

AGENDA MAY 15-17, 2007

SHORT COURSES

Tuesday, May 15, 2007

8:00 AM - 4:30 PM	“Troubleshooting HPLC Systems” John Dolan
8:00 AM - 4:30 PM	“Practical Advice for Developing Better GC Methods” Daron Decker
8:00 AM – 4:30 PM	“Introduction to cGMP’s: A Systems Based Approach” David Bliesner
9:45 AM	Morning Break
12:00 – 1:00 PM	Lunch
2:30 PM	Afternoon Break

Wednesday, May 16, 2007

8:00 AM - 12:00 PM	Short Courses continue
9:45 AM	Morning Break
12:00 – 1:00 PM	Lunch

SPRING SYMPOSIUM

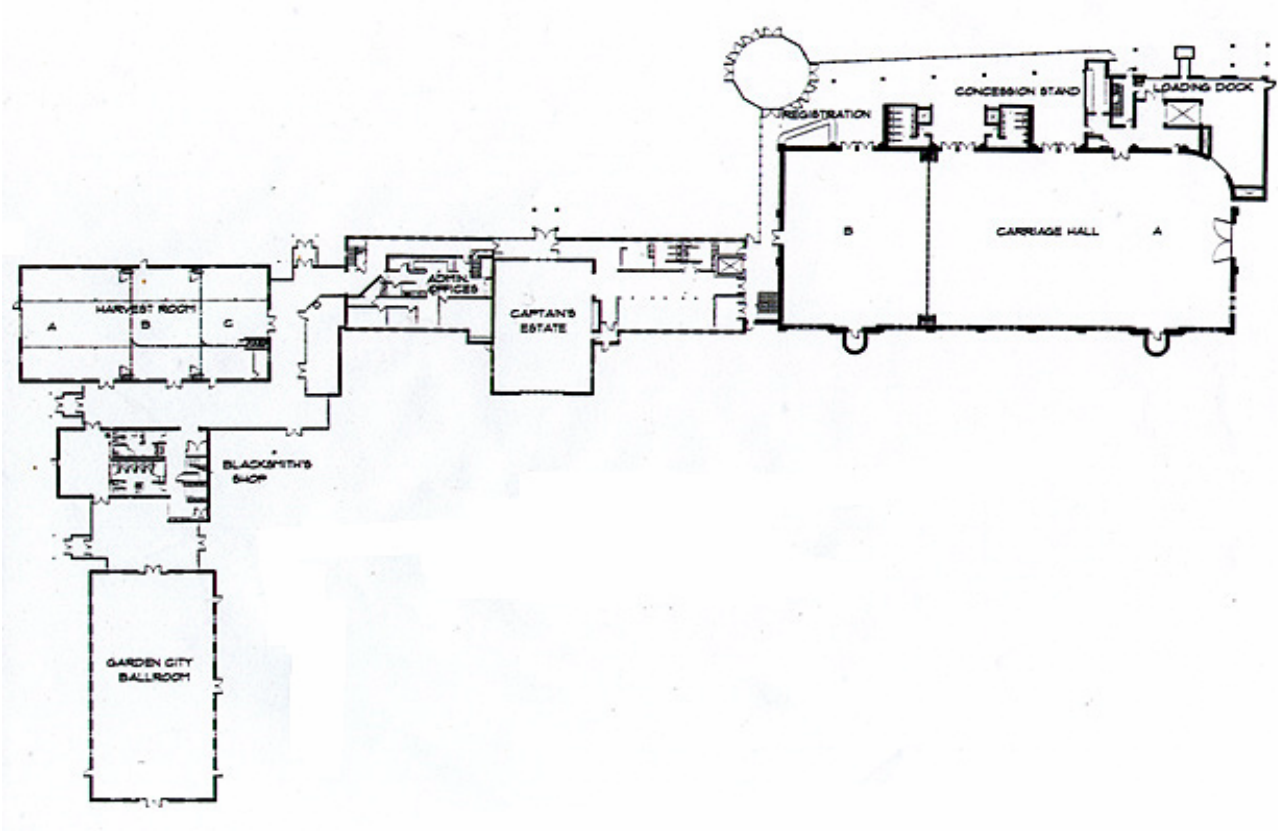
Wednesday, May 16, 2007

12:30 PM - 5:30 PM	Exhibits open
1:00 PM - 5:00 PM	Vendor Seminars
1:00 PM – 4:00 PM	Special Topics Sessions
1:00 PM - 5:00 PM	Posters to be displayed
4:00 PM - 5:00 PM	Authors asked to be with their posters
3:30 PM – 5:30 PM	Reception in Exhibits Area

Thursday, May 17, 2007

7:30 AM - 8:30 AM	Registration
8:30 AM - 10:30 AM	Opening Session
8:30 AM	Welcome and Introductory Remarks
8:45 AM	Palmer Award Presentation
8:55 AM	Undergraduate Research Grant Presentation
9:00 AM	Keynote Address
10:00 AM	Refreshments in Exhibits Area
10:30 AM - 4:30 PM	Oral Presentations
10:00 AM - 4:00 PM	Vendor Exhibits Open
12:00 PM - 1:00 PM	Lunch
3:00 PM – 3:40 PM	Refreshments in Exhibits Area and Prize Drawing
3:00 PM – 3:40 PM	Authors asked to be with their posters
5:30 PM	Annual Business Meeting

MAP OF THE EARLE BROWN HERITAGE CENTER

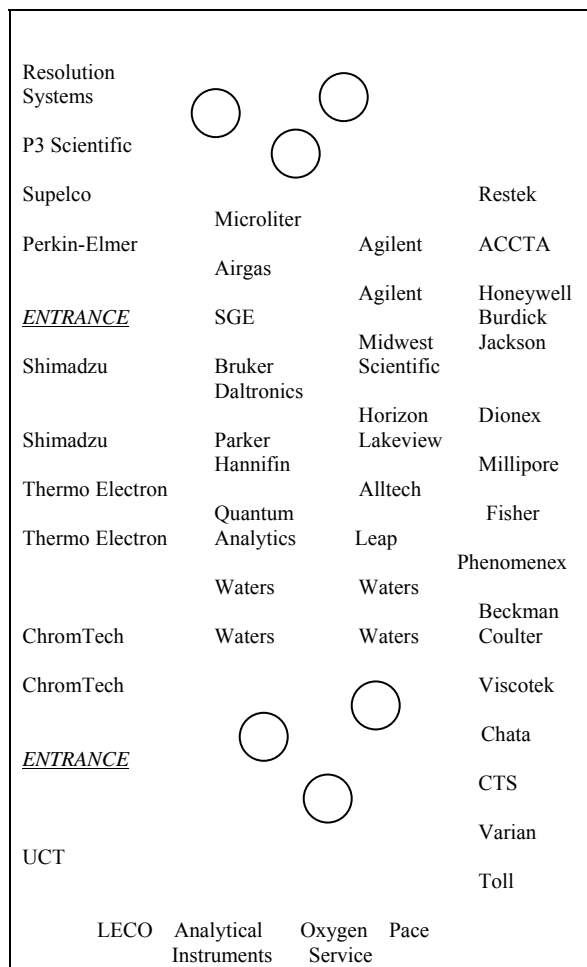


EXHIBITORS AND EXHIBIT FLOOR PLAN

ACCTA, Inc.
 Aerotek
 Agilent Technologies
 Airgas
 Alltech Associates
 Analytical Instruments
 Beckman Coulter
 Bruker Daltonics, Inc.
 Chata Biosystems
 Chromatography Tech. Services
 ChromTech, Inc.
 Dionex Corp.
 Fisher Scientific
 Honeywell Burdick & Jackson

Lakeview
 LEAP Technologies
 Leco Corporation
 MicroLiter Analytical Supplies, Inc.
 Midwest Scientific Inc.
 Millipore, Lab water Division
 Oxygen Service
 Pace Analytical Services
 Parker-Hannifin
 Part 3 Scientific
 Perkin Elmer
 Phenomenex
 Quantum Analytics

Resolutions Systems, Inc.
 Restek Corp.
 SGE Inc.
 Shimadzu Scientific Inst.
 Supelco
 Teledyne Tekmar
 Thermo Electron
 Toll Company
 United Chemical Technologies
 Varian
 Viscotek
 VWR Scientific Products
 Waters Corporation



— SPECIAL TOPIC SESSIONS —
Wednesday Afternoon, May 17

A new feature at the 2004 MCF Spring Symposium was the “Special Topic Sessions” on Wednesday afternoon. The Special Topic Sessions will address practical laboratory topics in GC, HPLC and MS. The intent is to provide topics of general interest and current utility to local chromatographers by leaders in each Special Topic area.

The sessions will be approximately 45 minutes in length, and focus on practical topics. After a brief introduction to a topic, the moderators will open the discussion for comments and questions.

Schedule for Wednesday afternoon, May 16

Special Topic Sessions --- Captain's

- 1:00pm HPLC Special Topic Session
 John Dolan, Bioanalytical Systems and LC Resources
 Daniel Marchand, Univ of WI – River Falls
- 2:00pm GC Special Topic Session
 Daron Decker, Agilent
- 3:00pm MS Special Topic Session
 Cliff Jacoby, 3M
 Cleston Lange, 3M

Schedule for Wednesday afternoon, May 16

Time	Captain's Room	Tack A (lower level)	Tack B (lower level)
1:00	HPLC Special Topics John Dolan, Dan Marchand & John Kern	Automated Eluent Preparation Mike Riley, Chata Biosystems	
1:30			The use of Comprehensive GC*GC TOFMS in Analyzing Complex Samples Mark Greenbaum, LECO Corporation
2:00	GC Special Topics Daron Decker & Rick Rossiter	Progress in Understanding Reversed Phase Retention Mechanisms: Designing Orthogonal HPLC Stationary Phases Richard A. Henry, SUPELCO	Advanced Macromolecular Characterization employing Tetra Detection SEC/GPC Shawn Welch, Viscotek
2:30			Design of High Purity Blends for Chromatography Utilizing Six Sigma Methodology Tony Kemperman, Honeywell Burdick & Jackson
3:00	Agilent	Characterizing Volatiles in Viscous Liquids and Solids using Thermal Extraction GC/MS Bob Freeman, Quantum Analytics	Care of Capillary Inlet Systems John Kroska, Lakeview Inc.
3:30			Shimadzu
4:00	Resolution Systems	Maintaining Gas Stream Purity and Improving Analytical Performance – S/N Ratio for GC/MS, FTIR, FID	Galaxie Data Systems Chad Bullard, Varian Inc.

TECHNICAL PRESENTATIONS – THURSDAY, MAY 17, 2007

7:30

REGISTRATION - LOBBY

8:00

GENERAL SESSION:

Introduction / 28th Anniversary / Palmer Award / Undergraduate Award

9:00

**Keynote Address: Professor Jim Jorgenson, University of North Carolina
“Ultra-High Pressure Liquid Chromatography”**

10:00

EXHIBITS AND POSTERS - CARRIAGE HALL

Time	Captain's Room	Tack A (lower level)	Tack B (lower level)
	HPLC	GC	LC- MS x Proteins
11:00	*** FOCUS SPEAKER *** Dr. Ron Majors, Agilent Technologies	*** FOCUS SPEAKER *** Dr. Robert Mustacich, RVM Scientific	HPLC Method Development for the “Proteome Lab High Capacity LC10” Immunoaffinity Column L. Anderson 6
11:20	High Throughput and High Resolution HPLC: Smaller Particles and Faster Separations 2	Changing GC Practice with Low Thermal Mass Technology 4	Large-Scale ID of Proteins in Whole Saliva of Oral Cancer Patients via 3D Peptide Separations & Tandem MS H. Xie 7
11:40	UPLC - Tandem Mass Spectrometry in the Food and Biotechnology Industries 3 J. Dalluge	The Role of EGA-GC/MS in the Characterization of Additives and Contaminants in Polymeric and Naturally Occurring Matrices 5 R. Freeman	Prediction of Peak Overlap Frequency Using Independent Doublets Model D. Andreyev 8
12:00	LUNCH (12:00-1:20)	LUNCH (12:00-1:20)	LUNCH (12:00-1:20)
	HPLC – Method Dvlpmt	Drug Analysis	LC-MS x Proteins
1:20	Effect of Mobile Phase pH and Buffer on Peak Shape and Loading Capacity in the Separation of Zwitterions J. Li 9	Residual Methamphetamine Contamination in Former Clandestine Labs Using HPLC-MS M. Bevan 13	*** FOCUS SPEAKER *** Dr. Carlos Gartner Harvard Medical School
1:40	Designing a Bullet-Proof QC Method S. Anderson 10	Quantitation of Aminoglycosides in Tissue and Serum Using HPLC-MS/MS S. Marimanikkuppam 14	Development of Catch-and-Release (CAR) Reagents for Quantitative Proteomics Analysis 17
2:00	The Effect of Water Quality on Chromatographic Performance M. Tarun 11	Quantitative Analysis of Corticosteroid in Equine Joint Fluid S. Park 15	Mass Spectrometric-Based Methods for Clinical Biomarker Discovery and Validation L. Higgins 18
2:20	Chiral Selector Screening and Regeneration of Zirconia Chiral Stationary Phases D. Nowlan 12	Subcellular Distributions of Leucyl-Doxorubicin Using Micellar Electrokinetic Capillary Chromatog with Laser Induced Fluor Detn Y. Wang 16	Differential Expression of Sera Protein Biomarkers for Ovarian Cancer J. Andersen 19
2:40	2006 MCF Undergraduate Awardee – Ryan Thurber Winona State University “DETECTION OF SULFONAMIDES IN WASTEWATER VIA HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY AND SOLID-PHASE EXTRACTION” Captain's Room 20		
	BREAK (3:00-3:30)		
	HPLC - Selectivity		Proteins: LC-MS & Proteins: HPLC & New HPLC Detection
3:30	The Role of Chemical Selectivity in Chromatography Y. Zhang 21		HDMS – A Powerful, New, High Efficiency Ion Mobility-TOF Hybrid MS System R. Upham 24
3:50	Retention Effects for Cationic Analytes on PerFluoroPhenyl (PFP) Stationary Phases M. Bicking 22		Protein-Polymer Interaction by HPLC J. Yaeger 25
4:10	Molecularly Imprinted Polymer Extraction – Compound- and Class- Specific SPE C. Santasania 23		Evaluation of Sensitivity and Linearity of Condensation Nucleation Light Scattering Detector D. Oberreit 26

AN INNOVATIVE APPROACH TO LOW MASS, ZERO DEAD VOLUME CONNECTION OF FUSED SILICA COLUMNS

Robert Freeman (Restek) Poster 1

UNIQUE COLUMN ALTERNATIVES FOR THE DETERMINATION OF EXPLOSIVES AND PROPELLANT RESIDUES VIA HPLC-UV

Robert Freeman (Restek) Poster 2

A NEW POLARITY SCALE FOR GC PHASES

Dan DiFeo (SGE Inc.) Poster 3

ON-LINE AND OFF-LINE APPLICATION OF MICRO-SPE

Dan DiFeo (SGE Inc.) Poster 4

NEW APPROACHES TO THE GC INJECTION PORT

Dan DiFeo (SGE Inc.) Poster 5

LC/MS SOLVENT BLENDS STABILITY

Tony Kemperman (Honeywell, Burdick and Jackson) Poster 6

EVALUATION OF THE ANALYTICAL ADVANTAGES OF NEW STATIC AND DYNAMIC HEADSPACE TECHNOLOGY

Jeff Sheriff (EST Analytical, Fairfield, OH) Poster 7

EVALUATION OF UV AND LIF DETECTION FOR CE-SDS PURITY APPLICATIONS FOR PRODUCT RELEASE AND STABILITY TESTING OF RECOMBINANT MONOCLONAL ANTIBODIES

Chad Wieneke (PDL Biopharma) Poster 8

COMPARISON AND ASSESSMENT OF WATER PURIFICATION TECHNOLOGIES USING ION CHROMATOGRAPHY

S. Mabic (Millipore Corp.) Poster 9

UV TECHNOLOGIES IN LAB WATER PURIFICATION SYSTEMS

S. Mabic (Millipore Corp.) Poster 10

ACHIEVING SUB-2 μ m PERFORMANCE AT MODERATE PRESSURES USING FUSED-CORE™ PARTICLE TECHNOLOGY

Carmen T. Santasania (Supelco / Sigma-Aldrich) Poster 11

EVALUATION OF 4-PROPYLAMINOMETHYL-BENZOIC ACID MODIFIED SILICA (4-PAMBAS) AS A CATION/ANION EXCHANGE HPLC STATIONARY PHASE.

Asanka Wijekoon (Kent State Univ, Dept of Chemistry) Poster 12

HIGH SPEED CHROMATOGRAPHIC ANALYSIS USING SUB-2 MICRON HPLC PACKINGS

Mark Jacyno (Grace Discovery Sciences, Deerfield, IL) Poster 13

EFFECT OF INADVERTENT THERMAL DEGASSING OF DISSOLUTION MEDIA: FINDINGS FROM METHOD TRANSFER

Peju Odunusi (Cima Labs Inc.) Poster 14

TROUBLESHOOTING GC HEADSPACE AUTOSAMPLER CONNECTIONS

Gregory Schmidt (Boston Scientific) Poster 15

Abstract # 1

*** KEYNOTE ADDRESS ***

ULTRA-HIGH PRESSURE LIQUID CHROMATOGRAPHY

JAMES W. JORGENSON

Department of Chemistry, University of North Carolina, Chapel Hill, NC 27599-3290

The history of HPLC has seen a progression in the use of columns packed with particles of decreasing size. Decreasing particle size has led to smaller values of the plate height and faster optimum velocities. Due to pressure limitations of conventional HPLC equipment this trend has translated, not into columns of increasing separation efficiency, but instead into shorter columns offering faster analysis times. The 400 bar pressure limit of conventional HPLC technology is an arbitrary limit. The use of ten-fold higher pressure allows the use of columns 40 cm long, packed with 1 micron particles, delivering 250,000 theoretical plates with column void times of a few minutes.

Significant amounts of heat can be generated in pumping solvents at optimum velocities through such a highly restrictive bed of particles. In a column of conventional diameter (4.6 mm), this heat will result in significant axial and radial temperature gradients, which will lead to excessive band spreading. Packed capillary columns can be used to reduce this difficulty. The design and performance of systems capable of isocratic and gradient elution liquid chromatography in packed capillary columns at ultra-high pressures will be described. Results of UHPLC separations of small organics, peptides and proteins will be discussed.

“Ultra High Pressure Reversed Phase Liquid Chromatography in Packed Capillary Columns”, J.E. MacNair, K.C. Lewis, and J.W. Jorgenson, *Analytical Chemistry*, 69, 983-989 (1997).

“Ultra High Pressure Reversed-Phase Liquid Chromatography: Isocratic and Gradient Elution Using Columns Packed with 1.0 μm Particles”, by John MacNair, Kamlesh Patel, and J.W. Jorgenson, *Analytical Chemistry*, 71, 700-708 (1999).

“The Use of 1.5 micron Porous Ethyl-Bridged Hybrid Particles as a Stationary Phase Support for Reversed-Phase Ultra-High Pressure Liquid Chromatography”, J. Scott Mellors, and James W. Jorgenson, *Analytical Chemistry*, 76, 5441-5450 (2004).

“In-depth Characterization of Slurry Packed Capillary Columns with 1.0 micron Nonporous Particles Using Reversed Phase Isocratic Ultra-High Pressure Liquid Chromatography”, Kamlesh D. Patel, Anton D. Jerkovich, Jason C. Link and James W. Jorgenson, *Analytical Chemistry*, 76, 5777-5786 (2004).

“Linear Velocity Surge Caused by Mobile-Phase Compression as a Source of Band Broadening in Isocratic Ultrahigh-Pressure Liquid Chromatography”, A. Jerkovich, S. Mellors, J.W. Thompson, and J.W. Jorgenson, *Analytical Chemistry*, 77, 6292-6299 (2005).

“Improved Protein Recovery in Reversed-Phase Liquid Chromatography by the Use of Ultrahigh Pressures”, John Eschelbach and J.W. Jorgenson, *Analytical Chemistry*, 78, 1697-1706 (2007).

BIOGRAPHICAL SKETCH

James Jorgenson was born in Kenosha, Wisconsin in 1952. He received his undergraduate education at Northern Illinois University where he received a B.S. in Chemistry in 1974. Following this he entered graduate school at Indiana University, where he worked in the research group of Professor Milos Novotny, and received a Ph.D. in Chemistry in 1979. His Ph.D. research concerned two principal areas; the study of mammalian pheromones, and the development of new detection schemes for liquid chromatography.

Dr. Jorgenson joined the faculty of the University of North Carolina as an Assistant Professor of Chemistry in 1979. He was promoted to Associate Professor in 1985, Professor in 1987, appointed the Francis P. Venable Professor of Chemistry in 1994, and William Rand Kenan, Jr. Distinguished Professor of Chemistry in 1999. He was Chair of the Chemistry Department from 2000 to 2005.

Among the honors he has received are the American Chemical Society Analytical Division Award in Chemical Instrumentation in 1992, the Martin Medal of the Chromatographic Society in 1992, elected a Fellow of the American Association for the Advancement of Science in 1992, the American Chemical Society Award in Chromatography in 1993, the Golay Medal in 1994, the Eastern Analytical Symposium Award in Separation Science in 1995, the Torben Bergman Medal of the Swedish Chemical Society in 1996, the Anachem Award in 1996, the Dal Nogare Award in 1998, the Esselen Award for Chemistry in the Public Interest in 2004, the Pittsburgh Conference Analytical Chemistry Award in 2005, and the American Chemical Society Award in Analytical Chemistry in 2007.

Professor Jorgenson is one of the originators of capillary electrophoresis, with his first publications on this topic appearing in 1981. His current research interests include ultra-high pressure liquid chromatography, multidimensional separations, microscale separations coupled to mass spectrometry, and the design of detectors for chromatography and capillary electrophoresis.

Abstract # 2

*** HPLC FOCUS SPEAKER ***

HIGH THROUGHPUT AND HIGH RESOLUTION HPLC: SMALLER PARTICLES AND FASTER SEPARATIONS

RONALD E. MAJORS, PH.D.,

Agilent Technologies, 2850 Centerville Road, Wilmington, DE 19808 USA

There are many driving forces for further developments in HPLC column technology: productivity gains, improvement in the quality of analysis; lowering costs; analysts facing smaller, more complex samples; the increasing importance of biologically-derived molecules; and the widespread use of LC/MS are some of the more important drivers. HPLC column technologists have responded to these needs with the development of columns specifically addressing these challenges. For example, one of the driving forces in improving laboratory productivity is to provide higher speed assays. New column formats have been specifically designed for high-throughput while maintaining separation performance. To achieve high speed and high throughput operation coupled with the need for high efficiency and resolution, the HPLC column can take on one of several formats:

- 1) Shorter column lengths (to reduce analysis time) packed with small porous particles (to maintain resolution); nowadays, sub-two micron particles are of increasing interest.
- 2) Longer column lengths (to increase efficiency) packed with even smaller porous and non-porous particles (to maintain resolution), the so-called "Ultra-high Pressure HPLC".
- 3) Narrower internal diameter columns (to increase sensitivity in sample-mass limited situations) packed with small porous particles (to maintain resolution).
- 4) Columns packed with small superficially porous particles that allow the rapid separation of biomolecules such as proteins.

Columns designed with new stationary phase formats such as monoliths and in-situ created stationary phases that show much promise in high speed and miniaturized chip-based systems.

This lecture will review some of these column developments and provide some guidance on how to choose the appropriate column to meet the increasing challenge of high-speed, high-sensitivity and high-resolution separations. A look at future directions in HPLC column technology will be presented.

Abstract # 3

**ULTRA-PERFORMANCE LIQUID CHROMATOGRAPHY /TANDEM MASS
SPECTROMETRY IN THE FOOD AND BIOTECHNOLOGY INDUSTRIES**

JOSEPH J. DALLUGE, KERI LYN ROSS, JAMIE A. KOEHLER, BREANN A. STOLTZ, and
TRAVIS T. TU

Cargill Incorporated, Minneapolis, MN 55440

Analysis of chemicals in biologically-derived, food and agricultural products requires both sensitive and selective analytical techniques. Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) is currently the gold standard for the determination of biological metabolites, non-volatile food components, and other analytes of interest found at trace levels in complex mixtures. As analytical throughput becomes paramount from the standpoint of expediting discovery, characterization of novel metabolic activities, rapid determination of health-beneficial natural products, and establishment of intellectual property, Ultra Performance Liquid Chromatography (UPLC), a new category of analytical separation science that retains the practicality and principles of HPLC while increasing the overall attributes of resolution, sensitivity and speed, represents a significant development in support of these activities. This talk will include discussion of a cross-section of analytical methodologies based on UPLC-MS/MS developed to address problems in the food and biotechnology industries.

Abstract # 4

***** GC FOCUS SPEAKER *****

CHANGING GC PRACTICE WITH LOW THERMAL MASS TECHNOLOGY

ROBERT MUSTACICH
RVM Scientific, Santa Barbara, CA

One of the main trends in analytical instrumentation is increased productivity. Regarding GC analyses, shortening the overall analysis cycle is central to increasing productivity, typically measured as an output per unit input. In an analytical laboratory this might be stated as the number of analyses/day for the laboratory, or the cost/analysis. Often, the GC temperature programming cycle constitutes the bulk of the analysis cycle time. Newer technologies such as Low Thermal Mass (LTM) GC technology are becoming common to accelerate this analysis step. LTM technology was developed in the laboratories of RVM Scientific for the temperature programming of GC instruments with low power consumption. For productivity applications, the large heating efficiency of LTM technology has directly translated into fast heating and cooling for faster analyses. LTM technology also squarely addresses the needs of other emerging trends in instrumentation such as smaller size and mobility. This presentation will provide an overview of LTM GC technology with a survey of applications showing how this technology is changing GC practice.

Abstract # 5

THE ROLE OF EVOLVED GAS ANALYSIS (EGA) – GC/MS IN THE CHARACTERIZATION OF ADDITIVES AND CONTAMINANTS IN POLYMERIC AND NATURALLY OCCURRING MATRICES

ROBERT FREEMAN

Quantum Analytics, Foster City, CA 94403

Evolved gas analysis (EGA) is based upon heating the sample and detecting vapors as they evolve from the sample. A plot of sample temperature versus detector response is called a thermogram. The thermogram provides insight into the sample complexity and indicates the temperature zones where compounds of interest are found. It also provides information about the optimum pyrolysis temperature for the sample.

Often, the EGA-MS thermogram itself can be used to identify a simple polymer or co-polymer. The average mass spectrum of the EGA is compared to the average mass spectra of a number of polymers. The search will eliminate many candidates and yield a short list of possible polymers. Absolute identification can then be obtained via pyrolysis.

Extracted ion chromatograms (EIC) are used to ascertain peak “purity”, determine the presence or absence of specific compounds (i.e., contaminants and additives) and enhance the quantitation of specific compounds.

The Frontier multi-functional pyrolyzer (Frontier Laboratories Ltd.) is ideal for performing EGA. The vertical micro-furnace ensures that the sample is heated in a reproducible manner. The initial temperature can be as low as 40°C and the inert, zero dead volume sample path assures compound integrity. The %RSD for an EGA profile is on the order of 0.11; therefore, shifts in the EGA profiles are significant and can be used to detect subtle differences in sample composition. EGA thermograms from several sample types including, ceramics, polymer blends, paper, and hydrocarbons will be presented. Each EGA will be examined and the information content used to characterize the constituents (contaminants, additives, polymer material, etc.) of the sample

Abstract # 6

**LC METHOD DEVELOPMENT FOR PROTEOME LAB IGY-12 HIGH CAPACITY LC10
IMMUNOAFFINITY COLUMN**

LORRAINE ANDERSON

University of Minnesota. Department of Biochemistry, Molecular Biology and Biophysics, Minneapolis, MN
55455

In serum and plasma, the dynamic range of proteins spans 10 orders of magnitude and a few proteins in high abundance mask potential biomarkers. To increase the likelihood of identifying biomarkers, we remove some of these high abundance proteins prior to LC-MS/MS. We are currently using the ProteomeLab IgY-12 High Capacity LC10 column (Beckman-Coulter) for immunoaffinity partitioning of these fluids.

I have developed an LC method for the 12.7x 79.0 mm LC10 column using HP Series 1100 quaternary pump HPLC. This LC method is a modification of a method supplied by Beckman Coulter for non-Beckman Coulter LC Systems. The immunopartitioning of plasma and cerebrospinal fluid has been very reproducible.

Abstract # 7

LARGE-SCALE IDENTIFICATION OF PROTEINS IN WHOLE SALIVA OF ORAL CANCER PATIENTS VIA THREE DIMENSIONAL PEPTIDE SEPARATIONS AND TANDEM MASS SPECTROMETRY

HONGWEI XIE AND TIMOTHY J. GRIFFIN

University of Minnesota, Department of Biochemistry, Molecular Biology and Biophysics, Minneapolis, MN 55455

Human saliva is an easy-accessed and non-invasive diagnostic fluid for cancer and diseases. Identifying protein biomarkers in saliva is useful for early diagnosis of oral cancer and a valuable clinical tool for patient follow-up. Here we report for the first time a full characterization of proteins in saliva taken from clinic patients diagnosed with OSCC using a novel proteomic method with extensive separation at the peptide level by a combination of preparative isoelectric focusing (IEF) using free flow electrophoresis (FFE) and strong cation exchange (SCX) fractionations prior to microcapillary reversed-phase liquid chromatography-tandem mass spectrometry (μ LC-MS/MS) analysis.

Our three dimensional (3-D) peptide separation method extensively fractionates peptide mixtures and enables high confident identification of proteins derived from both soluble and cellular fractions of whole saliva with the aid of peptide pI filtering. Peptides separated first by preparative IEF are compatible with a second SCX fractionation step. The effectiveness of peptide fractionation by SCX was clearly demonstrated by the different μ LC-MS/MS chromatogram patterns of SCX fractions eluted at different salt bumps from FFE fractions. Five times more peptides and proteins were identified using our 3-D separation method compared to a 2-D separation method.

Our proteomic method generated a catalogue of over 1000 proteins from the soluble fraction and 1900 proteins from the cellular fraction of saliva with high confidence, including low-abundance proteins and membrane proteins. Over 134 proteins from 34 different bacteria were also identified from saliva pellets. Coupling this method with peptide stable isotope labeling (such as the iTRAQ reagents) has tremendous potential for detecting salivary marker of oral cancer.

Abstract # 8

PREDICTION OF PEAK OVERLAPS FREQUENCY IN ELECTROPHEROGRAMS USING THE INDEPENDENT DOUBLETS MODEL

DMITRY ANDREYEV AND EDGAR ARRIAGA

Department of Chemistry, University of Minnesota, Minneapolis, MN

Peak overlaps often complicates analysis of data containing a multitude of uniform peaks. For example, electropherograms of individual particles typically have 102 - 103 peaks in a 10-min time window. The number of the observed peaks (m) usually is lower than number of true events (n) since some events are not individually resolved. The existing approaches to predict peak overlaps are not ideal. For example the Statistical Overlap Theory (SOT), requires a priori information about n .

Here, we suggest a new and direct approach for prediction of the number of peak overlaps. The following assumptions are made: (i) the only kind of overlaps are doublets and (ii) the probability of two given peaks forming a doublet remains the same for any pair of events in a given time interval. Under these assumptions, P , the conditional probability of observing m peaks when n true events are present can be defined mathematically. Is not trivial to find the maximum of P as a function of n analytically, but it can be found numerically.

The independent doublets model was applied to an electropherogram of polystyrene microspheres ($m = 450$, time window 358 s, average peak width 50 ms, minimum spacing between maxima of non-overlapping peaks 50 ms). The calculated number of doublets (15) was found to be in good agreement with number of experimental overlaps (17), estimated from increase in peak height. In contrast, the SOT predicted a significantly higher number of peak overlaps (54), when m is taken as an approximation of n .

Abstract # 9

Effect of Mobile Phase pH and Buffer on Peak Shape and Loading Capacity in Separation of Zwitterions

JIANWEI LI, BIN CAI, CARMEN SNAZA, WENDY FLEMING, THOMAS KEENE, DEANNA LANE
Medtronic Inc, Minneapolis, MN 55432

This presentation describes the results and conclusion of effect of mobile phase pH and buffer on the peak shape and column loadability in the separation of zwitterions by RPLC. Zwitterion molecules can be neutral or charged depending on the mobile phase pH, and it is expected that the pH-dependent charge state of analyte can have a significant effect on the peak shape and column loading capacity based on extensive studies by David McCalley and others. Different types of columns and mobile phases (pH and buffer) are used to elute a model analyte (baclofen) by both gradient and isocratic conditions. The peak shape and column loading capacity are then evaluated based on separation conditions (column and mobile phase). It is concluded that the best peak shape and loading can be obtained if the mobile phase pH is adjusted to the isoelectric point (PI) of the analyte.

Abstract # 10

MAKING A BULLET PROOF QC METHOD

STEVE ANDERSON, JOE TOKOS, AND LARRY FELICE

SurModics, Eden Prairie, MN

Methods for the QC laboratory are often needlessly cumbersome and not robust. They are usually a product of R&D chemists who write methods that are suitable to support early product development. Though suitable for that purpose, they are not revisited to create methods that are ready for routine product clearance prior to transfer to the QC lab. Product development support and routine product clearance are different activities and require different methods.

Minimal additional development and thorough validation can lead to much more robust methods, methods that are much simpler and more likely to pass system suitability. Implementation of single point calibration, internal standards, and disposable labware are just some examples of changes that can pay big dividends in cutting rework and increasing throughput for years to come.

Abstract # 11

THE EFFECT OF WATER QUALITY ON CHROMATOGRAPHIC PERFORMANCE

M TARUN, S MABIC

Research and Development - Bioscience Division, Millipore Corporation

Reversed phase HPLC is the most widely used HPLC technique, owing to its applicability to separate a wide range of compounds. When optimizing for HPLC parameters, the use of high purity solvents and reagents is extremely important. Water quality becomes all the more critical when it is realized that it is used not only as a mobile phase, but also in the preparation of samples, standards, and blanks. In routine HPLC runs, if the mobile phase solvent composition never becomes strong enough to elute organic contaminants that have been adsorbed or absorbed on the column, the contaminants could accumulate at the head of the column thereby significantly affecting chromatographic performance. The presence of trace organics in the water that is used for HPLC experiments thus ultimately affects the results of LC analyses, especially if the analytes of interest are present in very low levels.

This study presents the combination of technologies utilized to produce ultrapure water that is suitable for use in HPLC analyses. It focuses on UV photo-oxidation technology to effectively reduce organic molecules in water down to low ppb levels (measured as total organic carbon, or TOC). The benefit of using such high purity water will be illustrated by comparing how two sources of the weak mobile phase affects chromatographic performance (e.g., appearance of ghost peaks, peak tailing, loss of resolution) after repeated injection of a pharmaceutical mixture: a commercially available HPLC-grade bottled water that has no TOC specifications, and a freshly delivered ultrapure water with a TOC level of < 5 ppb.

Abstract 12

CHIRAL SELECTOR SCREENING AND REGENERATION OF NOVEL BRUSH AND POLYSACCHARIDE-TYPE ZIRCONIA CHIRAL STATIONARY PHASES

CLAYTON V. MCNEFF¹, BINGWEN YAN¹, KELLY JOHNSON¹, DANIEL NOWLAN¹,
SHENGXIANG JI², THOMAS R. HOYE².

¹ZirChrom Separations, Inc. 617 Pierce St., Anoka, MN 55303, ²University of Minnesota, 207 Pleasant Street SE, Minneapolis, MN 55455

The separation of enantiomeric compounds is a vital area of research in a world where there are an increasing number of chiral molecules being produced and distributed for biological and pharmaceutical applications. Many of the challenges faced in chiral method development stem from the inability to quickly and cost effectively analyze the performance of a set of chiral selectors for a given racemic set. Herein is described a different approach to chiral screening using a single column. By taking advantage of the unique Lewis acid-base surface chemistry of zirconia, suitably designed chiral stationary phases (CSPs) can be attached, removed, and then reproducibly reattached without unpacking the column. This allows the chromatographer to change to a different CSP or regenerate a column using only the HPLC instrument. Specifically, the CSPs investigated in this work are Pirkle type phases and 3,5-dimethylphenylcarbamate coated cellulosic phases. The ability to regenerate both types of phases, switch between phases, and example separations of compounds of interest will be discussed.

Abstract # 13

ASSESSING RESIDUAL METHAMPHETAMINE CONTAMINATION IN FORMER CLANDESTINE LABORATORIES USING LC-MS

MARTIN J. BEVAN (Minnesota Department of Health, Public Health Laboratory, St. Paul, MN 55164),
KATE GAYNOR (Minnesota Pollution Control Agency, St. Paul, MN 55155).

In the past decade, a dramatic increase in the abuse of the illegal stimulant drug methamphetamine (Meth) has occurred in Minnesota. Meth is often made by the conversion of ephedrines (frequently found in cold medicines) using common household chemicals. The conversion process or “cooking” can be readily accomplished via several well established methods and is often carried out in small clandestine labs (Clan Labs) which can create both environmental and public health hazards. In a partnership with the Minnesota Pollution Control Agency (MPCA), the Minnesota Department of Health’s (MDH) Public Health Laboratory Division (PHLD) investigated several former Meth Clan Labs to determine if Meth existed as a “persistent contaminate” after cooking and smoking had ceased.

To accurately sample the broad range of materials encountered in a Clan Lab, we employed several different sampling techniques including surface wipe sampling (SWS), total Meth extraction (TME), vacuum particulate collection (VPC), and active air monitoring (AAM). The collected samples were analyzed for methamphetamine by LC-MS. The results indicated widespread contamination of almost all surfaces throughout the Clan Lab. In addition, we found that methamphetamine readily penetrated or became embedded in carpets, fabrics, latex paint, and unsealed porous surfaces.

Abstract # 14

QUANTITATION OF AMINOGLYCOSIDES IN HUMAN LUNG TISSUE AND SERUM USING HPLC-MS/MS.

SUDHA MARIMANIKKUPPAM, CULLIN BACHMEIER AND GREGORY JANIS

Medtox, New Brighton, MN

Aminoglycoside antibiotics are a challenging group of compounds for quantitative chromatographic analysis due to their high polarity and susceptibility to detrimental ionization effects. Traditional extraction methodologies fail to adequately isolate these compounds from matrix components which result in suppression of their ionization in an ESI source. We have developed extraction and analysis strategies and techniques to quantitatively measure aminoglycoside antibiotics and other highly polar compounds in biological matrices. We will present our experiences with extracting and analyzing polar compounds by HPLC-MS/MS analysis including various protein precipitation methods¹, solid phase filtration, carbon-based solid phase extraction, normal phase HPLC-ESI-MS/MS and ion-paired² HPLC-ESI-MS/MS.

References:

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Abstract # 15

QUANTITATIVE ANALYSIS OF CORTICOSTEROID IN EQUINE JOINT FLUIDS

SEIJIN PARK (Univ. of Minn., Center for Mass Spectrometry and Proteomics, Minneapolis, MN, 55455),
MARY BOYCE & ERIN D. MALONE (Univ. of Minn., Veterinary Medical Center, St. Paul, MN 55108)

Navicular syndrome is a chronic, progressive disorder of horses, characterized by pathological changes developing in the navicular area. Approximately a third of all horses with chronic forelimb lameness have pain localized to the navicular area. Direct injection of corticosteroids onto the navicular bursa (NB) has recently been reported to have an 80% short term success rate. Even though NB injections with corticosteroid have been shown to be effective, NB injections are challenging to perform. Alternatively, indirect injections onto the distal interphalangeal joint (DIPJ) can be used for the treatment if the drug will move into the NB. Multiple studies have demonstrated that there is no direct communication between the DIPJ and the NB. However, a recent study has suggested diffusion of a corticosteroid (methylprednisolone acetate) across the distal tarsal joints.

The purpose of this study is to develop a sensitive analytical method that can detect a corticosteroid from the small amount of synovial fluid and to provide an evidence of the drug movement from DIPJ to NB. We spiked known amount of triamcinolone acetonide, therapeutically used corticosteroid, into normal equine synovial fluids. The synovial fluids were centrifuged and added with the internal standard (Triamcinolone-6-d₁ Acetonide-d₆). Compounds of interest were partitioned four times by liquid-phase extraction. Samples were first absorbed on the C₁₈ trap (5mm×300µm ID) and separated by 150mm×75µm ID C₁₈ column with linear gradient from 20% to 75% acetonitrile/0.1% formic acid at the flow of 0.4µl/min. Column effluent was introduced to a triple quadrupole instrument via an ESI source. The mass spectrometer was operated in MRM mode. Obtained data were processed with Analyst® software (Applied Biosystems). The nano-LC combined with mass spectrometer made it possible to analyze as little as 0.42µl-equivalent joint fluid and we are analyzing synovial fluids from DIPJ and NB with this method.

Abstract # 16

INVESTIGATING INCORPORATION, TRANSFORMATION AND SUBCELLULAR DISTRIBUTIONS OF N-L-LEUCYL-DOXORUBICIN WITHIN HUMAN UTERINE SARCOMA TUMOR CELLS USING MICELLAR ELECTROKINETIC CAPILLARY CHROMATOGRAPHY WITH LASER INDUCED-FLUORESCENCE DETECTION

YAOHUA WANG, EDGAR A. ARRIAGA

Department of Chemistry, University of Minnesota, Minneapolis, MN

The incorporation of N-L-leucyl-doxorubicin (LeuDOX), a prodrug of doxorubicin (DOX) into tumor cells, its transformation to DOX, and its subcellular distributions determine the cytotoxicity of this prodrug. We used micellar electrokinetic capillary chromatography with laser-induced fluorescence detection (MEKC-LIF) to investigate the incorporation, transformation and subcellular distributions of LeuDOX within human uterine sarcoma cell line, MES-SA, and its DOX resistant variant, MES-SA/Dx5 cell line.

The MEKC-LIF method using 4 mM CTAB and 50mM tricine (pH=8.5) as separation buffer could detect as low as 6.6×10^{-6} attomoles of DOX and 11×10^{-6} attomoles LeuDOX within 5 minutes.

The amounts of DOX and LeuDOX in the whole lysate were examined after treating both cell lines with 1) same amounts; 2) same IC values of DOX or LeuDOX for 12 hrs. The results showed that the total amount of the drug incorporated into cells was proportional to the amount of treatment while the percent of LeuDOX transformed to DOX is doubled in MES-SA/Dx5 cells than in MES-SA cells, which suggested that the transformation of LeuDOX rather than the incorporation played a key role in the cytotoxicity of the prodrug.

As for the subcellular distributions of LeuDOX in Dx5 cell line, the highest amount of DOX was found in nucleus enriched fraction where the highest ratio of DOX to LeuDOX was occurred, too. The highest amount of LeuDOX was found in both high density organelle fraction which includes most mitochondria and nucleus enriched fraction while the highest ratio of LeuDOX to DOX was found in cytosolic enriched fraction.

Abstract # 17

*** MASS SPECTROMETRY FOCUS SPEAKER *** DEVELOPMENT OF CATCH-AND-RELEASE (CAR) REAGENTS FOR QUANTITATIVE PROTEOMICS ANALYSIS

CARLOS GARTNER
Harvard Medical School

Relative quantification of expressed proteins in a biological sample is generally performed by one of two methods. When the sample can be cultured, stable isotope labeling with amino acids in cell culture (SILAC) has proven a powerful method. However, this strategy is not easily applicable to samples derived from tissue samples. In those cases, chemical labeling [e.g., isotope coded affinity tags (ICAT)] is usually the tool of choice. With respect to the ICAT strategy, a reagent allowing for the use of immobilized avidin rather than its monomeric counterpart would be preferential due to cost and simplicity of the procedure. We describe here the development of a new reductively cleavable reagent which facilitates the relative quantification of thousands of proteins from only tens of micrograms of starting protein. The ligand features a novel disulfide moiety that links biotin and a thiol-reactive entity. The disulfide is stable to reductive conditions employed during sample labeling, but is readily cleaved under mild conditions using tris-(2-carboxyethyl) phosphine (TCEP). This unique chemical property allows for the facile use of immobilized avidin in a manner equivalent to the use of conventional reversible-binding affinity resins. Target peptides are bound to avidin resin, washed rigorously, then cleaved directly from the resin, resulting in simplified sample handling procedures and reduced nonspecific interactions. The new reagent allows for parallel processing of samples for facile quantitative analysis by LC-MS/MS.

Abstract # 18

MASS SPECTROMETRIC-BASED METHODS FOR CLINICAL BIOMARKER DISCOVERY AND VALIDATION

LEEANN HIGGINS

University of Minnesota, Department of Biochemistry, Molecular Biology and Biophysics, 140 Gortner Laboratory, 1479 Gortner Avenue, St. Paul, Minnesota 55108, Phone: 612-625-2280, FAX: 612-625-5780,
Email: higgi022@umn.edu, <http://www.cbs.umn.edu/msp/>

Thousands of clinical laboratory tests are available for the diagnosis of human disease and detection of normal and abnormal physiological states, yet many disease states progress to advanced stages before symptoms appear due to the lack of adequate tests for the early detection of diseases. Cancer, neurological disorders, organ transplantation and sepsis are examples of diseases and conditions that proceed to stages for which there are few cures, due to late detection. Advances in mass spectrometry technologies in the last decade for the detection of proteins have accelerated the search for biomarkers of diseases in the quest for early detection. Advantages offered by mass spectrometric-based techniques for biomarker detection are analysis speed, sensitivity and mass specificity; disadvantages include dynamic range limitations for component detection and inter-individual variability in protein expression levels, which can confound method validation.

Current methods for biomarker investigation will be reviewed, which include screening of serum and other sample types with protein mass spectrometric patterns and measurement of relative protein expression using isotope labeling techniques. Examples of failures and successes towards biomarker discovery will be discussed. A targeted approach for biomarker validation using a combination of LC-MS and mass specificity will be introduced.

Abstract # 19

DIFFERENTIAL EXPRESSION OF SERA PROTEIN BIOMARKERS FOR OVARIAN CANCER IDENTIFIED BY DIGE, ITRAQ, AND MASS SPECTROMETRY

JOHN D. ANDERSEN (Univ of MN, Dept. Lab Med Path),

FEIFEI S. XUE (Univ of MN, Dept. Lab Med Path),

LORRAINE B. ANDERSON (Univ of MN, Dept. Biochem, Molec Biol, Biophy), LEEANN HIGGINS

(Univ of MN, Dept. Biochem, Molec Biol, Biophy),

BRUCE A. WITTHUHN (Univ of MN, Dept. Biochem, Molec Biol, Biophy),

TODD W. MARKOWSKI (Univ of MN, Advanced Genetics Analysis Center) and

AMY P.N. SKUBITZ (Univ of MN, Dept. Lab Med Path)

Ovarian cancer is the fifth leading cause of cancer death for women in the U.S., due in part to the lack of adequately sensitive and specific biomarkers for a diagnostic blood test. The discovery of novel biomarkers is hindered by the presence of a small number of highly abundant proteins that comprise ~95% of serum total protein.

In this study, we analyzed 60 serum samples from patients with ovarian carcinoma and 60 normal controls. We compared two different affinity columns for their ability to remove highly abundant proteins from serum samples: a multiple affinity removal system (MARS) column (Agilent) and a ProteomeLab IgY 12 affinity column (BeckmanCoulter). We then compared two mass spectrometry-based techniques for biomarker identification: Differential In Gel Electrophoresis (DIGE) and iTRAQ.

For DIGE analysis, low abundance protein from cancer or control patients was labeled with cy5 red fluorescent dye and cy3 green fluorescent dye, respectively. The samples were combined and resolved by 2D SDS-PAGE. Proteins were visualized with the Typhoon scanner and analyzed using the Decyder software package. In the case of the MARS column, ~1200 proteins were visualized, of which 11% were 2-fold or more upregulated in the cancer vs. control serum samples. Whereas in the case of the IgY 12 column, ~870 proteins were visualized, of which 9% were up-regulated 2-fold or more in the cancer vs. control serum samples. The same sample sets were subjected to iTRAQ labeling, whereby the proteins were trypsin digested into small peptides, then labeled with isobaric amine-specific mass tags. The peptides were separated by 2D HPLC and analyzed by tandem mass spectrometry (MS/MS). Nineteen proteins were found to be differentially expressed between the two sample sets, regardless of the affinity column used. Current studies are underway to determine the usefulness of the proteins as biomarkers for ovarian cancer.

Abstract # 20

**DETECTION OF SULFONAMIDES IN WASTEWATER VIA HIGH-PERFORMANCE
LIQUID CHROMATOGRAPHY AND SOLID-PHASE EXTRACTION**

RYAN THURBER AND JEANNE FRANZ

Winona State University, Chemistry Department, Winona, MN, 55987

Studies have shown that the majority of antibiotics administered to humans are not metabolized, and are introduced into the wastewater system largely unaltered. This brings to light the possibility that these antibiotics may be passing through the wastewater system and into the environment where countless types of bacteria are exposed to them and contributing to their increasing resistance. In this research, a method was developed to identify and quantify sulfonamides, a common and broad class of synthetic antibiotics. This method involved the use of solid-phase extraction to bring the concentration up from the ppt-ppm range, and high-performance liquid chromatography to prove the existence and quantity of individual sulfonamides. Once developed, this method was used to test different types of wastewater treatment plants to determine if they were effective at removing sulfonamides from wastewater. As predicted, sulfonamides were found in approximately 16% of water samples ranging from .11-12.03 $\mu\text{g/L}$ with no less than 70% removal in each case. Several sludge samples were also analyzed, and sulfonamides were found at 1.26 $\mu\text{g/g}$. This leads to the conclusion that sulfonamides are not commonly encountered in the wastewater treatment system, and when they are present, are removed effectively by the treatment process or captured in the sludge.

Abstract # 21

THE ROLE OF CHEMICAL SELECTIVITY IN CHROMATOGRAPHY

YU ZHANG, PETER W. CARR

Department of Chemistry, University of Minnesota, Minneapolis, MN

The ultimate goal of any separation is to achieve acceptable resolution (R_s) in the shortest time. As the most essential metric of separation power in chromatography, R_s can be expressed in terms of three parameters efficiency (N), selectivity (α) and retention (k'), where the most significant impact comes from selectivity. A small change in selectivity leads to a big change in resolution. However, there a great variety of columns for liquid chromatography (LC) are now available with dramatically different selectivities. Many attempts have been made to understand and measure the selectivity of reversed-phase columns. One new and powerful approach is "hydrophobic subtraction method" developed by Snyder, wherein the stationary phase is characterized by five selectivity parameters (H , S^* , A , B and C). Application of this model to retention data for various solutes and columns can provide new insight into the nature of different solute-column interactions and their relative importance in affecting sample retention and separation. It is particularly useful for the selection of column of either "equivalent" or "orthogonal (different)" selectivities.

This talk will focus on the chromatographic characterization of selectivity for two types of stationary phases recently developed in this lab based upon a hyper-crosslinked (HC) platform. By modifying silica surfaces using a multi-layer, two-dimensional, orthogonal reaction polymerization method, both the new HC phases thus synthesized are extremely stable under low pH (<2) and high temperature (100-150 °C) conditions. Their reversed phase selectivities have been extensively studied by the Snyder hydrophobic subtraction method and both show rather different selectivities compared to conventional bonded octadecylsilane (ODS, C18) phases. By derivatizing the surface aromatic groups with various functional groups, a family of stationary phases with a wide variety of chromatographic selectivities from one ultra-stable platform is being developed.

**RETENTION EFFECTS FOR CATIONIC ANALYTES ON PFP STATIONARY
PHASES IN VERY DILUTE ACID MOBILE PHASES**

MERLIN BICKING, ACCTA, Inc., St. Paul, MN 55125

RICHARD HENRY, Supelco, Division of Sigma-Aldrich, Bellefonte, PA 16823

While pentafluorophenyl (PFP) stationary phases have become popular recently, a fundamental understanding of the retention mechanism has proven to be elusive. These phases have the ability to produce both normal (water is strong) and reverse phase separations (water is weak), depending on the mobile phase composition. In this study, we will summarize our initial efforts to study the effect of acid and organic concentration on the retention of two cationic analytes - creatine and creatinine. Surprisingly, the results indicate that retention is controlled by both acid concentration at low (i.e., < 0.01%) levels as well as acetonitrile content in the range from 20 to 50%. The cations show normal phase behavior as a function of acetonitrile content, while a hydrophobic marker exhibits conventional reverse phase behavior under identical conditions. Increasing the acid concentration reduces retention of cations, suggesting the presence of a complementary ion exchange-type mechanism. These results highlight the unique aspects of this particular phase. If the retention mechanism and important separation variables could be more fully understood for PFP, its usefulness could increase dramatically.

Abstract 23

MOLECULARLY IMPRINTED POLYMER EXTRACTION: COMPOUND SPECIFIC TO CLASS SPECIFIC SOLID PHASE EXTRACTION

CARMEN T. SANTASANIA , CRAIG R. AURAND, OLGA SHIMELIS,
DANIEL SHOLLENBEGGER AND DAVID S. BELL

Supelco/Sigma-Aldrich, 595 N. Harrison Road, Bellefonte, PA 16823

In most analyses, the greatest challenge is sample clean up prior to analysis. Traditional approaches of sample clean-up have focused on liquid-liquid extraction or solid phase extraction. Many of these solid phase extraction (SPE) materials provide a general sample clean-up and lack specificity and selectivity for the analyte of interest. Molecularly imprinted polymers (MIPS) have recently been introduced as selective sorbents for specific compounds or specific classes of compounds.

The sample prep methods described in this talk will focus on the use of these MIPS. Selectivity is introduced during MIP synthesis in which a template molecule, designed to mimic the analyte, guides the formation of specific cavities or imprints that are sterically and chemically complementary to the target analyte. Unlike other SPE phases that do not have specific retention for one compound or class of compounds, the high selectivity of the MIP allows for cleaner samples with less interferences.

In this talk, SPE methods for the determination of chloramphenicol and beta-agonists will be described. This MIP technology can be used for determinations of specific compounds and also can be used for sample clean-up of classes of compounds with similar arrangements of interacting functional groups. Examples of this more general use of MIPS will also be described.

Abstract 24

HDMS – A POWERFUL, NEW, HIGH EFFECIENCY ION MOBILITY SEPARATION HYBRID MASS SPECTROMETRY SYSTEM

ROGER A. UPHAM

Waters Corp, St. Paul, MN

This presentation will describe a novel application of high efficiency ion mobility separation of molecules. This application involves IMS-MS data acquired using a novel Quadrupole/TWIMS/oa-ToF mass spectrometer, operated with a nano electrospray ion source. The TWIMS (Travelling Wave Ion Mobility

Spectrometer) is a stacked-ring ion guide, operated at elevated pressure, with opposite phases of an RF voltage applied to adjacent plates to provide radial ion confinement. A continual sequence of dc pulses is superimposed on the confining RF to provide "waves" which propel ions through the gas. The ions are separated based on their mobility through a high pressure of nitrogen gas. A description will provided of the use of the system for the analysis of intact proteins of different molecular masses and obtained IMS-MS of data for a number of possible conformers. There will also be a description of analysis of smaller proteins, including MS/MS followed by IMS separation of the protein fragment ions. In additional experiments we have looked at protein species highly contaminated with detergents and stabilisers, such as PEG, and have investigated the potential use of IMS as a means to allow rapid separation of proteins from the polymeric species, prior to analysis by mass spectrometry.

Abstract 25

PROTEIN-POLYMER INTERACTIONS BY HPLC

JASON YAEGER

SurModics, Eden Prairie, MN

HPLC can be an essential tool in the separation of proteins in complex matrixes. Often these matrixes not only need quantitative analysis of proteins and polymers, but also characterization of the interactions between them. By using a variety of separation techniques, and specific methods of detection, we can learn a lot about these interactions.

Abstract # 26

EVALUATION OF SENSITIVITY AND LINEARITY OF A NEWLY DEVELOPED HPLC DETECTOR, THE QUANT TECHNOLOGIES QT-500 CONDENSATION NUCLEATION LIGHT SCATTERING DETECTOR (CNLS D)

DEREK OBERREIT (Instrument Development Engineer, Quant Technologies Inc. Blaine, MN, 55449),
LAURENCIA KYARIGA (Applications Chemist, Fluid Measurements Inc. White Bear Lake, MN, 55110)

A new aerosol-based detector using a patented high-sensitivity water-based condensation particle counter (WCPC) as the sensor was developed and evaluated. Similar to other aerosol based detectors (ELSD, Charged Aerosol) column eluent is nebulized and dried. As analytes elute the column, the size of the dry particles will increase. Changes in the dry aerosol size are measured using a sensor. Unlike other aerosol-based HPLC detectors the CNLS D WCPC sensor does not introduce drift or background noise.

A variety of trace amounts of analytes were analyzed utilizing RP and NRP- HPLC techniques. Isocratic and gradient volatile mobile phases programs were used. Detection limits as low as 0.1 ng (on column mass) and Quantitation Limits as low as 0.5ng (on column mass) were observed. Linearity curves had R² values of 0.990 – 0.999.

The analyte volatility and thermal stability contribute to the size and the quality of the analyte signal. Detector evaporator temperature affects the detector response and background noise during the analyses. Non-volatile residue in the mobile phase affects the baseline noise level of the chromatogram. Most Mass Spec grade solvents have less residue and hence less baseline noise.

Poster – 1

AN INNOVATIVE APPROACH TO LOW MASS, ZERO DEAD VOLUME CONNECTION OF FUSED SILICA COLUMNS

ROBERT FREEMAN, MICHAEL GOSS, BRAD RIGHTNOUR, WILLIAM GROVE, MATT LININGER,
PAUL SILVIS, AND GARY STIDSEN

Restek Corporation

A common problem when joining fused silica columns together is obtaining a secure and leak tight seal. There are many different types of connectors available on the market today that allow the user to repair a broken column, or connect a transfer line or guard column to an analytical column.

Metal type connectors are often used and offer a secure connection, but it is often difficult to obtain a good, zero dead volume union. Press-Tight® connectors offer an inert connection, but these can disconnect when subjected high temperatures, pressures and turbulence in the GC oven. There is a new type of connector that offers both a secure connection at high temperatures and pressures with zero dead volume.

This new connection features a small union that will not disconnect after repeated heating and cooling cycles. This poster will illustrate the effectiveness and advantages of the new connector. We will show that the new connector gives the analyst a reliable seal, zero dead volume in the flow path, and has a lower thermal mass than standard metal type connectors.

Overall, GC/MS method appears to be more sensitive, however it requires derivatization, transfer to a volatile solvent, and SIM does not allow for the identification. The sensitivity of various HPLC/DAD/ESI-MS TOF and GC/MS methods as well as the application to flaxseed extracts will be presented.

Poster – 2

UNIQUE COLUMN ALTERNATIVES FOR THE DETERMINATION OF EXPLOSIVES AND PROPELLANT RESIDUES VIA HPLC-UV

ROBERT FREEMAN, JASON THOMAS, RICK LAKE, AND BECKY WITTRIG

Restek Corporation

The presence of explosive and propellant residues in the environment is a topic of common concern. These compounds are persistent in the environment, at ambient conditions, exhibiting little natural degradation. EPA 8330 is a test method for the determination of trace amounts of nitroaromatics, nitramines, and nitrate esters by means of liquid chromatography. The method uses reversed phase HPLC and dual wavelength UV detection (210 & 254nm). The method was recently revised in October 2007 and expanded to include three additional analytes: nitroglycerine (NG), pentaerythritol tetranitrate, (PETN), and 3,5-dinitroaniline (3,5-DNA). EPA 8330B now covers seventeen analytes that are commonly found in explosive and propellant residues.

We recently assessed various stationary phases for retention and selectivity of the new analytes in the revised method. Separations on all columns were accomplished with a simple, isocratic water:methanol mobile phase. Since the test method stipulates both primary and confirmation analyses, numerous columns were evaluated for selectivity differences such that an effective primary and confirmation column pair could be identified. This poster will illustrate the effectiveness and advantages of these stationary phases. We will show columns combinations that perform well in a primary-confirmation pair as well as illustrate several unique alternatives.

Poster – 3

A NEW POLARITY SCALE FOR GC PHASES

PAUL WYNNE, DAN DIFEO, PETER DAWES

SGE Inc, 2007 Kramer Lane, Austin TX 78758, USA and SGE Analytical Science,
7 Argent Place, Ringwood VIC 3134 AUSTRALIA

Traditionally, the polarity of GC phases has been represented on a qualitative polarity scale in one dimension. While different presentations have been adopted by different manufacturers, the information content has remained essentially the same in each case.

We present here a three-dimensional GC polarity scale that considers the selectivity of the GC phase towards any analyte as being dependent on at least three types of interactions: van der Waals forces, hydrogen bonding (or proton donor and acceptor type bonding) and bonding between π - and n-type orbitals. Because the scale is in three dimensional space, it can easily show that a phase may exhibit elements of all bonding types or a dominance of one. It also illustrates what the phase is not. The one dimensional polarity scale does not show different types of interactions and also does not show that if one bonding type becomes dominant then such dominance must logically be at the expense of at least one of the other bonding types. This information is readily conveyed in a three dimensional format.

The new polarity scale is able to convey a more detailed description of GC phase polarity with great simplicity. By using a qualitative graphic approach, three-dimensions of information can be provided to the chemist without the added complexity of descriptive terminology. The additional information content of the new polarity scale also allows the chromatographer to more reliably select a GC phase for their specific analyte and therefore aid in column selection across applications.

Poster – 4

ON-LINE AND OFF-LINE APPLICATION OF MICRO-SPE

DAN DIFEIO, PETER DAWES, ERN DAWES, PAUL WYNNE

SGE Inc, 2007 Kramer Lane, Austin TX 78758, USA and SGE Analytical Science, 7 Argent Place,
Ringwood VIC 3134 AUSTRALIA

Solid-phase extraction (SPE) has revolutionized sample preparation methodology for diverse sample types. In many cases, variations on the technique offer enhanced recovery, greater opportunity for speciation and a reduction in solvent and sample consumption. Unlike solvent extraction, SPE is generally easy to automate for off-line use. The simple adaptation of SPE for on-line use has not been as successful as its uptake for off-line applications.

Recognizing that the primary purpose of most SPE methods is solvent exchange (e.g. the extraction of an aqueous sample for GC inlet) or some form of matrix clean-up (e.g. desalting or removal of endogenous materials), the further development of SPE for on-line use becomes a directed exercise. Miniaturization of SPE into the sample injection system allows a much smaller portion of the sample to be extracted and for the whole of the extract to be injected onto the chromatographic column for separation. In many cases, such an approach allows the same level of sample concentration to be achieved as is possible with off-line conventional SPE. Providing the miniaturization does not approach the same scale as the pseudo-plates of the sorbent, micro-SPE may be readily adapted from established SPE methods.

Using simple applications, we demonstrate the usefulness of micro-SPE for different combinations of sample type and sorbent chemistry prior to analysis by GC or LC techniques.

The micro-SPE apparatus is suitable for on-line use in GC, HPLC and other chromatographic techniques and for sample preparation prior to other analytical techniques including immunoassay and off-line non-destructive spectroscopic analysis by NMR, IR and other methods.

Poster – 5

NEW APPROACHES TO THE GC INJECTION PORT

DAN DIFE0, PETER DAWES, DAVID MELVILLE, PAUL WYNNE

SGE Inc, 2007 Kramer Lane, Austin TX 78758, USA, and SGE Analytical Science, 7 Argent Place,
Ringwood VIC 3134 AUSTRALIA

The application of gas chromatographic methods is limited by the efficiency of vaporization and sample transfer from the injection port to the head of the column. The most common injection port design is based on flash vaporization and split or splitless transfer to the column. The technique is less effective for thermally labile analytes and, because the high temperatures of operation provide sufficient energy to exceed the activation energy, less effective for analytes that are prone to catalytic decomposition on the liner body or packing. Considerable effort has been directed to the design of injection port liners that overcome the undesirable aspects of flash vaporization and splitless residence times.

While on-column injection overcomes some of the discrimination and other difficulties associated with flash vaporization, it is not useful for split sample inlet and has limited appeal for use in routine applications.

We describe here alternative approaches to GC sample introduction based on the deactivation of the injection port liner and the principles under which the liner functions. The advantages of alternative injection techniques are described for polar and labile analytes.

Poster – 6

LC/MS SOLVENT BLENDS STABILITY

A. KEMPERMAN, K. SNOBLE, R. DUBEY, S. LORENZ, N. FOX,
J. PRZYBYTEK, J. WELCH

Honeywell, Burdick and Jackson, Muskegon, MI

LC/MS Spectroscopy is used for the determination of protein structural information. One important factor required to obtain quality structural information via LC/MS is the performance of the LC solvent blend systems used prior to the MS detector. The effect of chemical noise on LC/MS sample detection performance was described elsewhere. This poster describes a LC/MS study of several common organic acid modified solvent blend formulations and a GC/MS and titrimetric study comparing their relative stabilities. The results show a significant variation in the stability of several commercially available solvent blends.

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Poster – 7

EVALUATION OF THE ANALYTICAL ADVANTAGES OF NEW STATIC AND DYNAMIC HEADSPACE TECHNOLOGY

JEFF SHERIFF

EST Analytical, Fairfield, OH

Static Headspace technology is a common technique used in the analysis of volatile organic compounds in Analytical and Research Laboratories. Although Headspace analysis is considered to be an easy to perform technique which produces reliable analytical results, there are still several challenges to this technology. Some of the biggest challenges include sensitivity, speed, temperature concerns and the mode of sample transfer to the GC.

This paper will compare and evaluate new technologies incorporated into a single multifunctional Static and Dynamic Headspace Sampler to overcome these challenges. Traditionally Static Headspace analyzers inject a single aliquot of the headspace vapor onto the GC column which is either controlled by time or by filling a fixed volume loop but not both.

This new Headspace technology gives the user the flexibility to perform not only both of the routine static headspace time based and volume based injections but also dynamic sweeping and pre-concentration injections for improve sensitivity up to 100X within the same sequence. This expanded flexibility allows existing validated methods to be easily transferred for regulatory work and also produces analytical advantages for research work. Several examples will be presented utilizing these various injection techniques for various applications.

Poster – 8

**EVALUATION OF UV AND LIF DETECTION
FOR CE-SDS PURITY APPLICATIONS
FOR PRODUCT RELEASE AND
STABILITY TESTING OF RECOMBINANT MONOCLONAL ANTIBODIES**

CHAD WIENEKE¹, ANTOINETTE BENNAARS¹, HOLLY YIP²,
XINFENG ZHANG², BRIANSCHMIDT²

¹PDL Biopharma, QC Analytical Technologies and ²PDL Biopharma, Analytical Sciences

CE-SDS is recognized as an important analytical tool in the biopharmaceutical industry to support analytical product characterization, process development, product release and stability testing. CE-SDS may be applied for automated quantitative analysis of biopharmaceutical products and provides several advantages in comparison to conventional SDS-PAGE analysis including:

Automation

High-speed separation

Enhanced resolution

Direct, on-line quantification using either UV or laser induced
fluorescence (LIF) detection

PDL Biopharma has evaluated CE-SDS methods using both UV and LIF detection and each approach demonstrates the ability to provide data with acceptable sensitivity and precision. While the sensitivity of the LIF method is intriguing, there are additional factors to consider when using LIF detection to ensure that consistent responses are acquired across laboratories over time. These factors include optimization of the fluorophore derivatization procedure and LIF detector calibration.

Poster – 9

**COMPARISON AND ASSESSMENT OF WATER PURIFICATION TECHNOLOGIES
USING ION CHROMATOGRAPHY**

STÉPHANE MABIC, ELODIE CASTILLO, AND ICHIRO KANO

Research and Development, Lab Water Division, Millipore, St Quentin-Yvelines, France

Poster – 10

UV TECHNOLOGIES IN LABORATORY WATER PURIFICATION SYSTEMS

ICHIRO KANO, DANIEL DARBOURET AND STÉPHANE MABIC

Research And Development, Lab Water Division, Millipore, St Quentin-Yvelines, France

Poster – 11

**ACHIEVING SUB-2 μ m PERFORMANCE AT MODERATE PRESSURES USING FUSED-CORE™ PARTICLE TECHNOLOGY
ACHIEVING SUB-2 μ m PERFORMANCE AT MODERATE PRESSURES USING FUSED-CORE™ PARTICLE TECHNOLOGY**

RICHARD A. HENRY, DAVID S. BELL, WAYNE K. WAY, RUSSEL GANT,
PAUL ROSS, HILLEL K. BRANDES, WILLIAM CAMPBELL AND
CARMEN T. SANTASANIA

Supelco/Sigma-Aldrich, 595 N. Harrison Road, Bellefonte, PA 16823

Recent advances in high-performance liquid chromatography (HPLC) columns have focused on various approaches to increase the speed of analysis. Monolithic columns, for example, were introduced for their potential for use at high mobile phase velocities due to decreased mass transfer effects over conventional fully porous particles. More recently, ultra-high performance chromatography on sub 2 μ m particles have provided increased speed and efficiencies over conventional particles at the expense of high backpressures and, at times, column ruggedness. Fused Core™ Technology is the next significant breakthrough in column technology aimed at reducing analysis time through increased efficiencies.

The Fused Core technology breakthrough is based on a solid 1.7 μ m core particle fused to a 0.5 μ m outer shell of porous silica. The reduced intraparticle flow path results in significantly less diffusion within the confines of the analytical column. The result is similar column efficiencies to columns packed with sub 2 μ m particles with about half the backpressure. Consequently, sub 2 μ m-like efficiencies can be realized using conventional instrumentation. In addition to the kinetic advantages of the Fused Core particle, the larger overall particle size and narrow particle size distribution lead to improved packing mere configurations. These improvements then lead to greatly improved ruggedness of the columns.

In this report we introduce the Fused-Core Technology and discuss its advantages through fundamental kinetic discussions and chromatographic examples. Improved speed, sensitivity, overall separation power and ruggedness will be highlighted.

Poster – 12

EVALUATION OF 4-PROPYLAMINOMETHYL-BENZOIC ACID MODIFIED SILICA (4-PAMBAS) AS A CATION/ANION EXCHANGE HPLC STATIONARY PHASE.

ASANKA WIJEKON, MAHINDA GANGODA, ROGER B.GREGORY

Kent State University

The preparation and characterization of 4-propylaminomethyl-benzoic acid modified silica (4-PAMBAS) for use as a cation/anion exchange stationary phase in high performance liquid chromatography (HPLC) is described. 4-PAMBAS was prepared by reacting aminopropyl modified silica with 4-carboxybenzaldehyde (4-CBA) and reducing the resulting Schiff base with sodium cyanoborohydride. Elemental analysis indicated a coupling efficiency of 91 %. The average surface density of bonded groups was $2.9 \mu\text{mol/m}^2$. The structure of 4-propylaminomethyl-benzoic acid modified silica was confirmed by $^{13}\text{C} \{^1\text{H}\}$ cross polarization magic angle spinning NMR. The chromatographic properties of the 4-PAMBAS stationary phase were explored with mixtures of basic organic compounds, basic amino acids, substituted aromatic sulfonates and a series of n-alkanoic acids. At pH 3.0, basic compounds are unresolved and co-elute near the void volume, while aromatic sulfonates are retained and are well-resolved. When the pH of the mobile phase is increased to 7.0, the aromatic sulfonates are now unresolved and elute at the void volume, while the basic compounds are retained and are well-resolved. The 4-PAMBAS stationary phase therefore acts as an anion exchanger at pH 3.0 and a cation exchanger at pH 7.0. Hydrophobic interactions also contribute to the retention of analytes as indicated by the separation of a homologous series of n-alkanoic acids at pH 7.

Poster – 13

**HIGH SPEED CHROMATOGRAPHIC ANALYSIS USING SUB-2 MICRON HPLC
PACKINGS**

MARK JACYNO, RENO NGUYEN, SCOTT ANDERSON

Grace Discovery Sciences, Deerfield, IL

Today's laboratories are under greater demand to accomplish more in less time. Large pools of lead candidates in drug discovery labs and shorter project timelines have required greater sample throughput in less time. To help meet this demand, major product advances have been made to increase HPLC throughput and speed up analysis times. Reducing particle size and using shorter column lengths shorten analysis times without sacrificing resolution. Using sub-2 micron HPLC packings increases performance allowing even shorter columns and faster analysis times. Combined with the right column format the advantages of sub-2 micron HPLC packings can be realized on the latest high-end systems as well as conventional HPLC systems

Poster – 14

**EFFECT OF INADVERTENT THERMAL DEGASSING OF DISSOLUTION MEDIA:
FINDINGS FROM METHOD TRANSFER**

MATT DERUYTER, NANCY HIRSCH, TATYANA MAKSIMOVA AND
PEJU ODUNUSI

CIMA LABS INC., 7325 Aspen Lane, Brooklyn Park, MN 55428

Variable data between analysts were observed during the dissolution method transfer of a coated drug (processing intermediate). Effects of several parameters on the dissolution rate of the coated drug were investigated. The parameters investigated included particle size of the material, types of weighing vessels used, different analysts and the addition of the dissolution media to cool or warm baths. We present here data generated during the transfer and investigations.

The data indicated that the variability was attributed to the thermal degassing effect of the dissolution media. The temperature of the dissolution bath when the media was added to the vessels had a significant effect on the rate of release. Dissolution media added to a cool bath mimicked degassing and reduced the rate of release of the active ingredient. However, dissolution media added to a warm bath increased the rate of release. Observable gas bubbles in the media, on the sides and bottom of the vessels likely prevented the mounding of the coated drug at the bottom of the vessels, hence the increase in the rate of dissolution.

Degassing of the media by sparging with helium prior to running the dissolution test was found to provide acceptable variability. The method transfer was repeated with degassed media and all results met the criteria set, hence the method transfer was successful.

Poster – 15

TROUBLESHOOTING GC HEADSPACE AUTOSAMPLER CONNECTIONS

GREGORY SCHMIDT AND BRADFORD ROBERTS

Boston Scientific, Minneapolis, MN

Static headspace technology is a common technique used in the analysis of volatile organic compounds in Analytical and Research Laboratories. Although Headspace analysis is considered to be an easy to perform technique which produces reliable analytical results, there are still challenges to this technology. One challenge includes temperature concerns in the sample transfer to the GC.

The flow path for volatiles from the Agilent 7693 Headspace unit includes a heated transfer line connected to an unheated ZDV union, and then to the inlet of the GC. In this work, temperatures at the ZDV union were raised to >100°C to improve transport to the GC inlet. The instrumentation used: Agilent 7693 Headspace unit dedicated for use with a GC 6890 for analysis of volatile compounds or semi-volatiles such as dimethylformamide.

This paper will demonstrate that heating the entire flow path, from the head space unit to the injector, improves both peak shape and recovery levels for dimethylformamide, and also prevents carryover. Further, the choice of sample solvent also influenced chromatographic performance. Performance was compared for methanol and dichloromethane.

UPLC MS/MS Measurement of Cysteine During Fermentation

Jamie Koehler, Joseph J. Dalluge, and Keri Lyn Ross
Biotechnology Development Center and Global Food Technology Group,
Cargill Incorporated

A method has been developed for the rapid determination of total cysteine in fermentation media. The method is based upon ultra performance liquid chromatography/electrospray tandem mass spectrometry (UPLC/ESI-MS/MS). Standard cysteine mixtures, fermentation media, and enzyme assays were analyzed by UPLC/MS/MS with multiple reaction monitoring in positive ionization mode. Samples are reduced with dithiothreitol to convert all present cystine to cysteine as well as to prevent cysteine from oxidation. The proposed UPLC separation protocol allows for the near baseline separation of cysteine from cystine in under 15 minutes for monitoring reduction efficiency. Precisions for cysteine measurements range from 5% to 10% RSD, calibration curves are linear from 0.1 ppm to 150 ppm, and limits of detection in the low ppb range are achievable.

Poster – 17

**MEASUREMENT OF AMMONIUM BY ION CHROMATOGRAPHY
IN HIGH SODIUM CONCENTRATION**

SHARON KLEIN AND JIHONG COLE-DAI

Department of Chemistry and Biochemistry, South Dakota State University, Brookings, SD 57007

Chemical analysis of Antarctic ice cores provides valuable information on the history and dynamics of the atmosphere environment. The ice core samples contain trace amounts of ammonium, sodium, and other ionic chemical species. High concentrations of sodium may interfere with the accurate and precise measurement of ammonium, due to insufficient resolution of their chromatographic peaks. The extent of the interference is investigated in this project by quantitative determination of the ammonium concentration in solutions of various ammonium and sodium concentrations. A criterion of 20% relative error of measured ammonium concentration is used to determine the threshold of sodium concentration that results from unacceptable resolution of ammonium and sodium peaks.

THE HISTORY OF THE MINNESOTA CHROMATOGRAPHY FORUM

The MCF was born in the minds of a few Minnesota area chromatographers in early 1978. It took final shape on St. Patrick's Day in 1978 over a pitcher of green beer. In 1979, a constitution was written and passed by the members, declaring the objectives of the MCF to be "...to maintain and promote for education, discussion and exchange of information with respect to all fields of chromatography."

Toward these objectives, four technical meetings were held, including a notable address by Dr. Les Etre and culminating in the 1979 Spring Symposium and Exhibition. Chromatographers from the Dakotas, Iowa and Wisconsin joined those from Minnesota to present, discuss, agree and disagree about varied aspects of chromatography. The Spring Symposium was highlighted by the keynote address presented by Professor Barry Karger of Northeastern University. Twenty-five years later, the objectives of the MCF remain the same, but the group has grown considerably to a current membership of about 600 chromatographers.

The MCF is an independent, non-profit organization incorporated in the State of Minnesota. The constitutionally mandated officers and Board of Directors, elected by the membership and supplemented by an active committee structure, chart the course of the MCF. The names of the current Governing Board and Committee members are listed in Subsequent sections. Past Presidents of the MCF include:

1978-79	Peter Carr	1993-94	Julie Carver
1979-80	Larry Bowers	1994-95	Sandy McDonald
1980-81	Mark Brenner	1995-96	Dave Ehresman
1981-82	Jon DeVries	1996-97	Lars Pekay
1982-83	John Mahrevka	1997-98	David Whitman
1983-84	Steve Anderson	1998-99	Luke Charpentier
1984-85	Susan Price	1999-2000	Ravi Ravichandran
1985-86	Fred Gustafson	2000-01	Rebecca Wittrig
1986-87	Steve Pierson	2001-02	Ward Swanson
1987-88	Kay Olson	2002-03	Brian Leafblad
1988-89	Craig Markell	2003-04	Brenda Tjelta
1989-90	Jim Broge	2004-05	Liesa Shanahan
1990-91	Peter Johnson	2005-06	Franz Rolvaag
1991-92	Anne Ochs	2006-07	Chris Marquardt
1992-93	Gary Reineccius		

Since the most important function of the MCF is to provide area chromatographers with an opportunity to expand their knowledge of separation science, program planning stresses quality and variety of invited speakers. Each year, four evening meetings are held, with invited speakers ranging from local experts to leading international chromatographers. Past speakers have included Robert Mooney, Dr. Eileen F. Bostwick, Dr. Brian A Bidlingmeyer, Dr. Mark Schure, Dr. Glenn Ouchi, and Dr. Mark Konings.

A three-day Spring Symposium and Exhibition has been held in the Twin Cities each year. The Spring Symposium includes short courses, posters and papers by regional chromatographers and invited speakers, exhibits and talks by vendors and a keynote address.

The one and one-half day short courses vary in topic and level. In the past, this has included basic courses such as Capillary GC, taught by Walter Jennings, that are directed at beginners in the technique, while courses such as Advanced HPLC, taught by Lloyd Snyder, are aimed at more experienced chromatographers. Course subjects are selected after reviewing an annual survey of the members for course preferences.

The papers and posters, usually about 35 in number, give area chromatographers a chance to present their work. Each talk lasts about fifteen minutes with five minutes for questions and are grouped into several sessions with respect to subject. Each year, the keynote address is given by a recognized leader in their field of expertise. Recent speakers include: Thomas Chester, *Recent Changes, Current, Advantages, and New Directions for Supercritical Fluid Chromatography* (1998), Daniel Armstrong, *Aggregation Effects on Chiral Recognition* (1999), Dr. Art Mosely, *Nano-Scale Capillary LC-MS-MS for Proteomic Characterization* (2000), Professor Csaba Horvath, *Capillary Electrochromatography of Peptides and Proteins* (2001), and Professor Edward S. Yeung, *High Throughput Chemical and Biochemical Measurements* (2002).

A highlight of the Spring Symposium is the presentation of the Palmer Award, Named for L.S. Palmer (1887-1944), former professor at the University of Minnesota and author of an early chromatographic publication on carotenoids and related pigments. The Palmer Award recognizes contributions to the science of chromatography and to the Minnesota Chromatography Forum. Palmer Award recipients include;

1980	Les Ettore	1992	Gary Reineccius
1981	Larry Bell	1993	Dennis Johnson
1982	Don Hagen	1994	Ed Yeung
1983	Walter Jennings	1995	John Freeburg
1984	Peter Carr	1996	Jim Broge
1985	Lloyd Snyder	1997	Wils Bergstrom
	Larry Bowers	1998	Pat Sackett
1986	Susan Price	1999	Steve Pierson
	Mark Brenner	2000	Peter Johnson
1987	James Fritz	2001	David Ehresman
1988	Jon DeVries	2002	John Dolan
1989	Shoukry Khalil	2003	Daron Decker
1990	Kay Olson	2006	Ronald Majors
1991	Craig Markell		

Additional activities of the MCF include an annual donation to the University of Minnesota library for the purchase of chromatography publications and the MCF Undergraduate Research Award --a financial award presented to an undergraduate involved in chromatography research.

In 1987, the MCF hosted the ASTM Committee E-19 on Chromatography Meeting, in Minneapolis. The MCF also hosted the 25th Annual International Symposium, *Advances in Chromatography*, in August 1988. In 1994 the MCF hosted the International Symposium on Column Liquid Chromatography - HPLC '94.

As in the past, the MCF will continue to stress continuing education and information exchange among area chromatographers as a means of fulfilling a strong commitment to the advancement of chromatography.

2006-2007 MCF GOVERNING BOARD AND COMMITTEE CHAIRS

President	Chris Marquardt Cargill 12900 Whitewater Dr. MS 109 Minnetonka, MN 55343 952-984-0854 (o) chris_marquardt@cargill.com	3rd Year Director	Deanna Lind Boston Scientific 3 SciMed Place MS C140 Maple Grove, MN 55311 763-494-1594 (o) deanna.lind@bsci.com
Past President	Franz Rolvaag Lifecore 3515 Lyman Blvd. Chaska, MN 55318 763-514-8160 (o) franz.rolvaag@lifecore.com	2nd Year Director	Steve Anderson Surmodics. 9924 W. 74th. St. Eden Prairie, MN 55344 952-947-3523 sanderson@surmodics.com
President-Elect	Joshua Letze Boston Scientific Two Scimed Place Maple Grove, MN 55369 763-494-1648	1st Year Director	Janiece Hope Cargill 2301 Crosby Road Wayzata, MN 55391 (952) 742-3882 Janiece_Hope@cargill.com
Secretary	Crystal Weech Cima 7325 Aspen Lane Brooklyn Park, MN 554328 763-488-4847 crystal.weech@cimalabs.com	Newsletter	Glen Tipton 3M Pharmaceuticals Bldg. 260-4N-12 St. Paul, MN 55144 651-575-1149 (o) 651-733-0174 (f) grtipton@mmm.com
Secretary- Elect	Becky Bilek Braun 110001 Hampshire Ave. S. Minneapolis, MN 55438 952-995-2640 rbilek@braunintertec.com	Symposium Committee	Leeann Higgins Univ. of MN Dept. of Biochemistry 1479 Gortner Avenue St. Paul, MN 55108 612-625-2280x55 (0) 612-625-5780 (f) higgi022@umn.edu
Treasurer	Dan Howman Pace Analytical 1700 Elm Street SE., Suite 200 Minneapolis, MN 55414 daniel.howman@pacelabs.com	Education Committee	Jamie Koehler Cargill PO Box 5702 Minneapolis, MN 55440 952-742-3018 jamie_koehler@cargill.com
Webmaster	Ken Brown 3M Drug Delivery Systems Division 260-4N-12 St Paul, MN 55144-1000 651-737-2190 (o) 651-733-0174 (f) catchall@minnchrom.org	Meeting Support	Janice Jopke CCS Events 6611 Countryside Dr Eden Prairie, MN 55346 952/934-5082 (o) 612/934-6741 (f) csevents@comcast.net

COMMITTEES

PALMER AWARD COMMITTEE

Franz Rolvaag, Chair

The Palmer Award is named in honor Leroy Sheldon Palmer (1887-1944), former Professor and Head of the Division of Agricultural Biochemistry at the University of Minnesota, and author of a major chromatographic work on carotenoids and related pigments. The purpose of the award is to recognize and encourage the art and science of chromatography. The function of this committee is to select an outstanding chromatographer from among nominees submitted by the chromatographic community. The award is presented annually at the Spring Symposium.

NEWSLETTER

Glen Tipton - Chair

Statement of Purpose: The newsletter shall function to bring together the interests of the MCF membership by publishing articles which promote the objectives of the MCF through the exchange of information related to all areas of chromatography.

The newsletter promotes the goals of the MCF by publishing monthly issues from September through June to publicize meetings, introduce people and offer an opportunity for the exchange of technical information. The responsibility for the content of the newsletter with the Newsletter Committee under the direction of the MCF Board.

EDUCATION COMMITTEE

Jamie Koehler, Chair

Committee Members: Gibbes Bailie, Bill Cameron, Stephanie Drier, John Freeburg, Ananda Henly, Scott Henrich, Jamie Koehler, Laurencia Kyariga, Jeanette Morrison, Tammika.Shedd, DeWayne Townsend

This past year the MCF has been proud to offer *five* courses since the 2005 Spring Symposium. These offerings included the following:

- The annual 3-day course in “Beginning HPLC”, co-taught by Dr. David Johnson (3M) and Dr. Larry Felice (Surmodics) and supported in a “hands on” laboratory session by our vendors (Agilent, Shimadzu, Waters), was offered in October. This class will be offered again this fall, October 24-26, 2007.
- In November we offered two courses taught by Dr. John Dolan of LC Resources, “Advanced HPLC Method Development” and “LC/MS.” We will start offering this course every other year, look for it in the fall of 2007.
- The annual 3-day course in “Beginning Gas Chromatography”, taught in conjunction with Dr. Gary Reineccius (Univ. of MN) and John Freeburg (Midwest Scientific) at the University of Minnesota, was offered in January. This class will be offered again this winter, January 9-11, 2007.

- This winter we also held the MCF Design of Experiments course, instructed by Lars Pekay of General Mills. We hope to offer this class again in the near future.

Looking ahead . . .

We are excited to announce that this fall we will be offering a new course in the mix, "Introduction to Proteomics", the course will be instructed by Dr. David Carr of Bioanalytical Technologies, November 1-2, 2007.

Other News . . .

For the past five years I have chaired the education committee. After this symposium, I will be stepping down as chair, with a baby on the way this summer, I thought this would be a great time to let someone else lead the committee. Please welcome Jamie Koehler (Cargill) as the new education chair. Jamie has been co-chairing the committee this past year in preparation for becoming chair.

WE ALSO WANT TO EXTEND SPECIFIC THANKS TO THE VENDORS WHO PARTICIPATED IN THE BEGINNING HPLC COURSE AND/OR THE SYMPOSIUM GC TROUBLESHOOTING COURSE. THEY INCLUDE: AGILENT, LAKEVIEW ASSOCIATES, SHIMADZU, THERMO, AND WATERS. GASES FOR THE SYMPOSIUM GC LAB WERE DONATED BY TOLL. THANK YOU! REMEMBER TO SUPPORT THEM; THEY ARE DIRECTLY RESPONSIBLE FOR KEEPING YOUR COURSE COSTS DOWN!

Please be sure to include specific class topic requests in this year's survey.

Upcoming classes are determined directly from your input.

Thanks for all your support!

UNDERGRADUATE AWARD COMMITTEE

Paul Jackson - Chair

Committee Members:

Committee Members: John Freeburg, Tom Flock-Johnson, Richard Rossiter, DeWayne Townsend

The MCF Undergraduate Research Award is designed to encourage undergraduate students to conduct scientific research. Students submit proposals for short projects in which chromatography or some separation technique plays an important role. A panel of judges reviews the proposals and the student submitting the best proposal is awarded the sum of \$4000. The 2006 MCF Undergraduate Research Award was presented to Mr. Ryan Thurber of Winona State University for his proposal related to detection of sulfonamides in wastewater via HPLC and solid phase extraction. His sponsor was Professor Jeanne Franz, Ph.D., Department of Chemistry. Mr. Thurber will be presenting his results at this year's spring symposium on Thursday, May 17th.

Each year the MCF UGA committee looks for new ways to promote this award at colleges around the five-state area. Members interested in participating in this committee as well as suggestions for reaching prospective students more effectively are greatly appreciated.



Undergraduate Research Award Recipients

<u>Year</u>	<u>Name</u>	<u>Institution</u>	<u>Advisor</u>	<u>Topic</u>
2006	Ryan Thurber	Winona State University	Dr. Jeanne Franz	Detection of Sulfonamides in Wastewater via HPLC and Solid Phase Extraction
2005	Christian Herrild	Marquette University	Dr. Chieu Tran	Chiral Ionic Liquids as Stationary Phases in Gas Chromatography
2004	Claire Long	University of South Dakota – Vermillion	Dr. Miles Koppang	LC-EC Analysis of Amino Acids on Diamond Electrodes
2003	Jeremy King	University of Wisconsin – River Falls	Dr. Daniel Marchand	Development of a HPLC-ECD Method for Flavonoids in Cord Blood
2002	Molly Warnke	Hamline University	Dr. Eugene Smith	Arson Analysis by Headspace Enrichment and GC Using Simplex Optimization
2001	Christopher Field	Drake University	Dr. Mark Vitha	A Linear Solvation Energy Relationship Study of Amphiphilic Block Copolymers as Separation Enhancers in Capillary Electrophoresis
2000	Michael J. Kammerer	University of Iowa	Dr. Sonya Franklin	Myelin Basic Protein – Extraction and Purification
1999	Kevin Peterson	St. John's University	Dr. Kate Graham	GC-MS Analysis of Biologically Active Diterpenoids from Goldenrod in Plant-Insect Interactions
1998	Lee Stanek	Winona State University	Dr. Thomas Nalli	GPC Analysis of Polycyclohexane Oxide Produced in Iodonium/Methoxyphosphine Co-initiated Visible Photopolymerizations
1997	Jonathan Schroden	University of Minnesota – Duluth	Dr. Donald Poe	Supercritical Fluid Chromatography with PEEK Columns
1996	Joseph T. Aronson	University of Wisconsin – Eau Claire	Dr. Feimeng Zhou	Speciation and Trace Analysis of Heavy Metals with Flow Injection Fast Scan Voltammetry
1995	William Checkal	The College of St. Benedict / St. John's University	Dr. Kate Graham	The Use of Bio-Assay Guided Fractionation in the Isolation and Characterization of Novel Antifungal Drugs from Fungal Sources
1994	Eric Codner	University of Minnesota – St. Paul	Dr. Margaret Davis	Application of Field Flow Fractionation to Pollen Purification
1993	Jennifer J. Bailey	Carleton College	Prof. James Finholt	Development of an Efficient Method to find the Optimum Conditions for an Ion-exchange Separation
1992	Mark A. Aubart	University of Minnesota-Minneapolis	Dr. Louis Pignolet	Homogeneous Catalysis of H ₂ – D ₂ Equilibration by Mixed Transition Metal-gold Cluster Compounds

1991	Joanne Kramer	College of St. Catherine	Dr. Kathleen Tweeten	Development and Application of a HPLC Method for C50 Carotenoids from Halophylic Archaeobacteria
1990	Brad Bacles	University of Minnesota - Minneapolis		Affinity of Polyene Sterol Complexes
1991	Joanne Kramer	College of St. Catherine	Dr. Kathleen Tweeten	Development and Application of a HPLC Method for C50 Carotenoids from Halophylic Archaeobacteria
1988	Steven Holmgren	St. John's University	Fr. John Klassen	The Resolution of Hydroxy Acids Using HPLC
1987	Mark Heintz	University of North Dakota	Dr. J.W. Diehl	Dibenzofuranyl and Dibenzo(p)dioxinyl Substituted Polysiloxanes as Stationary Phases for Capillary GC
1986*	Christoph Pasch	University of Wisconsin – River Falls	Dr. David Rusterholz	Derivatives for the Separation of Racemic Mixtures Using Chiral HPLC / 2-biphenylcarboxamides

* (First award) Organized by Louis Haddad and Craig Markell.

SYMPOSIUM COMMITTEE

LeeAnn Higgins - Chair

Committee Members:

Peter Johnson – Program
 Steve Albrecht - Vendors / Exhibits
 Jan Jopke - Meeting Coordination

Nate Otte – Publicity / Facilities
 Sandy McDonald – Program
 Lori McNamara

Program: this subcommittee invites the keynote and focus speakers, coordinates the solicitation of papers and organizes the technical program. Program subcommittee members include:
 Dan Marchand, Sandy McDonald and Dan Dohmeier.

Exhibits: This subcommittee solicits vendors and vendor technical presentations, arranges vendor booth space and assigns exhibit space.

Publicity/Facilities: This subcommittee is responsible for publicizing the Spring Symposium through the creation of a poster that is sent to colleges, universities, and vendors, and by creating a brochure about the Spring Symposium that is sent to the MCF membership. The subcommittee also coordinates the audiovisual needs of the Symposium, the seating, food, refreshments, and the job board, designed to serve the needs of employers and prospective employees.

Chair: The Chair budgets, establishes timetables, meets with the MCF Board and coordinates all efforts in meeting the budgets and timetables for the Spring Symposium.

 The Symposium Committee members meet about bimonthly from Sept – May to plan, organize, and discuss details of the next Spring Symposium. The details of past events are reviewed for strong and weak points and possible changes and improvements for future events. New features in 2004, which reoccur in 2007, were live entertainment for the Wednesday afternoon reception and the popular, standing-room only Special Topics Discussion sessions. New in 2007 is a prize drawing during the Thursday afternoon reception. Any new ideas and comments are welcome, so please submit your Evaluation sheet at the Symposium or contact the committee chair directly

**NOTICE: Minnesota Chromatography Forum, Inc.
Annual Business Meeting**

**Thursday, May 17, 2007
Earle Brown Heritage Center**

- AGENDA:**
1. Approval of the minutes of the May 2 Board Meeting
 2. Treasurer's Report – Daniel Howman
 3. Committee Reports:
 - A. Spring Symposium - Leeann Higgins
 - B. Education - Jamie Koehler
 - C. Palmer Award - Franz Rolvaag
 4. Old Business
 5. Election Results – Josh Letze
 6. New Business - MCF Members Comments
 7. Adjourn

2007 EXHIBITORS

VENDOR

ACCTA

P.O. Box 25602
Woodbury MN 55125

Aerotek

4105 Lexington Ave. N. Sutie 300
Arden Hills, MN 55126

Agilent

13767 Danbury Path
Rosemount, MN 55068

Airgas

6191 McKinley Street NW
Anoka MN Knowles

Alltech Associates

2051 Waukegan Road
Deerfield IL 60015

Analytical Instruments

1200 Mendelssohn Ave Suite 50
Minneapolis, MN 55427

Beckman Coulter, Inc.

6550 Upper 28th Street
Oakdale, MN 55128

Bruker Daltronics

40 Manning Road
Billtrica MA 08121

Chata Biosystems

323 South College Ave #5
FT. Collins, CO 80524

Chromatography Tech. Services

11975 Portland Ave. So. Ste. 116
Burnsville, MN 55337

ChromTech, Inc.

P.O. Box 24248
Apple Valley, MN 55124

Dionex

5889 Cardinal Ridge Trail
Prior Lake MN 55372

Fisher Engineering

4500 Turnberry Drive
Hanover Park, IL 60103

Honeywell Burdick & Jackson

1953 S. Harvey St.
Muskegon, MI 49442

CONTACTS

Sales: Merlin Bicking
(651) 731-3670
(651) 730-0965 FAX

Sales: Eric Burgraff
651-415-6630
651-482-0463 FAX

Sales: Dan Thunselle
877-481-1306
951-322-3617 Fax

Sales: Richard Schinkowsky
763-712-5114

Sales: Deanna Rentner
800-255-8324
847-948-0215 FAX

Sales: Rob Palmquist
(952) 929-1996
(952) 929-1895 Fax

Sales: Brian Thoreen
(651) 253-1891

Sales: Joe Gill
978-663-3660
978-667-5993 FAX

Sales:

Sales: Greg France
952-767-1065
952-895-8493 FAX

Sales: Steve Pierson
(952) 431-6000
(952) 431-6345 Fax

Sales: Rod Brown
952-226-4140
952-226-4145 FAX

Sales: Mark Denn
800-955-9999 x9241
(952) 226-5217 FAX

Sales: Lou Avvisati
(630) 640-6683
(630) 924-73

VENDOR

Lakeview

23960 Fillmore St.
East Bethel, MN 56005

LEAP Technologies

PO Box 969
Carrboro, NC 27510

Leco

3000 Lakeview Ave.
St. Joseph, MI 49085

MicroLiter Analytical

3680 Burnette Park Drive Suite C
Suwanee GA 30024

Midwest Scientific

8535 Central Ave NE
Blaine, MN 55434

Millipore

16612 90th Ave N
Maple Grove, MN 55311

Oxygen Service

1111 Pierce Butler Route
St. Paul, MN 55104

Pace Analytical Services

1700 Elm Street Ste. 200
Minneapolis, MN 55414

Parker Hannifin

242 Neck Road
Haverhill, MA 01835

Part 3

1281 Helmo Ave N
Oakdale MN 55128

Perkin Elmer

W11450 620th Ave.
Prescott, WI 54021

Phenomenex

411 Madrid Avenue
Torrance, CA 90501

Quantum Analytics

363 Vintage Park Drive
Foster City CA 94404

CONTACTS

Sales: John Kroska
(612) 978-8012
(763) 786-3005 Fax

Sales: Mike Sloan
(815) 838-5595
(815) 838-6407 Fax

Sales: Mark Greenbaum
773-935-3173
773-935-6013FAX

Sales: Monica Howland
770-932-6565
770-932-6652 FAX

Sales: John Freeburg
763-785-1048
763-785-9725FAX

Sales : Renee Vrublely
763-420-4263
763-315-4263 FAX

Sales: Bruce Nasser
(651) 644-7273
(651) 603-8109 Fax

Sales: Tom Halverson
(612) 607-6398
(612) 607-6444 Fax

Sales: Mark Deisting
800-343-4048
(763) 420-9225 Fax

Sales: Shelley Paipal-Umland
651-738-2728
651-714-9360 FAX

Sales: Kevin Tyvoll
(763) 420-7669
(763) 420-9225 Fax

Sales: Karen Brauneck
310-212-0555
310-328-7768

Sales: Bob Freeman
916-947-6223

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CONTACTS

Sales: Marc Elliott
616-392-8001
616-396-7520

Sales: John Baker
(765) 759-5569
(765) 759-5619 Fax

Sales: Larry Ballard
(800) 945-6154
(512) 836-9159 Fax

Sales: John Campbell
(877) 698-7923
(913) 888-8388

Sales: Tom Williams
513-829-6385
513-829-5874 FAX

Sales: Brian Bertsch
513-229-7027
763-785-9725

Sales: Rob Shulfer
847-310-0140
847-310-0145

Sales: Karen Rodning
(763) 551-5342
(763) 551-5387 Fax

Sales: Gina Douget
(414) 406-6174
(215) 785-1226 Fax

Sales: Ronald Kausak
(612) 207-1444
(612) 822-4504 Fax

Sales: Amer Ebied
281-433-2655
519-668-2466 Fax

Sales: Tracie Will
(612) 824-0205
(612) 822-4504 Fax

Sales: Barb Chinnock
(508) 478-2000
(508) 872-1990 Fax
(508) 482-2674 Fax

NOTES: