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The Column

the interactive e-publication for the global separation science industry

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The Role of Two-Dimensional Gas Chromatography in Arson Investigations

The Column spoke to Nadin Boegelsack and Gwen O’Sullivan from Mount Royal University in Calgary, Canada, about their work creating a workflow for use in the two-dimensional gas chromatography (GC×GC) analysis of ignitable liquid residues in arson investigations.

—Interview by *Kate Jones*

Q. You recently published a paper detailing a three-step workflow for a retention time indices system for use in the two-dimensional gas chromatography (GC×GC) analysis of ignitable liquid residues (ILR) for arson investigations (1). What is novel about this approach and what benefits does it offer over other methods?

Nadin Boegelsack: Alignment is a key step, and a common challenge, in comprehensive two-dimensional gas chromatography–mass spectrometry (MS) data management.

When we started method development for ILR analysis, there was no standard approach to aligning chromatograms in both dimensions, or for calculating resolution in the second dimension. The use of Kovats indices in the first dimension is relatively common to allow overlays between GC and GC×GC chromatograms. The challenge arises with the second dimension, as existing methods require thermodynamic models or isovolatility curves, which are very involved approaches. An additional challenge is defining the starting measurement point

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(zero-value) in the second dimension. This is often achieved by relating each compound to its bracketing *n*-alkanes, which requires several calculation steps. We wanted to simplify this time-consuming approach for an easier transition to routine analysis by making it more user-friendly and allowing the whole system to be condensed into three steps: defining zero across the whole chromatogram, obtaining first-dimension retention index (RI) with a single calculation, and obtaining second-dimension RI with a single calculation. We therefore developed this RI system to address alignment in an inexpensive, quick, and user-friendly fashion by combining two well-known RI systems from GC. The combination of Kovats and Lee indices correlates seamlessly to the ASTM ILR classification scheme, which bases light, medium, and heavy product classification on an *n*-alkane range (Kovats indices), and compound grouping with polarity and aromatic ring number (Lee indices). Both RI systems have readily available standard mixtures due to their popularity in GC analysis, are inexpensive, and store easily.

Q. What challenges did you encounter when developing this method and how did you overcome them?

Gwen O'Sullivan: GC×GC is more widespread in academia than industry, so

there is very little standardization in how to approach handling data across a wide range of applications. As a result, we needed to evaluate the workflow for ease of use, the RI system for its flexibility and performance, and then ensure it was applicable to ILR analysis. Making the RI system user-friendly was achieved by building on existing single dimension GC RI systems. The standards were readily available at low cost, and the systems themselves had been validated over decades of GC applications. Evaluating flexibility and performance was more challenging, as published results displayed variability depending on GC system setup and no acceptable error margins had been published—especially for the second dimension. We established criteria based on theoretical expectations and requirements for general application to evaluate performance against, and evaluated flexibility based on the potential area covered in the chromatogram, as well as options to expand the range of compounds covered regarding boiling point and polarity. The application to ILR analysis was demonstrated by the development of the ILR classification map in relation to ASTM E1618 (2) and its practical application to a variety of neat ignitable liquids, as well as a fire scene sample.

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Q. What challenges exist when analyzing wildfire samples?

GO: Wildfires provide exceptionally difficult scenes to investigate. ILRs are typically present at low concentrations and are dispersed; natural compounds are present at very high concentrations and can have structural similarity to ILR compounds. The remoteness of the locations and efforts to extinguish these fires can result in the destruction of evidence. Limitations in sensitivity of analytical equipment lead to false negative reports, frequently contradicting canine detection on scene. Advances in analytical power (for example, using GC×GC–MS) allow laboratory analysis to keep up with canine detection rates and provide fewer ambiguities before court. Critical issues in the field of wildfire investigation are: the presence of ILR at low levels; complex matrices; competitive adsorption; interferences/pyrolysis background; subjective determination and judgment; and the need to determine error rates for court cases. With the prevalence of wildfire increasing globally, it is even more pressing to address these knowledge gaps within the field of wildfire forensics.

Q. What can chromatography reveal about how wildfires impact the environment?

GO: In the last decade, large-scale wildfires

have become increasingly prevalent across North America, Europe, and Australia. Although wildfires can be caused by natural phenomena, such as lightning strikes, the majority are caused by human activity. The cost of human-induced fires has risen significantly as the frequency, severity, and associated damages grow. Climate change causes earlier snow melts and overall drier conditions, exacerbating fire risk. Investigations into the origin and cause of wildfires are important for understanding the direct role of humans, the influence of climate change, and the development of necessary and appropriate forest management strategies. Chromatography contributes significantly to our understanding of wildfire initiation and propagation, enhances investigative processes, increases prosecutions and allocation of liability, and contributes information that informs policy and wildfire management.

Q. You have also recently developed a method for optimizing the analysis of ILRs using flow-modulated comprehensive GC×GC (3). Please could you talk a little about this research.

NB: We wanted to utilize the selectivity and sensitivity offered by GC×GC–MS to address the particular challenges associated with wildfire analysis: separation of target

compounds from matrix interferences, increasing sensitivity to avoid false negatives, and offering a seamless transition to the currently used standard (ASTM E1618).

When looking at existing method development papers, we noted that few went into details for hardware optimization, that is, column selection and modulator setup, beyond phase chemistry and modulation period. Flow-modulated GC×GC is often considered complicated, mostly due to the need for establishing a suitable flow equilibrium specifically for the method. In this paper, we provided a comprehensive overview of the most important factors to be considered and suggested a template workflow on how to achieve the desired results, with efficient decision-making based on our approach to an ILR method.

For column selection, this covers phase chemistry and its effect on selectivity, column dimensions and column order with their effect on the flow equilibrium, and orthogonality and film thickness with their effect on selectivity and effective chromatography area usage. We observed effects on total percentage of chromatogram area covered (with consideration of wraparound peaks, since they can lead to more demanding manual data processing), as well as separation and resolution in each dimension, and total peak capacity for

the system with the potential number of distinguishable peaks.

In the modulator setup section, we first looked at establishing a suitable flow ratio between the columns to ensure good peak shapes across the chromatogram for consistent integration. This flow ratio was also applied to the column selection process to keep results comparable. Second, we investigated carbon loading and dilution effect to ensure adequate sensitivity of the method. Establishing flows across the 7-port valve connecting the columns depends on the flow ratio and its resulting individual column flows. They need to be balanced with the bleed line to allow consistent loop fill, which in turn is responsible for carbon loading and peak shapes in the second dimension. Lastly, the flow equilibrium in the 3-port valve determines the detector split. To achieve the optimum split ratio, transfer line dimensions can easily be optimized with a modelled approach and the manufacturer-provided flow calculator.

The hardware optimization is not usually mentioned in method development approaches, despite its significance. Changes to columns and lines require system downtime, which may be a contributing factor. Regardless, following very general GC method recommendations for method parameters (for example, oven ramp) after the



hardware optimization resulted in us meeting all target separations for the verification, which consisted of a certified standard mixture and simulated wildfire debris.

Since real wildfire matrix is more varied and complex, parameter optimization was still required. We used design of experiment (DoE) to evaluate the three most significant parameters under the same performance criteria as the column selection process. The resulting optimized method successfully addressed the associated challenges (separation from interferences, increased method sensitivity, seamless transition to current ASTM standard) on real wildfire debris samples. Without our previous work on retention indices, the evaluation of resolution across different column combinations or parameter settings would have been incredibly challenging. They also allowed us to create an ILR classification map relating to the ASTM classification scheme, which provides a very user-friendly visual comparison of different samples and has been applied to various wildfire debris samples in our research.


Q. What projects are you working on next?


GO: The biggest area for growth in the application of GC×GC analysis for fire debris lies in interrogating the large volumes of

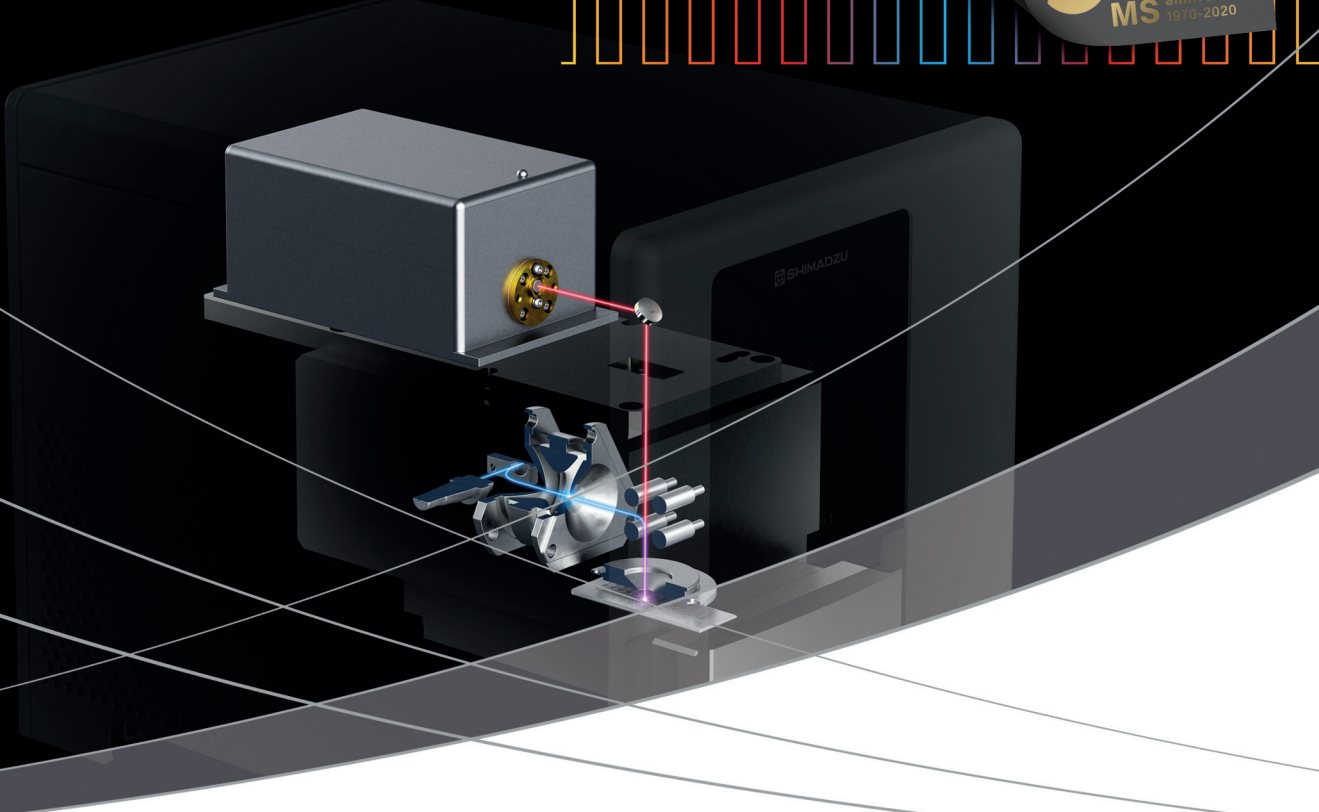
data generated from each analysis. The compositional characteristics information with each analysis is unequalled. However, the size and complexity of the data make data analysis a challenge. The work in this area has begun, including the development of new software and workflows for non-targeted screening and targeted analysis. Techniques such as chemometrics assess both chromatography and spectral data (for example, total ion counts, total ion spectrum, extracted and summed ion counts) using tools such as principal component analysis (PCA), hierarchy cluster analysis, and partial least squares (PLS) regression to reduce and rationalize data processing outputs. Our work continues in this area and we are looking forward to publishing our work in the early new year.

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1. N. Boegelsack, C. Sandau, D.W. McMartin, J.M. Withey, and G. O'Sullivan, *Journal of Chromatography A* **1636**, 461717 (2021).
2. ASTM E1618-14, *Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry* (ASTM International, West Conshohocken, Pennsylvania, USA, 2014) www.astm.org
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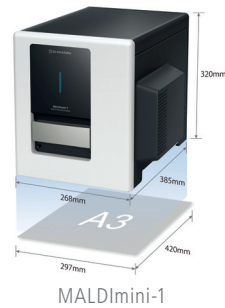


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Nadin Boegelsack is a passionate analytical scientist with 10+ years of experience working internationally in industry and academia. She is

currently finishing her Ph.D. at the University of Saskatchewan (Canada) while working as an instructional assistant at Mount Royal University (Canada), exploring solutions to

analytical challenges in forensic and environmental sciences.



Gwen O'Sullivan is Associate Professor and Chair of the Department of Earth & Environmental Sciences at Mount Royal University. Her areas of expertise

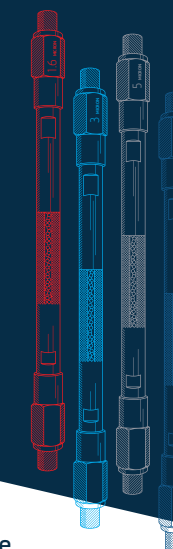
include environmental chemistry, environmental forensics, and contaminated land and groundwater.

She has over 20 years of experience in a variety of environmental and criminal forensic projects, including legal sampling, chemical fingerprinting, and statistical evaluation of data for source identification of ignitable liquid residues, persistent organic pollutants (POPs), petroleum hydrocarbons, methane, nitrates, and dioxins in water, soils, and sediments.

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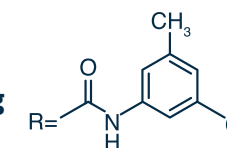
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ZooMS Identification of Australian Fauna

Thermo Announces PharmaFluidics Acquisition

Thermo Fisher Scientific (Waltham, Massachusetts, USA) has announced the acquisition of Belgium-based PharmaFluidics, the developer of the μ PAC range of micro-chip-based chromatography columns.

The micro-pillar array portfolio of columns enhance liquid chromatography (LC) performance in biomarker, proteomics, and drug research and development applications. The columns are formed by carefully etching away the interstitial volumes out of a solid silicon wafer, leaving an array of pillars. The resulting channels are folded onto a small footprint by concatenating bed segments with optimized flow distributors that limit peak dispersion. This creates a stationary phase support structure that is organized in a reproducible, ordered pattern.

“LC is a key analytical technique across numerous applications, and its accuracy, reproducibility, and robustness is critical to the delivery of meaningful results,” said Kent Davidson, Vice President and General Manager, High Performance Chromatography Solutions, Chromatography and Mass Spectrometry, Thermo Fisher Scientific. “Columns are an essential part of these workflows, and the next-generation μ PAC columns push this element to a new level through innovative pillar array technology, improving the quality of data.”

For more information, please visit: <https://corporate.thermofisher.com> or www.pharmafluidics.com

Australia is home to an extremely rich and unique animal life, with more than 85% of its terrestrial mammal species being found nowhere else. Famous members of Australia’s wildlife, such as kangaroos, wallabies, koalas, and wombats, play key roles in the ecosystems they inhabit, and were important both culturally and for survival to aboriginal communities, as their bones were used as raw materials for the creation of tools and other artefacts. Researching these animals and the artefacts derived from them can give valuable information on early human activity on the Australian continent, as well as insights into biodiversity shifts and the reasons for them.

Animal remains are frequently uncovered in Australia, dating from the late Pleistocene to the historical period. However, the continent’s harsh environmental conditions, along with other factors such as scavenging by marsupial carnivores, frequently results in many highly fragmented, morphologically unidentifiable bone fragments. This, combined with the scarceness of reference materials and osteological similarities between species, complicates the study of animal remains from Australian sites.

Zooarchaeology by mass spectrometry (ZooMS) offers a means to improve taxonomic identifications of fragmented bone material at sites around the world, and presents an exciting opportunity to address these challenges in Australian contexts. ZooMS is a high-throughput, proteomics-based approach that uses differences in the collagen type I (COL1) protein sequence to identify remains. COL1 is the most abundant protein in bone, skin, antler, and dentine. Researchers used matrix-assisted laser desorption–ionization tandem time-of-flight mass spectrometry (MALDI-TOF-MS) and liquid chromatography–tandem mass spectrometry (LC-MS/MS) to analyze a selection of modern reference samples taken from known collections and archaeological specimens consisting of 2922 bone fragments (1).

Researchers identified novel peptide markers for 24 extant marsupial and monotreme species, which allowed for genus-level distinctions between these species utilizing the ZooMS methodology. The utility of these new peptide markers was demonstrated by using them to taxonomically identify bone fragments from a nineteenth century colonial-era pearlshell fishery at Bandicoot Bay, Barrow Island, in Western Australia. Researchers believe the suite of peptide biomarkers presented in the study, which focus on a range of ecologically and culturally important species, have the potential to significantly amplify the zooarchaeological and paleontological record of Australia.—L.B.

Reference

1. C. Peters et al., *R. Soc. Open Sci.* **8**, 211229 (2021).

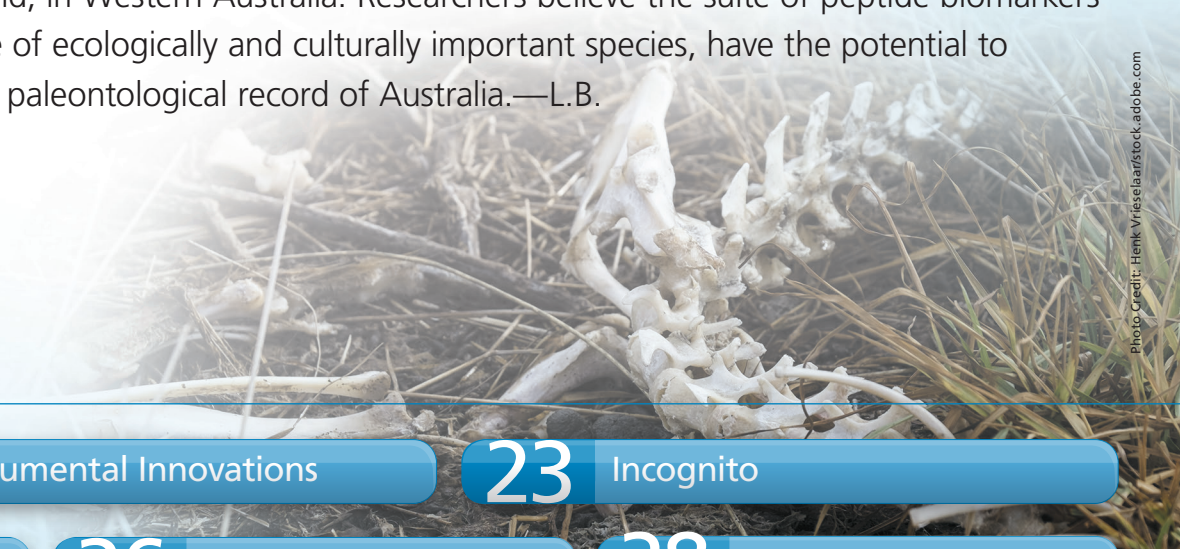


Photo Credit: Henk Vrieselaar/istock.adobe.com



Mülheim Water Award 2022 Open For Submissions

Sartorius Receives Bioprocessing Award

Sartorius (Göttingen, Germany) has won the “Overall Best Bioprocessing Supplier” award at the Europe Bioprocessing Excellence Awards 2021. According to award organizer IMAPAC, the awards seek to recognize exceptional bioprocessing experts, organizations, and technologies that facilitate biomanufacturing excellence at enhanced speed, reduced cost, and superior quality across Europe and the rest of the world.

“We’re honoured to be named ‘Overall Best Bioprocessing Supplier’ at this year’s Bioprocessing Excellence Awards. We remain committed to helping our customers create better patient outcomes through the development of new and improved therapies,” said Christopher Mach, Head of Key Account Management at Sartorius.

The award ceremony was held in conjunction with Biologics Contract Manufacturing Europe 2021 and 4th Biologics World Nordic 2021 conference events, and René Fáber, Head of BPS Division, Sartorius, accepted the award virtually on behalf of Sartorius.

For more information, please visit: www.sartorius.com

Submissions for the 2022 Mülheim Water Award open on 1 December, kicking off a three month application period for the award and its €10,000 prize money. The award, which will be given for the eighth time in 2022, recognizes practical research and development projects, as well as the implementation of innovative concepts in drinking water supply and water analysis methods. Submissions will close on 28 February 2022.

The Mülheim Water Award was first presented in 2006. Since then, the award has been presented seven times and over 150 applications from 22 different European countries have been received, covering the entire spectrum of water management topics.

The award is sponsored by the water supplier, RWW Rheinisch-Westfälische Wasserwerksgesellschaft mbH and Gerstel GmbH & Co. KG, both located in Mülheim an der Ruhr, Germany. The IWW Centre for Water will coordinate the application and selection process.

The theme for the 2022 award is “innovations for a sustainable water industry

and a safe and secure drinking water supply”. Applications are open to both natural and legal persons, groups of persons, and institutions from Europe. Submissions of innovative, practically relevant concepts, as well as application-ready solutions that will help address future challenges in drinking water systems and water analysis methods are sought.

There is a two-stage application procedure beginning with a short online application. The initial application form is available at: <https://muelheim-water-award.com/en/apply/>

From the pool of submitted applications, a jury of experts will shortlist the most promising submissions for progression to stage two, where further application documents will be requested.

The award ceremony of the Mülheim Water Award 2022 officially takes place during the

conference dinner of the 5. Mülheimer Wasseranalytisches Seminar (MWAS 2022) on 14 September 2022 in Mülheim an der Ruhr.

“We need innovative water treatment processes and environmentally friendly analysis techniques to take us to the next level of sustainability—Gerstel actively supports and takes part in that process,” said Holger Gerstel, Owner and General Manager of Gerstel.

For more information, please visit: www.muelheim-water-award.com/en/



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Agilent Announces Green Lab Sponsorship

Agilent Technologies Inc. (Santa Clara, California, USA) has announced a sponsorship deal of My Green Lab, a leading nonprofit organization dedicated to improving the sustainability of scientific research. Agilent has committed to a “Transformative Level” sponsorship and is the first institution to do so.

Sustainability is becoming a critical strategic element in science and industry, and there is a growing need for laboratories to consider their environmental responsibilities. My Green Lab was formed in 2013 to lead and unify scientists, vendors, designers, energy providers, and others in a shared drive to improve the social and environmental responsibility of scientific research.

“Environmental sustainability drives many of our innovations, enabling us to be a more holistic partner for our customers, helping them meet their own sustainability goals while at the same time increasing their productivity, lowering costs, and making their labs safer, healthier environments in which to work,” said Darlene Solomon, Agilent Senior Vice President, and Chief Technology Officer.

For more information, please visit: www.agilent.com

Walsh University Dedicate Shimadzu-Funded Analytical Laboratory

Walsh University (North Canton, Ohio, USA) and Shimadzu Scientific Instruments (Columbia, Maryland, USA) have dedicated the Center for Analytical Excellence Laboratory, a collaborative project that includes the installation of nearly \$1.2 million worth of new analytical equipment, at a recent event. The event was attended by Walsh University and local high school students and faculty, Yoshiaki Maeda, President of Shimadzu Scientific Instruments, and other company representatives, members of the business community, and elected officials.

The event also featured a celebration luncheon, a ribbon-cutting ceremony, science activities in the laboratory, student and faculty research presentations, and a science career fair. The ongoing partnership between Walsh University and Shimadzu was developed through the Shimadzu Partnership for Academics, Research, and Quality of Life (SPARQ) programme, which is designed to build long-term relationships between Shimadzu and academic institutions.

Shimadzu provided analytical solutions covering a range of techniques, including liquid and gas chromatography instruments for analyzing volatile and nonvolatile organic compounds, an inductively coupled plasma instrument for metals analysis, and equipment for a materials analysis laboratory. Supporting Walsh faculty and student research, the Center for Analytical Excellence Laboratory will also serve as a resource for collaborative projects within the business community, and for local companies that need to analyze samples but may not be able to make the investment in such instrumentation.

“The Center for Analytical Excellence Laboratory creates an opportunity for faculty and students that is unparalleled in the region,” said Dr. Tim Collins, Walsh University President. “Shimadzu’s generous donation will help position Walsh as a university that rivals the capabilities found at large research institutions—but with the benefits of the personal touch of a teaching campus.”

Walsh University received contributions for the laboratory from several benefactors

in the region, including a grant from Shimadzu of more than \$550,000 towards the state-of-the-art instrumentation. In addition, Shimadzu will provide internship opportunities and unlimited access for faculty, staff, and students to consult with Shimadzu scientists and industry experts on projects for the life of the partnership.

“Walsh University’s proven dedication to drive scientific research for the betterment of students, the local community, and organizations around the country reflects a true commitment to education. We’re proud to award this grant and excited about the prospects this relationship provides both Walsh and Shimadzu,” said Yoshiaki Maeda, President of Shimadzu Scientific Instruments.

To learn more about the partnership between Shimadzu Scientific Instruments and Walsh University please visit: www.ssi.shimadzu.com or <https://www.walsh.edu>



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Peaks of the Month



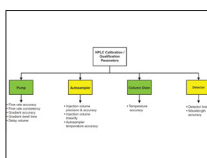
- **The LCGC Blog: The Beauty of the Quadrupole Mass Analyzer**—The quadrupole mass analyzing device is now accessible to many analytical chemists as a detector in either HPLC or GC instruments due to their increasingly accessible price point. While it's not vital that we understand the working principle of these detectors, insight into their design and operation can help enormously when planning or optimizing analyses or troubleshooting issues. [Read Here>>](#)



- **Automated High-Resolution Semi-Preparative Gradient Recycling Liquid Chromatography: Principles, Design, and Applications**—A gradient twin-column recycling liquid chromatography (GTCRLC) process has been developed and applied for the isolation of a single polycyclic aromatic hydrocarbon (PAH), chrysene, present in a complex PAH mixture. [Read Here>>](#)



- **Intact Mass Analysis of Biopharmaceuticals as its Own Unique Application**—Intact mass analysis is becoming increasingly useful for characterizing biologics. We describe the current application of intact mass analysis, including quantitation, sequencing, and structural characterization. [Read Here>>](#)



- **It's Qualification, But Not As We Know It?**—Qualification and calibration of HPLC chromatographs is a regulatory requirement, but how prescriptive should guidance be? [Read Here>>](#)



- **But My Peaks Are Not Gaussian! Part 3: Physicochemical Causes of Peak Tailing**—What leads to an asymmetric peak shape? Physicochemical phenomena can help chromatographers identify whether the cause of asymmetry has a chemical or physical origin. [Read Here>>](#)



- **Analyzing Glycerol in Biodiesel Using SPE and HPLC-RI**—Researchers have developed a viable alternative to existing GC methods for glycerol determination in biodiesel using SPE–HPLC-RI. [Read Here>>](#)

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News In Brief

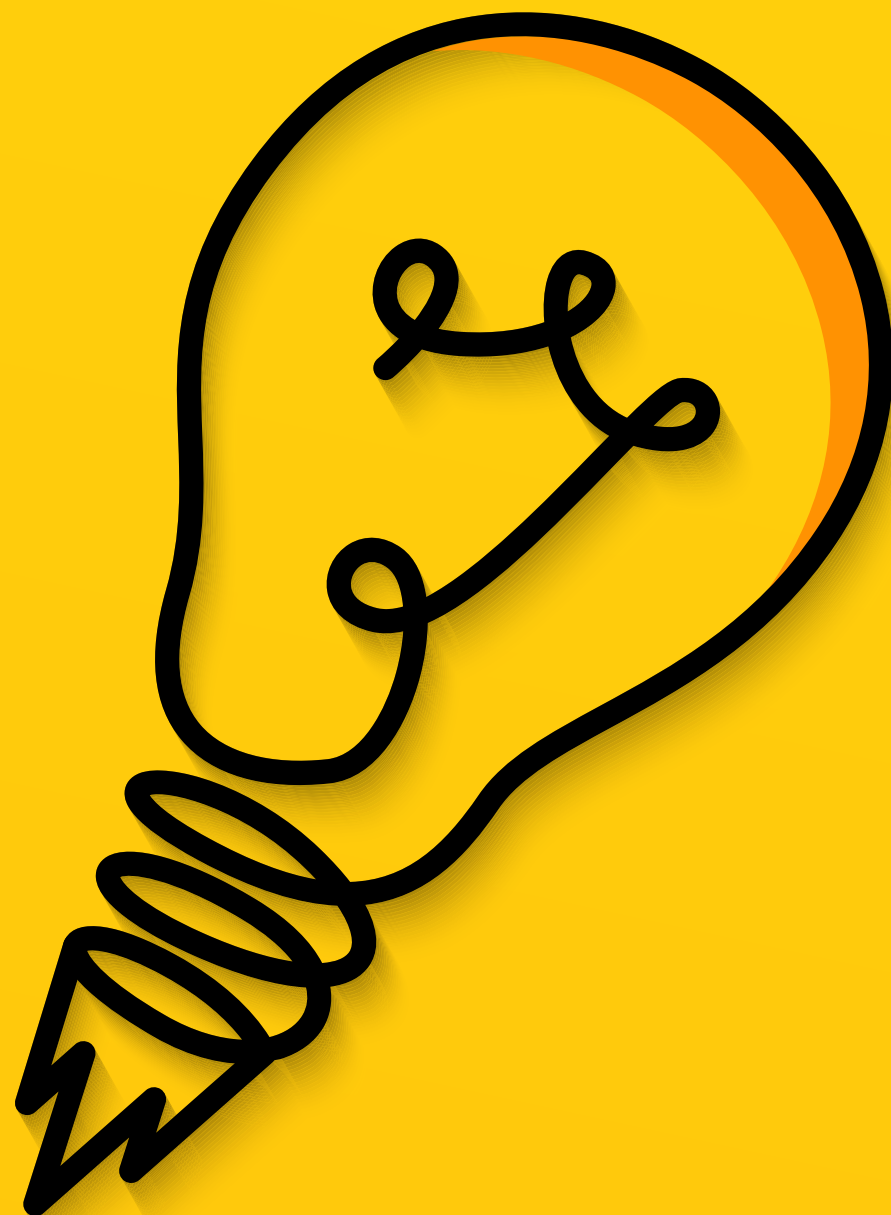
Cellumed Co. Ltd., (Seoul, Korea) and Artes Biotechnology GmbH (Langenfeld, Germany) have announced a development and license agreement aimed at the generation of microbial production cell lines, processes, and documentation for the cGMP manufacturing of two enzymes required in the manufacturing process of mRNA vaccines. For more information, please visit: <https://artes-biotechnology.biz>

Dotmatics Ltd (Bishop's Stortford, UK), an informatics software provider, has announced a partnership with RockStep Solutions (Portland, Maine, USA), a developer of software tools for managing data and operations for in vivo research. The collaboration combines the real-time access to all research data afforded by the Dotmatics Platform with RockStep's in vivo study management software "Climb", enabling a fully integrated workflow that will save researchers' time and improve ease of operation. For more information, please visit: www.rockstepsolutions.com/climb-dotmatics/

Life sciences company Labcorp (Burlington, North Carolina, USA) has launched a new laboratory in Singapore, with plans to expand its staff by adding about 90 research and engineering jobs to its 350-strong team in Singapore. The roles include nursing coordinators to facilitate decentralized clinical trials, as well as data engineers to develop artificial intelligence technologies to assist in diagnosing diseases. For more information, please visit: <https://www.labcorp.com/labcorp-opens-new-integrated-laboratory-strengthens-bioanalytical-services-asia-pacific>



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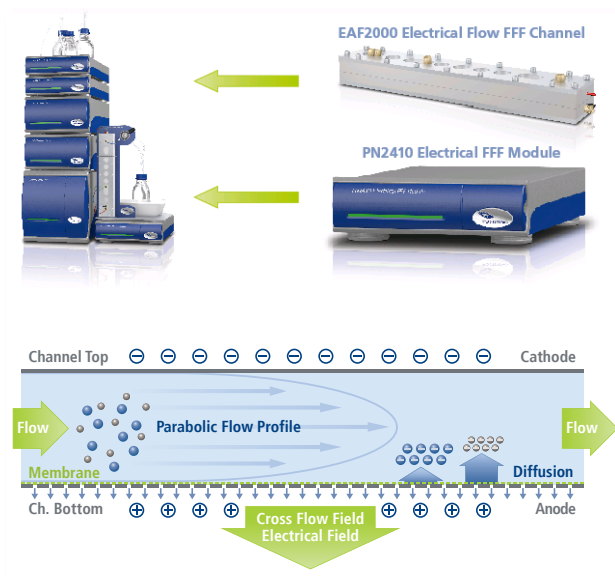
FFF Products

Electrical/Asymmetrical Flow Field-Flow Fractionation

Traditional separation technologies for biopharmaceutical and nanoparticle applications provide particle size or molar mass distributions as the final result. However, it is clear that particle and molecular charge play a primary role in many applications such as protein aggregation, polymer flocculation, particle agglomeration, and in pharmaceutical formulations in general. The Postnova EAF2000 instrument using electrical asymmetrical flow field-flow fractionation (EAF4) technology allows the particle size or molar mass distributions to be further differentiated and transformed into charge distributions. This identifies charge heterogeneities present within the different size and molar mass fractions and will help to aid research or establish more efficient product development processes.

The instrument works by combining the principle of electrical and AF4 in one system. According to the company, this instrument is a key tool, particularly for protein research, because existing techniques for zeta-potential are limited by concentration and are batch techniques giving an average value for all components in the solution. The EAF2000 can determine the zeta-potential of each individually separated component, such as protein monomer and dimer (or higher aggregates) or antibody monomer and fragments–aggregates.

<https://www.postnova.com/product/systems/eaf2000-electrical-af4.html>



LNP Gene Vector Analysis System

Comprehensive biophysical characterization of lipid nanoparticle (LNP) formulations for the delivery of nucleic acids may be achieved using Wyatt Technology's FFF-MALS system, which combines an Eclipse field-flow fractionation instrument, a DAWN multi-angle light scattering instrument, an Optilab refractive index detector, and industry-standard modules from Agilent Technologies, such as the Infinity II pump, autosampler, UV detector, and fraction collector.



This system performs size-based separation and online analysis of LNPs to provide high-resolution size distributions with quantitative particle concentrations. In addition, it can determine encapsulation efficiency and, using a proprietary method, the size-dependent payload distribution—the amount of encapsulated nucleic acid as a function of particle size.

All of these are measured in a single, automated run that takes about one hour, with no special sample preparation or reliance on reagents and highly skilled technicians, according to the company. In addition to basic characterization, the system is useful for stability studies, including stability in biological matrices such as plasma and formation of protein corona.

In addition to online analyses, size-based fractions may be collected for offline analyses, such as cryo-EM, HPLC, or in vitro/animal studies to evaluate how size affects biological functionality.

www.wyatt.com/LNP



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GC Products

Multi-Gas Enabled Thermal Desorbers

Helium is a rapidly dwindling non-renewable resource and is becoming more difficult to source, causing a disruption in supply and an increase in cost. For sustainability and to protect laboratories against future helium shortages, Markes International has launched the world's first range of multi-gas-enabled thermal desorption (TD) systems that can be used with helium, nitrogen, or hydrogen as carrier gas in TD–GC–MS workflows.

Markes' thermal desorbers have been externally tested and validated so that the full TD–GC–MS analytical instrumentation and workflow can be configured to work safely with all three gases.

Multi-gas thermal desorbers confer many benefits. Hydrogen offers faster chromatographic separations, shorter TD methods, and lower temperature separations than helium, drastically increasing laboratory throughput, extending consumables' lifetimes, and extending maintenance intervals. Switching from helium to hydrogen saves up to 90% for each cylinder, according to the company. A hydrogen generator eliminates the costs of cylinders together with their delivery, rental, and system maintenance, and removes the need to install expensive pressurized gas supply lines and cylinder cages in new or refurbished laboratories.

Switching to multi-gas systems is easy because existing columns and consumables can be used, allowing fast adoption in busy laboratories, according to the company.

<https://chem.markes.com/LCGC/Multi-Gas>

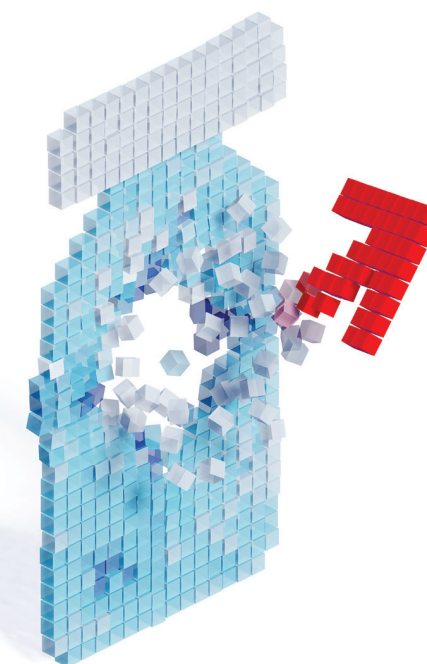


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ThermoFisher
SCIENTIFIC



TOF-MS for GC and GC×GC

The new BenchTOF2 mass spectrometer is recommended for tackling challenging GC and GC×GC applications because of the ability to perform target and non-target analyses on a single platform. This novel instrument builds on a combination of sensitivity, spectral quality, selectivity, speed, and stability, with innovative performance improvements in both hardware and software. Analysts can benefit from improved detection limits (<20 fg OFN) with full spectral information, meaning a wide range of trace-level species can be detected in a single analysis.



BenchTOF2 also allows retrospective searching for new compounds of interest. An extended dynamic range across five orders of magnitude allows accurate quantitation of high-concentration compounds, while maintaining low detection limits, removing the need for dilutions or repeat analyses and increasing productivity.

The company reports that the BenchTOF2 is fully certified for use with hydrogen carrier gas, resulting in faster chromatographic separations, reduced running costs, a faster return on investment, and mitigation of gas supply issues.

Incorporated into BenchTOF2 is Tandem Ionisation, a patented technique that enables simultaneous hard and soft EI ionisation, providing complementary spectra for characterization of targets and unknowns in a single run without hardware changes or additional method development.

ChromSpace software can be used for full instrument control and data processing and the BenchTOF2 can be coupled to all popular GC systems.

<https://chem.sepsolve.com/BTOF2>

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{image almost to scale}

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Ion Chromatography

Disinfection Byproducts with Hydroxide Eluents

Metrohm is a trusted manufacturer of high-precision instruments for chemical analysis and provides many solutions, particularly for the determination of ionic compounds in water using ion exchange chromatography. One class of these compounds is water disinfection byproducts—ions formed during the disinfection of drinking water. The oxyhalides chlorite, bromate, and chlorate are known to be health hazards and must be monitored according to multiple standards (for example, US EPA 300.1 A/B, DIN ISO 10304-4-1997).

The Metrosep A Supp 18 anion exchange column is the first Metrohm column able to separate and determine standard anions and oxyhalides in one run using a hydroxide eluent at room temperature. The company reports that this column excels with a very high resolution between chlorite, bromate, and chloride, resulting in low detection limits for oxyhalides. Next to the specific hydroxide selectivity of the stationary phase, very high separation efficiency is achieved with the compact packing of the small 3.5- μm hydrophilic polymer-based particles. The column has a pH range of 3–13 and is compatible with common organic modifiers.

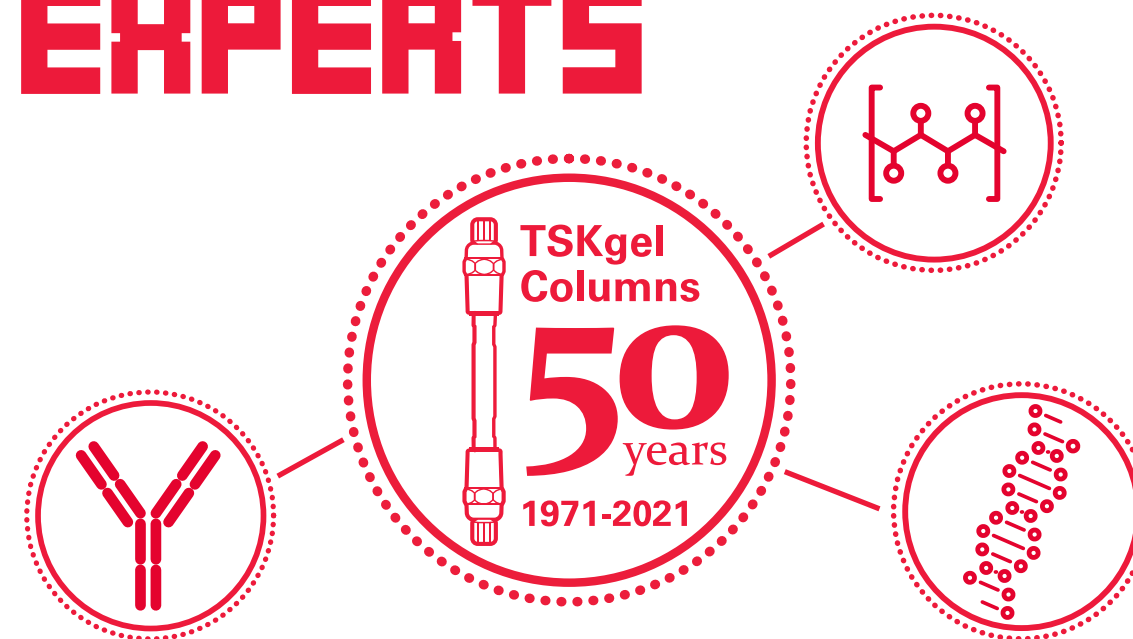
In addition, the unique selectivity of the Metrosep A Supp 18 column allows the determination of haloacetic acids in water samples.

www.metrohm.com/



TOSOH

#CHROMATO GRAPHY EXPERTS



CELEBRATING 50 YEARS OF HPLC INNOVATION

- **1971** - Launch of first TSKgel size exclusion column
- **1987** - Launch of the current gold standard for mAb analysis: TSKgel SW_{xL}
- **2019** - Launch of TSKgel FcR-III A columns for fast assessment of mAb ADCC activity
- **2021** - Find out more about our milestones and how you benefit from them: bit.ly/TSKgel-50

TOSOH BIOSCIENCE



LC Products

Online and At-Line Process Analysis

The Agilent 1260 Infinity II Prime Online LC System is a dedicated process analytical technology (PAT) tool for online and at-line process analysis via LC applications.

Supporting the current trends within the pharma and biopharma industry towards continuous manufacturing and real-time release testing, the 1260 Infinity II Prime Online LC System enables automated process monitoring of critical process parameters (CPPs) and critical quality attributes (CQAs), providing real-time data for greater control and faster understanding of processes.

The system supports direct injections for fast process monitoring. The company reports that it also features retain-sample functionality for dilutions (up to 1:1000), automated sample preparation steps, multimethod analysis, or sample archiving purposes for offline quality control.

As well as offering a variety of sampling options, the system also introduces hybrid injection technology to the LC world. Alongside classic flow-through injection, the company has included "Feed Injection" as a novel LC injection technique, guaranteeing seamless method transfer and the capability to mediate strong sample diluent effects.

The company reports that the Online LC System seamlessly operates with the Agilent Online LC Monitoring Software, which is designed for process applications and coordinates sampling and sample analysis. This reportedly allows for intuitive sample scheduling to quickly set up sampling events and provides data visualization tools such as trending plots to easily monitor the process.

Agilent 1260 Infinity II Prime Online LC System | Agilent



Liquid Flow Meter for LC Applications

Determination of flow rates in liquid chromatography has traditionally been a tedious, inexact, and manual task. But not any longer.

The new Biotech Liquid Flow Meter can continuously and accurately determine inline flow rates in any mode of liquid chromatography without interfering with the system, according to the company. Being compatible with most solvents, with a wide pH range, and pressures up to 3 MPa (435 psi), it is reportedly the perfect tool for high-precision measurement of flow rates ranging from 0.001 to 5 mL/min. The company report that it is very easy to use, small enough to fit into the hand or lab coat pocket, and does not weigh more than a cell phone. The Biotech Liquid Flow Meter comes pre-calibrated and can be connected inline in any orientation to continuously measure inline flow rates of liquids, for instance, during qualification of chromatography instruments. From its USB interface and modern PC application, data can also be recorded as time series, which may be very helpful when identifying flow rate variations during troubleshooting of pumps, check valves, or other malfunctioning components in liquid chromatography equipment.

Learn more about the Biotech Liquid Flowmeter AB-40010 at:
www.biotechfluidics.com/products-sensors-flowmeter/



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Chiral Chromatography Solutions

Expanding its range of innovative chiral chromatography solutions, Daicel Chiral Technologies announces the introduction of Chiralpak IK, adding new selectivity capabilities to the Daicel portfolio.

This new immobilized selector builds on the success of Chiralpak IG, using the same chiral derivative but with a cellulose polymer.

In laboratory testing, the company reports that Chiralpak IK adds new separations and enhances some separations already achieved using IG and other selectors.

From November 2021, Chiralpak IK will be available in 5- μ m particle size, extending into other sizes over the coming weeks.

Daicel will continue to add new immobilized columns in 2022, offering more ways to achieve successful enantiomeric separations. According to the company, the extended life and new selectivities of Daicel's robust immobilized columns ensure their increasing success.

By continuously extending its range of robust immobilized columns, Daicel now offers more than 2000 chiral products, giving analysts a broad range of enantiomeric separation answers. Each new product builds upon Daicel's 40 years of experience specializing in chiral chromatography.

chiraltech.com



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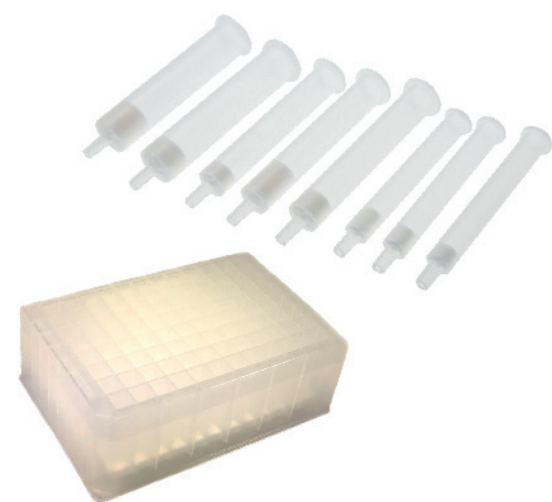
HILIC SPE for Separation of Polar Compounds

Hilicon's iSPE-HILIC cartridges and 96-well plates are designed for solid-phase extraction (SPE) sample preparation of polar/hydrophilic compounds in hydrophilic interaction liquid chromatography (HILIC) mode. They can be used for enrichment and purification of glycopeptides, glycans, PSP toxins, and many other polar compounds.

The packed stationary phases are charge modulated hydroxyethyl amide silicas that are covalently bonded with neutral, positively charged, and negatively charged hydrophilic functional groups. Therefore, the polar compounds are enriched or purified by combined interactions, hydrophilic interaction, hydrogen bonding, and weak electrostatic interaction between the analytes and stationary phase. The selectivity is specific, and the procedure is relatively simple. It is applicable to high-throughput analysis of large sample quantities. One of the applications using iSPE-HILIC is selective and efficient enrichment of glycopeptide or glycans prior to detection and identification by mass spectrometry (MS) analysis. It has also been used to purify polar phospholipids classes from nonpolar lipid classes before LC-MS analysis.

As well as the HILIC SPE products, the company offers a broad range of innovative HILIC columns based on polymeric and silica particles, which allows sustainable and reproducible HILIC separation in wide pH ranges.

www.hilicon.com



Nitrogen Gas Generator Solution

Peak Scientific has recently released the latest addition to their Infinity XE series, the Infinity XE 60. Peak has designed a nitrogen gas generator that can have its capacity expanded on site, to increase flow rates to meet a laboratory's requirements (up to 520 litres per minute). Conveniently, this expansion can fit in the same footprint in your laboratory so analysts will not notice a reduction in workspace.

With Intelligent Membrane Management (IMM) incorporated into the generator, the Infinity XE 60 is a more sustainable, environmentally friendly on-site gas generation solution, according to the company. The IMM adjusts the compressed air requirements based on gas consumption and target purity. By doing this, depending on the setup, IMM can help to reduce compressor run hours, which can increase the generator's service life while reducing the carbon footprint.

The Infinity XE 60 is designed to support multiple instruments and applications, including LC-MS, ELSD, NMR, FT-MS, and sample evaporators. By supporting multiple instruments, Peak has designed a nitrogen generator that can reportedly help analysts to reduce their laboratory's costs and environmental impact and provide a single solution for customer's nitrogen gas needs.

<https://www.peakscientific.com/products/nitrogen/infinity-xe-60xx-membrane-nitrogen-generator/>



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UHPLC System for Proteomics Analyses

Low-flow liquid chromatography (LC) users have typically faced challenges with insufficient robustness and reproducibility, in addition to complex workflows requiring significant experience and training. The Thermo Scientific Vanquish Neo Ultra-High Performance Liquid Chromatography (UHPLC) System provides biopharmaceutical, academic, and contract research laboratories performing proteomics analyses with



an all-in-one nano-, capillary-, and micro-flow LC system for high sensitivity LC–mass spectrometry (LC–MS) workflows that is easy to set up and delivers long-term robustness.

For ultimate flexibility and performance, the Vanquish Neo UHPLC System uses Thermo Scientific ProFlow XR active flow control pump technology to deliver excellent retention time precision from nano up to 100 $\mu\text{L}/\text{min}$ at pressures up to 1500 bar, according to the company. In addition, productivity is accelerated through fast sample loading and column equilibration, which improves sample throughput while enhancing MS utilization. To further meet the needs of laboratories performing 24/7 large sample cohort analysis, the company reports that maintenance-free 1500 bar-compatible ceramic valves and near-zero dead volume Thermo Scientific Viper and nanoViper fittings minimize leaks for long-term robust operation. The UHPLC system also features the Thermo Scientific Vanquish User Interface, which provides remote system monitoring and control with automated procedures and guided instrument maintenance, supporting users of all levels of expertise to get high-quality results.

www.thermofisher.com/vanquishneo

Nano-UHPLC System

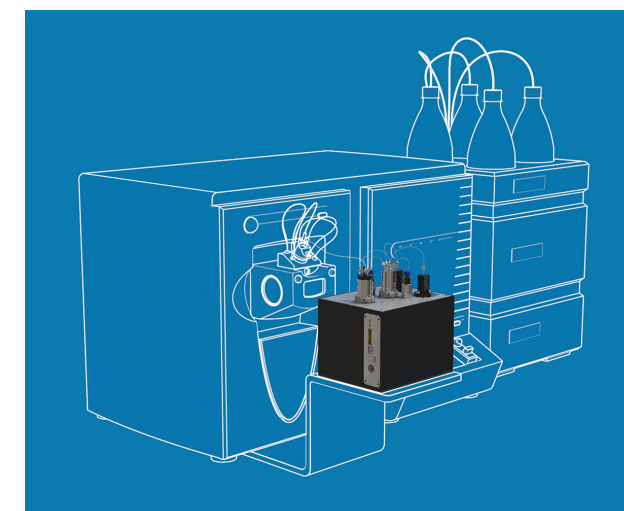
Traditional analytical laboratories have large instruments that take up a lot of space and resources. Thinking smaller and being smaller are linked with “greener” laboratories. Smaller instrumentation leads to smaller everything else: reagents, sample sizes, electricity usage, space requirements, and waste.

An entire chromatographic unit in just a few pounds, the True Nano UHPLC offers precision and flexibility with the industry-leading quality of VICI

components, according to the company. With True Nano 360 μm fittings, stable flow rates as low as 10 nL/min , and pressures up to 1500 bar, the system provides split-free injections, allowing direct, on-column detection.

The smaller fittings facilitate the increased back pressure and low flow rates. Columns packed with smaller particles allow for an increase in theoretical plate height, and short or no transfer lines with small diameters inside and outside of the system reduce dead volume, speeding up analyses and reducing band broadening. Available in varying pump configurations, VICI can help analysts configure a tool to help them meet their needs.

www.vici.com



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Analyte–Metal Surface Interactions

The Waters Acquity Premier System is a liquid chromatography (LC) system that, when paired with Acquity Premier Columns, is designed to alleviate the problem of analyte–metal surface interactions when analyzing metal-sensitive organic acids, organophosphates, oligonucleotides, phosphopeptides, acidic glycans, and phospholipids by reversed-phase and hydrophilic interaction chromatography.



The enabling technology behind both the Acquity Premier System and Acquity Premier Columns is MaxPeak HPS, a hybrid organic–inorganic surface technology, which forms a barrier between the sample and the metal surfaces of both the LC system and column. MaxPeak HPS eliminates the need for surface passivation, which can take days and cost thousands of dollars in wasted time, sample, and effort, according to the company.

The company reports that the Acquity Premier System reduces the time from sample to results and improves analyte recovery and assay-to-assay reproducibility to give separation scientists excellent assurance in the integrity of their qualitative and quantitative analytical results.

Acquity Premier System Product Page

UHPLC Column Hardware

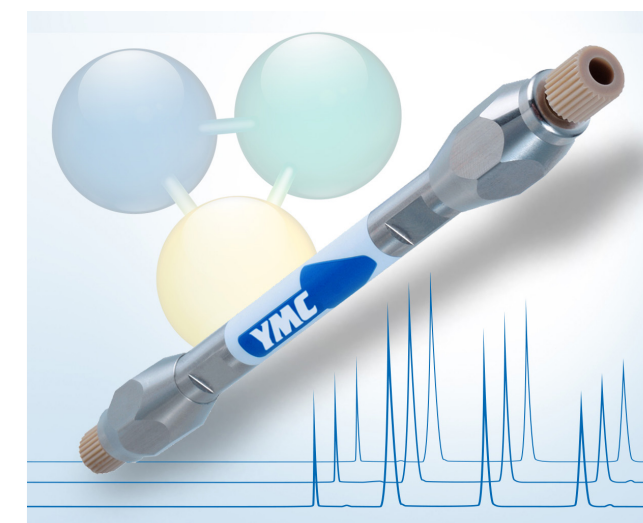
YMC-Triart UHPLC columns with 1 mm i.d. for LC–MS use extend the YMC-Triart portfolio! In combination with MS and a corresponding UHPLC system excellent sensitivities can be achieved with very low sample amounts/concentrations. These new 1 mm UHPLC columns fill the gap between YMC's MicroLC- and UHPLC columns.

According to the company, the reproducible hardware, which is particularly challenging for such small inner diameters, is accompanied by the excellent lot-to-lot reproducibility provided by the YMC-Triart stationary phases, ensuring truly reproducible results. As a result of the organic–inorganic hybrid silica base material, YMC-Triart stationary phases with 1.9- μm particle size are robust and flexible; a pH range of 1–12 as well as temperatures up to 90 °C can be used.

These 1 mm UHPLC columns are dedicated for laboratories that routinely use LC–MS and need to deal with very small concentrations where high sensitivities are important, for example, in pharmaceutical R&D, in academia, or in the environmental and food sectors.

Initially, 1 mm YMC-Triart C18 UHPLC columns are available and are suitable for a wide variety of small molecules, peptides, peptide mapping, or oligonucleotides.

<https://ymc.eu/d/brDmC>



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Sample Preparation Products

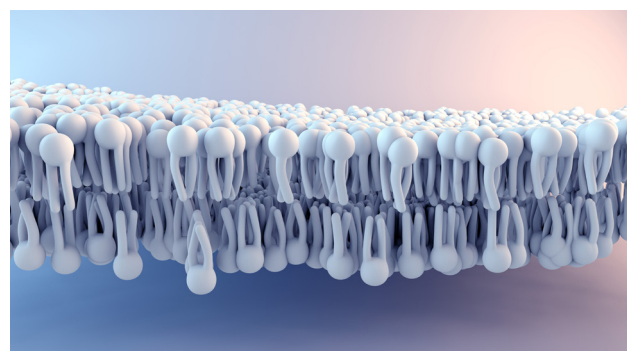
Phospholipid Removal Plate

Porvair Sciences has introduced a new and improved version of its Microlute PLR phospholipid removal microplate designed to enhance the flow of samples through a proprietary hydrophobic PTFE filter to maximize the recovery of analytes.

The Microlute PLR combines Porvair's expertise in filter/porous plastic manufacturing and microplate manufacturing. Unlike traditional loose-filled products, the solid interconnected network of evenly distributed pores in a Microlute PLR plate allows biological fluids to flow smoothly and consistently throughout the filter, according to the company. As a result, the microplate is able to efficiently remove a wide range of phospholipids from plasma and serum samples and increase the sensitivity and integrity of your UHPLC/HPLC methods.

Testing shows that the new plate offers greater than 99% removal, with high levels of recovery and reproducibility for a wide range of acidic, basic, or neutral analytes. The company reports that laboratory scientists will benefit from the easy-to-use Microlute PLR protocol, enabling them to remove phospholipids and recover analytes every time reliably and reproducibly. High-throughput studies will reportedly benefit from the reproducible, high performing sample preparation workflow of the Microlute PLR.

www.microplates.com/microlute-plr/



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Microlute® is the next generation range of composite polymer-based technologies that delivers on reliability and reproducibility of standard chromatography sample preparation methods.

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Microlute® SLE
for Supported Liquid Extraction

Microlute® PPP
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Software Products

Environmental Analysis Sorbent

Analysis of extractable petroleum hydrocarbon (EPH) is important to assess the risk posed by petroleum products in the environment. Solid-phase extraction (SPE) with heat-treated silica gel is the traditional approach for the fractionation of aliphatic and aromatic hydrocarbons. Deactivation of the hygroscopic silica gel often leads to inconsistent results, incomplete separation, and low recoveries. UCT's Enviro-Clean Fusion Ag⁺ is a novel sorbent designed to help overcome these challenges. The sorbent consists of silver ions anchored onto solid support that provides optimized selectivity, capacity, and performance. Aromatic hydrocarbons form a charge-transfer complex, with silver ions ensuring complete separation of aliphatic from aromatic fractions without a breakthrough. Fusion Ag⁺ requires less solvent consumption and reduced extraction times when compared to silica cartridges with a similar configuration. Also, using acetone instead of dichloromethane as the elution solvent for the aromatic fraction is environmentally friendly. The company reports that it offers accurate testing results with superior reproducibility. Recoveries were primarily in the 80 to 110% range with a relative standard deviation (RSD) less than 10%. Sorbent batch-to-batch consistency was ensured by rigorous QC testing. This product is available in three configurations; 500 mg, 1 g, and 2 g sorbent packed in a 6-mL cartridge.

<https://www.unitedchem.com/product-category/spe/environmental/enviro-clean-fusionag>

Software-Assisted Design Space Modeling

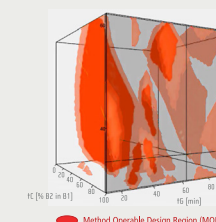
Design space modeling has supported industry practitioners in their efforts for a fast and comprehensive understanding of chromatographic challenges for a long time. More recently, design space modeling also finds application as a tool for the qualified characterization and comparison of separation systems. If—for instance—instrumentation, sample, and method conditions remain identical, a direct comparison of the design spaces of different columns can be made and the differences between the stationary phases can be evaluated holistically. Following that approach, DryLab Design Space Modeling also yields the knowledge and scientific understanding necessary to move existing applications to new hardware or to redesign analytical methods altogether.

DryLab models method operable design regions (MODRs) for them to comprise multi-attribute method target profiles (MTPs). The resulting design spaces then include critical method attributes for one or more peaks of interest. The attached figure shows the MODR within a design space as a combination of red, three-dimensional bodies. The red areas represent method conditions where the pre-defined critical method attributes are met—attributes such as overall critical resolution, but also peak specific characteristics such as minimum resolution, retention time windows, peak asymmetries, and peak widths.

Design space modeling has become an important pillar of analytical operations—and it can be expected to become increasingly relevant in a data-driven industry 4.0.

empower.drylab.com

Multi-Attribute MODRs



DryLab Design Spaces model multiple, peak specific Critical Method Attributes such as

- Overall Critical Resolution
- Minimum Resolution of Peak(s)
- Peak Retention Time Ranges
- Peak Asymmetries
- Peak Widths

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Chromatography—It's Just About Following the Recipe, Right?

A great chromatographer does more than simply follow the method.

I have heard analytical science and the chromatographic disciplines compared to “cookery” or “recipe following” so many times during my career. Frankly, these references have always been slightly disparaging, perhaps meant to demean the degree of knowledge and skills that are required to become a chromatographer. As a career chromatographer, it goes without saying that I disagree with this perspective in the main.

I've also had a lifelong fascination with fine cuisine and restaurant kitchens. The art and science of producing good food is fascinating, and in the professional kitchen, just as in a chromatography laboratory, knowledge, skills, and experience are combined to produce, at the very least, fit for purpose results. Professional chefs do not follow recipes, but they do take guidance from their creative leader or executive chef. There is an enormous amount of discipline and care required to

consistently produce the best results, the most exquisite food. There are a number of skills that must be mastered and built into “muscle memory”, from basic food preparation to fundamental cooking techniques. There is equipment—often quite complex equipment—that must be learned and mastered to produce the best results. It is said that a new range or stove takes at least a month to produce consistently good results, and that even simple changes, such as a new make of saucepan with a different material of construction or thickness of base, must be “learned” over time. But of course, there is also “art”, the combination of basic skills, techniques, and equipment, a passion and a palette for good food, and a mastery of the combination of ingredients, flavours, visual acuity, and production techniques, all needed to produce the perfect dish. Is this really so different from the skills, passion, and flair that is required

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to become a good chromatographer? I really don't think there is so much of a gap. To say that we are "recipe followers" would be like saying Escoffier was a "cook", clearly incorrect. A well performing kitchen has roles and responsibilities that can be mapped very closely to a high-end restaurant kitchen, and indeed beyond the kitchen and into the dining room. It's worth a few column inches to compare the professional kitchen to the laboratory to give us some further perspective on how teamwork and the vital aspects of each role can combine to produce either beautiful food or beautiful separations!

Georges Auguste Escoffier (1846–1935) was a French chef, restaurateur, and writer, who popularized French cooking techniques by simplifying and modernizing styles codified by Marie-Antoine Carême (1784–1833), an early practitioner of the French *haute cuisine* school. He published the seminal work *Le Guide Culinaire* and, together with César Ritz, rose to prominence at the London Savoy Hotel, as well as the Ritz Hotel in Paris. Escoffier elevated the chef's profession by demanding cleanliness, discipline, and silence in the kitchen, drawing on his military experience to develop the *brigade de cuisine* system, a hierarchical system of management and team working still in evidence in many of the world's best kitchens to this day. The various roles within this system can be mapped to those

of the analytical laboratory, the interactions within the team and the requirements for each role bearing an interesting comparison with those in our own working environment.

Garçon de cuisine (Kitchen porter) is the foundational level of the *brigade de cuisine*, receiving deliveries, performing the most basic preparatory tasks such as trimming vegetables, and ensuring all ingredients are available at the correct cooking "stations". It will take no imagination to translate these activities to those of the "technician" or "stores" person in the laboratory. Seemingly a lower ranking job, anyone working in a laboratory will know that without the correct reagents and consumables to hand, the laboratory quickly grinds to a halt. It is to this person that we often turn when deadlines are tight or new supplies or a new consumable are required, as they hold the fundamental relationships between the laboratory and our suppliers and are key to ensuring that the chemist is correctly provisioned. It is worthy of note that whilst the *Garçon de cuisine* was rarely formally trained, many have, through hard work and the development of empirical skills, risen to become great chefs.

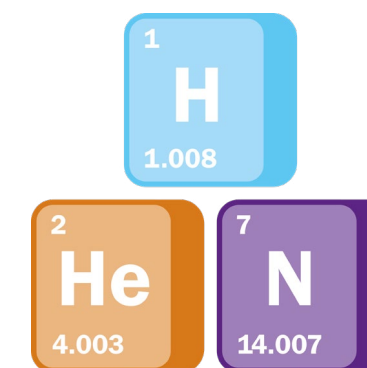
Plongeur (dishwasher) and *marmiton* (pot washer) are the backbone of the kitchen brigade system. Without the requisite plates and dishes, cleaned to a highly professional standard, no service to

the table will occur. The plates and dishes are the blank canvas onto which the various chefs will apply their artistry. Without the required cooking vessels and implements, no cooking can be performed. Well, I think we all know what can happen if our laboratory glassware is not cleaned to exacting standards, and one might also imagine that the requirement for our laboratory equipment to perform correctly is also the responsibility of the instrument technicians or metrology department.

Kitchens are typically arranged into "stations" or "sections", each of which oversee various different food types or dishes. These various sections will all have a *chef de partie* (senior station chef), *demi chef* (deputy to the *chef de partie*), and *commis chef* (under chef of the station, usually a trainee) according to seniority and experience. These roles will do everything from preparation of the basic foodstuffs to turning out the final product from that section. It is typical in many laboratories to have specialists within the various chromatographic disciplines of liquid chromatography (LC), gas chromatography (GC), and spectroscopy. It is also not unusual to have various levels of expertise within these sections, all working together to produce high quality analytical data, from the production of extracts and sample solutions to the final acquired and analyzed data.

Give your lab a massive boost

Multi-Gas thermal desorbers



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Apropos of nothing other than a romantic fascination with the brigade system, and to highlight the number of specialities within the kitchen brigade, here are some of the various stations that one might encounter in a larger professional restaurant:

- Sauté chef (*saucier*)—produces sautéed items and their sauces. Often the most respected position of the stations, this critical role will also often oversee the quality and presentation of the food as it transits the “pass” between the kitchen and the dining room.
- Fish chef (*poissonnier*)—undertakes fish butchering and the production of fish dishes.
- Roast chef (*rôtisseur*)—responsible for roasted and braised meats and butchering.
- Grill chef (*grillardin*)—produces all grilled foods.
- Fry chef (*friturier*)—responsible for fried items.
- Entrée preparer (*entremetier*)—produces hot appetizers and soups, vegetables, pastas, and starches.
- Soup chef (*potager*)—produces soups and cold sauces and works closely with the sauté chef.
- Pantry chef (*garde manger*)—manages cold foods, salads, cold appetizers, pâtés, and other charcuterie items.
- Pastry chef (*pâtissier*)—responsible for baked goods: pastries, cakes, breads, and desserts.

As a *commis chef*, one will learn to prepare the fresh ingredients and the rudiments of good cooking. The *demi chef* will be well established after several years of experience and will be doing the majority of the cooking, whilst the *chef de partie* will be finishing dishes and ensuring good quality and timeliness. The position of sauté chef will often be the pinnacle of a chef’s career, taking years to master and constantly evolving to reach new heights. Again, it takes no real explanation from me that the necessity for the study and mastery of the basics followed by many years of experience building to a point where one can recognize what is good and what is below par is perfectly aligned with the various chromatographic specialisms.

Also worthy of comparison is the role of the sauté chef and our colleagues in quality assurance. The sauté chef will inspect each of the dishes as they move out of the kitchen, ensuring that the presentation and taste meet the required standards—not too different from the compliance and assurance role within a laboratory organization.

Three further *brigade de cuisine* roles that are worthy of special note are the *tournant*, the *aboyeur*, and the *chef de cuisine*.

The *tournant* is the spare, but very safe, pair of hands who can float within the kitchen and lend a hand at whichever station is under pressure. These chefs are typically



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long serving and skilled at many, or all, of the station specialisms. As such, they are an invaluable resource and often the glue that holds together a successful service. I'm sure we all know a few laboratory *tournants*, without whom our laboratory organization would cease to function in an efficient manner!

The *aboyeur* controls the flow and timing of food from the kitchen and is the link between the front of house and kitchen staff. They ensure dishes for each table come to the pass at the same time and flow out to the dining room as quickly and harmoniously as possible. This often means close liaison with the *chefs de partie* to ensure timing and coordination. I liken these folks to a good project manager who can assess where pinch points may occur and ensure that sufficient resources are allocated to various departments to avoid critical time delays. A good laboratory *aboyeur*, or project manager as we might know them, can be worth their weight in gold, keeping the various aspects of sample preparation, data acquisition, and analysis flowing towards the final report.

Finally, the *chef de cuisine* (or executive chef in very large organizations) is responsible, along with other team members and suppliers, for creating new dishes and setting standards in the kitchen. They are the creative mastermind, inspiring new dishes and ideas that the *chefs de partie* will help to create.

It is usually their name above the door. They will also liaise with restaurant owners as well as formally managing the whole kitchen team. This is the typical laboratory manager role, and the responsibility for new method development or method improvement lies with them, as well as the role of formal people management and reporting out to other departments. A *chef de cuisine* is typically highly creative, absolutely focused on quality, and, although not always on the face it, fiercely protective of their workforce. Does that describe your own *chef de cuisine*?

The introduction of the brigade system saw a paradigm shift in the restaurant business. This team enabled the introduction of à la carte dining as we know it today, enabling a wide range of dishes to be offered and, no matter the diner's choices, to arrive at the table at the same time. The modern laboratory also strives to bring together various types of analytical data to present a high-quality product and enable the wider organization or client to make informed choices regarding their product or development decision or their treatment options.

What is true is that none of these folks follow a step-by-step recipe, instead, using their skills, experience, and the guidance of the *chef de cuisine*, they produce dishes that are exactly to the specification. Whilst we may have more formal recipes, such as

protocols, standards operating procedures (SOPs), or laboratory instructions, we can also act as a brigade, with the various functions working together to produce a quality product at the required time, time after time. But one thing must be realized, the production of a good chromatographer, just like a good chef, requires time and patience to master the basics, develop your craft, and eventually create wonderful new methods and approaches. There is no shortcut and you must seek mentors who can teach and inspire you. If you don't have the patience, if your *chef de partie* is unwilling to show you the hands-on essentials, or your *chef de cuisine* is uninspiring, it may be time to find a new brigade!

By all means follow the recipe. Let's face it, the production of consistent medicines is a little more important than the production of consistent steaks, but always try to retain a desire to produce a perfectly cooked, beautifully presented, piece of data.

Just one further piece of advice, don't be tempted to taste test as you go—just learn to know, as any good grillardin will tell you, when something is over- or undercooked!!

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Expanding Capacity in Gas Chromatography Columns

Chris English, Restek, Bellefonte, Pennsylvania, USA

My previous article for *Practical GC* in the September issue of *The Column* focused on compound capacities of three different stationary phases using a wide range of compound polarities to determine overload as a change in symmetry. How much sample can be put on a column was addressed for the 0.53 mm internal diameter (wide-bore) columns. Since capacity is the maximum amount of sample on a column before peak distortion occurs and is directly proportional to column dimensions, this article will expand on this evaluation to include narrow-bore and microbore columns with 0.25 mm, 0.18 mm, and 0.10 mm column internal diameters.

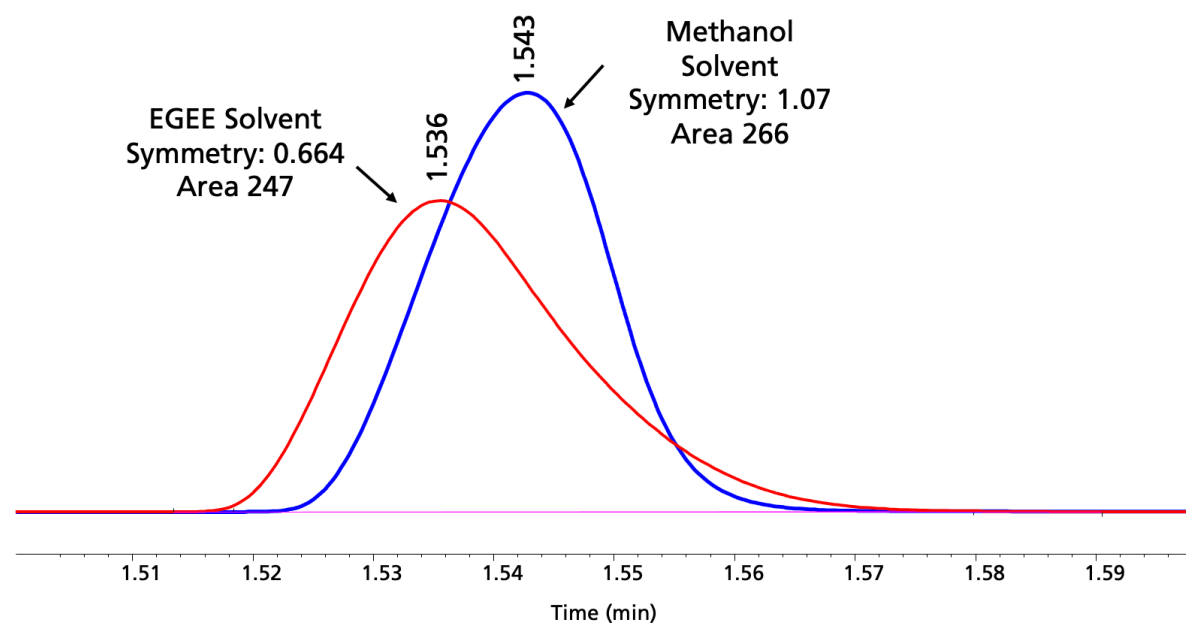
Too much sample on a column results in retention time shifts and poor resolution of closely eluting analytes. Currently, there is no agreed upon peak shape that defines overloading in specific terms (1,2). We chose symmetry values where an imperceptible but measurable amount of fronting occurs, that is, a symmetry value equal to or greater than 1.05. To determine slight changes in peak fronting (overload), Gaussian peak shapes are required because tailing peaks bias the

results. In our previous article we found that capacity for a 0.53 mm internal diameter (i.d.) column on three different phases ranges from 1 ng on-column to 2000 ng on-column (3). Compounds with poor solubility in a stationary phase overloaded at low concentrations, and using thin film columns increased solvent effects for specific compounds (4,5). In this article we will evaluate other column internal diameters and use other solvents to determine capacity.



Figure 1: An alternative solvent was chosen to investigate peak tailing with several compounds. Split ratios were 100:1 with EGEE and 200:1 with methanol. Peak distortion prevented an accurate measurement of acetonitrile using EGEE as a solvent.

1701-Type Stationary Phase 20 m x 0.18 mm x 0.18- μ m Comparing Two Different Solvents with Acetonitrile at 50ng On-Column Concentration



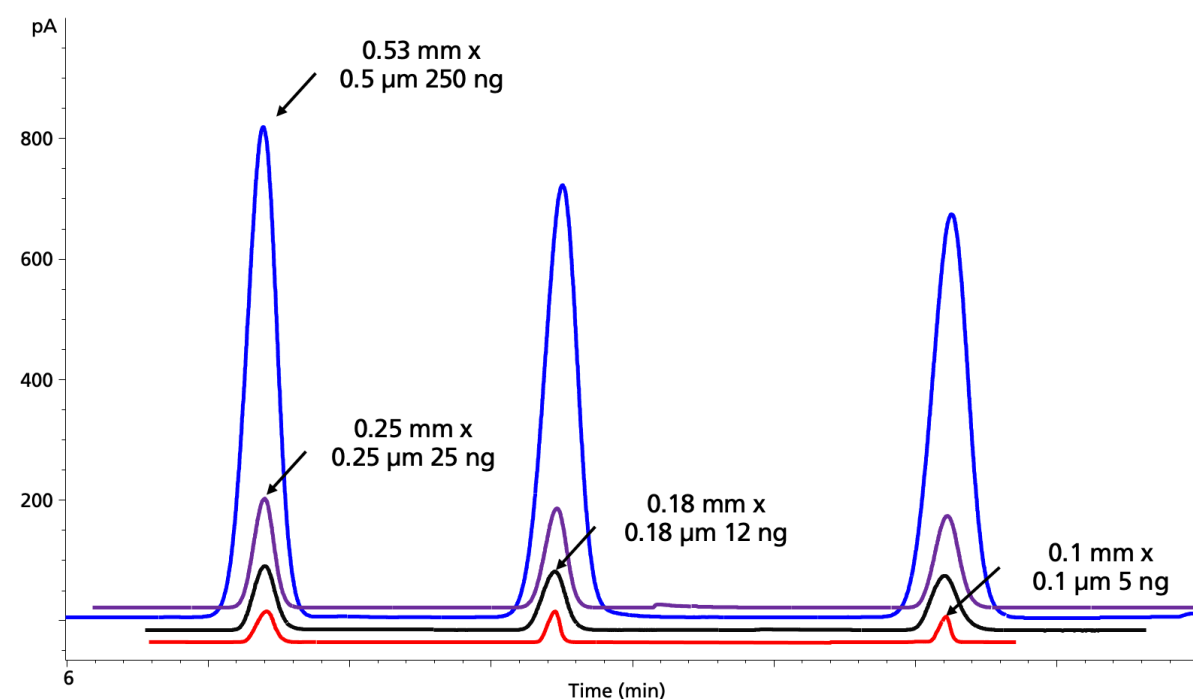
Experimental Design

The same three phases in our previous work were chosen: 100% polydimethylsiloxane (PDMS) (1-type phase), polyethylene glycol (PEG) (wax phase), and a 14% cyanopropylphenyl–86% polydimethylsiloxane phase (1701-type phase) (3). In addition to the 30 m \times 0.53 mm, 0.5- μ m d_f column, three other dimensions were evaluated: 30 m \times 0.25 mm, 0.25- μ m d_f , 20 m \times 0.18 mm,

0.18- μ m d_f , and 10 m \times 0.10 mm, 0.1- μ m d_f (all Restek). The column dimensions are common stock items and have similar phase ratios (β values). Our choice of compounds is designed to cover highly polar (acetic acid), polar (1-butanol), mid-polarity (butanal, 2-butanone, acetonitrile, and chloroform), and nonpolar (benzene, cycloheptane, octane, octene, and octyne). Using data from previous work, we chose 2-ethoxyethanol

Figure 2: Capacity for the C8 compounds on the four column dimensions evaluated. Retention times were different between columns, and chromatograms were stretched and stacked for peak height comparison purposes.

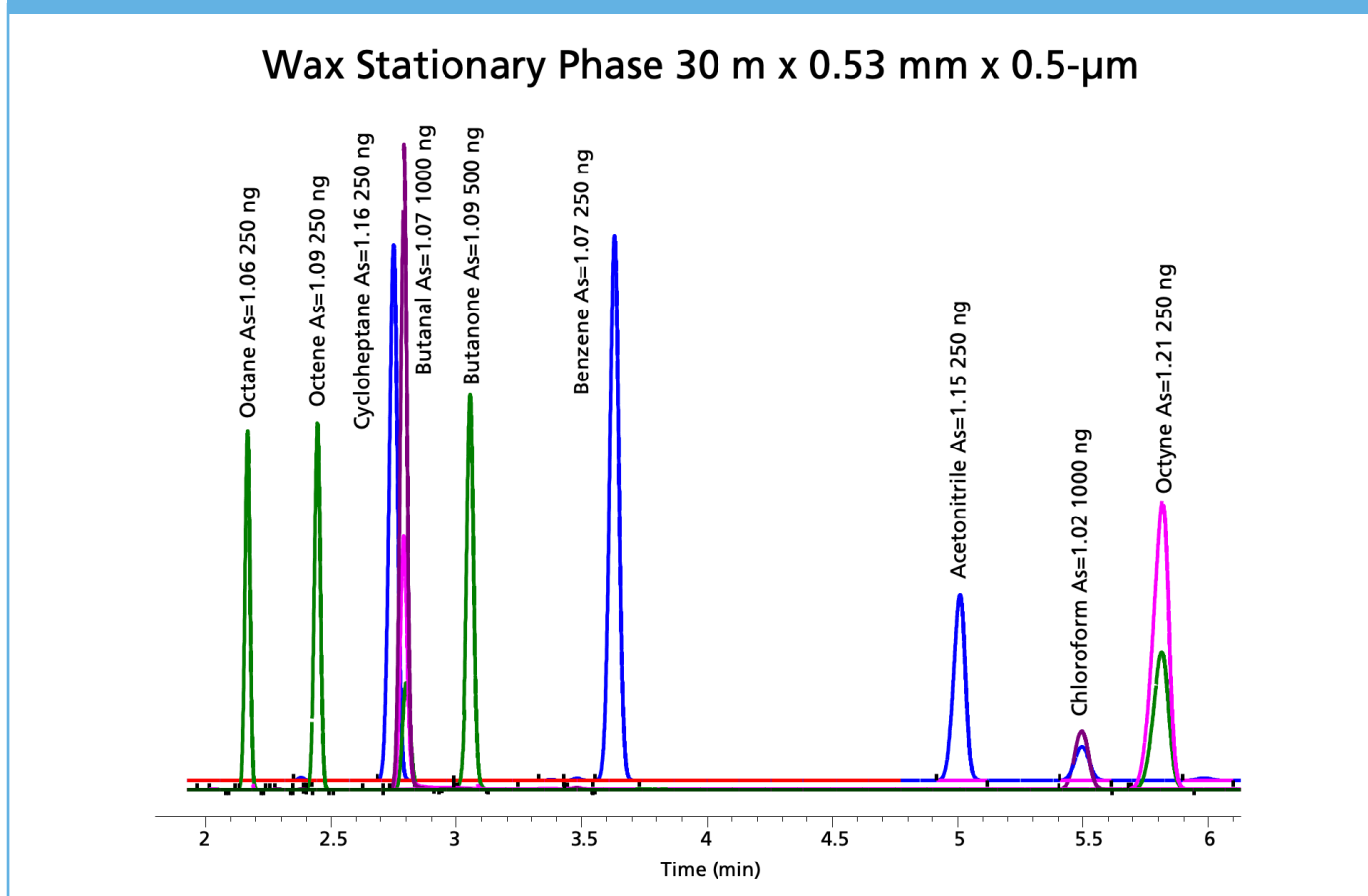
Octane, Octene and Octyne Capacity for 1-Type Stationary Phases



(EGEE) as a solvent with split rates of 1:25 or higher and evaluated methanol as a solvent with high concentration standards (10,000 ppm) to minimize the impact on peak shape. The 2-ethoxyethanol exhibits excellent solubility for a wide range of compounds. All compounds were added to this solvent, except acetic acid, which was dissolved in water. Oven conditions started at 40 $^{\circ}$ C with a hold time of 10 min, followed by a 30 $^{\circ}$ C/

min to 330 $^{\circ}$ C, with a final hold of 2 min. Compounds eluted during the hold time, allowing a more controlled comparison. Six split ratios of 25:1, 50:1, 100:1, 200:1, 400:1, and 500:1 were used for the three narrow-bore columns. Sample concentrations were adjusted from 100 parts per million (ppm) to 15,000 ppm, and on-column concentrations ranged from 1 to 500 ppm. Capacity experiments were run on a flame

Figure 3: Capacity for specific compounds on a typical wax phase, which range from 125 ng on-column to 1000 ng on-column. Chloroform did not saturate at 1000 ng on-column and butanol (not shown) exhibited tailing, which prevented an accurate capacity measurement.



ionization detector (FID) and samples were analyzed at the same concentration on-column using different splits and starting concentrations, in addition to methanol standards for specific compounds to verify performance (Figure 1).

Carboxylic Acid Analysis: Analyzing acetic acid required water as the solvent, different concentrations, and a change in the wax column starting temperature to elute

acetic acid in a reasonable amount of time. Propionic acid was used for the evaluation of the 0.53 mm column, but was dropped for subsequent work since its performance mirrored acetic acid. Oven conditions for the wax columns were adjusted to 120 °C isothermal, with a final bakeout to 230 °C, while the 1701 and 1-type columns remained at 40 °C. Final on-column concentrations for acetic acid ranged from 0.25 to 20 ppm.

Data Analysis: We defined an overloaded analyte as one with an Agilent Chemstation symmetry value of 1.05 or greater.

Chemstation values are sensitive because they are not measured at 5% or 10% peak height and, therefore, they do not inversely correlate to USP or FDA asymmetry calculations.

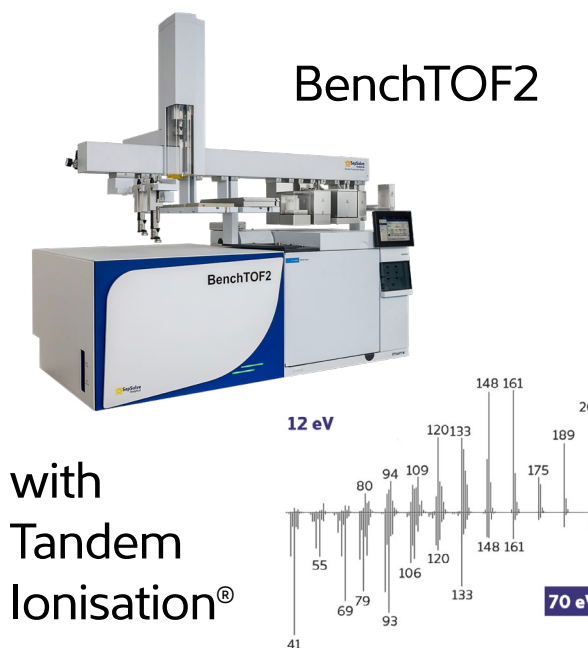
Results and Discussion

One of our goals in expanding our work to include the narrow-bore columns was to address peak shape issues with compounds such as chloroform, butanol, and acetonitrile. Tailing analytes significantly increased the area for the second half of the peak, which made capacity calculations using symmetry impossible. For compounds that are soluble in methanol, a standard was made at a concentration of 10,000 ppm, thereby allowing a high split ratio. In Figure 1, this alternate solvent was chosen to evaluate acetonitrile using a 200:1 split with 50 ng on-column concentration. Interestingly, 2-ethoxyethanol as a solvent produced a tailing peak using a split ratio of 100:1 for the same final concentration on-column. Peak areas for acetonitrile were within 10% between the two solvents and there was a noticeable shift in retention not associated with column overload and most likely a solvent effect (3), since the EGEE solvent wets the stationary phase and evaporates from



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the head of the column after acetonitrile. This means acetonitrile interacts with the stationary phase and the EGEE solvent, whereas methanol would evaporate before acetonitrile moves down the column. Unfortunately, chloroform and butanol exhibited peak distortion and not classical fronting, and therefore accurate column capacity values could not be determined. Future work with a thicker film column may reduce the solvent effects and allow a more accurate measure of capacity. Acetic acid could be detected at 0.25 ng on-column using a 1-type phase in the 0.1-mm i.d. column, but peak shapes for this compound were generally poor on the three phases evaluated. Nonpolar analytes, such as octane, octene, and octyne, exhibited classical fronting peaks and were easily measured on the three phases. For example, Figure 2 shows the capacity and concentrations for these three compounds on the four different

column formats. Figure 3 is a collection of chromatograms overlaid with nine of the 11 compounds presented, with concentrations ranging from 125 to 1000 ng on-column using the wax-type stationary phase in the 0.53-mm i.d. format. Chloroform was not saturated and butanol exhibited tailing, which prevented an accurate capacity measurement.

Conclusions

Ranges for peak capacity are based on the solubility of the analyte in the stationary phase. Results from evaluating the 1-type phase using the four column formats ranged from 0.25 to 2000 ng on-column and the 1701-type phase ranged from 2 to 2000 ng on-column. Figure 3 represents the high end of the capacity range for the wax-type phase; when using the 0.1 mm i.d. format, compounds exhibited distorted or fronting peak shapes at 5 ng on-column. Surprisingly, solvents even at high split ratios impacted

peak shape, as shown in Figure 1. Nonpolar compounds, such as benzene, cycloheptane, octane, octene, and octyne, produced classical fronting peaks and were easily measured on all column phases and formats. Future work with different solvents and thicker film columns may increase our ability to determine overloading of intermediate and more polar analytes using symmetry calculations.

Acknowledgement

Special thanks to Jaap de Zeeuw and Christopher Rattray (Restek) for their technical advice and review.

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Chris English has managed a team of chemists in Restek's innovations laboratory since 2004. Before taking the reins of the laboratory, he spent seven years as an environmental chemist and was critical to the development of Restek's current line of volatile GC columns. Prior to joining Restek, he operated a variety of gas chromatographic detectors conducting method development and sample analysis. Chris holds a B.S. in environmental science from Saint Michael's College, USA.

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IMSC2022 Event Preview

The International Mass Spectrometry Conference (IMSC2022) will be held 27 August–2 September 2022 in Maastricht, The Netherlands. Here is a sneak preview of what to look forward to.

The 23rd International Mass Spectrometry Conference (IMSC2022) will take place from **27 August to 2 September 2022** in The Netherlands, in the beautiful town of Maastricht. It is the largest conference on mass spectrometry in Europe, attracting between 1500 and 2000 participants active in science, industry, and societal institutions from all over the world. IMSC is organized once every two years by the International Mass Spectrometry Foundation (IMSF), with its location alternating between Europe and other continents.

The aim of the 2022 IMSC conference will be to enhance interaction between both the topics covered by mass spectrometry (MS) and between the different generations of scientists within the global community. The main programme is organized around five parallel main sessions, with over 40 topics across five conference days, covering

fields such as fundamentals, proteomics, imaging, metabolomics, ion spectroscopy, forensics, structural biology, geology, clinical applications, and food and doping control. Workshops, tutorials, and vendor sessions will be organized prior to and during the conference, and there will be abundant time and space for informal discussions and meetings in a spacious central area within the conference centre.

It is the second time that the IMSC will be held in The Netherlands, after the successful 12th conference in 1991 in Amsterdam. The Dutch Society for Mass Spectrometry organizes the conference with a local organizing committee that represents a well-connected and active community from universities, knowledge institutes, and industries. “We are delighted to organize IMSC 2022 in Maastricht, bringing together the global mass spectrometry community in all its diversity,” said chair of the conference

Nikolai Siroki/istockphoto.com



Professor Ron Heeren of Maastricht University and M4I, the Maastricht MultiModal Molecular Imaging Institute. “Especially after such a long period of restrictions, leading to the unfortunate cancellation of IMSC 2020 in Rio, everybody is looking forward to meeting in person again, and all informal exchange of ideas and social interactions that make our community what it is today.”

Professor Albert Heck of Utrecht University and the Netherlands Proteomics Centre and Professor Manfred Wuhrer of Leiden University Medical Centre and Chair of the Dutch Society for Mass Spectrometry are co-chairs of the conference. Along with Heeren, these strong proponents of mass spectrometry in various fields of application will drive the organization of the conference, embedding it securely in the Dutch mass spectrometry community and promoting it through their extensive international networks. The Dutch Society for Mass Spectrometry is an experienced and active organization, organizing national meetings twice a year, covering a wide range of topics and offering a platform for young scientists and new fields.

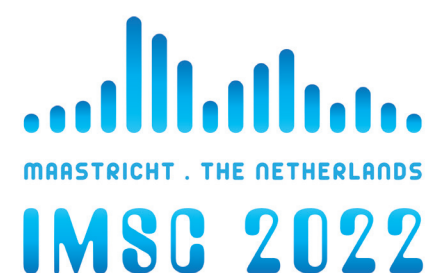
Although a small country, The Netherlands has a long tradition and a very active community in mass spectrometry, reflected by supposedly the highest density of mass spectrometers in the world. Maastricht was selected as the conference venue, not only for

its attractive and enjoyable atmosphere with history, art, and culture but also because of its central location in the heart of Europe, together with the excellent Maastricht Exhibition and Conference Centre (MECC), which is located within walking distance of ample comfortable and affordable hotel accommodation.

Maastricht is one of the oldest cities in The Netherlands, as you will discover when strolling through the historic inner city. You can discover the entire city centre on foot. Churches, city walls, monumental merchant houses, and big squares merge seamlessly, with a comprehensive and varied range of shops. Maastricht is an attractive city, visited by over 3 million tourists every year. It is traditionally a city of students, with its internationally focused university and medical centre. Maastricht is also a city of indulgence and culinary highlights. There will be ample opportunity to appreciate the picturesque surroundings and enjoy high-quality food in a relaxed atmosphere. Conference attendees can enjoy the beautiful nature and castles outside the city boundaries on foot, by bike, or by motorized tour. Maastricht houses a top-notch conference centre, which has hosted conferences of comparable size to the IMSC. The city has adequate hotel facilities in prices ranging from 35 Euro up to 220 Euro—all in close proximity to the conference centre. Maastricht is within easy reach from various

international airports, allowing easy access from across the globe. Moreover, Maastricht is only 2.5 h by train from the centre of Amsterdam.

For more information on the conference, and for registration and abstract submission, visit the website at imsc2022.com



Conference Chairs: Prof. Manfred Wuhrer, Prof. Ron Heeren, and Prof. Albert Heck

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The *LCGC* Blog: Don't Fear the Automation

Jonathan Shackman, Bristol Myers Squibb, New Jersey, USA

With modern chromatography modelling software, is it time to hang up our laboratory coats? Is resistance to software solving separations futile?

We live in a world with ever-increasing automation, from the simple (hello automatic coffee maker!) to the complex (I think I'll just let the car do the driving...or at least the parking). The pervasive, almost intelligent machines have seemingly always been a part of an analytical chemist's world. Are we ready to let programs and instruments take over the art of separations?

High Throughput on a 400 MHz Processor

Some of my favourite research time in Prof. Robert T. Kennedy's laboratory as a graduate student was spent writing programs to automate and speed up data collection and processing. For the most part, these activities did not make it into my thesis, but I sincerely believe they were

integral to my own experiments, as well as accelerating the research of others' projects. One particular program I worked on for many months (years?) was a means for rapid batch processing of the tens to thousands of electropherograms we were generating on a daily basis in the group. This was a tribute to the high-speed capillary electrophoresis being performed in Bob's laboratory. However, performing peak integrations serially and manually by poor grad students resulted in a painful bottleneck in terms of converting raw data to useful knowledge. With some additional features, such as peak deconvolution and statistical moment analysis, we ultimately published this work as "High-throughput automated post-processing of separation data" (1). The automated analysis was rapidly adopted and allowed the scientists to not only assess their data faster but also



to redirect their reclaimed time to more productive endeavours. I'm fairly certain that none of the students or postdocs felt they would be replaced by this program; it simply allowed scientists to focus on more interesting ideas and challenges. Indeed, the program is still used in the laboratory today, and, based on citations, is employed in other research laboratories, which is an interesting feat for a program whose metrics were benchmarked on a 400 MHz Pentium II computer.

All Aboard the *In Silico* Train

Upon joining Bristol Myers Squibb (BMS), I was introduced to some very cool software programs to aid in liquid chromatography (LC) method development, mainly in the achiral reversed-phase LC domain, such as the Molnar Institute's DryLab, S-Matrix's Fusion, and ACD's LC Simulator, to name a few. With only a couple of empirical LC runs, these modelling programs could map out predicted separations *in silico* across a wide design space. With a click of a button, we could potentially let the program "choose the optimum" separation, which we would then quickly discard. Why? Mainly because the programs at the time did not know what we humans really wanted. For example,

it was difficult to specify our needs, such as: only make gradient changes in this region; allow a step gradient here; keep the retention factors between 1 and 20; all while maintaining baseline resolution in under 5 min runtime...with re-equilibration. Usually, we were faster at defining these boundary conditions in our heads, mapping it into the program's gradient, and seeing what popped out visually, followed by empirical verification. However, these programs are making significant and rapid evolutions, with: better optimal design optionality; more dimensionality by increasing from one dimension (for example, gradient), to two (such as add-on temperature), to three (for example, add in pH); more separation modes to be able to model non-reversed-phase separations with the same facility as reversed-phase LC; and back-end analysis of robustness, sometimes with pre-templated reports. Regardless, the human has traditionally always been the conductor of the *in silico* express train, picking the input packages, keeping them on the rails, and deciding on the final destination. Sometimes wholly iterative method development will arrive at the same place as the modelling, albeit at a slower pace. However, sometimes the modelling program can reveal better-operating spaces, while your

iterative work only finds a locally ideal space that is not globally optimal. In this case, the automation not only removes the labour-intensive grunt work but also yields a better end-product that could save additional time down the road by being less prone to failure.

Speed Through Screens

We need a starting point for our separation before we can start modelling it. We could grab our personal favourite C18–mobile-phase combination or use a generic platform method (as fellow columnist Tony Taylor puts it, the potluck supper of analytical chemistry) (2) to see if there's a hit. Alternatively, we can use some sort of screening system, which could be as simple as a couple of columns and solvents, or quite exhaustive. Indeed, at BMS we have a comprehensive reversed-phase LC system capable of automatically running well over 100 conditions based on the MeDuSA paradigm (3). In these cases, automation results in single-button data acquisition with an overnight run. Although processing can be automated to some degree, at this stage data interpretation can be the primary bottleneck. Some efforts have been made in current commercial chromatography data systems (frequently as an add-on purchase) to facilitate this

evaluation, but it is still up to the human to decide what "optimal" actually means. Do we prioritize peak count? Efficiency? Resolution? Tailing factors? Does the answer change if I tell you the sample has 20 components but only 10 are relevant? Similar to the modelling case, the scientist is accountable for picking the right samples to inject, determining the results' relevancy, and usually defining the ultimate final separation conditions. Additionally, in the absence of human–data engagement, potential critical situations may be missed, such as a clean sample turning up a new unknown during screening (those pesky peak shoulders!). The bottom line is that my fellow scientists and I are extremely grateful we don't have to serially churn through setting up all these conditions, which gives us the time back to delve deeper into the data or work on other, more rewarding activities.

The Art of Chromatography

A more recent LC method automation system available today is feedback optimized screening, such as by ACD's Autochrom. In this implementation, we let the software choose the best screening condition, but also allow it to do its own final optimization on that condition through an automated modelling exercise.



Will this perfect marriage of screening and modelling result in the bench scientists hanging up their lab coats? I hope the previous paragraphs have already convinced you otherwise. Beyond the above arguments, there are just too many separation problems that require serious intervention beyond what current systems can handle. If my automated system relies on mass spectrometry to track peaks, what will it do when I need to do low UV detection (I'm still waiting for the perfect low wavelength cutoff and volatile buffer). What if the optimal condition is missed because of chelation effects that I could mitigate with a mobile-phase additive? Buffer concentration? Ionic strength? Alternative ion pairs? Uncommon columns? On-column degradation? New technology? Lest we forget, the human

is also in the driver's seat for proper sample preparation (it's more than just a separation!). We are both blessed and cursed to have so many selectivity levers in LC separations. There's still plenty of opportunity for artistry by those who study the field. Let automation give you the freedom to explore the many less-common ways of doing your separations. You may just find the next big thing.

This blog is a collaboration between LCGC and the American Chemical Society Analytical Division Subdivision on chromatography and separations chemistry.

This month's blog is dedicated to Christopher J. Watson, co-author of the first reference. Chris was an amazing friend, scientist, father, and so much

more, who was taken far too soon from this Earth.

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Jonathan Shackman is Associate Scientific Director in the Chemical Process Development department at Bristol Myers Squibb (BMS) and is based in New Jersey, USA. He earned his two B.S. degrees at the University of Arizona and his Ph.D. in chemistry from the University of Michigan

under the direction of Prof. Robert T. Kennedy. Prior to joining BMS, he held a National Research Council position at the National Institute of Standards and Technology (NIST) and was a professor of chemistry at Temple University in Philadelphia, Pennsylvania, USA. To date, he has authored more than 30 manuscripts and two book chapters. He has presented more than 30 oral or poster presentations and holds one patent in the field of separation science. Jonathan has proudly served on the executive board of the ACS Subdivision on Chromatography and Separations Chemistry (SCSC) for two terms.

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Website: www.chromacademy.com/channels/gc-training-courses/troubleshooting/gc-troubleshooter

GC Fundamentals

Website: www.crawfordscientific.com/training-consultancy/gc-training/gc-fundamentals

Applied Method Development for GC and GC–MS

19 January 2022

Online—virtual classroom

Website: www.anthias.co.uk/online-training-courses/gc-and-gc-ms-method-development

HPLC/LC–MS

HPLC Fundamentals

Onsite training

Website: www.crawfordscientific.com/training-consultancy/hplc-training/hplc-fundamentals

HPLC Troubleshooter

Website: www.chromacademy.com/channels/hplc-training-courses/troubleshooting/hplc-troubleshooter

Fundamental LC–MS

Website: www.chromacademy.com/channels/lc-ms/principles/fundamentals-of-lc-ms-video-training-course

LC–MS Introduction

Onsite training

Website: www.chromacademy.com/channels/lc-ms/principles/lc-ms-introduction

Applied Interpretation of LC–MS Mass Spectra

17–19 January 2022

Online—virtual classroom

Website: www.anthias.co.uk/training-courses/interpretation-mass-spectra-lcms

SAMPLE PREPARATION

Fundamentals of Solid-Phase Extraction (SPE) Mechanisms

Online training

Website: www.chromacademy.com/channels/sample-preparation/technique/fundamentals-of-spe-mechanisms

MISCELLANEOUS

Coping With COVID-19: Remaining Productive and Safe in the Analytical Laboratory

Online webcast from CHROMacademy

Website: www.chromacademy.com/channels/basic-lab-skills/technique/coping-with-covid-19-remaining-productive-and-safe-in-the-analytical-laboratory

Introduction to Infrared (IR) Spectroscopy

Online webcast from CHROMacademy

Website: www.chromacademy.com/channels/infrared/principles/introduction-to-infrared-spectroscopy

Please send your event and training course information to [Kate Jones](mailto:kjones@mjlifesciences.com) kjones@mjlifesciences.com



Event News

26–28 January 2022

The 17th International Symposium on Hyphenated Techniques in Chromatography and Separation Technology (HTC-17)

Het Pand Conference Center, Ghent, Belgium

E-mail: htc17@kuleuven.be

Website: <https://htc-17.com/>

5–9 March 2022

Pittcon 2022

Georgia World Congress Center, Atlanta, Georgia, USA

E-mail: info@pittcon.org

Website: <https://pittcon.org>

21–24 June 2022

Analytica 2022

Messe München, Munich, Germany

E-mail: info@analytica.de

Website: www.analytica.de/en/

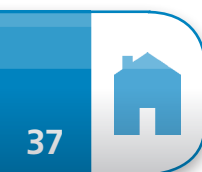
11–14 October 2022

The 18th International Symposium on Preparative and Industrial Chromatography and Allied Techniques (SPICA 2022)

Lisbon, Portugal

E-mail: secretariat@LDOrganisation.lu

Website: www.spica2022.org/



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The Column (ISSN 2050-280X) is the analytical chemist's companion within the dynamic world of chromatography. Interactive and accessible, it provides a broad understanding of technical applications and products while engaging, stimulating, and challenging the global community with thought-provoking commentary that connects its members to each other and the industries they serve.

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