Cover Story

10 What Will Be Happening at HPLC 2023?
As HPLC 2023 approaches, The Column spoke with the organizers, Oliver Schmitz and Michael Lämmerhofer, to find out what attendees can expect from the conference, which is a highlight in the chromatography calendar.

Features

2 Do Small Leaks in Your Gas Chromatography System Matter? Part 2
Chris English, Restek
Small leaks may compromise column lifetime, increase maintenance, cause retention time shifts, and decrease sensitivity at elevated temperatures, but do they matter?

13 Rising Stars of Separation Science: Raviraj Chandrakant Shinde
This month we interview Raviraj Chandrakant Shinde from India, about his work on the analysis of various food contaminants using chromatography and mass spectrometry, and the challenges involved in analyzing polar and ionic pesticides.

18 High Performance Liquid Chromatography Analysis of Cinnamon from Different Origin
Gesa J. Schad and Natsuki Iwata, Shimadzu Europa GmbH, Shimadzu Corporation
Quantifying the content of coumarin and cinnamaldehyde in cinnamon products

22 ASASS III Event Preview
The Third Australian Symposium on Advances in Separation Science (ASASS III) will take place 15–17 February 2023 in Hobart, Tasmania. Here’s a small glimpse of what you can look forward to.

Regularitys

7 News
The latest research news and news in brief

24 The LCGC Blog
The Rise of Automation—Evolve or Die?
Tony Taylor, Arch Sciences Group and CHROMacademy
An article from The Robot Report contrasted the performance of robots in industry against those of humans. The takeaways from the report are as follows.

Chris English, Restek, Bellefonte, Pennsylvania, USA

The previous instalment of “Practical GC” evaluated the impact of small leaks in the system that could be measured by the mass spectrometer but that would allow the system to operate and to even pass an instrument tune. The leak was measured as 23% nitrogen and 4% water and caused a slight retention time shift, a dramatic decrease in sensitivity, and measurable increase in bleed. The focus will now shift to evaluating several other stationary phases and determining if leaks are more detrimental to functionalized siloxane polymers, such as those containing phenyl or cyanophenyl.

In the last instalment (1) we determined that a leak in the system that does not result in an injection port shutdown and allows the mass spectrometer to perform a tune can still result in loss of sensitivity and increased bleed at higher temperatures. For example, a “1-type” stationary phase exposed to 23% nitrogen (m/z 28) and 4% water (m/z 18) when compared with the tuning compound’s base peak of m/z 69 dramatically increased in bleed when operated at the column’s maximum temperature for 180 min. Analysis under these conditions suggests the column would be irrevocably damaged with continued exposure to temperature in the presence of air/water. In our last experiment, the “1701-type” column was installed but a consistent leak could not be maintained while at the maximum programmable temperature of 280 °C. This article will revisit the “1701-type” column and evaluate an arylene-35% diphenyl column and demonstrate a way to test both columns with a consistent leak (Figure 1). Phases that contain arylene or silphenylene substituted...
into the siloxane backbone are more thermally stable since the polymer becomes less flexible (2), whereas cyano-groups are considered more sensitive to oxygen and water.

**Experimental Design**

Three stationary phases were evaluated: a “1-type” 100% polydimethylsiloxane (PDMS), a “1701-type” composed of 14% cyanopropyl-phenyl with the remaining 86% as PDMS, and an arylene-35% diphenyl with the remaining percentage as PDMS (all Restek). Column dimensions for the columns tested were 30 m × 0.25 mm, 0.25-µm and each column was installed into a gas chromatography–mass spectrometry (GC–MS) system (Agilent 7890 GC, 5975 MS) with a split/splitless inlet and 100:1 split, constant flow 0.5 mL/min (~1 psi @ 50 °C). The “1-type” GC program was 50 °C (hold 10 min), 10 °C per min to 200 °C (hold 10 min), 10 °C per min to 250 °C (hold 10 min), 10 °C per min to 300 °C (hold 10 min), 10 °C per min to 350 °C (hold 10 min), as shown in Figures 2 and 3. The “1701-type” GC program was 50 °C (hold 10 min), 10 °C per min to 200 °C (hold 10 min), 10 °C per min to 250 °C (hold 10 min), 10 °C per min to 280 °C (hold 10 min), 10 °C per min to 300 °C (hold 10 min), as shown in Figure 4. The arylene-35% diphenyl column was 50 °C (hold 10 min), 10 °C per min to 200 °C (hold 10 min), 10 °C per min to 250 °C (hold 10 min), 10 °C per min to 300 °C (hold 10 min), 10 °C per min to 300 °C.
The columns were operated to their maximum programmable temperature limits with and without leaks. A 1 m × 0.25 mm internal diameter guard column was installed in the split/splitless inlet connected to the analytical column using two press-fit connectors. The first press-fit connector was scratched along the side of the column to create a partial seal and the second press-fit connector was used to connect each column to the leaking press-fit. By maintaining the same leaking press-fit, each column could be evaluated with the same leak. Testing was performed without a leak and with a small leak. When evaluating the columns without a leak, the leaking press-fit and guard were carefully flushed using two overlaid extracted ion chromatograms using m/z 207 for the base peak of hexamethyldisiloxane (D3).
removed to be reinstalled for the next column evaluation. This allowed both the “1701-type” and the arylene-35% diphenyl to be tested with the same leak, as shown in Figure 1. The leak was determined by measuring nitrogen (m/z 28) and water (m/z 18) and comparing with PFTBA’s base peak of m/z 69 to determine air and water relative to the abundance of the tuning compound and reported as percentages. A 1000 µg/mL pesticide standard of disulfoton, o,o,o-triethyl phosphorothioate, dimethoate, sulfoatepp, methyl parathion, famphur, phorate, zinophos, and ethyl parathion dissolved in methylene chloride was used for this testing. The compounds have boiling points of between 200–400 °C and were monitored for response, retention, and peak shape to verify consistency between analyses and columns. The MS scan rate was m/z 35–550 without a solvent delay.

Discussion

An air and water check was performed as one method of determining a leak-free system for testing the “1-type” column where nitrogen was at 1.3% and water at 0.6%. The press-fit connector was loosened and after many attempts a small leak was created that was measured as 23% nitrogen and 4% water. The “1-type” column operated at a maximum temperature of 350 °C with a small leak, which resulted in a continually rising baseline when compared with leak-free analysis that demonstrated a baseline drop (Figure 2). With a leak-free system, the baseline stabilized and remained flat at the maximum programmable temperature. One striking comparison can be performed by overlaying two chromatograms—one with a

**Figure 3:** “1-Type” (100% polydimethylsiloxane) stationary phase 30 m × 0.25 mm, 0.25-µm operated at maximum temperature without a leak (blue trace) compared with a press-fit connector with a leak (black trace) measured as 23% nitrogen (m/z 28) and 4% water when compared with m/z 69 PFTBA GC–MS tuning compound. Since the GC–MS scan range is from 35 to 550 amu, only CO₂ (m/z 44) is present in the chromatogram and not water, oxygen, and nitrogen. This rise in CO₂ is another indication of a leak in the system as demonstrated in this extracted ion chromatogram (EIC).

**Figure 4:** Overlay of total ion chromatograms (TICs) comparing three different stationary phases operated at their maximum temperatures with an air leak for a total of 180 min. “1-Type” (blue trace), “1701-Type” (red trace), and an arylene-35% diphenyl (black trace) columns were tested. The arylene-35% diphenyl phase was most resistant to oxidative breakdown.
leak and the other leak-free using m/z 207, the base peak for hexamethyldicyclosiloxane (D3), as shown in Figure 2. A second example was performed by overlaying two extracted ion chromatograms (EICs) for m/z 44 (carbon dioxide); as the GC–MS scan range is from 35 to 550 amu it did not detect water (m/z 18), oxygen (m/z 16), and nitrogen (m/z 28). The increased presence of carbon dioxide is another indicator of a leak in the system (Figure 3). For the addition of the “1701-type” column and the arylen-35% diphenyl column to the experiment, two press-fit connectors were used in series where the first connector closest to the injection port was installed with a leak and the second connector allowed columns to be changed, meaning both columns could be tested with the same leak. Hours were spent creating a leak where the guard column did not disconnect at higher temperatures and the leak was not large enough to cause the inlet to shut down. The method that worked for creating a small leak required a clean cut of the fused silica guard column followed by running the smooth side of the ceramic cutting wafer lengthwise down the column end. This created a small notch at the end of the column, yet the column was sealed in the press-fit and did not come loose at higher temperatures and pressures. The leak was measured as 19.1% nitrogen and 1.9% water at 50 °C. The “1701-type” and arylen-35% diphenyl columns were tested with five consecutive analyses of the standard without a leak followed by 18 analyses of the standard with a leak. This meant that the three columns were exposed to 180 min at their maximum temperatures and could be directly compared with each other by overlaying the total ion chromatograms (TICs). Since the bleed products were different for each of the columns, the best comparison required the use of TICs and not specific ions, as shown in previous examples. Interestingly, the “1701-type” and “1-type” columns had similar bleed profiles after exposure to oxygen for their maximum temperatures, whereas the arylen-35% diphenyl column had significantly less bleed and less of an increase at maximum temperature.

Conclusions

Small leaks may compromise column lifetime, increase maintenance, cause retention time shifts, and decrease sensitivity at elevated temperatures. Both the “1701-type” and “1-type” columns exhibited high bleed at their maximum temperatures but similar bleed at lower temperatures, such as 200 °C. The arylen stabilized polymer showed the best resistance to oxidative breakdown and exposure to water at elevated temperatures.

Acknowledgements

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

References


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.

E-mail: chris.english@restek.com
Website: www.restek.com

Introducing Daicel’s New CHIRALPAK® IM

Backed by 40+ years of experience and innovation, Daicel Chiral Technologies offers more ways to achieve successful enantiomer separations.

- CHIRALPAK IM is the immobilised equivalent of CHIRALCEL OZ
- Robust to withstand all mobile phase combinations
- Designed for HPLC and SFC to improve methods for challenging separations
- 5 µm particle size available in October 2022

The best separations demand the best columns.
Visit us at chiraltech.com
Agilent Joins How2Recycle Programme

Agilent Technologies Inc. (Santa Clara, California, USA) has become a member of How2Recycle, a standardized labelling system that advises the public on recycling. Owned by the Green Blue Institute, Inc., the programme is comprised of brands who want their packaging to be recycled and are empowering consumers to do it through smart packaging labels.

By joining the programme, Agilent is offering its customers better information about packaging and the correct disposal of Agilent products. “We are increasing our effort in designing and developing sustainable product packaging as part of our commitment to the environment,” said Henrik Ancher-Jensen, Agilent’s president of order fulfillment and supply chain. “By adding the How2Recycle standardized label on our packaging materials, we eliminate the guesswork on what is recyclable, allowing our customers to correctly recycle.”

Agilent will have access to the official recycling labels to use on its packaging. Every package that features a How2Recycle label undergoes an individualized recyclability assessment based on detailed packaging specifications that the company sends How2Recycle via its member platform.

“To address a laboratory’s carbon footprint, we have to lead with actions that align our product R&D, development, manufacturing, packaging, delivery, and end-of-use towards those goals,” said Darlene Solomon, senior vice president and chief technology officer at Agilent Technologies. “By acting on the sustainability goals of our customers, we are able to deliver our brand promise in a meaningful way and we are committed to listening, learning, and working with outside agencies to improve and to drive positive change within our markets and industry.”

For more information please visit www.agilent.com
LCGC Announces the 2023 Winners of the Lifetime Achievement and Emerging Leader in Chromatography Awards

LCGC, the leading resource for separation scientists, is proud to announce that Peter J. Schoenmakers and Emanuela Gionfriddo are the winners of the 16th annual LCGC Lifetime Achievement and Emerging Leader in Chromatography Awards, respectively. Schoenmakers and Gionfriddo will be honoured in a symposium as part of the technical programme at the Pittcon 2023 conference in March 2023.

The Lifetime Achievement Award
The Lifetime Achievement in Chromatography Award honours an outstanding professional for a lifetime of contributions to the advancement of chromatographic techniques and applications.

Schoenmakers, the 2023 winner, is a professor of analytical chemistry and a former scientific director of the van’t Hoff Institute for Molecular Science at the University of Amsterdam. He is best known for his work in liquid chromatography (LC), starting with the theory of gradient elution in reversed-phase chromatography and its optimization, and continuing through his many contributions to the analysis of polymers and his pioneering work in developing two- and three-dimensional LC methods, particularly of polymers.

His earliest work focused on a general theory of gradient elution LC. His 1978 paper, “Gradient selection in reversed-phase liquid chromatography”, is a foundational work and has been cited 300 times.

Schoenmakers is unquestionably one of a small group of pre-eminent contributors to the development of two-dimensional LC (2D-LC). He has contributed extensively to areas such as the optimization of 2D-LC including his innovative work on Pareto optimization, three-dimensional LC (3D-LC) separations, the theory of resolution in 2D separations, and a quite novel method for determining the fractional coverage. Additionally, Schoenmakers has done seminal work on ways to minimize sample dilution.

Schoenmakers has also contributed significantly to the area of polymer characterization. Initially he focused on size-exclusion chromatography (SEC) and reversed-phase LC of homo polymers and co-polymers. He also studied “LC at the critical-composition” of polymers. He explored the use of multiple types of detectors, such as optical absorbance, multi-angle light scattering, and Fourier transform infrared detectors. His more recent work in polymer analysis has been his development and use of 2D-LC and 3D-LC. He is truly a pioneer in this field.

The Emerging Leader Award
The Emerging Leader in Chromatography Award recognizes the achievements and aspirations of a talented young separation scientist who has made strides early in his or her career towards the advancement of chromatographic techniques and applications.

Gionfriddo, the 2023 winner, received her Ph.D. in chemistry in 2013 at the University of Calabria, in Italy. Following work as a postdoctoral fellow and research associate at the University of Waterloo, in Canada, in 2018 she became an assistant professor of chemistry at the University of Toledo, in Ohio, USA, where she is the founding member of the Dr. Nina McClelland Laboratory for Water Chemistry and Environmental Analysis.

Gionfriddo’s contributions to separation science involve the implementation of alternative, green microseparation methodologies for extraction of small molecules from challenging biological and environmental samples. Her research focus has important implications for environmental monitoring of contaminants, exposomics studies in biological samples and living organisms, and targeted and untargeted metabolomics. Of particular relevance is her contribution in developing and testing biocompatible extraction phases for microextraction that have enabled the extraction of a broad range of molecules in complex media.

She currently serves on the University of Toledo Water Task Force and on the Scientific Council of Advisors for the State of Ohio’s Attorney General’s Office.

To nominate a candidate for the 2024 Lifetime Achievement and Emerging Leader in Chromatography Awards, scan here:
• **The LCGC Blog: Efforts to Increase Diversity and Equity in Introductory Chemistry**—Increased diversity in the workplace brings broader perspectives and innovation. Yet many chemical industries, including separation science, lack the diversity that is representative of the population. To fix this problem, we must start at the beginning of the pipeline. Read Here>>

• **Analytically Speaking Podcast Ep. 6: Sometimes “Negative Results” Turn Out To Be the Most Interesting Ones**—In this episode, podcast host Dr. Dwight Stoll talks with Dr. Adrian Clarke, Global Analytical Network Leader at Novartis Pharma in Basel, Switzerland. They discuss the origins of his interest in separation science, the evolution and maturation of supercritical fluid chromatography and its use in the pharmaceutical industry, and the dramatic growth in learning about separations of synthetic oligonucleotides that is happening right now. They also discuss the importance of training and continuing education in separation science, and Adrian shares his advice for young scientists just getting started in the field. Read Here>>

• **Building Small But Thinking Big**—The Column spoke to Brett Paull from the University of Tasmania in Hobart, Australia, about the development of truly portable LC systems for use in the pharmaceutical industry and in environmental analysis. Why are they necessary, and where can improvements still be made? Read Here>>

• **Authentication of Panax Ginseng-Based Herbal Teas Using “Chemical Markers” Strategy**—This article describes the method development involved in the authentication of nutraceuticals, particularly those containing Panax ginseng, which is popular because of possible positive effects on human health. Read Here>>

• **Miniaturized Liquid Chromatography Analysis of Dietary Supplements**—Researchers have evaluated three different miniaturized liquid chromatography (LC) instruments for the analysis of caffeine in dietary supplements. Read Here>>

---

**Shimadzu Collaborates with University Medical Center Göttingen**

Shimadzu (Duisburg, Germany) has announced a collaboration with the University Medical Center Göttingen (UMG) (Germany). The collaboration will focus on the development of new clinical laboratory methods using liquid chromatography–mass spectrometry (LC–MS) for therapeutic drug monitoring (TDM) analysis.

TDM plays an important role in providing precision medical care; TDM analysis measures the concentration of drugs currently in the blood to determine the next dosage of a drug. Working together with Prof. Andreas Fischer, Dr. Frank Streit, and other leading experts at UMG, new methods for multiple target groups of drugs will be developed and optimized so that clinical laboratories can switch methods quickly and easily. Speeding up this process will give doctors, clinics, and hospitals treating emergency cases quicker access to better test results, and ultimately lead to better medical care for emergency patients.

The collaboration with UMG takes place with the help and support of Shimadzu’s European Innovation Center (EUC) programme.

For more information please visit www.shimadzu.eu
What Will Be Happening at HPLC 2023?

As HPLC 2023 approaches, *The Column* spoke with the organizers, Oliver Schmitz and Michael Lämmerhofer, to find out what attendees can expect from the conference, which is a highlight in the chromatography calendar.

Q. HPLC 2023 will be held in Düsseldorf, Germany, from 18–22 June 2023. This seems a long time away, but as the organizers I am sure you both know the opening ceremony will be happening before we know it! Are there any deadlines on the horizon that delegates, speakers, sponsors, and exhibitors should be aware of?

Michael Lämmerhofer: All deadlines are listed on our homepage [www.HPLC2023.com](http://www.HPLC2023.com)

The most important deadline for speakers is the abstract submission deadline for orals, which is 16 January 2023. Submissions after this deadline cannot be considered for oral presentations.

Delegates can register at a reduced fee from now until 20 April 2023. Last but not least, sponsors and exhibitors should register now to get the best booth locations.

Q. You mentioned that one of your goals is to raise awareness of industrial scientists who use chromatography at HPLC 2023. Can you tell us more about that?

ML: We have a plenary speaker from the chemical industry who will tell us the concepts of Industry 4.0 and how to make the analytical lab in chemical industry fit for the future. In addition, we will have a full-day parallel session on high performance liquid chromatography (HPLC) in the chemical industry, with talks and discussion groups from experts within that industry. These industrial sessions and discussion topics are organized in close cooperation with two analytical chemists from the chemical industry—Stefan Lamotte, BASF and Matthias Pursch, DOW—who...
know the burning issues and questions in the industrial analytical chemistry laboratory.

**Q. Is there anything else new or different in next year’s programme?**

**OS:** HPLC Tube and Science Slam, which were introduced at HPLC 2019 in Milan, Italy, as new attractive formats for the younger generation, will be special features in the programme. In addition, we are trying to encourage more women as speakers. At the moment, we have already selected 26 women as plenary or keynote speakers. Moreover, we want to give the best poster award a new face and better visibility.

**Q. What aspects of the conference will be of value to young scientists?**

**ML:** In addition to being inspired by a variety of invited and renowned speakers, and communicating directly with them, we will also install a job board to help young scientists find employment in industry or at research institutions. Companies will get the opportunity to recruit the most talented young scientists at the conference. There are also plenty of attractive features for young scientists, such as short courses on various topics of separation science, mass spectrometry, ion-mobility spectrometry, and data processing. Within the scientific programme, we will offer a number of tutorials that will present an overview of hot topics in the field of analytical science.

**Q. How important are poster presentations at HPLC 2023? Are you doing anything new to increase visibility of posters?**

**OS:** In order to highlight the value of the poster presentations in the process, we will give the shortlisted preselected posters of each day the opportunity to pitch in a short oral presentation for the award, and the final poster prize winner selected by a jury will be able to briefly present their poster in the final closing session. The three best posters will receive prize money. The other seven poster prize winners will receive the usual prize money of former HPLC conferences. Thus, we hope for a nice mixture of scientific entertainment and scientific presentation of results at the conference.

**Q. Does the HPLC conference have a value that is distinct from that of other conferences? Why do you think the European conference is held in such high regard?**

**ML:** HPLC can be described as the world’s largest leading conference in the field of separation science and hyphenated techniques. The number of participants at the European HPLC conferences is usually always above that of other conferences, and the exhibition substantially larger than comparable symposia. It features a good mix of scientific, technical, tutorial, and short course programme options, making it a perfect meeting for young and
established scientists, industry delegates, and vendors. As coronavirus restrictions will be relaxed in June 2023, we again expect over 1200 participants for HPLC 2023. These high numbers may be due to the strong chemical and pharmaceutical industry in Europe—particularly in Germany, with the industrial Rhine-Ruhr region in close vicinity to Düsseldorf.

Q. Anything else participants should be aware of?
OS: Düsseldorf is known for having the longest bar in the world. A whole street long. So talking about science will not end at the end of each day at HPLC 2023 in Düsseldorf!

Michael Lämmerhofer, Professor at the University of Tübingen, Germany

Oliver S. Schmitz, Full Professor at the University of Duisburg-Essen, Germany

Q. In today’s digital world—particularly given the many virtual events held since the start of the pandemic—do you think people need to be convinced of the value of attending an in-person scientific meeting?
OS: No, I think we have all found in the last two years that the personal contact, the conversations between and after the presentations, and the conference atmosphere are all essential for triggering the necessary inspirations in science.

Q. Putting together a large conference is a lot of work. How will you judge if it was a success?
ML: If the participants have a good time, enjoy the lectures, actively participate, and give us good feedback, then the conference was a success. There is no need to have a big financial surplus; if there is any, it will go to a nonprofit society (the German Chemical Society GDCh) for the promotion of young scientists of the Separation Science Working Group of the GDCh.

Website: www.hplc2023.com
Email: hplc@gdch.de
Chairmen: Michael Lämmerhofer and Oliver S. Schmitz
Host city: Duesseldorf, Germany; https://www.hplc2023-duesseldorf.com/visit-duesseldorf
Venue: Conference Center Duesseldorf; https://www.hplc2023-duesseldorf.com/venue
Abstract deadline: 16 January 2023 (oral); 2 May 2023 (poster)
Deadline for early bird registration fee: 20 April 2023
Rising Stars of Separation Science: Raviraj Chandrakant Shinde

This month we interview Raviraj Chandrakant Shinde from India, about his work on the analysis of various food contaminants using chromatography and mass spectrometry, and the challenges involved in analyzing polar and ionic pesticides.

—Interview by Kate Jones

Q. When did you first encounter chromatography and what attracted you to the subject?

A: I was a master’s student in the Department of Agrochemicals and Pest Management at Shivaji University, Kolhapur (Maharashtra, India) and visited UPL Limited, Thane (formerly known as United Phosphorus Ltd.) for a one month training programme as part of my studies in 2015. It was here that I saw chromatographic and mass spectrometry instruments up close for the first time, and it was an unforgettable and awesome moment in my life. I understood it to be a very interesting technique that provides an accurate analysis of various compounds. Because of my good fortune, I was able to receive my initial training from Mayur Aitawade (theoretical topics) and Prem Naik (theoretical themes proven with a practical approach), who taught me these techniques very nicely and with proper demonstration. I will be eternally grateful to the team at UPL Limited., Thane, for the excellent training in analytical techniques and formulation. I am especially grateful to respected Madhukar Deshmukh (Shivaji University, Kolhapur) and the team at UPL, Thane.
Raviraj Chandrakant Shinde has a bachelor’s degree in chemistry, and a master’s degree and Ph.D. in agrochemicals and pest management (chemistry). To further continue his work and to gain more knowledge in his research subject, he has enrolled in a master’s in chemistry at Singhania University in India. His work is mainly focused on the development of new analytical methods for the analysis of various food contaminants. He has developed several analytical methods and successfully validated for the multi- as well as single residue analysis of different food contaminants such as pesticides, veterinary drugs, antibiotics, food adulterants, and mycotoxins using different chromatographic (LC and GC) and mass spectrometric techniques (triple quad, single quad, QTOF-MS, orbital trap). He has received many national and international awards, such as the Eurofins Foundation “Testing for Life” Award in 2021 in Boston, USA; the Herbalife Nutrition Award in 2022 at Scottsdale, Arizona, USA, from AOAC International; the InSc-Young Researcher Award-2021; the INSO-Emerging Scientist Award-2022; and various other awards and recognitions.

(Q: Can you tell us more about your Ph.D. thesis?
A: I completed my Ph.D. at the Department of Agrochemicals and Pest Management, Shivaji University, Kolhapur, under the guidance of P.D. Shiragave, Devchand College, Arjunnagar, Maharashtra, India. My work was mainly focused on the development of new analytical methods for pesticide residue analysis in cereals, pulses, nuts, and processed products. I have developed analytical methods using various sophisticated analytical techniques such as liquid/gas chromatography (LC/GC) with mass spectrometry (MS), and also performed qualitative analysis by atmospheric-pressure matrix-assisted laser desorption–ionization high resolution mass spectrometry (AP-MALDI–HRMS). My Ph.D. work included the development of new techniques for the analysis of food contaminants.
of an analytical method for highly polar herbicides, a novel method for multiresidue analysis of thermally unstable fungicides (captan, captafol, and folpet analysis by LC–electrospray ionization [ESI]-MS/MS without any degradation), and development of a large-scale multiresidue analytical method for the analysis of 400+ pesticides and mycotoxins in fatty matrices using LC–MS/MS and GC–MS/MS. These methods were thoroughly validated to AOAC-SMPR and SANTE guidelines and complied with regulatory requirements. These methods are useful for regulatory enforcement, as well as high-throughput commercial residue testing purposes.

Q. What chromatographic techniques have you worked with?
A: I have employed various chromatographic and mass spectrometric techniques in my research, where I have developed many analytical methods for the wide nature of compounds. I am interested in research focused on food analytical chemistry, with a special emphasis on analytical method optimization, development, and validation for the multi- as well as single residue analysis of different food contaminants such as pesticides, antibiotics, veterinary drugs, food adulterants, and mycotoxins. I have used different chromatographic techniques such as high performance liquid chromatography (HPLC) or gas chromatography with basic (UV–vis, fluorescence, flame ionization detection [FID]) and advanced detectors, including triple quadrupole, LC–quadrupole time-of-flight [QTOF]-MS, and LC–orbital trap.

Q. Your research focus currently lies in the analysis of various food contaminants using chromatography and mass spectrometry—what specifically attracted you to this area of research?
A: My research is focused on the analysis of various food contaminants such as pesticides, antibiotics, veterinary drugs, food adulterants, and mycotoxins. The analysis of these food contaminants is very critical. Sometimes it is very simple, but sometimes it can be very difficult, especially for some challenging compounds. It depends on the nature of the targeted compounds and the nature of the food commodity. Consequently, this is a very interesting and challenging part of food contaminant analysis, as although we are analyzing the same compounds, if we are changing the food
commodity (matrices), the development/modification of an extraction method is very vital.

For example, if we are analyzing pesticides from fruits (grape, mango, and orange), after the extraction, we need to perform clean up with only primary secondary amines (PSA). However, if we are doing this for complex matrices, such as nuts, cereals, or pulses, we need to use other clean-up sorbents, such as C18 along with PSA (freezing step for fatty matrices). So, nothing is always ready to go here, but we always need to look for a new or improved way to take the next step. This is a very interesting task in food contaminant analysis using chromatographic techniques and attracted me towards this field.

Q. You have recently published a paper on the analysis of polar and ionic pesticides in grape and pomegranate using LC–MS/MS (1). What are the challenges you faced during the development of a method for the analysis of these challenging to analyze highly polar and ionic compounds? How did you establish this method?

A: Globally, various multiresidue methods (QuEChERS- or ethyl acetate-based) are used for the extraction and analysis of multiclass pesticides in a single method. However, polar and ionic compounds cannot be satisfactorily recovered by regular multiresidue sample preparation workflows or in multiresidue analytical methods because these compounds are predominantly soluble in polar solvents such as water or methanol. These ionic and highly polar compounds mainly include herbicides such as glyphosate, glufosinate and their metabolites, and a few other pesticides such as ethephon and fosetyl-Al (with its metabolite phosphonic acid). Analysis of these pesticide residues is very challenging on reversed-phase column chemistry (octadecylsilane) because they get eluted in the void volume with reversed-phase columns. To improve the retention of these compounds on reversed-phase column chemistry requires derivatization with 9-fluorenylmethylchloroformate (FMOC-Cl). This derivatization step increases the molecular weight as well as changes the chemical nature of the targeted compounds, which helps to increase the chromatographic retention on the reversed-phase column. However, derivatization with the FMOC-Cl step is tedious and lengthy. On the other hand, the direct analysis (without derivatization) requires a specific instrument in the laboratory, for example, an ion chromatographic system, which increases the cost.

To overcome these obstacles, I developed new and improved analytical methods for the analysis of these difficult-to-analyze, highly polar, and ionic compounds. There was a significant challenge during this development; various column chemistries are available on the market to improve retention. However, when it comes to the practical on instrument, it is difficult to analyze all compounds along with their metabolites in a single chromatographic run using a base-to-base separation. However, I optimized each and every step, from mobile phase, chromatography (selection of column chemistry), mass spectrometry parameters (optimization of source parameters), and sample preparation workflow (without derivatization). This was a very interesting and challenging task. Finally, a simple method was developed that allows for the simultaneous analysis of parent compounds and their metabolites in a single chromatographic run with separation. This method is suitable for regulatory testing of the targeted compounds because they provide satisfactory repeatability and reproducibility.

Q. Another of your recent papers presents for the first time a method for the multiresidue analysis of multiclass pesticides in sesame seeds using LC–MS/MS and GC–MS/MS (2). What does this method offer over existing ones? Could this method be used in the analysis of other foodstuffs?

A: In 2020, public exposure to chemical contaminants through sesame seeds drew serious regulatory attention. A new regulation of the European Union 2020/1540 (22 October 2020) was brought into being, commanding that all imported sesame consignments from India must be analyzed for the residues of ethylene oxide along with pesticides. Therefore, this study was focused on sesame seeds for the development and validation of an analytical method for multiclass pesticides only.

Pesticide analysis from fatty matrices has been a difficult task for every chemist or scientist in the food industry, and sesame seeds are tricky. Sesame seeds (fatty matrix) contain higher proportions of lipids, posing challenges for pesticide extraction and analysis in...
routine samples. The issues were high matrix effects during analysis and a decrease in LC–MS/MS sensitivity caused by co-extracted lipid deposition in the ion source. Previous methods targeted a limited number of pesticides or required a time-consuming and lengthy sample preparation workflow. To overcome these obstacles, LC–MS/MS and GC–MS/MS were used to develop a simple method for extracting a large number of pesticides (400+) from sesame seeds.

This method is very simple and robust, and it can be used in a food testing laboratory. Furthermore, given the method’s performance, it can be used for pesticide residue analysis in other oil seed matrices as well.

Q. You have also developed a simple, rugged, and short method for the analysis of patulin (3). This method is recognized by the Food Safety and Standards Authority of India (FSSAI) for inclusion in the official food testing manual. How difficult was this method to implement?
A: Patulin, a toxic metabolite of fungus that is predominantly found in apples and its processed products, can pose severe health hazards to humans (patulin-producing pathogen is *Penicillium expansum*). Given its toxicological significance, many regulatory bodies have imposed stringent restrictions on the levels of contamination of this mycotoxin. Therefore, analysis of patulin from apple and its processed products is important.

A simple, rapid, sensitive, and robust analytical method was developed that ensures high throughput analysis of patulin within 5 min. The method does not involve any lengthy and complicated clean-up steps; and so it is less expensive and more time-effective compared with previously published methods. The developed method is aligned with the analytical quality control criteria and complies with the requirements of CODEX, EU, FSSAI, and US FDA.

The method has been adopted by the nodal-government agency-Food Safety and Standards Authority of India in their official food testing manual (4). The method can be implemented in regulatory and commercial food testing laboratories. Considering its accuracy, repeatability, and reproducibility, the method is fit-for-purpose for testing of patulin in apple and its processed products.

Q. Anything else you would like to add?
A: I am always thankful to my family (Shinde, Taur, Shejul, and Jadhav) for their tremendous support throughout the whole journey. I am also thankful to all my professors, teachers, and all my friends for believing in me.

References
Coumarin and cinnamaldehyde are main ingredients in cinnamon products—often used as a common spice or herbal remedy. As well as their proven health benefits, they both also exhibit potential adverse effects. This article introduces a method for the pretreatment and analysis using high performance liquid chromatography (HPLC) to simultaneously quantify the content of coumarin and cinnamaldehyde in cinnamon products from different origin.

Cinnamon is a common spice and herbal remedy used around the world, and has many health benefits associated with it. The reddish-brown powder is ground from the dried bark of an evergreen tree in the Lauraceae family, and is often added to baked goods, tea, curries, and meat dishes as a spice. Apart from its positive effects on digestion, stomach health, intestinal regulation, and detoxification, it has been reported to be effective in preventing or improving diabetes, as a result of its hypoglycemic and lipid-lowering potential (1,2). Cinnamon also contains natural antioxidants that could reduce the risk of cancer and the signs of ageing, and is a common treatment for inflammatory diseases and amenorrhea in traditional Chinese medicine (2,3).

Despite these clinical benefits, there is also evidence that cinnamon may exhibit some adverse effects. One of the main components of cinnamon, coumarin, has been shown to have hepatotoxic and
carcinogenic effects, and as a result, the European Food Safety Authority has determined the tolerable daily intake (TDI) of coumarin to be 0.1 mg per kg of body weight (4). In addition, cinnamaldehyde—another main component—was determined to be an irritating and sensitizing component that may exhibit teratogenic effects (3). Consequently, determining the concentration of these compounds in cinnamon is considered an important part of quality control.

The main types of cinnamon are *Cinnamomum verum* (Ceylon cinnamon) and *Cinnamomum cassia*, both containing coumarin and cinnamaldehyde in varying concentrations, depending on the origin of the spice. This article describes a method for using high performance liquid chromatography (HPLC) to simultaneously quantify the content of coumarin and cinnamaldehyde in cinnamon samples from different regions.

**Analysis of Coumarin and Cinnamaldehyde in a Mixed Standard Solution**
A mixed standard solution containing 0.5 mg/L of each of the analytes of interest—coumarin and cinnamaldehyde—was prepared in acetonitrile and analyzed using the conditions listed below. Coumarin was detected at 280 nm, near the maximum absorption wavelength.
While the maximum absorption wavelength for cinnamaldehyde is 287 nm, taking into consideration sensitivity and separation from contaminants in the cinnamon sample, cinnamaldehyde was detected at 320 nm. Figure 1 shows a typical chromatogram of the separation obtained at both detection wavelengths.

**Method: Analytical Conditions of the Determination of Coumarin and Cinnamaldehyde:** System: Nexera Lite HPLC (Shimadzu); column: 150 mm × 3.0 mm, 3-µm Shim-pack GIST-HP C18 (Shimadzu); flow rate: 0.8 mL/min; mobile phase: A) water B) acetonitrile; time program: 50%B (0–2 min) → 60%B (4 min) → 100%B (4.01–5.00 min) → 50%B (5.01–10.00 min); column temp.: 25 °C; injection volume: 5 µL; detection (photo diode array [PDA]): channel 1 (coumarin) λ = 280 nm, channel 2 (cinnamaldehyde) λ = 320 nm.

**Results:** Repeatability of the method was determined based on retention time and peak area from six successive analyses of the 0.5 mg/L mixed standard solution. Linearity was evaluated by producing calibration curves using standard solutions within a concentration range of 0.013–1 mg/L for coumarin and 0.5–40 mg/L for cinnamaldehyde. With correlation coefficients \( r^2 = 0.9999 \) or greater and variation of < 0.1% in retention time and < 0.5% in peak area for both analytes, good linearity and repeatability of the assay was established.

**Sample Analysis: Coumarin and Cinnamaldehyde in Commercial Cinnamon Products**

Commercially available samples of *cinnamomum cassia* produced in Vietnam and *cinnamomum verum* produced in Sri Lanka were analyzed after extraction with acetonitrile following the pretreatment protocol shown in Figure 2. Lipids were removed using a dispersive solid-phase extraction (dSPE) cartridge. The cartridge eliminated the need to carry out conditioning before loading samples, which simplified the process. The supernatant was filtered through a 0.2 μm membrane filter and diluted with acetonitrile before analysis by HPLC. Figure 3 displays the obtained chromatograms of the quantification of coumarin and cinnamaldehyde in commercial cinnamon samples. The supernatant obtained from extraction from *cinnamomum cassia* required a 200-fold dilution, while the *cinnamomum verum* extract was diluted by a factor of 2.

**Table 1:** Results of the quantitative analysis of coumarin and cinnamaldehyde in cinnamon samples

<table>
<thead>
<tr>
<th></th>
<th>Coumarin</th>
<th>Cinnamaldehyde</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity (mg/L)</td>
<td>Concentration (mg/g)</td>
<td>Quantity (mg/L)</td>
</tr>
<tr>
<td>Cinnamomum Cassia</td>
<td>0.190</td>
<td>3.79</td>
</tr>
<tr>
<td>Cinnamomum Verum</td>
<td>0.148</td>
<td>0.03</td>
</tr>
</tbody>
</table>

**Recovery Test:** To verify the effectiveness of the pretreatment protocol, a recovery
A fast and simple HPLC method for the simultaneous analysis of two main compounds in ground cinnamon was developed. Using a C18 column, baseline separation of the target compounds was achieved within 4 min, with good repeatability and proven linearity within the range of 0.013–1 mg/L for coumarin and 0.5–40 mg/L for cinnamaldehyde. A simple pretreatment protocol was proposed that offered efficient extraction and appropriate sensitivity to ensure the detection wavelength was optimized to achieve good separation from contaminants. This method offers quick qualitative and quantitative analysis for the quality control of cinnamon products.

**References**

1. N. Iwata, Shimadzu Corporation Application News No. 01-00233-EN (Shimadzu Corporation, December 2021).

**Gesa Johanna Schad** graduated with a diploma in chemical engineering from the Technical University NTA in Isny, Germany, in 2004 and as a master of science in pharmaceutical analysis from the University of Strathclyde in Glasgow, UK, in 2005. She worked until 2006 as a consultant in HPLC method development and validation in an analytical laboratory of the FAO/IAEA in Vienna, Austria. She gained her doctorate for research in pharmaceutical sciences at the University of Strathclyde in 2010 and was employed as an HPLC specialist in the R&D department at Hichrom Ltd in Reading, UK, from 2009. Since 2013, she has worked as a HPLC product specialist and since 2015 as HPLC Product Manager in the analytical business unit of Shimadzu Europa in Duisburg, Germany.

**Natsuki Iwata** graduated with a diploma in chemistry and materials technology from Kyoto Institute of Technology in Kyoto, Japan, in 2010 and a MEng in chemistry and materials technology from the graduate school of Science and Technology, Kyoto Institute of Technology in 2012. She has worked as an HPLC product specialist in the Shimadzu global application development centre since 2012 and the solutions centre of excellence of Shimadzu Corporation in Kyoto since 2022.

**Table 2: Recovery rates of the extraction (n = 6) of coumarin and cinnamaldehyde from cinnamomum cassia**

<table>
<thead>
<tr>
<th>N</th>
<th>Recovery Rates (%)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Coumarin</td>
<td>Cinnamaldehyde</td>
</tr>
<tr>
<td>1</td>
<td>103.0</td>
<td>91.7</td>
</tr>
<tr>
<td>2</td>
<td>100.2</td>
<td>91.5</td>
</tr>
<tr>
<td>3</td>
<td>106.9</td>
<td>95.5</td>
</tr>
<tr>
<td>4</td>
<td>110.4</td>
<td>95.7</td>
</tr>
<tr>
<td>5</td>
<td>110.6</td>
<td>95.6</td>
</tr>
<tr>
<td>6</td>
<td>105.1</td>
<td>91.4</td>
</tr>
<tr>
<td>Average</td>
<td>106.0</td>
<td>93.5</td>
</tr>
<tr>
<td>(%RSD)</td>
<td>(3.9%)</td>
<td>(2.4%)</td>
</tr>
</tbody>
</table>
ASASS III Event Preview

As we move back into a world of face-to-face meetings, we take a look at upcoming conferences—the Third Australian Symposium on Advances in Separation Science (ASASS III) will take place 15–17 February 2023 in Hobart, Tasmania. Here’s a glimpse of what you can look forward to.

The Third Australian Symposium on Advances in Separation Science (ASASS III) will take place 15–17 February 2023 in Hobart, Tasmania.

The first Australian Symposium on Advances in Separation Science (ASASS I) was held in December 2008 at the University of Tasmania, Hobart, the state capital of the beautiful island of Tasmania, Australia. The inaugural symposium, then known as the ACROSS Symposium on Advances in Separation Sciences, was chaired by Paul Haddad and was timed to mark the 60th birthday of its inaugural Chair. This first symposium was a great success, attracting more than 150 separation scientists from over 18 countries. The symposium was held over three days, delivering lively discussion and presentations covering all aspects of cutting-edge separation science, all within a beautiful and stimulating environment.

ASASS II was held from 29 November to 2 December 2016, and was once again held in Hobart, on this occasion on the historic waterfront, opposite the beautiful and world-famous Salamanca Place. The symposium, chaired by Brett Paull, once again opened its doors to an international delegation of separation scientists, both young and old, for three days of great science and social interaction. With close to 140 delegates, spanning local Australian based PhD students to internationally renowned experts, together originating from over 25 different countries, the meeting was an exciting mix of youth and experience, making for a hugely educational, productive, and entertaining experience for all.

After a further hiatus of six years, exacerbated most recently by Covid-19, the organizing committee are once again delighted to be able to announce the call for delegates and supporters of ASASS III. After missing the enjoyment and dynamism of face-to-face symposia for over three years, the time has come for the separation science community across Australia, the wider Australasian region, and indeed international delegates to come together and enjoy the benefits of our vibrant and popular
symposium series. Keeping with emerging tradition, the organizers will once again host ASASS in Hobart, Tasmania, as a location easily reached nationally, and enthusiastically supported internationally. ASASS III will also continue the ambitions of the symposium to date and will include an exciting variety of separation science, from advanced materials to advanced instrumentation, and spanning industrial analysis to the environmental sciences. With a significant number of stimulating plenary, keynote, and invited presentations to kick off each exciting session of contributed papers, ASASS III will provide all delegates with a rewarding symposium, with ample opportunity for networking and social interactions. With presentations from across the entire separation science community, from early career researchers and research students to industry-based scientists and internationally renowned leaders, ASASS III will provide opportunities for all to share their science, with a commitment to diversity and the support of early-career researchers. ASASS has at its core a desire to provide a unique and inspirational experience, with value for money and a vibrant and exciting social programme guaranteed.

Changes to the traditional symposium format at ASASS III will involve the inclusion of a full day’s programme dedicated to innovation and the future state of separation science, which will give particular opportunity to early- and mid-career researchers (EMCRS) to showcase the direction their research is taking the field of separation science.

The symposium programme will include a dedicated EMCR-industry collaboration session focused on success stories arising from academic-industry collaboration on separation projects, which is to be followed by a panel-led discussion and social networking event, to further enable future academic-industry collaboration and pathways to research translation and impact. ASASS III will be held at one of Hobart’s premium event and conference venues, the Hobart Function and Conference Centre (HFCC), which enjoys a waterfront location like no other; situated directly adjacent to Hobart’s historic Salamanca district, the conference centre extends 100 m along Elizabeth Street Pier, overlooking the sparkling water of the Derwent, the centrepiece of Hobart’s bustling waterfront community.

All symposium rooms boast floor to ceiling windows so delegates and guests enjoy Hobart’s wonderfully clear natural light, or at night during the symposium gala dinner, the bobbing lights of boats in the harbour.

Abstract submissions for oral and poster presentations are now welcome, covering all aspects of separation science, including, but not exclusive to, “Materials for separation science”, “Separation technology and instrumentation”, “Applications of separation science”, and “Future state separation science”.
The LCGC Blog: The Rise of Automation—Evolve or Die?

Tony Taylor, Arch Sciences Group and CHROMacademy

An article from The Robot Report contrasted the performance of robots in industry against those of humans. The takeaways from the report are as follows.

In the article from The Robot Report (1), five areas were highlighted where robots perform better than humans: a) handling tedium, b) extreme sensing, c) strength and speed, d) unwavering focus, and e) perfect, objective recall.

The article also highlighted three areas where humans still outperform robots: a) empathy, b) flexibility, and c) acceptability and trust.

It strikes me that considering these points within the laboratory context, we may not be taking full advantage of the positive aspects of robotics, or indeed working to address the negative aspects. This is especially true when considering sample preparation and sample manipulation.

Most laboratory workers with more than just a few samples to process will be familiar with the daily routine of preparing samples, setting up equipment, running a system suitability, checking fitness for purpose, and then starting the “batch” of samples just before leaving work for the evening. Modern instruments have very much reduced the amount of finger crossing required on the journey home and that sense of anticipation as one enters the laboratory the next morning to see if the “run has worked” or not, but those feelings of anxiety have not been completely eliminated.

Automated robotics have the power to change this paradigm, with samples being prepared in a “just-in-time” fashion so that the analysis can begin at the start of the day, and much of it will be done as we leave work—preparation and analysis times permitting. We could then even rack up enough samples to do the
same thing overnight, hence improving our throughput, if required.

Of course, we need the automated solutions to be very robust and reliable, and this is perhaps where the industry needs to evolve, to ensure that our engineering is optimized to the point at which fallibility is not a consideration. Again, most of us are familiar with situations where campaigns have been halted overnight due to a vial not being gripped or picked up properly, or a vial tolerance that has caused the autosampler to reject a particular sample, or even simply to drop the vial. Here evolution in the flexibility and “learning” of the instruments could be improved. Provided that the rejected vial does not lie in a dangerous or obstructive position, the robot should be able to move to the next operation and simply flag the failure in the batch report, provided the sample is not a key system suitability or quality control (QC), which may render the remainder of the analyses invalid.

There is a myriad of situations in which robotics can be used for sample preparation in high performance liquid chromatography (HPLC) and will produce a better result than the human laboratory worker might otherwise manage. Operations such as sample dilution (including serial dilution), filtration, and derivatization are all possible. There are sample robotics that can handle weighing, mixing, and even centrifugation, yet I don’t see these solutions being employed in many HPLC laboratories and I wonder why? I can state with certainty that the robot will follow a sample preparation or extraction protocol with much greater precision than a human if it has been properly “trained”. It will also follow the standard operating procedure (SOP) every time. Can we honestly say that we follow the SOP for every sample preparation that we undertake? Always shaking for the precise time, using the same method of agitation, using the same spot in the sonic bath, weighing or pipetting using exactly the correct technique—I could go on! Robots are infallible in terms of recall and repeatability, we are not.

While many sample preparation or extraction protocols in HPLC are relatively straightforward, there are those that are not so simple, such as liquid–liquid extraction (LLE) protocols. Method development is a time-consuming and costly activity, involving the systematic exploration of variables, with the aim of finding the optimum set of conditions for a robust method that yields data of an appropriate quality and cost. In method development, it is vital to vary the parameters under investigation in a stepwise fashion, automatically, and tightly fix all other parameters, so that the effect of varying a target parameter isn’t obscured by random variations elsewhere. Manual sample preparation is a significant source of unwanted variation and automating sample preparation—making method development a more straightforward and predictable process—spares a great deal of time. With more analysts taking a design of experiment (DoE) approach (where several parameters are adjusted simultaneously in a systematic way) to method development, automated sample preparation is a logical partner to reduce these unwanted variations but again, I don’t see widespread implementation and it really puzzles me as to why.

Could it be that we consider sample preparation for HPLC too simple? Not worthy of automation because it can be quickly and easily achieved within the sample preparation laboratory? Ask yourself how many batch failures or laboratory investigations have been related to issues with sample preparation?

You will note that up to this point I have concentrated on liquid chromatography for this discussion. I think the gas chromatography (GC) marketplace may be slightly different, more evolved even. There are several manufacturers producing advanced robotics systems for the preparation and manipulation of samples prior to GC analysis, and the complexity of the systems is much more advanced than I typically see allied to HPLC instruments. Furthermore, the systems are fully integrated and can inject the sample into the GC system, with just-in-time sample preparation capabilities. These systems are equipped with many tools that enable weighing, mixing, shaking, heating, centrifugation, solvent evaporation, and a host of other options. This enables operations such as sample dilution, addition of internal standards, derivatization, LLE, solid-phase microextraction (SPME), SPE, and other micro-extraction techniques to be automated.

Even in situations where large sample volumes were traditionally used, such as environmental analysis, automation has been made possible because of advances in mass spectrometric detection technology and sensitivity, which can be achieved using detectors such as triple quadrupole (QQQ) and quadrupole time-of-flight (QTOF) instruments. Essentially, the increase in detector sensitivity enables much smaller volumes of samples to be processed without compromising limits of detection or quantitation. Of course, this reduction in sample volume also means that the automated version of these techniques is not only greener (lower volumes of organic extraction solvents), but is achieved within the chromatographic run time, enabling “just-in-time” sample processing for all but the most complex preparation or extraction protocols.

Perhaps this latter point highlights a potential issue with the interfacing of HPLC to robotic auto sampling procedures. With the advent of ultrahigh-pressure liquid...
chromatography (UHPLC), chromatographic run times are typically very short, and lengthy sample preparation protocols would not match the time frame of the separative phase and the overall analysis time may be extended. Here the “batch preparation” robotics with multi-head probes used in bioanalysis (mentioned earlier) may have the edge because the overall campaign time may be reduced. However, when the benefits of improved fidelity, reproducibility, and unattended operation (from fully integrated robotic solutions) are considered, even one sample at a time approaches may still be of considerable benefit in HPLC analysis. Miniaturized sample preparation protocols can be very rapid indeed when using modern automated systems with optimized processing workflows.

So, what is preventing further implementation of robotic solutions in the GC laboratory, or the wider scale adoption of robotic approaches in the HPLC laboratory?

Below, I’ve listed reasons typically cited for low adoption of automation, specifically regarding sample preparation for chromatography analysis, as well as a short response for each.

- **Not enough budget for automation?**
  
  **Did you carry out a return on investment (ROI) calculation that included an improvement in right first time and a reduction in solvents and the energy to power fume hoods if these are used as part of your current sample preparation routine?**

- **Tried automation before and it didn’t deliver the promised benefits?**

  **Did you work with the manufacturer of the robotic system to fully explore the time, materials, and cost savings?**

- **My process couldn’t be automated**

  **I rarely encounter a sample preparation routine that cannot be automated with modern laboratory systems. As I’ve said above, SPE, LLE, weighing, sonication, centrifugation, heating, shaking, dilution, desorption, evaporation, reconstitution, and a host of other tasks can be completed using automated systems.**

- **My sample preparation is very straightforward, and I can’t justify the spend to automate something so simple**

  **Did you work with the manufacturer of the robotic system to fully explore the time, materials, and cost savings? Did you consider what that analytical chemist or technician could be doing while they aren’t doing sample preparation?**

- **Automated approaches can’t meet my sensitivity requirements**

  **This is invariably not the case when the sensitivity of modern mass spectrometry and other detectors are considered, and the workflow optimized to result in a sample of low enough volume to produce a robust detector response.**

- **I don’t trust automation; it adds more complexity and susceptibility**

  **Nothing is more complex or susceptible as a human. The repeatability, accuracy, and relative infallibility of modern robotic systems for sample preparation may be worthy of suspicion for their unerring performance, but I would find it difficult to question their susceptibility.**

I have perhaps left the most important emerging driver for automation to last, that of the Environmental, Sustainability, and Governance (ESG) agenda. As a society we are becoming “greener in our mindsets”, and while we may consider the major business wins in the green agenda to lie outside the laboratory, believe me, these initiatives are coming to a laboratory near you very soon. The reduction in power, solvent volumes, and exposure of analytical staff to potentially harmful solvents and reagents are clear when using automated platforms. They fit the ESG agenda perfectly and allow us to clearly demonstrate that we take the future of the planet very seriously! There are several analytical “greenness” calculators available; however, one that I particularly favour is from a collaborative group that includes the sample preparation impact of the method and can be found at this link (2): https://mostwiedzy.pl/pl/wojciech-wojnowski,174235-1/agreeprep

Revisiting the title of this piece, do we need to evolve or die? Of course, it’s a fact of every species on the planet. How true is this of automation of sample preparation for chromatographic applications? Well okay, I like to use a shocking title to draw in the reader, however, the flexibility of modern automated systems and the range of tasks they can complete has been revolutionized, even in the past 10 years. Their unflawing accuracy and repeatability is proven, and their ability to reduce worker exposure to both tedium and hazardous reagents or operations can also be clearly demonstrated.

Do we then just fear the “rise of the robots”? Surely not in our modern society, and I encourage you to further investigate the upsides of automation that, hopefully, I’ve been able to point out in this blog.

**References**

1.  https://www.therobotreport.com/5-things-robots-better-humans/


**Tony Taylor** is Chief Scientific Officer of Arch Sciences Group and Technical Director of CHROMacademy.

**Website:** www.chromatographyonline.com
# Training Courses

## GC

**GC Introduction**  

**GC Troubleshooter**  

**Operating and Understanding GC**  
Website: [www.crawfordscientific.com/training-consultancy/gc-training/gc-fundamentals](http://www.crawfordscientific.com/training-consultancy/gc-training/gc-fundamentals)

**GC Headspace**  
Website: [www.crawfordscientific.com/training-consultancy/gc-training/gc-headspace](http://www.crawfordscientific.com/training-consultancy/gc-training/gc-headspace)

**Hands-On Complete GC and GC–MS**  
16–20 January 2023  
The Open University, Milton Keynes, UK  
Website: [www.anthias.co.uk/training-courses/complete-handson-GC-GCMS](http://www.anthias.co.uk/training-courses/complete-handson-GC-GCMS)

## HPLC/LC–MS

**Understanding HPLC**  
Website: [www.crawfordscientific.com/training-consultancy/hplc-training/hplc-fundamentals](http://www.crawfordscientific.com/training-consultancy/hplc-training/hplc-fundamentals)

**HPLC Troubleshooter**  
Website: [www.chromacademy.com/channels/hplc-training-courses/troubleshooting/hplc-troubleshooter](http://www.chromacademy.com/channels/hplc-training-courses/troubleshooting/hplc-troubleshooter)

**Fundamentals of LC–MS**  
Website: [www.chromacademy.com/channels/lc-ms/principles/fundamentals-of-lc-ms-video-training-course](http://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](http://www.chromacademy.com/channels/lc-ms/principles/lc-ms-introduction)

## Hands-On HPLC Theory, Methods, and Maintenance

23–24 January 2023  
The Open University, Milton Keynes, UK  
Website: [www.anthias.co.uk/training-courses/handson-LC-theory-maintenance](http://www.anthias.co.uk/training-courses/handson-LC-theory-maintenance)

## SAMPLE PREPARATION

**Fundamentals of Solid-Phase Extraction (SPE) Mechanisms**  
Online training  

**Hands-On Solid-Phase Microextraction**  
17 March 2023  
The Open University, Milton Keynes, UK  
Website: [www.anthias.co.uk/training-courses/handson-SPME](http://www.anthias.co.uk/training-courses/handson-SPME)

## MISCELLANEOUS

**Introduction to Infrared (IR) Spectroscopy**  
Online webcast from CHROMacademy  
Website: [www.chromacademy.com/channels/infrared/principles/introduction-to-infrared-spectroscopy](http://www.chromacademy.com/channels/infrared/principles/introduction-to-infrared-spectroscopy)

**Validation, Verification, and Transfer of Methods for Biopharmaceutical Analysis**  
6–9 March 2023  
Online—virtual  
Website: [https://mournetrainingservices.com/method-validation-biopharma-course/](https://mournetrainingservices.com/method-validation-biopharma-course/)

---

**Please send your event and training course information to Kate Jones kjones@mjhlifesciences.com**
## Event News

### 1–3 February 2023

**The 10th International Symposium on the Separation and Characterization of Natural and Synthetic Macromolecules (SCM-10)**

Amsterdam, The Netherlands

**Email:** info@scm-10.nl

**Website:** http://scm-10.nl

### 1–3 March 2023

**Analytica China**

Shanghai, China

**Email:** info@analyticachina.com

**Website:** www.analyticachina.com

### 11–14 April 2023

**ANAKON 2023**

Vienna, Austria

**Email:** office@anakon2023.at

**Website:** www.anakon2023.at

### 17–22 September 2023

**6th International Mass Spectrometry School**

Cagliari, Sardinia

**Email:** gianluca.giorgi@unisi.it

**Website:** www.spettrometriadimassa.it/imss2023/#hero
Mission Statement
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.

Whilst every effort is made to ensure the accuracy of the information supplied, MultiMedia Healthcare LLC accepts no responsibility for the opinions and statements expressed.

Custom Reprints: Contact Brian Haug at MJH Life Sciences. E-mail: bhaug@mmhgroup.com

© 2022 MultiMedia (UK) LLC Limited all rights reserved. No part of the publication may be reproduced in any material form (including photocopying or storing it in any medium by electronic means and whether or not transiently or incidentally to some other use of this publication) without the written permission of the copyright owner except in accordance with the provisions of the Copyright Designs & Patents Act (UK) 1988 or under the terms of the license issued by the Copyright License Agency’s 90 Tottenham Court Road, London W1P 0LP, UK.

Applications for the copyright owner’s permission to reproduce any part of this publication outside of the Copyright Designs & Patents Act (UK) 1988 provisions, should be forwarded in writing to Permission Dept. e-mail: ARockenstein@mjhlifesciences.com. Warning: the doing of an unauthorized act in relation to a copyright work may result in both a civil claim for damages and criminal prosecution.