LC GC

LC GC's CHROMacademy powered by element

CHROMTALKS 2023: TROUBLESHOOTING CHROMATOGRAPHY WORKFLOWS

A Virtual Symposium

Monday & Tuesday, May 22 & 23, 2023
9 am – 3:30 pm EDT

Event Overview

Following the enormous popularity of our past ChromTalks symposia, the event is back! This year, our focus is on helping you troubleshoot your chromatographic workflows, from sample preparation through detection, for both liquid chromatography (LC) and gas chromatography (GC). Our expert talks will provide you with both specific troubleshooting tips as well as explanations of the underlying processes to guide you through both making the best choices for your separation up front as well as fixing problems that may arise later. These tutorial talks—and the live question-and-answer (Q&A) periods with our experts—will address the doubts you have and clarify points you didn't even realize you had misunderstood! A few hours at ChromTalks will save you days or weeks of headaches down the road. Attendance is free. Don't miss it!

Key Learning Objectives

1. ChromTalks 2023 will answer the questions you have—and the questions you should have but don't—about your LC and GC workflows
2. Our experts will guide you through troubleshooting common problems in chromatography
3. You'll learn key fundamentals that will empower your own problem-solving skills
4. The more you understand about your instruments, the easier your work becomes
5. A few hours at ChromTalks will save you days—or weeks—in the lab.

Sponsored By:

Biotage

LECO EMPOWERING RESULTS

SARTORIUS

scion INSTRUMENTS

VUV ANALYTICS SCIENCE IN A NEW LIGHT

Day One Agenda - Troubleshooting Gas Chromatography

Morning session:

9:00 am EDT	**Current Issues with Sample Preparation for Gas Chromatography** *Tom Brettell, Cedar Crest College* Sample preparation is a key step in any analysis. There are a wide variety of techniques available for the preparation of samples prior to analysis by gas chromatography (GC). Among recent developments are faster, greener extraction methods and microextraction techniques that have been applied to a variety of sample matrices. High-throughput workstations for faster preparation and analysis of a large number of samples have revolutionized sample processing in a variety of fields. The advantages and disadvantages as well as some key issues with modern sample preparation techniques for GC will be discussed.
9:30 am EDT	**GC Sample Introduction—The Achilles Heel of GC** *Mary Ellen McNally, FMC Corporation* The type of inlet to use for your gas chromatography (GC) analysis is a complex decision. The injection is the transition point in the analysis from the injector to the column, and unfortunately, no injection technique meets all required demands. Reproducibility, the absence of discrimination based on boiling point, polarity, or thermal stability, as well as an intact sample composition/concentration during injection, are all stiff requirements. Add in avoiding column overload and no additional band broadening effects, and the demands on the inlet are many. How to make the best choice of inlet technique from cool on-column, purged packed column, programmable temperature vaporizers, multimode and split/splitless will be explained. Operation, maintenance, and troubleshooting of GC inlets will also be included.
10:00 am EDT	**GC Column Selection: The Three Most Important Parameters to Consider** *Amber M. Hupp, College of the Holy Cross* In gas chromatography (GC), separation relies of analyte volatility and polarity. The choice of GC column is paramount to achieve good resolution in an efficient timeframe. In this talk, I will first give an overview of the separation mechanism in GC. Then I will discuss several important parameters to consider when selecting a capillary GC column. I will spend the bulk of the time describing column chemistry, which is the most important consideration when establishing a new GC method. Finally, I will show examples from my own research showing the importance of column selection.

Day One Agenda (cont.)

10:30 am EDT	**Principles of Operation, Maintenance, and Troubleshooting for Sulfur and Nitrogen Chemiluminescence Detectors with Gas Chromatography** *Randall Shearer, University of Colorado, Boulder, Colorado* Sulfur and nitrogen chemiluminescence detectors play important roles in the analysis of trace species as they are applied in the laboratories of a wide array of industries, from foods, flavors, and beverages to petroleum and petrochemicals. Each of these detectors is based on a similar mechanism involving ozone-induced chemiluminescence. Important detector attributes include high elemental specificity, high sensitivity, and a linear and equimolar response on an S or N basis. This allows the gas chromatographer to detect trace species of interest in complex sample matrices without having to achieve complete separation. This presentation will compare and contrast the mechanism of each detector and use this background to help guide best practices for detector operation, maintenance, and troubleshooting.
11:00 am EDT	**GCxGC: It's Not That Hard—Really!** *Melissa Dunkle (presenter), Pascal Pijcke, Matthijs Ruitenbeek, and George Bellos, all at Dow Benelux BV, Terneuzen, The Netherlands* Over the years, gas chromatographic (GC) instrumentation and software platforms have become more robust, reliable, and user friendly, which has eased the implementation of comprehensive two-dimensional GC (GCxGC). However, even with these advances, GCxGC has not become commonplace in industrial settings, where 1D separations still dominate the landscape for (routine) applications. This presentation will focus on how to deal with some of the pain points encountered with GCxGC—especially for new users—while showing that it's really not that hard, once you get the hang of it.
11:30 am EDT	**Question-and-answer session with all the speakers in the session**
12:15 pm EDT	**Session ends**

Day One Agenda (cont.)

Lunchtime Training Session from CHROMacademy:

1:00–1:45 pm EDT	**Troubleshooting GC Sample Introduction** *Colin Towers, Element Lab Solutions, Strathaven, UK* GC instrumentation is mechanically relatively simple and robust. However, there are a few key areas where problems regularly arise. One of the most important parts of a GC method is getting your sample onto the column for analysis. If you get this wrong, the data will be incorrect and you'll be left with a difficult problem to troubleshoot. This presentation will therefore focus on the process of GC sample introduction and where it can go wrong—from issues with the autosampler components, to contamination, then on to the inlet. Topics addressed will include needles, plungers, contamination, septa (bleed and leaks), liners (contamination, adsorption), and split/splitless inlets.

Afternoon session:

2:00–3:30 pm EDT	**GC Best Practices, Tips, Tricks, and Applications from our Sponsors** *Leading suppliers provide advice on GC fundamentals and troubleshooting, including best practice, tips, tricks, and optimizing GC for specific applications and types of analysis.*

Day Two Agenda - Troubleshooting Liquid Chromatography

Morning session:

9:00 am EDT	**Key Issues with Sample Preparation for Liquid Chromatography** *Douglas E. Raynie, South Dakota State University* Surveys have shown that errors made during sample preparation procedures are among the largest sources of error in chromatographic separations. For example, in liquid chromatography, lack of attention to sample preparation may introduce particulates into the column, alter the solvent strength of the mobile phase, or render the analytes of interest less detectable. In this presentation, we will look at relationships of solvent selection considerations with peak shapes and injection considerations (solute focusing) and other serious chromatography offenses created by poor sample preparation considerations.
9:30 am EDT	**Essentials of LC Troubleshooting—Some Problems Just Never Go Away** *Dwight R. Stoll, Gustavus Adolphus College* The sophistication and reliability of instrumentation for liquid chromatography has improved dramatically over the past few decades and continue to improve today. However, even with the best available instrumentation we continue to see some of the same old problems appearing now that we have seen for decades. In other words, some problems just never go away. In December of 2021 I started an "Essentials of LC Troubleshooting" article series for LCGC that discusses many of these persistent problems. In this presentation I will touch on the highlights of this series, including: 1) best practices for effective troubleshooting; 2) pressure problems; 3) peak shape and width problems; and 4) baseline problems. For each topic I will discuss fundamental principles that underpin our troubleshooting approach and use experimental data to illustrate the problems and solutions where possible.
10:00 am EDT	**Listen to Your LC Instrument—It Is Trying to Tell You Something!** *Merlin K. L. Bicking, ACCTA, Inc.* Your LC instrument is always talking. Are you listening? Modern instruments are producing several streams of data and successful users have learned to use that information to their advantage. Troubleshooting a problem becomes much easier when you know how to use this information. This presentation will focus on two of those information streams—pressure and retention time. Many users do not have a complete understanding of the origins of, and influences on, retention time in HPLC, which makes retention time changes more difficult to troubleshoot. This presentation will take a more global approach to understanding these changes by examining some of the influences on retention, including our primary diagnostic tool—pressure. Examination of pressure plots can reveal a great deal about a particular separation, and we will show examples of how to use this information in troubleshooting retention-time problems. In addition, we will explore the use of change ratios and other practical tools for diagnosing the cause of the retention changes.

Day Two Agenda (cont.)

10:30 am EDT	**Leveraging Optimal Performance of Charged Aerosol Detection Through Optimization and Troubleshooting Techniques** *Imad A. Haidar Ahmad, Merck & Co., Inc.* In recent years, charged aerosol detection (CAD) has become a valuable tool for fast and efficient quantitative chromatographic analysis of drug substances with weak UV absorption. Some of the well-known applications of CAD are analyses for organic and inorganic counterions, sugars, and lipids. CAD is known to respond uniformly to a broad range of analytes; CAD also has other strengths, including ease of use, reliability, and wide dynamic range. The reluctance associated with utilizing CAD is diminished by understanding both the non-linear detection response and the sensitivity of the detector to all non-volatile compounds. In this talk, we will highlight some of the ways that CAD in particular is different from UV-absorbance detection, including non-linear response to analyte concentration, and sensitivity to mobile-phase impurities. We will show how careful optimization of parameters and troubleshooting can lead to successful development of analytical methods, nearly on par with that or conventional UV absorbance detector.
11:00 am EDT	**Beyond the HPLC Column: Understanding Non-Column Factors That Influence Peak Widths and Shapes in High-Efficiency Separations** *M. Farooq Wahab, University of Texas at Arlington* In HPLC, we are trained to closely examine columns, their surface chemistry, and mobile-phase interactions. However, factors that occur outside the column can affect the separation process and distort peak shapes. Examples include connection tubing and detection parameters, data sampling rate, and response times of different embedded digital filters. In particular, the impact of response time and data sampling rate on peak shapes remains a subject of substantial interest for high-efficiency separations. For example, what are the response times for HPLC-UV detection parameters, and what do they represent in signal processing? How many data points should we sample per peak? Addressing these questions and understanding the digital filters used in commercial chromatographs can help mitigate peak shape distortions caused by data acquisition software. Through simulated and real examples, this discussion will also focus on the conceptual understanding of sampling rates, response times, and various denoising processes used in commercial liquid chromatographs. These factors can become significant in low signal-to-noise ratio environments.
11:30 am EDT	**Question-and-answer session with all the speakers in the session**
12:15 pm EDT	**Session ends**

Day Two Agenda (cont.)

Lunchtime Training Session from CHROMacademy:

1:00–1:45 pm EDT	**The Top Seven Autosampler Problems and How to Avoid Them** *Dr Dawn Watson, Element Lab Solutions, Strathaven, UK* Introducing your sample consistently into the HPLC system is essential for reproducible analytical results. The role of the autosampler is to do just that. So what happens when it goes wrong? This presentation will detail the top seven autosampler issues you are likely to encounter. We'll explain how to spot these problems, how to fix them, and how to prevent them in the first place, so that you can avoid having to troubleshoot. Topics addressed will include autosampler designs, integral loop autosamplers design and operation, injection valve anatomy, mechanical autosampler issues, and troubleshooting and avoiding carryover.

Afternoon session:

2:00–3:30 pm EDT	**LC Best Practices, Tips, Tricks, and Applications from our Sponsors** *Leading suppliers provide advice on LC fundamentals and troubleshooting, including best practice, tips, tricks, and optimizing LC for specific applications and types of analysis.*

Speakers

Tom Brettell
Professor of Chemistry, Department of Chemical, Physical, & Forensic Sciences, Cedar Crest College, Allentown, Pennsylvania, USA

Tom Brettell, PhD, is a Professor of Chemistry and the Chair of the Chemical, Physical, and Forensic Sciences Department of Cedar Crest College, where he teaches analytical and forensic chemistry. He previously served for 31 years in the New Jersey State Police Office of Forensic Sciences, where, as the director, he oversaw the operation of the state's regional forensic laboratory system. He has been active in leadership positions in national and international forensic science professional organizations. Dr. Brettell has published 45 peer-reviewed publications, authored 9 book chapters on various topics of analytical chemistry, and has taught GC short courses for over 25 years.

Mary Ellen P. McNally
Global R&D Technical Fellow, Analytical Sciences, Stine Research Center, FMC Corporation

Mary Ellen McNally, PhD, is a Global R&D Technical Fellow at the Stine Research Center for FMC Corporation. She was employed by DuPont for 33 years before joining FMC. McNally has led teams on New Emerging Technologies as well as a team of inter-disciplinary scientists from three universities and DuPont on an NSF GOALI project. McNally was named to the Analytical Scientist Power List as one of the Top 50 most influential women in the analytical sciences in 2016 and has been announced as the 2021 Awardee of the Eastern Analytical Symposium Separation Science Award.

Amber M. Hupp
Associate Professor of Chemistry, College of the Holy Cross, Worcester, Massachusetts, USA

Amber Hupp, PhD, is an Associate Professor of Chemistry at the College of the Holy Cross in Worcester, Massachusetts, USA. She earned her B.A. from Kalamazoo College and her PhD in chemistry from Michigan State University. Dr. Hupp enjoys teaching a wide range of courses including environmental chemistry, general chemistry, and instrumental analysis. Her research group utilizes gas chromatography and chemometric methods to understand the fatty acid methyl ester content in biodiesel produced from different feedstocks as well as in biodiesel diesel blended fuels.

Randall (Randy) Shearer
Researcher, University of Colorado

Randall (Randy) Shearer, PhD, is a researcher at the University of Colorado, currently involved in the synthesis of novel cannabinoids and utilization of chemiluminescence detection for gas chromatography. Prior to joining the University of Colorado, he was the director of Analytical and Chemical Technologies at RES Kaidi where he helped to develop and certify jet and diesel fuels derived from biomass and natural gas. Dr. Shearer began his career at Shell R&D (Houston) and later worked in R&D and product management at Sievers Instruments (Ionics and GE) and Agilent. He's an inventor and has authored numerous publications is active in ASTM.

Speakers (cont.)

Melissa Dunkle
Senior Research Scientist, Dow Benelux, Terneuzen, the Netherlands

Melissa Dunkle, PhD, is a Senior Research Scientist at Dow Benelux, located in Terneuzen, the Netherlands. She recently joined the Hydrocarbons R&D Plastics Processes & Circularity group after nearly eight years in Analytical Science. In her current role, Dr. Dunkle focuses on various sustainability and circularity research topics, including the evaluation and characterization of circular materials and technologies. Prior to joining Dow, Melissa earned her PhD in 2007 under the direction of Prof. Luis A. Colón in the Department of Chemistry at the University of Buffalo. She then completed a two-year post-doctoral role at Ghent University under the direction of Prof. Dr. Pat Sandra. In 2009, Dr. Dunkle accepted a role as a GC Specialist at the Research Institute for Chromatography in Kortrijk, Belgium. Then in 2015, she joined Analytical Science at Dow Benelux. Her expertise includes liquid chromatography–high resolution mass spectrometry (LC–HRMS, MS/MS), gas chromatography coupled to various detector technologies (GC-FID, GC–MS, GC–(HR)TOFMS, GC-VUV), and multidimensional chromatography (LCxLC and GCxGC).

Colin Towers
Senior Technical Support Chemist, Element Lab Solutions, Strathaven, UK

After completing a degree in pharmacy, Colin Towers pursued a career in analytical chemistry. Following three years performing routine testing and three years instrument maintenance and calibration, he moved into a role as a method development and validation specialist in contract research. He gained 11 years of experience in developing methods for HPLC, LC–MS, GC, GC–MS and SPE in a high throughput commercial environment. In addition, he spent three years lecturing in forensic analytical toxicology and a further three years working as a community pharmacist. Colin has now been with Element for 11 years as a technical specialist, consultant, and trainer.

Douglas E. Raynie
Department Head and Associate Professor, Department of Chemistry and Biochemistry, South Dakota State University

Douglas E. Raynie is the Department Head of Chemistry and Biochemistry and an Associate Professor at South Dakota State University. His research interests include green chemistry, alternative solvents, sample preparation, high-resolution chromatography, and bioprocessing in supercritical fluids. He earned his PhD in 1990 at Brigham Young University under the direction of Milton L. Lee. Prior to joining SDSU, he worked in the Corporate Research Division of Procter & Gamble for eleven years. He received the L. S. Palmer Award from the Minnesota Chromatography Forum and is the "Sample Preparation Perspectives" columnist for LCGC.

Dwight R. Stoll
Professor of Chemistry, Gustavus Adolphus College

Dwight Stoll is a Professor of Chemistry at Gustavus Adolphus College in St. Peter, Minnesota, USA. He has authored or co-authored more than 90 peer-reviewed publications and six book chapters in separation science, as well as a recently released book on two-dimensional liquid chromatography. He has also written the monthly "LC Troubleshooting" column for LCGC since 2017. His primary research focus is on the development of two-dimensional liquid chromatography (2D-LC) for both targeted and untargeted analyses. Within this area he has made contributions on many aspects of the technique including stationary phase characterization, biopharmaceutical analysis, and new 2D-LC methodologies and instrumentation. He has taught several short courses on HPLC and 2D-LC in recent years at venues including Pittcon and the international HPLC conference series.

Speakers (cont.)

Imad Haidar Ahmad
Associate Principal Scientist, Analytical Enabling Technologies, Merck & Co. Inc

Imad A. Haidar Ahmad received his PhD from Florida State University under the mentorship of Dr. André Striegel. He completed his postdoctoral research with Prof. Peter Carr at University of Minnesota. He is currently an Associate Principal Scientist and supervisor in the Analytical Chemistry Enabling Technology group within the Analytical R&D department at Merck Research Laboratories (MRL).

Merlin K. L. Bicking
Senior Analytical Scientist, ACCTA, Inc.

Merlin Bicking, PhD, is a Senior Analytical Scientist at ACCTA, Inc. He has 40 years of experience as an analytical chemist in academia, contract research, independent testing, and consulting environments. His publications and presentations cover a wide range of topics, including liquid chromatography theory, method development, and application optimization. He also develops and presents technical training seminars for analytical laboratory staff.

M. Farooq Wahab
Research Engineering Scientist, University of Texas at Arlington

M. Farooq Wahab is a Research Engineering Scientist at the University of Texas at Arlington (UTA), in Arlington, Texas. He received his PhD from the University of Alberta, Canada. He worked as a postdoctoral fellow with Prof. Daniel Armstrong at UTA and assisted him in his successful start-up of a chiral HPLC column manufacturing company. His research interests include chiral analysis, development of detection technologies, and advanced signal processing for analytical chemistry, particularly an emerging technique, gas chromatography–microwave rotational resonance spectrometry. He serves on the editorial advisory board of Chromatographia and is a reviewer for several other journals. Dr. Wahab received the 2019 Young Investigator Award from the Chinese American Chromatography Association. The Journal of Separation Science included him in "Emerging Thought Leaders in Separation Science" in 2020. Wahab has authored more than 50 articles in peer-reviewed journals on various topics.

Dr Dawn Watson
CHROMacademy Product Manager, Element Lab Solutions, Strathaven, UK

Dawn Watson completed her PhD in synthetic inorganic chemistry at the University of Strathclyde, in Glasgow, Scotland, followed by postdoctoral research on small-molecule reaction kinetics at Princeton University in the United States. For several year she worked for a major instrument manufacturer, then in 2013 she joined Element and became a technical expert for CHROMacademy. She has expertise in various analytical techniques, including HPLC, GC, GC–MS, LC–MS, NMR, and molecular spectroscopy, as well as numerous wet chemistry and sample preparation techniques.