road, Teddington, Middlesex TW11 OLY, UK, hgi@lgc.co.uk

The need for global comparability of analytical results has traditionally been met through comparisons between laboratories or widespread use of reference materials (RMs). Traceable measurements depend on higher order calibrants, reference methods and reference materials which have fully evaluated measurement uncertainty and are traceable to internationally accepted references. In the speciation field, which faces a large number of sources of error during sample preparation, species separation and detection, species-CRMs are scarce and, therefore, urgently needed for quality control of the speciation measurements. This course will discuss the need and availability of "speciated" certified reference materials in the healthcare and environmental fields. Also, the requirements for the preparation and characterisation of CRMs for speciation analysis, arising from experience gained within a series of interlaboratory studies organized by LGC in collaboration with other national metrology institutes, will be addressed.

Keywords: Reference materials, standards, speciation analysis, quality assurance, inter-laboratory comparison, traceability, national metrology institutes, biological, clinical, environmental.

Heidi Goenaga-Infante graduated from the University of Havana, Cuba and was then awarded a MUTIS fellowship from the Spanish International Cooperation Agency (Madrid, Spain) at the University of Oviedo, Spain from which she



obtained her Ph.D. in 1999. She was a research assistant at the Micro and Trace Analysis Centre (MiTAC), University of Antwerp (1999-2003). Since 2003, she joined, as a senior research and development scientist, LGC's Specialised Techniques Area in Teddington, United Kingdom. Dr. Goenaga-Infante is currently a principal scientist at LGC. Her

research interests concern the combined application of elemental and molecular mass spectrometry with chromatography for heteroatom speciation analysis of dietary substances, supplements and clinical samples and the high accuracy determination of heteroatom-containing species in these substances by isotope dilution mass spectrometry, with the aim of characterising new "speciated" certified reference materials. She has undertaken and managed a wide variety of projects and studies including the coordination of CCQM (Consultative Committee on Amount of Substance) international intercomparisons in the speciation field.

ST-19 Proficiency Testing Issues Relating to Clinical ICP-MS. Christopher D. Palmer, palmer@wadsworth.org, and Patrick J. Parsons, Lead Poisoning/Trace Elements Laboratory, Wadsworth Center, New York State Dept. of Health, P.O. Box 509, Empire State Plaza, Albany NY 12201-0509

Proficiency Testing (PT) is an evaluation of participant performance against a pre-established criteria by means of interlaboratory comparisons. Typically, clinical ICP-MS laboratories will participate in multiple PT events per year, by testing PT materials (whole blood, serum, and urine matrices) for a suite of trace elements, at levels that would represent both background and occupational levels of exposure. We describe the operation of the NY State PT program for trace elements. Additionally, we will present some of the general requirements for clinical PT material manufacture; preparation, conformity assessment (stability and homogeneity), and target value assignment, all leading to ISO/IEC 17043 accreditation for PT scheme providers. We will also examine how a participant's performance may vary according to different PT schemes, or why there may be notable biases for assigned target values for different instrumental techniques (e.g., ICP-MS versus atomic absorption). Finally, we will discuss the problem identification resulting from a failed PT event, the mechanism of corrective action, and resolution from the perspective of a "large" commercial clinical ICP-MS laboratory.

Keywords: Proficiency testing, clinical laboratory, accreditation, performance, ICP-MS

Patrick J. Parsons received his Ph.D. from the University of London in 1983, following which he completed postdoctoral training at the National Institutes of Health. He is currently the Chief of Laboratory and Nuclear Chemistry, and the Deputy Director of the Division of Environmental Disease Prevention at the Wadsworth Center, New York State Department of



Health. As the Section Head of the Trace Elements Proficiency testing program he oversees the production of blood, urine and serum proficiency testing events.



Christopher D. Palmer received his Ph.D. from the University of Massachusetts, Amherst in 2001. He is a Research Scientist at the Wadsworth Center where he develops and maintains clinical ICP-MS methodology for trace element biomonitoring purposes. He is also responsible for the production of NY State blood and serum proficiency testing materials.

Workshop on New Plasma Instrumentation

WS-01 Workshop on New Plasma Instrumentation

A workshop featuring current plasma instrumentation will be presented during three afternoons to highlight the latest commercial plasma instrumentation and related products.

The workshop will be divided into three main sections: plasma source (ICP) atomic emission spectroscopy, plasma source (ICP) mass spectrometry, and plasma accessories (e.g., chromatograph interface, electrothermal vaporizer, laser and spark ablation, special nebulizers, preconcentration and sample introduction equipment, special adapter kits) and sample preparation. Representatives from major plasma companies and/or exhibitors will present brief technical descriptions and discussions of their new product developments and design philosophy. Each company will provide product literature, application notes, and presentation summaries. Participants will be encouraged to question representatives. The afternoon program is planned to parallel the exhibition and poster sessions. There is no charge to attend the workshop.

Keywords: New instrumentation, product description, ICP-AES, ICP-MS, accessories

Manufacturers' Seminars

Seminar abstracts and schedules to be announced.

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