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Volume 41 Number 11 December 2023

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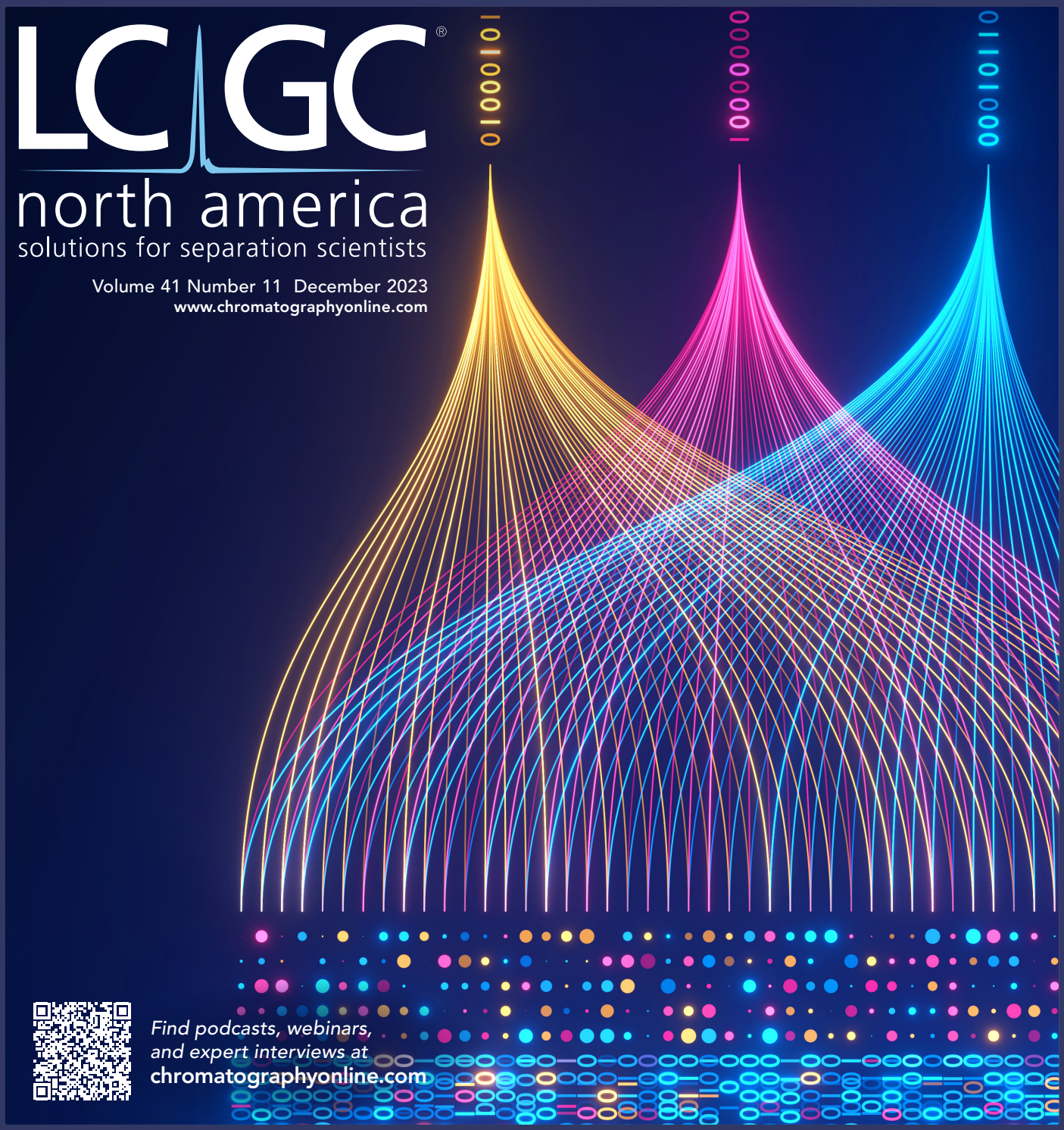

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2023–2024

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LCGC North America (ISSN 1527-5949 print) (ISSN 1939-1889 digital) is published monthly by MultiMedia Healthcare, LLC., 2 Clarke Drive, Suite 100, Cranbury, NJ 08512, and is distributed free of charge to users and specifiers of chromatographic equipment in the United States and Canada. LCGC is available on a paid subscription basis to nonqualified readers in the United States and its possessions at the rate of: 1 year (13 issues), $91.00; 2 years (26 issues), $163.00; in Canada and Mexico: 1 year (13 issues), $116.00; 2 years (26 issues), $182.00; in all other countries: 1 year (13 issues), $170.00; 2 years (26 issues), $304.00. Periodicals postage paid at Trenton, NJ 08650 and additional mailing offices. POSTMASTER: Please send address changes to LCGC, P.O. Box 457, Cranbury NJ 08512-0457. PUBLICATIONS MAIL AGREEMENT NO. 40612608, Return Undeliverable Canadian Addresses to: IMEX Global Solutions, P. O. Box 25542, London, ON N6C 6B2, CANADA Canadian GST number: R-124213133RT001. Printed in the USA.

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Note from the CEO

Mike Hennessy Jr.
President & CEO, MJH Life Sciences®

With 2023 drawing to a close, we're thrilled to present the 2023-2024 edition of our annual Resource Issue, a comprehensive guide featuring company profiles, product profiles, application notes, and a directory of companies.

We are also pleased to provide you with the quality editorial that you've grown to expect from *LCGC*. The effect of particle monodispersity in HPLC column performance is the topic of this month's "Column Watch," where we explore the impact of particle size distribution on chromatographic parameters, and discuss the trend towards improved monodispersity of silica particles for enhanced HPLC performance.

Nick Snow reflects on current state of sample preparation in GC in the first of a two-part "GC Connections." Discussing the results of a recent *LCGC* survey, Snow emphasizes the critical role of sample preparation in gas chromatographic analysis, setting the stage for a follow-up installment focusing on specific sample preparation techniques and their management.

"Focus on Food Analysis" examines automated aroma and flavor profiling of honey using high-capacity sorptive extraction. and demonstrates how this technique can combat food fraud by uncovering subtle differences between tested honey samples through automated statistical analysis.

Our peer-reviewed cover story, "Approaches to Accelerate Liquid Chromatography Method Development in the Laboratory Using Chemometrics and Machine Learning," explores innovative workflows and the implementation of computer-aided methods, emphasizing the role of machine learning in optimizing kinetic and thermodynamic parameters for liquid chromatography.

Finally, our featured article, "Multi-Analyte Method (MAM) for the Analysis of Agriculture Product Technical Ingredients and Formulated Products," provides valuable insights into pesticide industry laboratories. This article presents a multi-analyte method as a useful screening tool for regulatory laboratories, offering an efficient and accurate means of analyzing technical active ingredients used in formulated products.

As 2024 approaches, the world of separation science continues to advance at a dizzying pace, and we want you to know we are working hard to stay at the forefront of this scientific field and endeavor to keep our readers fully informed of the latest developments. We encourage you to explore these articles, columns, and features to stay informed and inspired by the cutting-edge developments in the field of chromatography.

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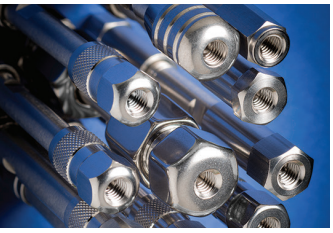

LC TROUBLESHOOTING

Troubleshooting Odds and Ends to Close Out 2023: Questions from the Mailbag, and Second Opinions

Similar to Stephen Covey's "Seven Habits of Highly Effective People," effective troubleshooters take a systematic approach to liquid chromatography (LC) troubleshooting and rely on several key principles to help them navigate the complex task of troubleshooting. In this installment I add to this list of principles described in prior installments with three specific ideas: 1) Ask—does it make sense?; 2) Get a second opinion; and 3) Join a chromatography discussion group to help grow your chromatography network.

Dwight R. Stoll

As 2023 comes to a close, I've been reflecting a bit on the problems with instrumentation that I've encountered this year, both through messages from folks in my professional network who ask me troubleshooting questions, and in my own laboratory. In the process of thinking about these various problems, a few themes have emerged, and I think it is worthwhile sharing them here because—at least on the timescale of a career spent working with instruments—they are relevant to all of us at some point. I'm a big fan of systems—systems-based approaches to tackling complex problems, and a systematic approach to problem solving. Maybe this is the training I had as a young scientist rubbing off on other parts of my work, but I think a systematic approach to liquid chromatography (LC) troubleshooting is very valuable because it leaves us with "results" that we can interpret and use to inform rational next steps in the troubleshooting process. If we don't follow a systematic approach, the only tool we have at our disposal is a trial-and-error approach, which is usually frustrating, and it can also be very expensive. In this installment, I elaborate on this point using troubleshooting cases I've encountered this year to emphasize important things to keep in mind, including getting a second opinion and the value of a robust professional network.

In the October 2020 installment of "LC Troubleshooting," I wrote about some essential principles of effective troubleshooting (1), referring to a manual developed by the U.S. Army that provides an introduction to logical troubleshooting (2). The list of principles included:

1. Change one thing at a time
2. Do no harm
3. Drawers are not repair centers
4. Build up knowledge about expected behavior
5. Documentation helps in the long run
6. Know when to ask for help

In this installment, I'd like to extend this list with two additional ideas:

7. Does it make sense?
8. Get a second opinion

Developing troubleshooting habits based on these principles can lead to more effective and efficient troubleshooting over time, reduce stress over instruments that don't work, and lead to significant savings in the cost of instrument operation.

Does It Make Sense?

A few troubleshooting cases this year have highlighted the importance of asking a basic question when considering potential causes of instrument malfunction, and potential solutions—does it (that is, the cause or solution) make sense? Part of what makes troubleshooting most analytical instrumentation challenging is that there are so many ways for things to go wrong. Given all these possibilities, it is also not hard to come up with a pretty long list of potential causes and solutions. Moreover, it is typically prohibitively expensive to actually evaluate all the potential causes and solutions in the laboratory. Thus, it is exceedingly important to critically evaluate each of them so that we can prioritize which ones from the list we will actually evaluate. Prioritizing the wrong possibilities can lead to a lot of wasted time, effort, and money working through the causes that are unlikely to be the real problem, and the solutions that are unlikely to work.

So, how do we actually evaluate the potential causes and solutions? I guess as individual troubleshooters we either need to have the expertise and knowledge needed to critically evaluate the

causes and solutions, or we need to seek out the expertise and knowledge from other individuals or sources. As I often say to my students, in 2023 the question is not whether information on a particular topic exists, but rather discerning which information is "good" (as in correct, accessible, and approachable), or "bad." Determining which troubleshooting advice is good or bad can be tricky, especially if it comes from a source unfamiliar to us. At a minimum, though, we can ask a simple, yet powerful question—does it make sense? A couple of months ago when I was playing around with ChatGPT, I asked it some LC troubleshooting questions. For example, I asked it: "What should I do if the pressure on my HPLC instrument is too high?" Some of the responses made good sense, including: 1) "Check the column: Make sure that the column is properly installed and has not been damaged. Check for any clogs or debris in the column that may be causing the pressure to increase"; and 2) "Check the flow rate: Verify that the flow rate is within the recommended range for the column and the instrument. If the flow rate is too high, it can cause increased pressure." On the other hand, some responses did not make sense at all. For example: "Check the detector: Verify that the detector is properly calibrated and functioning correctly. If the detector is not calibrated correctly, it can cause increased pressure." This simply has no basis in reality. How could calibration of a detector affect the pressure drop in an LC system? In this case, I would not seriously consider evaluating this potential cause of my pressure problems, and move on to other possibilities instead.

Another real-life troubleshooting example from the mailbag will help illustrate the value of asking—does it make sense? Earlier this year, a PhD chemist with considerable experience in LC reached out to me about a problem with a pump that had been elusive. They explained to me that the main symptom they observed was bubbles in the waste line of the pump during purging, and that after purging, the pump would slowly lose pressure to the point where the pump was unusable. They also indicated that the pump vendor suggested that the cause of these symptoms was that their method involved a high concentration of a commonly used organic solvent (and not tetrahydrofuran [THF], by the way). My first reaction upon hearing the facts of the case was that the stated cause of the symptoms simply did not make any sense. I could not imagine any physically- or chemically-based line of reasoning that would lead to this explanation. Although the suggested cause of the problem could possibly have been correct, it seemed improbable, and there would be many other explanations that I would put on a list before the one that was suggested. In the end, after further investigation, it turned out that the actual

cause of the bubbles was that the pump head was so loose that it was drawing air into the pump head from the back, and then pushing that air out of the head, which manifested as bubbles in the waste stream.

Get a Second Opinion, Particularly if the Fix is Going to Be Expensive

On two separate occasions in the past year, a troubleshooting exercise I've participated in has led to the conclusion that a very expensive repair would be required involving the replacement of an electronic board. In each case, the troubleshooting process was guided remotely by a person very knowledgeable about the particular instrument. In the first instance, I contacted a different person I knew that had intimate knowledge of the instrument, purely on a hunch that we should double-check to be sure we were not overlooking a different explanation for the cause of the symptoms. Indeed, the second person suggested a completely different potential cause, and it turned out that resolving this problem, which was entirely software-related, resolved the root cause and we did not have to replace any parts at all! Having had this experience, it was natural for me to seek a second opinion again a few months later when we encountered a different set of symptoms with the same instrument. Again, the suggested remedy was replacement of a very expensive electronic board. As in the first case, a second expert on the instrument suggested a different line of inquiry that ultimately concluded in finding that the observed symptoms were software-related and could be addressed at the level of the software, rather than replacing electronic components of the instrument.

No troubleshooter I know has a perfect track record, and sometimes the best of us misdiagnose. The point here is not that I think we can completely eliminate those mistakes and oversights. Rather, the lesson I've learned from these troubleshooting exercises is that if the conclusion of the exercise is that I'm going to spend a lot of time and money on a repair, then I will always get a second opinion moving forward. Much like it is common to get second opinions on health matters in cases where the suggested intervention is going to be painful, expensive, or both, we should also feel comfortable reaching out for a second opinion in cases of troubleshooting complex instrumentation where there is high potential for misdiagnosis and painful and expensive repairs.

Professional Networks Hold Tremendous Value—Grow Yours, and Use It to Help You Troubleshoot

I live in the upper Midwestern state of Minnesota in the United States, and we have a local (regional) professional organization for chromatographers and those with some affinity for chromatography called the Minnesota Chromatography Forum (MCF). I have been involved with MCF in various ways for approximately 20 years, but currently I chair the Symposium and Educational committees. At our most recent board meeting, we were discussing the trajectory of the organization and related factors including a lot of turnover in membership as we see longtime members retire, and many new people coming into the field. We also discussed how the Covid-19 pandemic has or has not affected how scientists and their employers interact with an organization like MCF. At one point during the discussion one of the board members asked rather pointedly—what is our response to people who ask what is the value of membership in MCF? In other words, why should they bother joining and regularly attend MCF events (typically three quarterly meetings and an annual symposium, in our case)? I think this is a fair question, especially in 2023, post-pandemic, in a time when it seems increasingly likely that people turn more to AI tools like ChatGPT for answers rather than consult the historical sources of expertise, such as books and expert sources like *LCGC* magazine. However, as I thought about the question after our board meeting, alongside of my thoughts about this installment of LC Troubleshooting, it occurred to me there is a deep and important connection here. A big reason to join a chromatography discussion group is that it can be a really efficient way to grow your professional network, and a network of 10, or 30, or 100 chromatographers that you can reach out to at the drop of a hat to get some troubleshooting advice is invaluable. Just think about this—an annual membership in the MCF costs a whopping $25. This fee buys you admission to quarterly MCF meetings where you get to know tens of other chromatographers in the area. There is a good chance that one of them will have good advice when you encounter the next problem with your LC instrument that you can't answer. If their advice saves you from a single visit by a service engineer, this will probably save you or your employer enough money to pay for a lifetime membership in the MCF.

On a related note, recently Dr. Jim Grinias and I interviewed Dr. Kevin Schug for *LCGC*'s podcast Analytically Speaking (www.chromatographyonline.com/analytically-speaking-podcast). I like to finish these conversations with some quick, light questions, and one of them is: "If you could wave a magic wand and solve any problem in your laboratory, what it would it be?" Kevin's answer (paraphrasing here) was that he would wish to have service contracts for all the instruments in his laboratory. As an academic researcher (Kevin is at the University of Texas at Arlington), instrument maintenance is an acute, persistent challenge because the organizations we typically seek funding from (as well as our host institutions), such as corporate sponsors and governmental agencies, are not fond of paying for instrument maintenance. My point in bringing this up here is that in laboratories where funds for service contracts simply are not available, hav-

ing a professional network to lean on for troubleshooting advice (and sometimes even loaning instrument parts!) is very valuable.

What Other Chromatography Discussion Groups Are There?

I don't have a comprehensive list of chromatography discussion groups around the world, but a considerable number of groups in the United States are listed at the SIS website: www.sisweb.com/referenc/sites/chmtgrop.htm. Some of the groups I have personally interacted with over the years are the Chromatography Forum of the Delaware Valley (CFDV; www.cfdv.org), the North Jersey Chromatography Discussion Group (www.njcg.org), and CASSS (www.casss.org). I especially encourage young scientists and new users of chromatography to find a discussion group and start building a network of separation scientists that they can reach out to in the future for troubleshooting or other professional advice.

Summary

In this installment of "LC Troubleshooting," I've discussed some habits and practices that can improve the effectiveness and efficiency of any troubleshooter. These include asking the question "does it make sense?" when considering the potential causes of a problem with an LC instrument, as well as potential solutions to solve the problem. Also, increasingly, I think that getting a second opinion on a diagnosis of an instrument problem is a good idea, especially when the solution that is suggested to solve the problem is expensive, intrusive, or both. Finally, I strongly encourage chromatography users to engage a local chromatography discussion group as a way of building a professional network of separation scientists. These networks are great resources for us to call upon when troubleshooting a specific LC problem, or looking for a second opinion.

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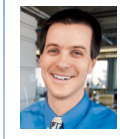

Dwight R. Stoll is the editor of "LC Troubleshooting." Stoll is a professor and the co-chair of chemistry at Gustavus Adolphus College in St. Peter, Minnesota. His primary research focus is on the development of 2D-LC for both targeted and untargeted analyses. He has authored or coauthored more than 75 peer-reviewed publications and four book chapters in separation science and more than 100 conference presentations. He is also a member of *LCGC*'s editorial advisory board. Direct correspondence to: LCGCedit@mmhgroup.com

COLUMN WATCH

The Effect of Particle Monodispersity in HPLC Column Performance

Many developments in silica particles used in liquid chromatography (LC) have been well documented over the years. The move from irregular silica to spherical silica, the decrease in particle size from >5µm particle in high performance liquid chromatography (HPLC) to sub-2-µm particle size in ultrahigh-pressure LC (UHPLC), the improved silica purity of type B silica over that of type A silica, and more recently the adoption of superficially porous particles compared to traditional fully porous particles. One area of development that has been discussed less, and is still open to debate, is the particle size distribution (PSD) of these chromatographic materials. In this article, we discuss the move towards improved monodispersity of silica particles for use in HPLC and how the use of monodisperse particles can impact the resulting chromatographic parameters such as reduced plate height and column impedance. On a practical level, we review how the reduction of PSD impacts efficiency, backpressure, and sensitivity.

Ken Butchart and Mark Woodruff

Chromatographic phases have come a long way since the early large pellicular particles that were in the range of 30–80 µm. In the 1970s, 10-µm and 5-µm spherical fully porous particles (FPPs) superseded pellicular particles and became the dominant particle of choice. Since then, particle sizes have been reduced still further with 3-µm and sub-2-µm diameters, adding a higher degree of efficiency to separations, albeit at the expense of increased backpressure.

Silica-based spherical FPPs have many benefits, including a high surface area, high physical strength, and robust nature of the columns produced. This is why silica remains today the number one support material for chromatographic stationary phases despite the availability of other materials such as carbon, polymeric supports, zirconia, and hybrid materials, which have all been evaluated over the years. Because of the high surface area and the highly reactive nature of amorphous silica, there are few limitations to the stationary phase types that have been bound to the surface to enhance selectivity. This combination of selectivity from the stationary phase and efficiency from the particle is paramount to any separation in high performance liquid chromatography (HPLC) and ultrahigh-pressure LC (UHPLC).

Particle Morphology

Many early silica powders developed for use in chromatography were generated by the destabilization and aggregation of silicate solutions. They possessed irregular morphology and broad particle size distribution coupled with high impurity levels from metal ion contaminants.

In 1968, Stober and associates (1) designed a process for growing non-porous silica particles. By mixing alkyl silicate with ammonium in a water-alcohol solution, the formation of spherical silica particles was achieved, with the ability to produce different particles sizes by altering the reaction conditions. The problem was that this produced non-porous particles only. Later developments led to surfactants and porogens being introduced as a modification to the Stober process (2), which allowed silica morphology to be manipulated, producing what we know today as fully porous silica particles.

Commercial silicas produced using variations of the modified Stober process tend to have a wide particle size distribution (polydisperse). Previously sub-2-um, monodisperse particles have been manufactured and been assessed in literature (3,4). However, the major drawback of these early attempts was the lack of scaling between particle sizes, meaning its use as a commercial phase was limited. By carefully manipulating the processing conditions, a new commercial, monodisperse FPP (MFPP) has been produced. This process has been tailored to produce particle sizes ranging from sub-2-µm through to 10 µm.

The difference in uniformity between polydisperse and monodisperse particles can be seen in Figure 1. The scanning electron microscopy (SEM) images of traditional HPLC particles and new monodisperse particles visually highlights the uniform size distribution of the new monodisperse particles.

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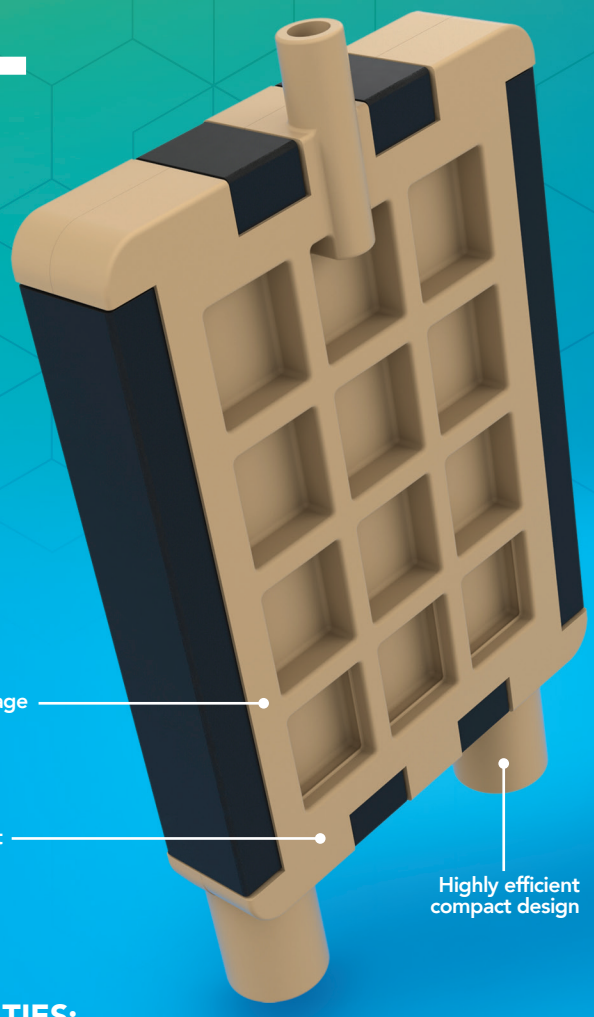

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distribution, techniques such as electrical zone sensing or laser diffraction are commonly used. To characterize the particle size distribution generated by either of these techniques, the ratio of $D_{90/10}$ may be applied to gauge the degree of size uniformity of the particles. The parameter D90 signifies the point in the size distribution, up to and including, 90% of the total volume of material in the sample is "contained." For example; if the D90 is 6 µm, this means that 90% of the sample has size of 6 µm or smaller. The definition for D50 is then the size point below, in which 50% of the material is contained. Similarly, the D10 is the size below, in which 10% of the material is contained. This description has long been used in size distribution measurements. As the particle size distribution for chromatographic silica moves towards monodisperse then the D90 and D10 values become closer together and the $D_{90/10}$ ratio moves toward a value of 1.

Using a Coulter Multisizer 3 (Beckman Coulter Inc.), an electrical zone sensing instrument, we measured two commercial 3-µm porous silicas and compared against the MFPP. Figure 2 shows the particle distribution of two commercial 3-µm spherical silica samples in comparison with the MFPP. As can be seen, both commercial silicas have a much broader size distribution than the monodisperse material, with one having a slightly smaller mean particle size and the other a slightly larger mean particle size than the 3 µm specified in the manufacturer's literature.

The $D_{90/10}$ values calculated from the size distribution data can be seen in Table 1, where the traditional particles possess $D_{90/10}$ values greater than 1.5. In comparison, the new monodisperse particles achieve a $D_{90/10}$ of 1.1. To this day, commercial silica columns containing particles with these high $D_{90/10}$ values (>1.5) are being successfully used with an acceptable level of column efficiency for the user. Theoretically, we should now expect these monodisperse particles to improve column performance.

Origin of Efficiency with a MFPP

Figure 3 shows a graph of the average column efficiencies obtained for twenty 150 x 4.6 mm columns packed with a 5-µm polydisperse C12 (internal Fortis Technologies bonding) with a $D_{90/10}$ value of 1.5. An average efficiency of 72,500 plates per meter (p/m) was achieved. A monodisperse 5µm particle exhibiting a $D_{90/10}$ of 1.1 with identical C12 bonding realized an average efficiency of 114,000p/m over 20 packed columns of identical dimension. Therefore, a gain in the region of 57% efficiency because of the difference in the monodispersity of the particles was achieved.

When the experiment was repeated with 20 columns of 3-µm particles, the average efficiency for traditional polydis-

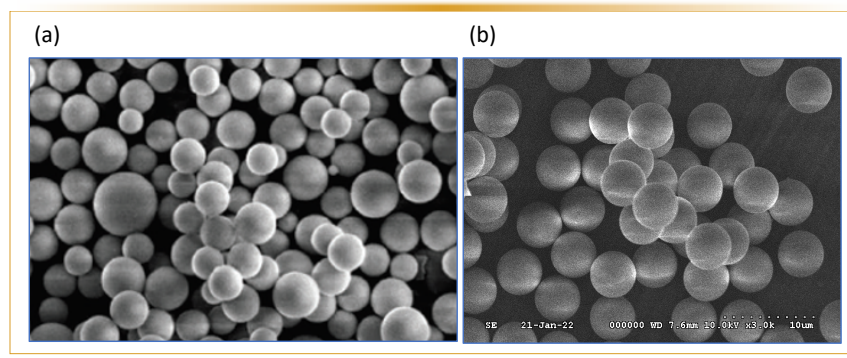

FIGURE 1: Particle SEM imaging, (a) commercial polydisperse silica particles, and (b) monodisperse particles (Fortis Technologies Ltd).

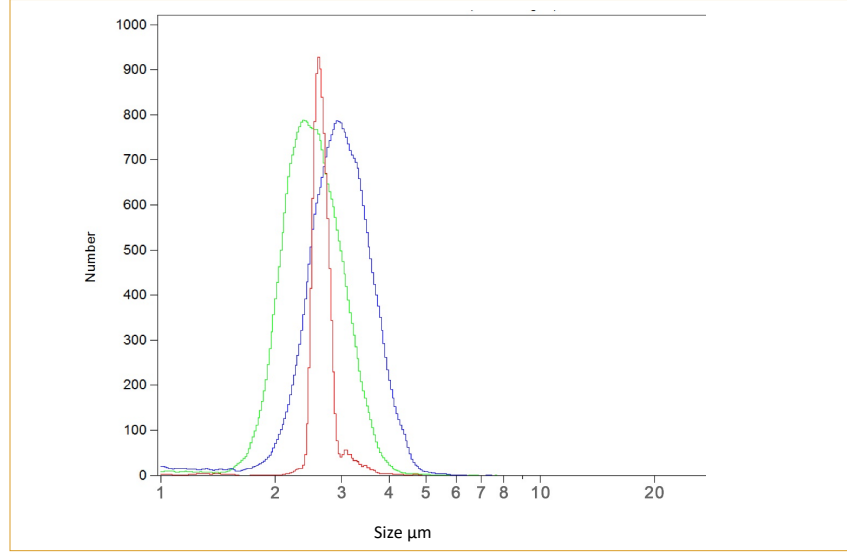

FIGURE 2: Particle size distribution measured by Coulter counter. Measuring aperture 50 µm, aperture current 800 µA, measurement diluent is Isoton II. Color Key: Green is commercial 3 µm silica sample A; Blue is commercial 3-µm silica sample B; Red is monodisperse commercial 3-µm silica sample. Resulting numbers are shown in Table I.

TABLE I: Results from Coulter counter analysis of three samples of commercial silica. Pore volume has to be similar so electrolyte concentration within the particle is the same.

	Monodisperse Silica	Commercial Silica A	Commercial Silica B
Mean particle size (d50)	2.66 µm	2.49 µm	2.97 µm
$D_{90/10}$	1.14	1.58	1.61
Pore volume ccg^{-1}	0.89	0.88	0.89

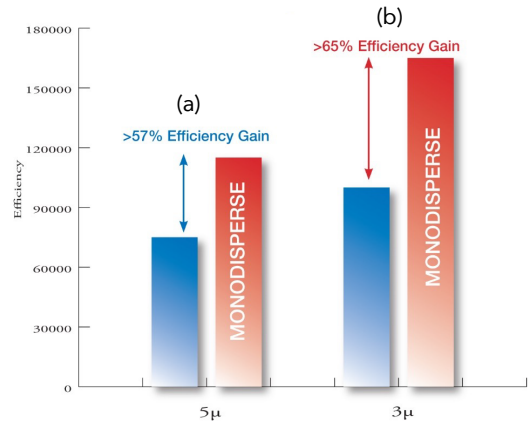

FIGURE 3: Efficiency gains over traditional porous particles. Each bar represents the Average of 20 packed columns of each material. (a) 5 µm traditional porous particles $D_{90/10}$ 1.5 and 5 µm monodisperse particles $D_{90/10}$ 1.1. (b) 3 µm traditional porous particle and 3 µm monodisperse particle. All columns: 150 x 4.6 mm; 60:40 acetonitrile:water, flow rate 1mL/min, wavelength 254 nm. Analytes: uracil, phenol, propiophenone, butyrophenone, napthalene.

perse 3-µm particles was 98,000 p/m. Monodisperse particles achieved 165,000 p/m on average.

Figure 4 shows a typical chromatogram from each batch of monodisperse and polydisperse columns or packed columns. Using the 3-µm C12 150 x 4.6 mm columns, efficiency is shown to be significantly higher on the monodisperse column, as would be expected. Efficiency is manifested chromatographically as improved resolution and sensitivity.

This research will continue to include multiple column diameters (that is, 3 mm and 2.1 mm i.d. columns) to ascertain the contribution from packing and to ensure it is representative across dimensions and is not adversely influenced by increased wall effects or the inherent difficulty in packing narrow bore columns.

So, What Causes This Efficiency Increase Seen in HPLC Columns Packed with Monodisperse Particles?

Assuming the particles are packed under controlled conditions and testing has been optimized (reduced dwell volume of system to a minimum), there will be three main parameters which contribute to lower reduced plate heights (5) (h_{min}) as defined in the van Deemter equation. The first term, A (Eddy diffusion) is the function of the size and distribution of the interparticle channels or nonuniformities in the packed bed. Secondly, the B term is inversely proportional to linear velocity so describes the molecules diffusion in the axial direction into and out of the pore structure. Finally, the C term which is proportional to linear velocity represents the mass transfer of the molecule from the solvent into the particle stationary phase and back again.

When superficially porous particles (SPP) with narrower particle size distributions came to market (6.7), it was widely assumed that their lower h_{min} counts were attributed to the narrower PSD, allowing the formation of homogenous packed beds and therefore, a reduced A term, coupled with a smaller impact from the rapid mass transfer of solutes into and out of the reduced porous shell structure. Further studies (8,9) have suggested that longitudinal diffusion into a lower pore volume could be a more significant contributing factor than was initially envisaged. With MFPPs, we should potentially see the same gains from the A term (Eddy dif-

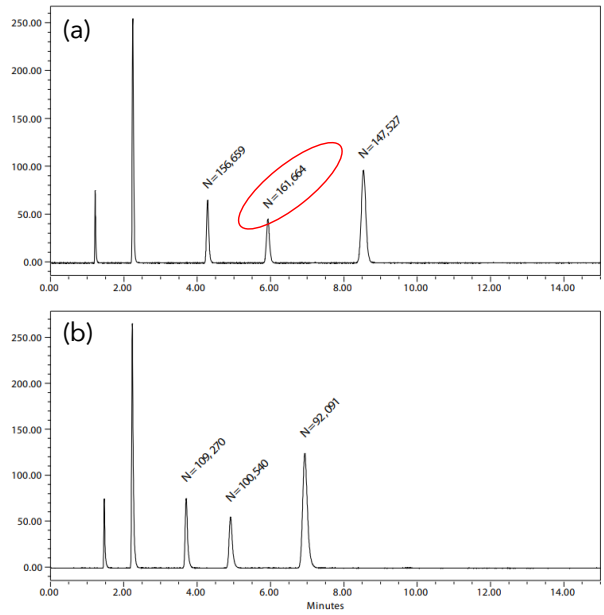

FIGURE 4: Chromatographic gain in efficiency. (a) 3 µm monodisperse C12, (b) 3 µm polydisperse. Both columns 150 x 4.6 mm, 60:40 acetonitrile:water, temperature: 25 °C, and a wavelength of 254 nm. Analytes: uracil, phenol, propiophenone, butyrophenone, naphthalene, and a wavelength of 254 nm. Axis labels are Time (min) for *x*-axis and Signal for *y*-axis.

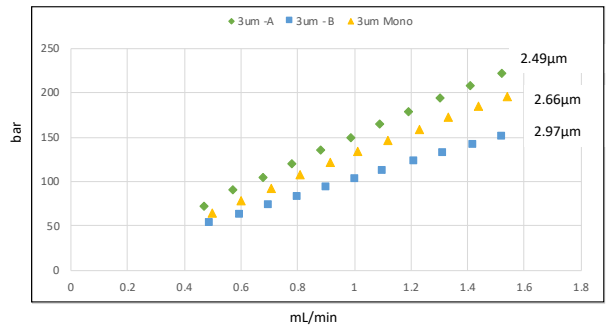

FIGURE 5: Impedance of two commercial particles and a monodisperse particle, all marketed as 3 µm in the manufacturer's literature. Size measured by Coulter counter.

fusion) as SPP particles first imagined. Since both have an almost identical level of monodispersity with $D_{90/10}$ values around 1.1.

If pore size, pore structure, pore diameter and mean particle size do not differ between MFPP and traditional porous particles in any significant respect, the flow path around a homogenous well packed column bed will have to be the main contribution to the much-enhanced efficiency seen for MFPP columns. Further study will be required to ascertain the individual contributions.

Effect of Monodispersity on Column Backpressure

By moving to monodisperse high efficiency particles, do we have a positive or detrimental effect on column backpressure?

There is evidence in the literature to suggest that it is difficult to differentiate between the contribution of PSD on the column backpressure as opposed to the impact of column packing process itself. Daneyko (10) suggested column packing was a high contributing factor. Cabooter (11) suggested a small PSD was key. Liekens (12), similar to Dewaele (13), fabricated mixed particle sizes to try and determine the impact of PSD, concluding that while there was no significant drop in h_{min} when used at optimum linear velocity, at higher elution speeds there was both a drop off in efficiency and also significant increase in backpressure.

Anecdotally, we see in our early evaluations that, although column packing processes impact on the resulting efficiency, there is a clear improvement in column performance when monodisperse particles are compared with polydisperse particles without any discernible increase in backpressure. These new MFPP in various particle sizes will aid in the evaluations since until now narrow PSD was only really available with SPP particles.

In our laboratory, we studied the column backpressure of commercial silicas, each possessing different PSDs (Table I). This was done by packing each silica under identical pressure and flow conditions, to ensure parameters such as sedimentation rates played no part in the ensuing results. Silicas were selected that possessed the same pore volume and pore diameter. From the results shown in Figure 5, we can see that although the mean particle size affects the backpressure, the degree of monodispersity ($D_{90/10}$) did not. This would tie in well with the opinions of Dewaele.

Gains in Loading Capacity

Because of the much higher surface area available on the MFPP than the SPP, sample loading can be increased without loss of peak shape, efficiency, and therefore, resolution. Currently, this is where we see limitations in the performance of SPP particles.

The monodisperse particles studied in this article have a surface area of 350 m^2/g thereby offering higher retention as well as increased loading capacity. When peak overload occurs, critical separations, such as a parent peak and its impurities, become compromised and the loading capacity is significantly reduced potentially up to a 10-fold difference.

A loading study was performed, for both SPP and MFPP, by steadily increasing the mass of sample that was injected

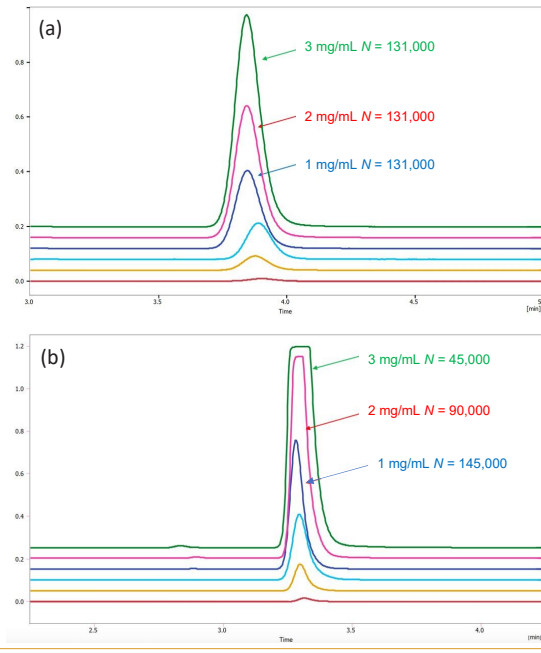

FIGURE 6: Loading study on (a) monodisperse fully porous particle, and (b) SPP particle. Compound: napthalene at 6 different concentrations. Both columns 150 x 4.6 mm, mobile phase 50:50 acetonitrile:water. Axis labels are Time (min) for *x*-axis and Voltage for *y*-axis.

onto the respective column (Figure 6). It can clearly be seen how efficiency drops rapidly as overload quickly occurs on SPP particles. The importance of this issue is significant when considering the development of analytical methods for any new molecular entity (NMEs) that will ultimately require purification once they move to a manufacturing process. The ability to move from a UHPLC sub-2-µm particle in initial method development to a 5-µm or 10-µm FPP in a production environment relies on the ability to have a scalable particle at a non-prohibitive cost that is available in multiple column diameters. Because of the processing costs involved in the manufacture of SPP materials, they are often not commercially available in larger preparative column dimensions, and those that are can be prohibitively expensive. By contrast, these new MFPP materials are produced by conventional manufacturing methods at no more expense than the current commercial silicas. Therefore, they can be supplied in all preparative column formats at a cost affordable to most production sites requiring this type of chromatography.

Is Efficiency Enough?

The primary goal of HPLC is the separation of one or more analytes within a mixture. Focusing on increasing efficiency alone is analogous to overcoming only part of the challenge posed by separation science.

Selectivity is an often-overlooked concept as the majority of analysts will start method development with the selection of a C18 stationary phase running a simple aqueous: organic mobile phase. Although this approach is great for simplicity and with a relatively high percentage success rate, it does not always allow for the separation of complex samples or closely related species. The separation challenges that lie ahead include isomers and molecules with near identical structural properties as well as complex samples from, for example, biological sources. These challenges require the resolving power of stationary phase selectivity.

If we can expand on the traditional stationary phase characteristics by introducing not just alkyl chain hydrophobicity (that is, C18 and C8), but by their halogenated, aromatic, or polar character, then these secondary mechanisms will promote greater analyte-stationary phase engagement. Figure 7 shows the separation of a diverse mixture of compounds on two, predominantly C18 alkyl chain phases, however one has aromatic secondary character and one has pentafluorophenyl (PFP) secondary character. It can be clearly seen that in some areas of the chromatogram the phases appear to be quite similar in selectivity, whereas with some of the components there are large shifts in resolution between analyte pairs. This is the type of movement that can then be tuned for the NME and its impurities in initial method development screening.

Further surface modifications produced by manufacturers to offer enhanced selectivity are far too numerous for the scope of this article and have been characterized in multiple ways, such as the Tanaka (14) protocol, Engelhardt (15) test or the Synder, Dolan (16) hydrophobic-subtraction model. The reader should take some time to review as much of this work as possible as it offers a good insight into the diversity available in stationary phase design over the years along with many good examples of application development to solve challenging separations

The more selectivity that can be imparted from the stationary phase the less demand there is to manipulate the mobile phase, temperature or pH range. Changes in the organic mobile phase component seem to have become somewhat limited in RP chromatography because of miscibility, safety, cost, and environmental impact of solvents. Fully porous monodisperse particles available with a wide range of orthogonal stationary phases will therefore allow the separation scientist to optimize selectivity and speed of analysis without the need for exhaustive method development or pushing the extremes of pH, additives or solvent mixtures.

This simplification leads to more robust, reproducible methods which is a productivity gain within our development process.

Conclusion

There have been many developments in particle technology over the years aimed to improve performance, result-

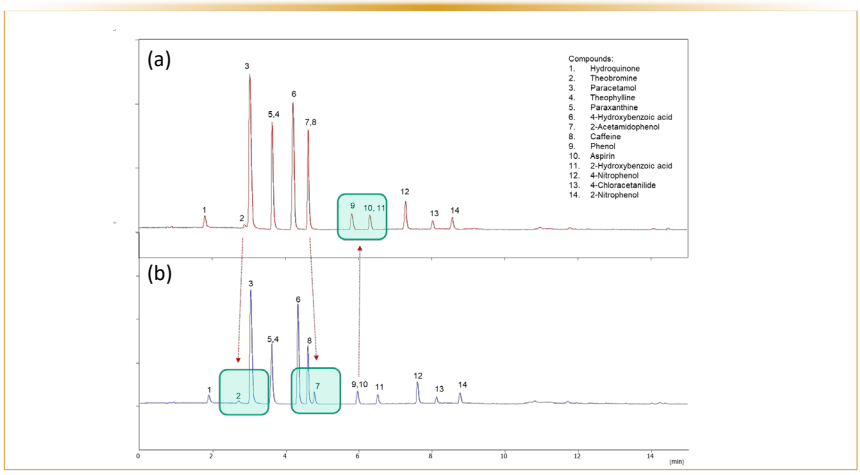

FIGURE 7: Selectivity on a (a) monodisperse C18/AR, and (b) a monodisperse C18/PFP. All columns are 3 µm 100 x 2.1mm. Mobile phase A: 10 mM ammonium formate pH 3.0, B: 10 mM ammonium formate pH 3.0 in a cetonitrile. Flow rate is 0.4 mL/min., wavelength is 254 nm, temperature is 40 °C. Compounds are: 1. hydroquinone, 2. theobromine, 3. paracetamol, 4. theophylline, 5. paraxanthine, 6. 4-hydroxybenzoic acid, 7. 2-acetamideophenol, 8. caffeine, 9. phenol, 10. aspirin, 11. 2-hydroxybenzoic acid, 12. 4-nitrophenol, 13. 4-chloracetanilide, 14. 2-nitrophenol. Axis labels are Time (min) for *x*-axis and Signal (voltage) for *y*-axis.

ing in chromatography with increased speed, efficiency, and sensitivity. However, these changes always appeared to provide a benefit at the expense of another parameter, whether that be the high backpressure trade-off for the efficiency gain in UHPLC, or the limited sample loading and cost trade-off made for the efficiency gain seen with SPP particles. With the development of a monodisperse fully porous particle (MFPP) there is now a commercially available silica support that brings performance benefits without associated drawbacks.

MFPP particles have now been optimized to a degree where commercialization has now been achieved. Improving the particle morphology has led to gains in efficiency over existing porous particles in the region of 50–60%, giving greater sensitivity and resolution. These gains are attributed to the reduced A-term as a result of the better flow path through the interstitial spaces in the packed column bed.

Our studies show when comparing equivalent 3-µm monodisperse packed beds and polydisperse packed beds we see no discernible backpressure differences that could be attributed to the improved monodispersity. By increasing monodispersity we have in effect simultaneously eliminated both small and large particles, and their respective contribution to increasing and decreasing back pressure is therefore canceled out. Hence, we see no net increase in backpressure.

By maintaining a fully porous structure in these monodisperse particles we show the high surface area leads to high sample loading capacity. Our loading studies showed a >4-fold increase in capacity on a simple pharmaceutical molecule when comparing to a high efficiency core-shell particle.

We acknowledge how even with the enhanced efficiency of a monodisperse particle, a choice of orthogonal stationary phases is critical to the more complex samples faced in the current pharmaceutical and biomolecule workflow. This topic will be covered in further articles.

Further study of these monodisperse particles will revolve around additional comparison with SPP, trying to understand all of the contributions leading to the source of the efficiency gains. Assuming the A-term, particle size and packing density are similar with both, what is the difference between C-terms? Also, can you compartmentalize the column packing process impact from the particle architecture contributions?

In summary, MFPP offers high efficiency, high surface area with homogenous well packed column beds for analytical through to preparative scale chromatography. Method development and method transfer can move seamlessly from initial UHPLC through to preparative scale production environment. In particular, 3-µm MFPP particles have been shown to offer gains in efficiency similar to UHPLC particles without a trade off in increased backpressure or loading capacity.

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ABOUT THE CO-AUTHORS

Ken Butchart is co-founder and Research Director of Fortis Technologies Ltd and has master degrees in Chemical Technology and Business from Bradford and Strathclyde Universities. Over the past 25 years he has worked directly in the chromatography industry, specifically on the manufacture of silica particles and the design and commercialization of stationary phases for HPLC. With a proven track record of successful product development, he has presented his research and development data at symposia worldwide. His continued focus remains on the improvement of particle and phase chemistry performance in order to provide the analytical chemist with the best tools for achieving their desired chromatographic separations.

Mark Woodruff co-founded Fortis Technologies Ltd in 2005, having spent 10 years developing stationary phases for what used to be the Hypersil brand. Mark worked as the Technical manager and the Global Product Manager for HPLC columns at Thermo Hypersil. Mark has a BSc and Masters in Analytical Chemistry as well as an MBA from Manchester University (UK). Mark is currently involved in the development and marketing of new columns from Fortis Technologies and has an excellent relationship with many key people in major pharmaceutical companies in order to ensure that the needs of the industry are met.

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GC CONNECTIONS

Reflecting on the Influence of the Current State of Sample Preparation on GC, Part 1

Nearly all gas chromatographic analyses are preceded by sample preparation, which is often the major determiner of success. Recently, *LCGC* surveyed its readers about their work in sample preparation, covering the overall state of the field, and the results were presented in the "Sample Prep Perspectives" column. In this installment, we discuss the impact of part 1 of this survey, which covers broad questions about who does sample preparation, the fields they work in, and their most important aspects and goals for sample preparation on the gas chromatographic analysis that follows. We see that sample preparation has been (and remains) a critical step in any gas chromatographic method. In a second installment, we will discuss the impact of specific sample preparation techniques and their management on gas chromatography, following the release of part 2 of the sample preparation survey.

Nicholas H. Snow

Sample preparation and sample injection are often the most challenging steps in a full gas chromatographic method. Gas chromatography (GC) is generally amenable to liquid and vapor phase samples, and in most applications, the sample must be in the vapor phase as it enters the column. This can be seen in the most common injection techniques, split and splitless, in which a liquid sample is injected using a syringe into an inlet where it is vaporized. Sample preparation for GC can be online, with direct interfacing, usually through a transfer line, between a separate sample preparation technique, such as static headspace extraction, or offline, with samples prepared separately, aliquoted into vials, usually as solutions, and injected as liquid samples.

Table I provides a summary of some sample preparation techniques commonly used with GC for solid, liquid, and vapor samples. Typically, for solid samples, the solid is dissolved or otherwise treated, followed by one of the techniques for liquid samples. Commonly, solid samples are dissolved in a solvent, but they can also be extracted using a number of instrumental techniques, including headspace extraction, accelerated solvent extraction, and microwave assisted extraction. Liquid and vapor samples can be injected "neat," but usually require sample preparation to remove nonvolatile or reactive sample components.

In *LCGC* surveys reported in 1991 (1) and 2023 (2), we see that sampling and sample preparation occupied over 50% of analyst time in a typical analysis, so the importance of sample preparation cannot be overlooked. In both surveys, analysts were also asked about the most important error sources in their methods, with the sample preparation-related responses of sample processing, operator and contamination combining for just over 50% of the responses in 1991 and about 37% in 2023. In 2023, calibration was stated as the largest single contributor to error, while in 1991, it was sample processing. This change was attributed to the rise in automation over the past 30 years. Interestingly, the major sources of error in most gas chromatographic methods are likely outside of the instrument, either in sample preparation and handling (which happens prior to the chromatographic analysis), or data analysis (which happens following chromatography).

Subtleties Fundamental to All Sample Preparation Techniques

From Table I, we see that nearly all sample preparation techniques used with GC involve a phase transition and phase equilibrium, just as GC depends on the theory of equilibrium between the mobile (vapor) and stationary (usually liquid or solution) phases. A brief review of phase equilibrium applied to GC and sample preparation will show principles common to all sample preparation techniques. The chemical equation and equilibrium constant expression for a phase transition, shown in equation 1, are among the simplest that describe a chemical process, but there are many subtleties.

$$A_{sample} \Leftrightarrow A_{extract} \quad K = \frac{[A]_{extract}}{[A]_{sample}} \quad [1]$$

The first subtlety is the consequences of the value of the equilibrium constant. This determines how much of the analyte can be transferred from one phase to the other in a single extraction step. In GC, a single extraction step is analogous to a single theoretical plate, as we saw last year in our discussion of the classical Golay equation (3,4). Figure 1 shows a comparison of several common extraction methods, including solid-phase microextraction (SPME), liquid-liquid extraction (LLE), and low-volume LLE. The mole ratio of a hypothetical analyte extracted from an aqueous phase into an organic phase is

plotted against log *K*. If *K* is large, indicating that the analyte is much more soluble in the organic phase than the aqueous phase (as is commonly seen in gas chromatographic methods, when extracting organic compounds from water or other aqueous systems into an organic phase), then a nearly complete, or quantitative, transfer of the analyte from the aqueous phase to the organic phase is possible.

The second subtlety, also seen in Figure 1, is seen in the relative volumes of the sample (in this case aqueous) phase and the extract (organic) phase. The micro-liquid-liquid extraction (MLLE) case is a low, but roughly equal, volume of both phases, volumes that would fit into a single autosampler vial; the high case is a large volume of aqueous phase extracted with a small volume of organic phase, while the low case is like MLLE, but using a smaller volume of organic phase and a large volume of aqueous phase. Finally, SPME represents an extreme volume difference with the very low-volume coated fiber immersed in a large volume of aqueous sample.

The third subtlety is that while equilibrium constants may be very large or very small, they are finite. This means that in any extraction system, even with a very large constant, some amount of the analyte will remain in the original phase. This challenge opens an additional idea that has not been explored much in the literature. If the equilibrium constant is small, multiple extractions form the same sample, using a fresh extraction phase, can result in appreciable extraction of the analyte when the separate aliquots of the extraction phase are combined.

A fourth subtlety is that for any chemical reaction or process, especially extraction, reaching equilibrium takes time. Even in the simplest of systems, a "dilute and shoot" procedure, the diluted liquid must be mixed. When diluting one liquid by another, how does the analyst know that the two liquids have fully mixed into a homogeneous mixture? Mostly, we count on indirect evidence based on our judgment of the time and vigorousness of mixing, appearance of the solution, and reproducibility of the data. The same questions apply to simply dissolving solids. Mostly, we determine that the solid has dissolved when we no longer see particles, and we also know that no solid dissolves instantly.

TABLE I: Sample types amenable to common sample preparation techniques for GC (S = solid, L= liquid, G = gas).

Technique	Sample Types
"Neat" injection	L, G
Dissolving in a solvent	S, L
Liquid-liquid extraction (LLE)	L
Solid-phase extraction (SPE)	L
Sorbent Extractions (SPME, SBSE)	L, G
QuEChERS	L
Headspace extraction (SHE, DHE)	S, L
Membrane extraction	L, G
Supercritical fluid extraction (SFE)	S
Accelerated solvent extraction (ASE)	S
Pyrolysis	S
Thermal desorption	S
Microwave assisted extraction (MAE)	S

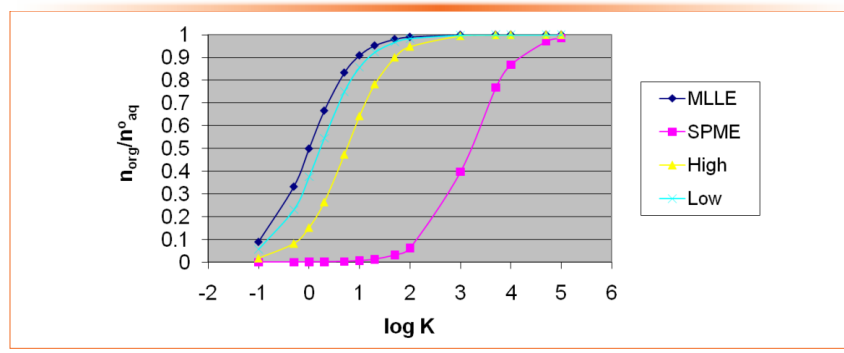

FIGURE 1: Comparison of some extraction methods. MLLE–liquid-liquid extraction with both phases of low volume; SPME–solid-phase microextraction; High–large volume of aqueous phase extracted with a small volume of organic phase; Low–like MLLE but using a smaller volume of organic phase and a large volume of aqueous phase.

In short, when diluting or making solutions, allow sufficient time for complete mixing.

We see, from the simplest of techniques, that all sample preparation methods require time to come to equilibrium. This amount of time may be on the same order of or longer than the time required for the chromatographic separation. Our fifth subtlety in all sample preparation techniques is kinetics. While equilibrium determines how much analyte we can transfer between phases, kinetics determines how long it takes. Due to the variety and complexity of most samples, the kinetics of extraction must be determined experimentally and may vary widely for seemingly similar samples.

Fortunately, coming back to the simplicity of equation 1, most extraction techniques follow or approximate first order kinetics. In the simplest case, we have individual particles partitioning between two phases. Extraction kinetics may be complicated by additional matrix components in the sample phase that bind or adsorb the analytes of interest. In any method development involving extraction, the kinetics of extraction should be carefully studied. A kinetic plot for a simple and fun extraction is shown in Figure 2. This is the extraction of fats from a chocolate bar using supercritical fluid extraction (5). In this case, time is measured by the volume of supercritical carbon dioxide delivered at constant flow. This is a quantitative extraction example, but it shows the key elements of extraction kinetics.

For the best reproducibility and maximum recovery, extraction should be allowed to proceed for long enough so that the amount extracted is expressed on the flat,

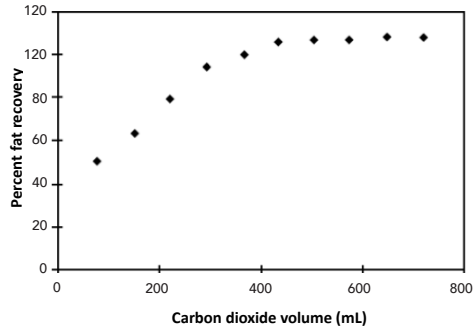

FIGURE 2: Extraction of fat from a candy bar using supercritical fluid extraction. Reprinted with permission from reference (5). Copyright 1996, American Chemical Society.

nearly horizontal part of the curve. Small variations in the extraction time or conditions will have the least effect on this portion of the curve. Shorter extraction times, where the curve is steeper, can be used with calibration, but at the risk of larger uncertainty and impact of minor variations in the extraction conditions.

A sixth subtlety is the experimental uncertainty inherent in the use of glassware for volumetric measurements and sample transfers. The most common experimental error that impacts all analytical methods, including GC, is the experimental error and overconfidence involved with any use of volumetric glassware, which is often assumed to be error-free, but is not. A second, often hidden, error is seen in the volume of the glassware itself, as pipets and flasks have increasing relative uncertainty as their volumes get smaller. This is discussed in detail in a previous column, where we see that smaller volume flasks and transfer pipets introduce larger experimental uncertainties, and that, with improved instrumentation over the decades, these uncertainties may eclipse those from the instrumentation or other method steps (6). In short, there is a tradeoff of increased experimental uncertainty when using serial dilution methods or any method that requires additional sample transfers and volume measurements, which use smaller solvent volumes and single-step dilution, which uses larger volumes.

In summary, six subtleties in sample preparation result from the seeming simplicity of equation 1.

- The equilibrium constant determines how much can be extracted.
- The relative volumes of the two phases determine how much can be extracted.
- Equilibrium constants, large or small, are finite. There is always some analyte in both phases.
- Dissolving and mixing take time.
- Extractions are subject to kinetics.
- Glassware has inherent experimental uncertainty.

Returning to the survey results, Raynie points out some interesting changes since 2003 that, along with the subtleties, provide some interesting challenges and observations about the current state and future of sample preparation for GC. In the survey, several shifts are seen since 2013, with new fields, including cannabis, cosmetics and personal care and a drop in the percentage of users reporting work in pharmaceuticals. In all industries, there are both increased regulation and pressure to "go green" with reduced solvent and consumable usage, miniaturization, more efficient laboratories along with lower detection limits and reduced experimental uncertainties (7). Interestingly, the subtleties can work against these pressures, as the kinetics of extraction often work toward longer extraction times and lower sample throughput, and the glassware uncertainty favors larger samples and more solvent usage. When evaluating techniques, users report the greatest importance in price, time, recovery and low contamination.

Sample preparation and GC are inherently connected. One cannot run a gas chromatograph without samples, and all samples require some amount of preparation, from the simplicity of loading "neat" samples into vials, through the complexity of on-line instrumental extractors, such as accelerated solvent extraction (ASE), supercritical fluid extraction (SFE), and headspace. All extraction techniques are subject to equilibrium, kinetics and experimental uncertainty, as is GC. With GC becoming simplified over the decades, the often-overlooked time, complexity, kinetics, and equilibrium involved in sample preparation may often be the most important and challenging steps in the full method.

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FOCUS ON FOOD ANALYSIS

Automated Aroma and Flavor Profiling of Honey Using High-Capacity Sorptive Extraction

Honey is prone to food fraud, where either a less desirable honey is misrepresented as a more desirable one, or honey substitutes are used to bulk the original product. This article demonstrates how high-capacity sorptive extraction can be used to extract aroma compounds spanning a wide volatility range from different honey samples. Automated statistical analysis was used to uncover subtle differences between the honey samples, to determine possible markers of authenticity and help to combat fraud.

Rachael Szafnauer

Honey is a natural aromatic sweetener comprising 95% water and sugars. The remainder comprises flavonoids, proteins, vitamins, free amino acids, and volatile organic compounds (VOCs), which give different honey varieties their distinctive characteristics (1). Unfortunately, honey is open to food fraud, where an inexpensive honey is misrepresented as a more desirable one, or honey substitutes such as cheap sweeteners or sugar syrups are used to add bulk to the original product (2). Traditional authentication techniques, such as solid-phase microextraction (SPME) and solvent extraction, are becoming obsolete because they involve time-consuming sample preparation and pollen analysis by specially trained analysts. As a result, a new type of analysis is required. This article demonstrates how a high-capacity sorptive extraction technique can be used to sample key aroma compounds in different types of honey samples to assess their potential for use as unique markers of authenticity. The different properties of the compounds in honey mean that sorptive phase combinations must be selected carefully to maximize uptake and achieve the most comprehensive profile.

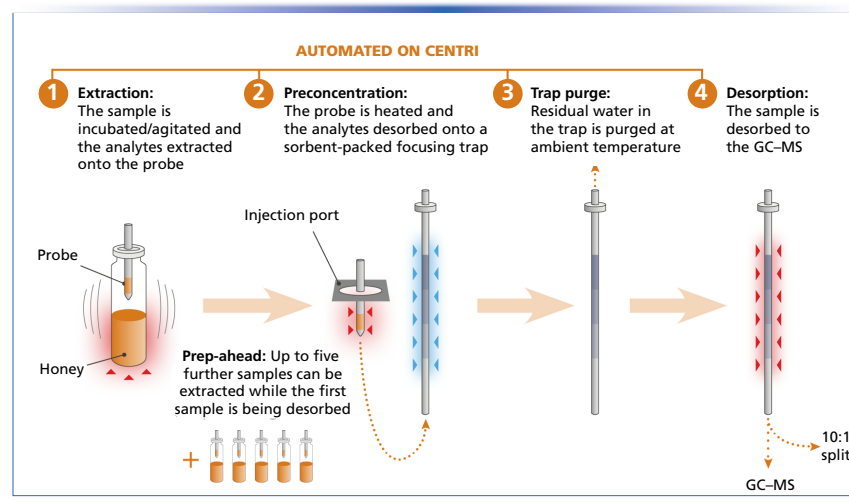

FIGURE 1: Automated sample extraction workflow on the Centri platform.

Experimental

The samples analyzed were five different varieties of honey, including a mass market honey, hobbyist honey, forest honey, Manuka honey, and Welsh honey. A sample of golden syrup was also used as a sixth sample for comparison. The samples were prepared in a 20 mL headspace vial containing 1 g of sample and 1 mL of water. Vials were crimp-capped and sonicated, and the analytes extracted from the headspace (Figure 1). For headspace extraction, HiSorb DVB/CWR/PDMS inert-coated (H4-AXAAC) probes were used, with the whole process being fully automated using a Centri 360 extraction and enrichment instrument (all Markes International). Each sample was analyzed five times for reproducibility, and the scan range on the gas chromatography–mass spectrometry (GC–MS) system was 35–450 *m/z*. The software used for data mining and chemometrics was ChromCompare+ (SepSolve Analytical).

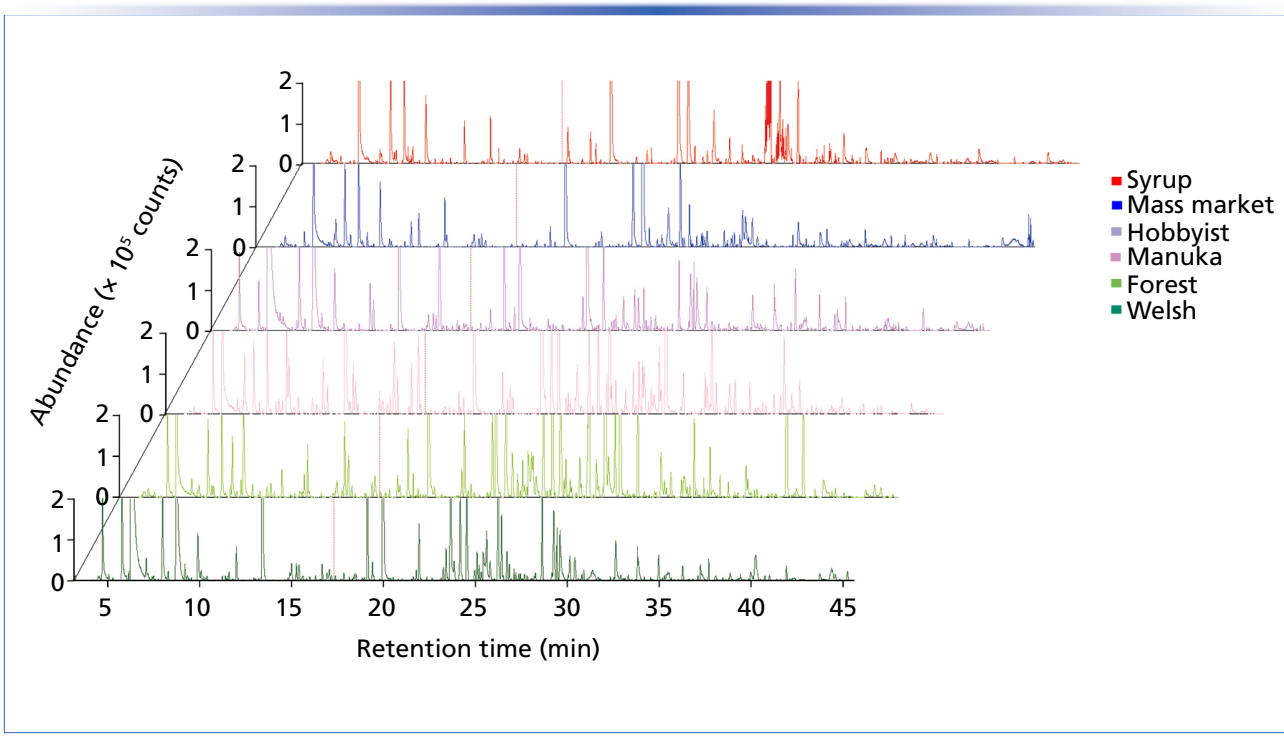

FIGURE 2: Aroma profiles of syrup (red), mass market (blue), hobbyist (purple), Manuka (pink), forest (light green), and Welsh honey (dark green).

Results

Aroma Profiling of Honey

The aroma profiles generated for each sample (Figure 2) indicated that some compound classes were common to all the honey samples, but others differed between samples. Each class contributed characteristic aromas; for example, ketones, common in all samples, confer buttery and nutty odors.

Multiple esters were discovered in all samples, which was not unexpected given that this group of compounds provides sweet and fruity aromas. Aldehydes were prominent, bestowing fresh, green, and herbal notes. Alcohols, which add fresh flavors to honey, were a large component. They can occur naturally or as a result of heat treatment during processing (3).

Furans, known for their sweet woody notes, are also common in honey and are typically present because of the dehydration of reducing sugars in the matrix. Their presence can also indicate that thermal processes and storage after collection from hives have degraded the sugars in the honey (2).

Overall, the honey profiles had common descriptors of fruity and sweet notes. However, naphthalene, an aromatic hydrocarbon commonly derived from coal, was also highlighted as an odor in one honey sample. This off-odor may have come from the smoke used by beekeepers to calm bees before removal of honey from hives (4).

Combating Food Fraud Using Markers

To distinguish the key variances between samples, a custom library of VOCs in each sample was created using the

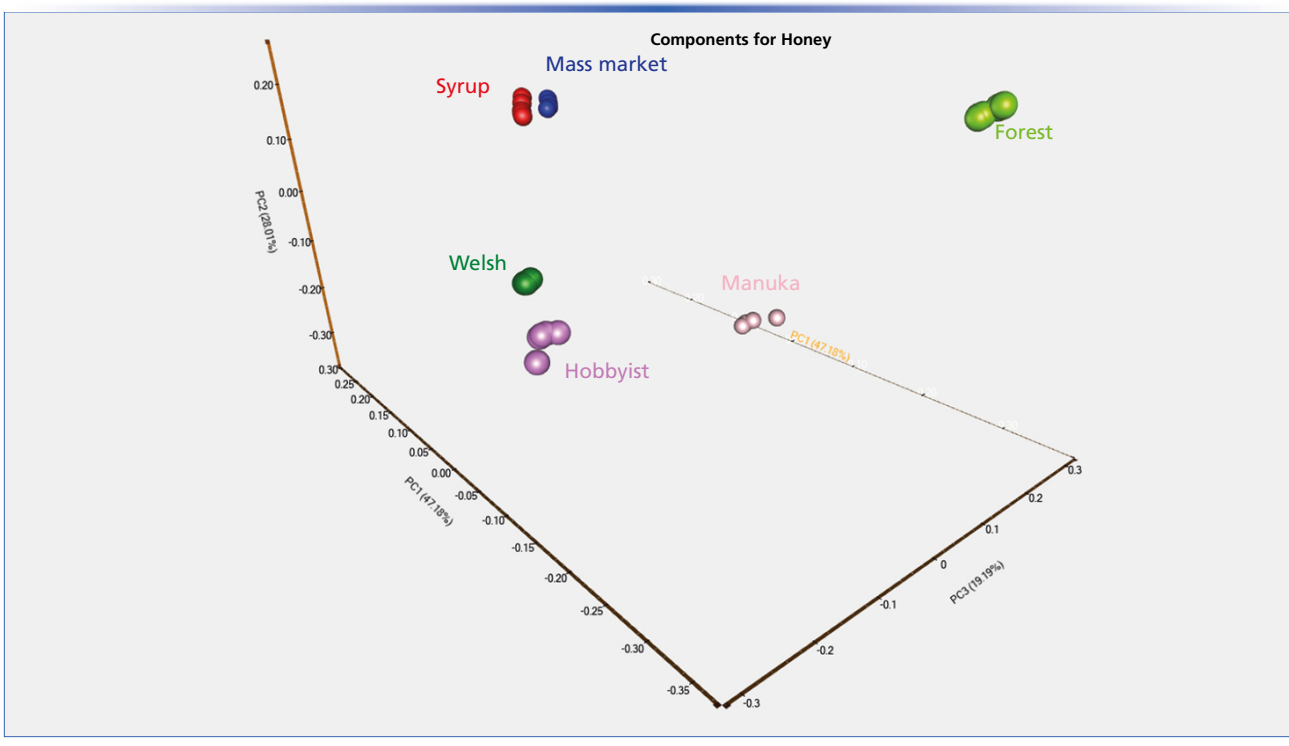

FIGURE 3: Principal components analysis score plot in the software for the comparison of honey varieties extracted using DVB/CWR/PDMS HiSorb.

software, and provided quantitative and qualitative analysis of one-dimensional (1D) and two-dimensional (2D)-GC–MS data. A total of 79 compounds with match factors above 850 and probabilities above 85% were used to generate the library. Alignment and comparison of the chromatographic data shown in Figure 2 led to the formation of a principal components analysis (PCA) score plot (Figure 3).

The mass market honey was very similar to the syrup sample, as they both cluster closely in the PCA plot, indicating that additional sugars may have been added to bulk the honey product. The Welsh and hobbyist honeys also had similarities, suggesting that they have a similar geographical origin. The Manuka and forest samples were the most distinctive varieties, which can be seen by the distance in the clustering compared to the other honey samples.

Twenty key aroma compounds were determined as differentiating significantly between the samples. Figure 4 demonstrates the abundance of these compounds by sample type. Compounds that are specific to a particular sample have the potential to be used as markers for declarations of sample origin.

The unique marker compounds found in the luxury Manuka honey are 1-methoxy-4-methylbenzene, which has a phenolic, minty aroma, 4′-hydroxyacetophenone, which has a floral, sweet aroma, methyl salicylate, which has a wintergreen, mint aroma, and o-methoxyacetophenone, which provides an anisic, almond, and cherry aroma. As these compounds were only present in the Manuka honey, they could be used

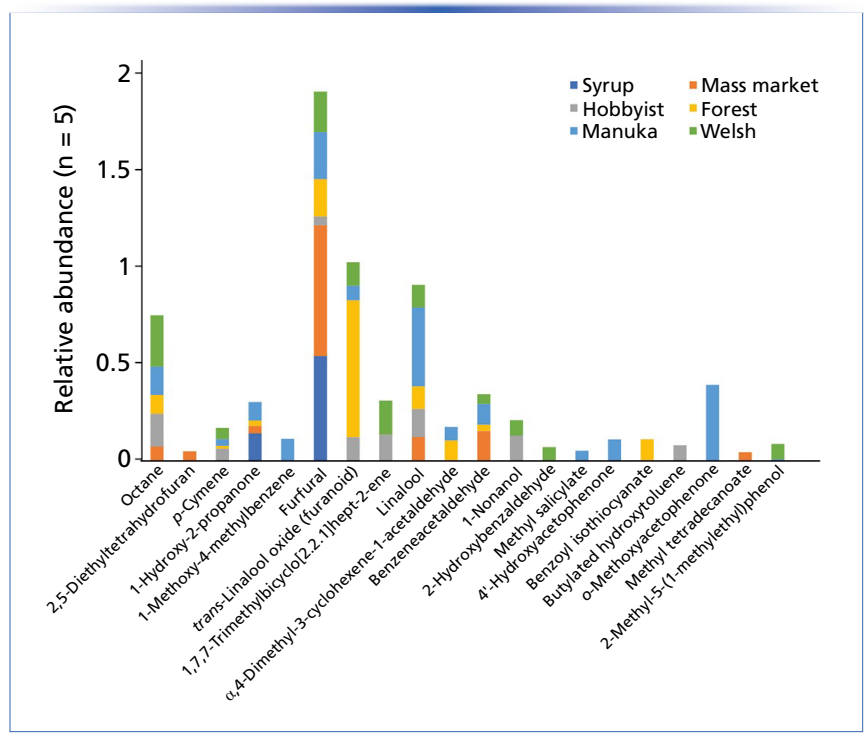

FIGURE 4: Comparison of relative abundance for the top 20 differentiating compounds identified in five honey varieties and a golden syrup.

as confirmatory markers when classifying samples of unknown origin that are labeled as Manuka.

Conclusions

Analysis of the VOC content of foodstuffs is important in the food and beverage industry, with a wide range of applications in research and development, quality control, shelf-life assessment, and the detection of food fraud. A common technique in food VOC profiling is sorptive extraction, wherein compounds partition from food onto a sorptive phase and are transferred to analytical instrumentation such as a GC–MS system for separation and detection (5). Sorptive extraction offers a highly sensitive, robust, and fully automatable extraction solution, and provides excellent results in GC–MS-based analysis of foodstuffs.

This article has demonstrated that sorptive extraction is a highly efficient technique for the VOC profiling of honey samples. The technique was used to extract a wide range of compounds, particularly those with key aroma and flavor properties.

This article has additional supplemental information only available online. **Scan code for link.**

ABOUT THE AUTHOR

Rachael Szafnauer received an MSci in forensic science from the University of South Wales, UK, where her final-year project focused on fingerprinting emerging psychoactive substances using advanced techniques such as GC×GC–TOF-MS, in collaboration with Markes International. She later took up the role of thermal desorption product specialist at Markes, providing technical and application support to the commercial team, before taking on her current role as product marketing manager and specializing in the development of applications using extraction and enrichment techniques for GC–MS.

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Approaches to Accelerate Liquid Chromatography Method Development in the Laboratory Using Chemometrics and Machine Learning

Liquid chromatography (LC) is the single largest analytical field in terms of people involved and money spent. LC is crucial for almost all public and private sectors and the technique has seen tremendous technological advancements. Nevertheless, separations are often performed under suboptimal conditions and technological capabilities remain unused. Because expert knowledge and method development time are increasingly scarce, methods are often inefficient. Exploiting the full technological capabilities of liquid-phase separation technology requires deep knowledge and great time investments. Method optimization strategies that can simultaneously optimize the large number of parameters involved are therefore of great interest to chromatographers. This review examines different workflows that have been designed and used to facilitate and/or automate method development. In particular, focus is paid to the implementation of computer-aided workflows for the optimization of kinetic and thermodynamic parameters in LC, as well as on the possibilities to conduct this in a closed-loop fashion. Finally, the opportunities to use machine learning to achieve these goals is addressed.

Gerben B. van Henten, Tijmen S. Bos, and Bob W.J. Pirok

Decades of research and development have allowed liquid chromatography (LC) to become the reliable and robust technique that we know today. This has paved the way for the development of increasingly advanced two-dimensional LC (2D-LC) methods with increased peak capacities, better use of mass spectrometry (MS), and new opportunities such as in-line reactions (1). For complex samples a 2D-LC method can be much faster than a one-dimensional (1D)-LC counterpart (2,3).

The development of 2D-LC has largely been driven by the growing interest of society and industry in understanding the increasingly complex and diverse samples, each with their own challenges associated.

The need to implement more powerful separation technologies is growing faster than the number of qualified users of multidimensional chromatography. The leading cause hampering further proliferation is the considerable amount of resources required for method development. This task is ever more daunting as method parameters are numerous and often interdependent, increasing the experience required to use all capabilities effectively.

This is illustrated in Figure 1, where the technological complexity (that is, the cost) is sketched against the information density (the benefit). There is a disproportionate increase in resources required to obtain further information. State-of-the-art separation technology such as 2D-LC can only make an impact if it can be efficiently employed.

To facilitate this, researchers worldwide have been designing strategies with software to simplify method development (4–6). These and other method development and optimization strategies capitalize on theoretical understanding to simplify the method development process, as well as maximize its potential—similar to the motto "work smarter, not just harder" (7). This review will map the method development process and identify categories of parameters that can be targeted. Different method optimization approaches that simplify or even automate sections of this workflow will then be examined. Finally, the opportunities to use machine learning to achieve in this context will be addressed.

Objectives of Chromatographic Method Development

Chromatographic method development is affected by the aim of the method. The optimization approaches for LC method development can be roughly classified as targeted and untargeted (8). Targeted approaches focus on specific analytes, whereas untargeted methods attempt to characterize the entire sample. This article will focus on untargeted methods, as targeted methods tend to have dedicated goals that cannot easily be generalized.

Generally, an analyst will start method development by using any information available on the sample chemistry, for example, Giddings' sample dimensions (9), literature, and other resources to select an initial column (often a general-purpose column available in the laboratory) and generic method parameters (a generic linear gradient).

After the initial instrument parameters are established, the method can be evaluated based on its ability to provide the required critical information. When the method does not provide this information, method parameters can be adjusted to improve the method. This

process is generally termed *optimization*. In chromatographic literature, the term *optimization* often represents divergent intentions based on the objective and the stage of the method development process. Indeed, "optimization" can refer to sample preparation (10), kinetic parameters that affect efficiency (11), the selection of a suitable stationary-phase chemistry (12), a reduction in analysis time (8), and so on. The common denominator in each case is that method parameters are adjusted with the aim of obtaining the desired information more effectively. Typically, this is evaluated through metrics that quantify the quality of separation, also known as *quality descriptors*.

To maximize the ability of a method to characterize a sample, untargeted approaches are often driven by such quality descriptors. For example, in the case where an analyst encounters a chromatogram saturated with peaks, maximization of the peak capacity (that is, the number of peaks that can be separated by the method) can be employed as a quality descriptor.

Untargeted optimization suffers from the vast number of adjustable parameters and the large interdependence between these. It is inherently difficult to predict whether a change in parameters will result in an improved method. Improving one metric (analysis time) may result in worsening another metric (resolution). Computer-aided development of a chromatographic method thus requires a balanced approach.

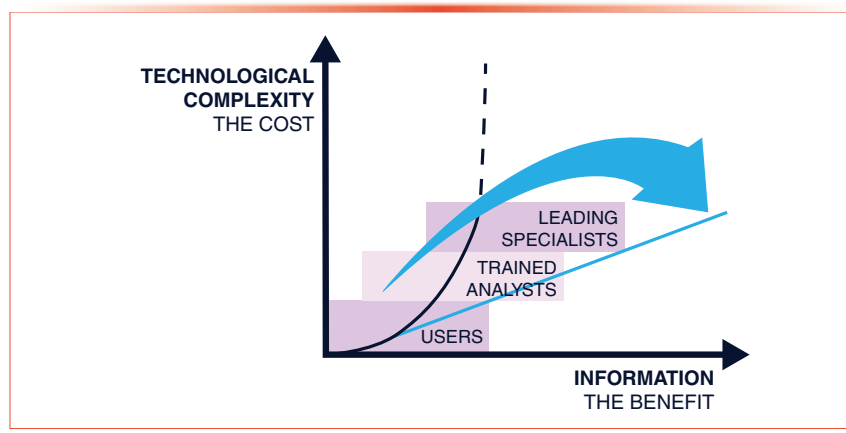

FIGURE 1: Sketch illustrating the disproportionate increase in technological complexity (cost) to acquire more information (benefit) from an analytical method.

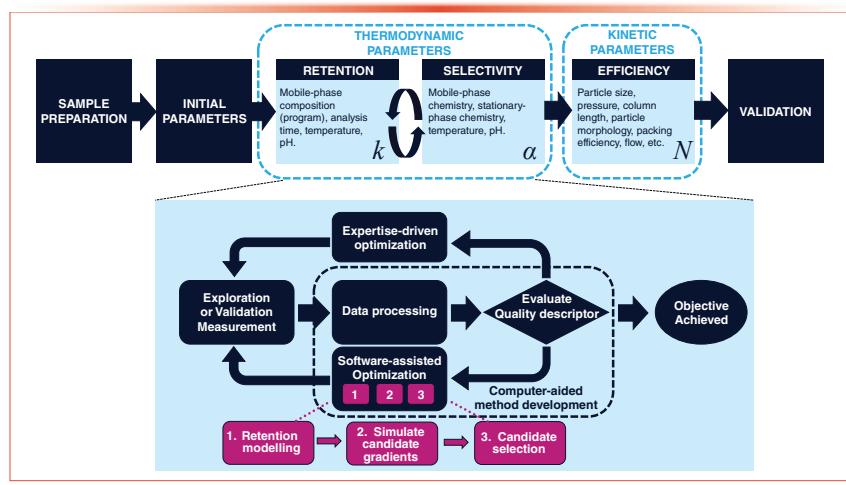

FIGURE 2: Example of a generic representation of a method development workflow.

Resolution to Adjust a Chromatographic Method:

One useful quality descriptor that quantifies the degree of separation, yet also instructs on possibilities to improve it, is the resolution (equation 1):

$$R_S = \frac{t_{R,2} - t_{R,1}}{2\sigma_1 + 2\sigma_2} \quad [1]$$

Here, the separation between analytes 1 and 2 is expressed with t_R and σ representing the retention time and peak standard deviation. When assuming that both analytes experience a similar column efficiency (that is, $N_1 = N_2$), we can rewrite equation 1 as follows (13):

$$R_S = \frac{\sqrt{N}}{2} \cdot \frac{\alpha - 1}{\alpha + 1} \cdot \frac{\bar{k}}{1 + \bar{k}} \quad [2]$$

Now, we have resolution quantified as the product of three contributions: efficiency (N), selectivity (α), and average retention factor (k). Equation 2 demonstrates that the only means to adjust the separation is to change something within either of these three parameters.

At this point, it is important to acknowledge the significance of sample preparation and the crucial effect this has on the method development workflow. Sample preparation tends to be highly sample dependent, and it has been extensively treated elsewhere (14,15). It is outside the scope of this review.

Equation 2 can be schematically expressed in the context of method development workflows as depicted in Figure 2, which represents a typical case often faced by chromatographers. After having used their experience to select an appropriate column and initial method parameters, chromatographers are recommended to first adjust the method conditions by altering thermodynamic parameters that affect retention and selectivity. For example, they may change the gradient program or—if necessary—adjust the selectivity dramatically by selecting a different column or mobile-phase chemistry.

Finally, they may opt to improve the efficiency or analysis time of the method by regarding the kinetic parameters. Different approaches that have been developed to facilitate method development through automated workflows or chemometrics will be reviewed in the following sections.

Kinetic Parameters

Improving the efficiency (that is, the theoretical plate number) of the separation provides a clear metric relevant to method optimization, as it increases the peak capacity for the separation and decreases the likelihood of coelution. For 1D-LC it is possible to predict how different parameters will affect the efficiency. Another method is the use of kinetic plots (16). However, the square-root dependence of R_s on N implies a rapidly diminishing return on investment.

In comprehensive two-dimensional liquid chromatography (LC×LC), kinetic optimization schemes can be applied to substantially improve the method. This is attributed to the additional parameters, including the second-dimension (^{2}D) column dimensions, and phenomena such as under-sampling (of the ^{1}D effluent) and ^{2}D sample-dilution (17). Vivo-Truyols and associates proposed the use of sample-independent Pareto optimization (PO) for LC×LC (18). In this chemometric-driven approach, multiple method parameters (column dimension, flow rates, gradient slope) were varied and the effects thereof were evaluated using (theoretically related) quality descriptors such as peak capacity, analysis time, and dilution factor. The authors demonstrated that optimizing the kinetic parameters can be highly rewarding and that this approach is suited to deal with tradeoffs between different objectives. The PO method provided optimal values for a large number of parameters, including ^{1}D and ^{2}D particle sizes and column diameters, while taking into account pressure restrictions.

This work was extended by the group of de Villiers (11,19,20), who applied the PO strategy for the chromatographic separation of a phenolic red-wine extract (19), implemented a predictive kinetic-optimization tool for a hydrophilic interaction liquid chromatography×reversed-phase liquid chromatography (HILIC×reversed-phase LC) separation of procyanidins (11), and used the same tool to compare reversed-phase LC×HILIC and HILIC×reversed-phase LC for phenolic analysis (20). In all cases, optimization of kinetic parameters provided higher peak capacities and produced valuable knowledge on the influence of under-sampling, dilution factors, and band broadening.

Thermodynamic Parameters

Selectivity in liquid chromatography is influenced by the thermodynamic parameters related to analyte interactions with the mobile and stationary phases. Changes in mobile phase composition, gradient program, stationary phase chemistry, pH, or temperature can significantly affect the distribution of peaks in a chromatogram. While modifying the mobile phase, pH, or temperature is relatively straightforward in theory, in practice this may not suffice to meet the objectives of the separation. Changing the stationary phase in principle requires a re-optimization of the mobile phase, making it more expensive in terms of effort (apart from the column cost), but generally provides broader changes in selectivity.

Various strategies and workflows have been described to select appropriate chemistries for both the mobile and stationary phases in LC. One such strategy involves optimizing the parameters through a Design-of-Experiments (DoE) workflow, in which sets of parameters (for example, mobile phase, temperature, pH) are varied and an extensive screening is performed, often on multiple columns, to determine the optimum approach. Similar approaches are commonly used in the industry (21). They may be efficient in terms of manpower required, but can be quite inefficient in terms of numbers of measurements, time, solvents, and instruments required.

For column selection, stationary phase characterization methods aim to simplify the end user's choice of stationary phases based on characterizing the physicochemical interactions with small-molecule probes. For an extensive review the reader is referred elsewhere (22). The best-documented example is the hydrophobic subtraction model (HSM) developed by Snyder, Dolan, and co-workers, which may be used to select columns with different selectivity (23).

Recently, workflows have emerged related to selectivity screening for 2D-LC method development. For example, Zhang and associates developed a multiple heart-cut 2D-LC setup for pharmaceutical purity testing that enabled automated screening of the ^{2}D selectivity (24). In other work, Wang and co-authors (25) used an automated column screening framework for analyzing chiral and achiral impurities, with online multiple heart-cutting 2D-LC; in a similar study, Zhang and co-workers described fast chiral and achiral profiling of compounds with multiple chiral centres by an LC–multicolumn-LC approach (26). Together with automated data acquisition, these workflows constituted a step forwards, but interpretation and processing of the data are still dependent on manual input and user experience.

Interpretive Automation

In an ideal scenario, a method development workflow must be capable of integrating all previously discussed parameters and optimizing these in an automated and interpretive manner. In this context, the word *interpretive* implies that the chromatograms are interpreted as a result of the elution of the individual analytes. In the workflow the peaks should be tracked and simple models may be established for each analyte—all of this in an unsupervised and automated manner. Based on the analyte models, many chromatograms can be simulated and the objective function can be optimized. Such a method development workflow has the potential to minimize manual input and the expertise required for developing complex methods.

Paradigm Shift in GCxGC Workflows: Unlocking the Potential of Complex Sample Characterization

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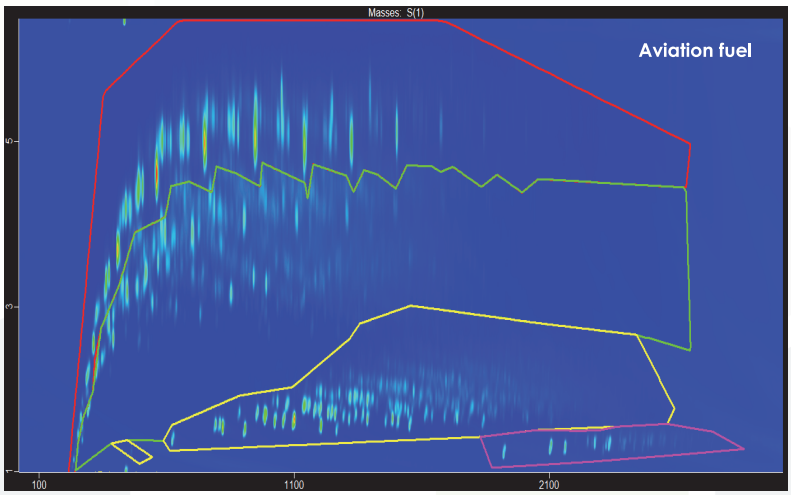

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Bos and associates developed the AutoLC approach, in which the mobile phase gradient was optimized in a closed-loop fashion (27). Based on an earlier theoretical LC×LC proof-of-principle study (28), and the pioneering work of Dolan and co-workers (29), Bos and co-authors designed an algorithm that could accommodate different chemometric optimization strategies. Two examples were presented: one using empirical retention modelling, and one using the machine-learning Bayesian optimization technique. In both cases, the algorithm developed multi-segment gradient programs to optimize the retention and selectivity of separations of a monoclonal antibody digest by LC–MS.

The examples above demonstrate the possibility of including computer-assisted optimization strategies for kinetic and thermodynamic parameters in automated workflows. However, despite the significant progress made, three major challenges remain that need to be addressed to achieve a closed-loop, interpretive algorithm for developing LC methods in a completely unsupervised and automated manner.

The first and arguably most important challenge is to define quality descriptors that can assess the quality of a separation. The success or failure of the optimization process critically depends on the objective function. The latter depends on quality descriptors that, in turn, may depend on chemical and physical parameters. The objective function serves as the primary driving force for every algorithm. Single-number descriptors, known as *chromatographic response functions* (CRFs), have been developed to quantify the critical information that the method is intended to provide. Various CRFs have been proposed in the past (30). Although many CRFs exist for 1D-LC, few are available for 2D-LC (31–36). Alvarez-Segura and co-workers recently included peak prominence in their CRF (33,34), Huygens and associates (35) introduced a CRF for 2D-LC based on peak purity, and Boelrijk and co-authors (36) presented a CRF based on connective components in graph theory to assess both separation quality and time. It has also been demonstrated that the selection of inappropriate quality descriptors can lead to suboptimal separations (27). Therefore, it is crucial to create CRFs that can effectively weigh quality descriptors, while the program provides quantitative information regarding various practical aspects of analytical queries, such as method time and sensitivity.

The second challenge is related to the data processing. The quality of the output of an algorithm relies strongly on the input data and the data processing. Any error in background correction, peak detection, or peak tracking can lead to cascading errors in later iterations (27). While MS provides information to reduce the errors in 1D-LC, data processing remains a significant challenge for LC×LC. This is especially the case for peak tracking, where an algorithm must establish the location of each analyte across multiple signals and account for incorrect clustering of coeluting peaks (37,38). In addition, it is difficult to quantitatively compare the performance of the large number of data processing approaches available (27,39).

The third challenge concerns the computation costs associated with the large number of simulations and the number of parameters that need to be optimized. Improving the performance of the algorithm either requires more measurements or more simulated data that resemble the experiments more closely. The former increases the amount of time and solvent needed, while the latter requires more computational power. Machine learning techniques may be employed to reduce the computation time required.

Machine Learning

Interest in machine learning methods is rapidly gaining momentum in the field of chromatography (7). The increasing computational power available, combined with the demand for advanced simulation tools, has led to the exploration of machine learning approaches for accelerated method development (40,41). Machine learning has the potential to enhance various stages of the chromatographic-analysis workflow, including baseline and noise correction (42), retention-time or retention-index prediction (43–45), peak detection (46,47), peak annotation (48), and classification (49). This section will focus on the utilization of machine learning to optimize the gradient program, which frequently relies on several of the aforementioned stages.

One type of machine learning is artificial neural networks (ANNs). The application of neural networks in chromatography has been explored in the past (50–53). In a recent study, Kensert and associates (42) investigated the potential use of reinforcement learning for selecting optimal isocratic scouting runs for retention modelling. The neural network was trained on simulated data using a Q-Learning algorithm and evaluated based on the accuracy of the retention predictions for 57 small-molecule analytes in situ experiments. While the work is promising, the algorithm from this study has only been tested on isocratic data for a specific reversed-phase LC setup with a limited number of simplistic molecules. A downside of the use of artificial neural networks is that they require a large amount of data for training. This can be mitigated with simulated data, but this carries a risk of the model not capturing the actual chromatographic space with sufficient accuracy (46).

Several authors have explored the potential of evolutionary algorithms to optimize gradient profiles (35,54). In particular, Hao and co-authors (54) used a genetic algorithm to optimize multi-linear gradient profiles for the separation of lignin-degradation products. They validated their simulations by comparison with experimental measurements and found good agreement. Huygens and associates (35) investigated evolutionary strategies for optimizing ^{1}D and ^{2}D separations through *in silico* experiments of an LC×LC separation of 100 experiments. Their genetic algo-

rithm required less than 100 experiments to reach a better CRF than a grid search of 625 experiments. This suggests that evolutionary algorithms have the potential to guide search-based method development, accelerate computational model-based method development, and deal with many parameters simultaneously. However, the authors simplified the experimental conditions by assuming perfect orthogonality, equal concentrations, and perfect Gaussian peaks. This raises concerns over the risk of oversimplification and the underutilization of the full range of variations in chromatographic conditions, as can also occur with neural networks.

One of the biggest limitations of applying machine learning is the required amount of data. An emerging optimization method that is less dependent on the amount of input data is Bayesian optimization, a type of machine learning tool that can optimize expensive-to-evaluate functions (expensive in terms of computation cost), even when there is a low amount of data available. Due to the compatibility with black box functions, only the score itself needs to be ascertainable, while the underlying mechanics or processes do not necessarily need to be understood or known. Boelrijk and co-authors investigated the potential use of Bayesian approaches for LC×LC in silico (36); they developed an unsupervised closed-loop algorithm to predict 1D-LC gradient profiles for a complex dyestuff mixture (55) and compared Bayesian optimization to retention modelling for automated method development (27). These studies demonstrated the versatility of Bayesian approaches and highlighted their applicability in method development workflows, with the added benefit of being less reliant on retention models and peak tracking. However, a potential limiting factor for LC×LC is the maximum number of parameters that can be optimized simultaneously. Due to the multivariate nature of the method, the number of necessary measurements for each additional parameter increases exponentially. The authors emphasized that the efficiency of Bayesian optimization is heavily dependent on the accurate definition of the objective function.

Machine learning approaches show tremendous potential to optimize the gradient program, yet the most advanced applications often have limitations that render them unsuitable for widespread use. This is partly due to the fact that these models can only handle scenarios that were present in the training data. Insufficient data can stem from a lack of appropriate standards or from insufficient resources to perform all the necessary measurements. One way to alleviate this issue is by employing simulated data, but this approach only works effectively if the simulated data closely resemble real-world situations, which is frequently not the case. With the rapid advancements in the field of machine learning, the issues currently encountered are expected to be addressed in the near future.

The Need for a Combined Effort

Driven by the ever-increasing need for more information, separation technology is advancing faster than our ability to use it to its full potential. Despite the enormous size and importance of the field, separations are often performed under suboptimal conditions and technical capabilities remain unused. For advancements in LC, such as 2D-LC, to make a significant impact, it is imperative that our community develops tools to fully exploit the current potential.

To this end, the chromatographic community must continue to deepen its understanding of fundamental concepts. This review has underlined the huge benefits and impressive advancements by scientists who have developed tools to streamline method development, focusing on kinetic or thermodynamic parameters—or both. Many of these advances are expressed as improved method development workflows that capture the experience and knowledge of the chromatographer.

However, the chromatographic community cannot do it alone. Indeed, we need the help of computers and information sciences. Automated LC method development workflows are rapidly advancing through the implementation of chemometric and machine learning algorithms. Nevertheless, despite the potential and promise of such algorithms to expedite method development, the challenges are still daunting and require a combined effort by the chromatographic and chemometric communities. Better ways must be found to mathematically express the chromatographic end goals, improve data analysis (peak detection), and invent smart algorithms that can interpret chromatographic data and combine these with fundamental kinetic (kinetic plots) and thermodynamic (retention models) concepts to create better methods. Meanwhile there is also the continuous need for chromatographers to deepen the understanding of physicochemical interactions of analytes inside our columns. The use of machine learning has recently been demonstrated by several groups to show great promise (35,55).

From the above-mentioned strategies it is clear that optimization workflows, especially for selectivity, remain heavily dependent on prior knowledge of the sample dimensionality or a trial-and-error approach. New machine learning algorithms should be able to optimize more parameters without needing unreasonable amounts of data. Other approaches may entail optimizing subsets of parameters at a time to lower the dimensionality of the problem or combining retention modelling with machine learning; in that way, aspects that can be well described do not need to be captured in the machine learning models. The practical challenges are mostly on the required number of measurements or computations. Every optimization process is quicker when starting with a good setup. By scanning various columns at the start, a good estimate can be made of which columns show potential and are candidates for orthogonal separation in 2D-LC.

Computer-aided method development for LC has been under development for some four decades and it has

failed to become common practice. However, with the rapid advances in LC, the gap between contemporary LC practice and potential has grown to a point where computer-aided method development is becoming a must. This need, in combination with the enormous growth in computational power and the dramatic advances in artificial intelligence, suggests that the time has come for computers to outperform analysts in LC method development.

Acknowledgments

This publication is part of the project Unleashing the Potential of Separation Technology to Achieve Innovation in Research and Society (UPSTAIRS) (with project number 19173) of the research program TTW-VENI, which is financed by the Dutch Research Council (NWO).

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Industry Insights: Thought Leadership

Empowering the AI-Driven Laboratory

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In November 2022, OpenAI's launch of ChatGPT transformed Artificial Intelligence (AI),[1] and more specifically, generative AI, from science fiction to an everyday reality. While there is no doubt that generative AI is going to be a disruptive technology across a multitude of industries, AI techniques like machine learning (ML) and deep learning have been utilized within the scientific laboratory setting for several years, particularly in drug discovery and development. In fact, there have been several highly publicized collaborations between pharmaceutical companies and AI technology providers that have started to yield results.[2,3] For example, In Silico Medicine entered clinical trials with the first new therapeutic where AI was used to identify the target and generate the design.[4] Other recent partnerships, like the new collaboration between Imperial College, BASF, and Sterling Pharma, are deploying AI to improve continuous manufacturing processes.[5, 6] While scientific organizations are recognizing and capitalizing on the potential of AI in many ways, there are still many challenges in the present day analytical laboratory that AI can be leveraged to overcome.. AI has the potential to change the way that we do science inside and outside of the laboratory but, before that ambition can be realized, organizations need to unlock their data to turn AI from science fiction into science.

Identifying the problem

The first step in leveraging AI is identifying the problem that needs to be solved and what data is available to inform the solution. AI is particularly well suited for data-rich processes where understanding the data that has been generated can inform and improve that process over time. For example, instrument telemetry data can be used to inform AI models that warn and ultimately even prevent common run failures. Analytical instruments produce vast amounts of telemetry data, instrument readouts that do not contain any intellectual property (IP) about what is being run, but describe the instrument conditions before, during, and after runs. Of course, the reason for instrumental analysis is the scientific data, and AI can help here too. AI algorithmic techniques like anomaly detection can help identify common analytical challenges in chromatographic data by monitoring for variances, such as spikes, baseline noise and retention time drift, from the typical patterns' baselines and peak detection. A human in the loop approach would allow chromatographers to confirm or reject the algorithm's suggestions that there is a trace impurity, gas in the line, or it is time for a new column. Over time, the algorithm learns how the lab-specific workflow, method, and compounds impact the various chromatogram properties, becoming more accurate and improving its recommendations.

Addressing common chromatographic challenges is one potential application AI-driven improvements in the laboratory, but there are numerous other opportunities. There are many tedious and human error-prone steps to follow in an analytical workflow that are better suited for AI-powered automation to perform, such as data review and instrument maintenance. Additionally, complex and highly manual tasks such as setting up runs for method development and data analysis that are currently done in third party applications such as Excel can benefit from AI solutions. Identifying a problem that AI could solve is likely the easiest step in the process. The harder challenge is getting the data.

Building the data set

The challenge is that to build AI, ML and advanced analytics solutions, data scientists need large data sets to train their models. Given the focus on generation of reproducible, high-quality data in science, analytical applications seem to be an obvious opportunity for AI. Unfortunately, the reality is that the scientific data needed to train AI models is often siloed in disparate systems. In recognition of these challenges and the growing value of data science, in 2016, the FAIR Guiding Principles for scientific data management and stewardship were published. They focused on the "machine-actionability" of data in that it needs to be findable, accessible, interoperable, and reusable.[7] Open standards like the Allotrope Ontologies and Data Model seek to break down these siloes by creating "linked data that standardizes experimental parameters so we can remove human error and enhance scientific reproducibility."[8] While Allotrope and other open standards like AnIML[9] and MZML[10] help to translate scientific data into a common ontology, the reality is that this solution does not address all the challenges organizations face.

Open standards, like MZML, are useful for further data processing enabled through third-party industry solutions, but as organizations appetite for AI-driven solutions grows, so will their need for complete, contextualized data sets. Conversion into a common ontology does not by itself enable organizations to aggregate and correlate processed results with the associated instrument telemetry data since it is often not saved with the result files. Given the variety of analytical techniques and other data sources like Laboratory Information Management Systems (LIMS) and Electronic Laboratory Notebooks (ELNs), it is difficult for organizations to make meaningful associations across all the different data types in their data lake through open standards alone.

Automating data pipelines

Rather than doing manual data conversion and upload, organizations need automated data pipelines that help them to bring data

from laboratory systems into their data lakes. With real-time data pipelines that upload, catalog, transform and store data in compliance with FAIR data principles, organizations can utilize different data science methodologies and tools. Dedicated pipelines also have the added advantage that telemetry data, scientific raw data and processed results can all be uploaded and associated, giving a complete picture of the data as it was acquired. Robust data pipelines ensure the data is fully contextualized and avoid what is often referred to as a data swamp - the less-than-ideal state where massive repositories of data exist but are unusable.[11]

As Kate Wearden recently described in The Digital Revolution: The Connected Lab of the Future, connectivity is critical to realizing the Lab of the Future. Today, multiple mechanisms exist to export data from Waters data systems to open standards or to map and parse data into custom data pipelines. While these solutions meet the needs of many customers today, they are often disconnected, manual processes. Our future vision is to eliminate the burden organizations face by maintaining these solutions and to provide contextualized data that is AI-ready through automated data pipelines.

Summary

We are only beginning to scratch the surface of what AI could do to improve laboratory operations and aid scientific discovery. Given the strategic importance of chromatography, there is significant potential to improve laboratory operations and derive insights by incorporating chromatography telemetry and analytical data into an organization's digital strategy. To realize this potential, organizations need to focus on how to FAIRify their data making it available for scientists and data scientists alike. Robust, automated data pipelines will bring data science to scientists so that they can use their data to solve problems that matter.

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Multi-Active Method (MAM) for the Analysis of Agriculture Product Technical Ingredients and Formulated Products

In regulatory laboratories, where potentially hundreds of different types of sample and active ingredients can be evaluated every year, a unique set of conditions provided by agricultural product manufacturers in their regulatory methods are faced with the decision to purchase a new and different analytical column or not follow the enforcement method provided by the manufacturer. Because of this, a multi-analyte method has been developed at the Irish Department of Agriculture, Food and The Marine, contributed to by the regulatory laboratories in Belgium and the Czech Republic, analyzing more than 70 active ingredients using high performance liquid chromatography (HPLC) and more than 35 active ingredients by ultrahigh-pressure liquid chromatography (UHPLC). The method has been designed for use by quality control laboratories and is suitable for determining a range of active substances in a wider range of formulated products as well as the technical AI itself. The method has been validated for linearity, precision, accuracy, and specificity for seven technical active ingredients as defined by FMC Corporation. The method and results are described in this article.

Jim Garvey, Olga Nováková, Olivier Pigeon, and Mary Ellen McNally

In the pesticide industry, regulatory laboratories are responsible for the quality of the material sold in the open market. Laboratories receive samples from the open trade that are sold in their individual countries. To this end, methodologies to analyze for the technical active ingredient (AI) and formulated products are available to them from the commercial agricultural product manufacturers' registration dossiers. The methods are part of the regulatory package submitted to countries for approval to sell and are the official registered methods for the AI and the formulated product.

AI analytical methods are typically liquid or gas chromatography-based and identify and quantify the AI in technical material and formulated products. These methods have one purpose: to determine if the product being produced and sold meets registered specifications. The commercial producer analyzes manufactured material before it is released to the open market to ensure it meets registered specifications, and worldwide regulatory agencies use these methods to check that material that is sampled from the open market also meets registered specifications.

So just how many registered AI methods are there? There are 1,055 active pesticide ingredients authorized for use in the United States alone (1), and worldwide active ingredient numbers are proportional (2). These active ingredients represent 20,000 marketed or formulated products, many with different concentration levels of the active or mixtures of two or more actives. In a small country such as Vietnam, there are products with more than 3,000 separate trade names registered.

For manufacturers, where only one product is made at a time, the analysis of what is produced to determine the acceptability of a product is routine. Generally, many batches or a continuous process are monitored with the company method which is set up in the site laboratory and maintained throughout the manufacturing campaign. Chromatographic columns that have been chosen for this methodology are specific for the analysis at hand, and can be routinely used for years, that is, the life of the product. The cost is trivial by comparison to the expense of supporting the manufacturing process.

Such is not the case in the regulatory laboratories, where potentially hundreds of different types of sample and active ingredients can be seen every year. With each set of unique conditions provided by the manufacturers, the regulatory laboratories are faced with the decision to purchase a new and different analytical column, or not follow the enforcement company method.

Because of this, a multi-analyte method has been developed at the Irish Department of Agriculture, Food and The Marine, contributed to by the regulatory laboratories in Belgium and the Czech Republic, analyzing more than 70 active ingredients using high performance liquid chromatography (HPLC) and more than 35 active ingredients by ultrahigh-pressure liquid chromatography (UHPLC). The method has been designed for use by quality control laboratories and is suitable for determining a range of active substances in a wider range of formulated products as well as the technical AI itself. The method has been validated for linearity, precision, accuracy, and specificity for seven technical active ingredients as defined by FMC Corporation.

The method and results are presented in this report.

Experimental

Reagents (Reagent Grade, Except as Noted)

Analytical standards, of known purity, stored in refrigerator or as per laboratory protocol.
Dicyclohexyl Phthalate (DCHP) > 97% pure – internal standard
Ethyl Acetate, HPLC grade
Acetone, HPLC grade
Acetonitrile, HPLC grade
Water, HPLC grade
Phosphoric Acid, HPLC grade
Formic Acid, MS grade

Apparatus

A high performance liquid chromatography (HPLC) system equipped with a constant temperature column compartment, a tempera-

ture-controlled autosampler, an autosampler capable of delivering 10 µL injections, a variable-wavelength UV detector or photodiode-array detector, and a digital integrator or other data handling device.

or

An ultrahigh-pressure liquid chromatography (UHPLC) system equipped with a constant temperature column compartment, a temperature-controlled autosampler, an autosampler capable of delivering 0.5 µL injections, a variable-wavelength UV detector or photodiode-array detector, and a digital integrator or other data handling device.

- HPLC column: Kinetex C18, 100 mm × 4.6 mm (i.d.) × 2.6 µm or equivalent.
- UHPLC column: Kinetex C18, 100 mm × 2.1 mm (i.d.) × 2.6 µm or equivalent, and precolumn Phenomenex C18, 2.1 mm i.d. or equivalent.
- Electronic integrator or data system
- Ultrasonic bath capable of being heated up to 50 °C
- Water bath
- Analytical balance
- Grinder/pulverizer
- Mechanical shaker
- Calibrated pH meter
- Organic PTFE filter, 0.22 or 0.45 µm

Standard Preparation

Internal Standard Stock Solution

Dissolve 80 mg of dicyclohexyl phthalate (to the nearest 0.1 mg) into a 100 mL volumetric flask and dilute to the mark with acetone. Internal standard solution concentration is approximately 0.08 mg/mL.

Calibration Solution

Weigh in duplicate about 10 mg of the required analytical standard to the nearest 0.1 mg in a volumetric flask (100 mL). Dissolve and fill to the mark with acetonitrile. This gives a standard concentration of approximately 100 mg/L, calibration solutions C_A and C_B.

Sampling

Ideally, an intact product should be taken from the market as intended for use with an end user.

Sample Preparation

Sample preparation is dependent on the

TABLE I: HPLC Mobile Phase Gradient

Time (min)	%A pH 2.0 to 2.2 Phosphoric or Formic Acid Adjusted HPLC Grade Water	%B Acetonitrile
0.01	65	35
10.0	15	85
16.0	15	85
16.4	65	35
18.0	65	35

TABLE II: UHPLC Mobile Phase Gradient

Time (min)	%A pH 2.0 to 2.2 Phosphoric or Formic Acid Adjusted HPLC Grade Water	%B Acetonitrile
0.00	65	35
4.00	15	85
6.00	15	85
6.01	65	35
7.00	65	35

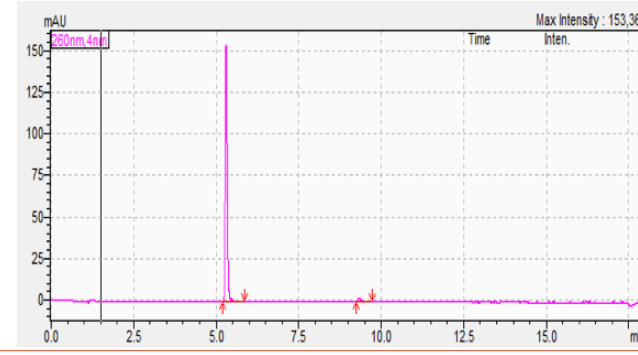

FIGURE 1: Example chromatogram of Spirotetramat using the HPLC conditions of the multi-analyte method, concentration is 150 g/L. Axis labels are Time (min) for *x*-axis and Signal (mAU) for *y*-axis.

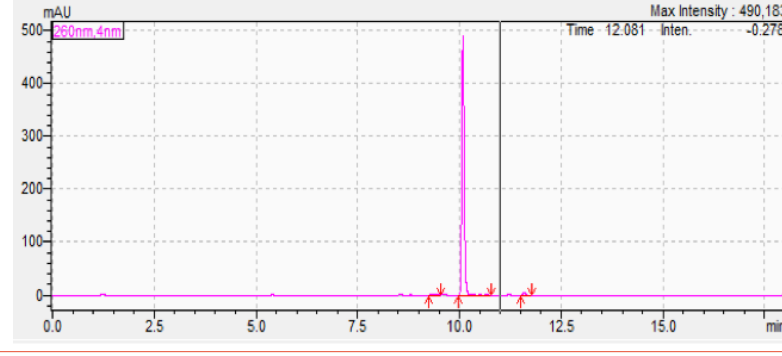

FIGURE 2: Example chromatogram of Pendimethilin, CS, using the UHPLC conditions of the multi-analyte method, concentration is 455 g/L. Axis labels are Time (min) for *x*-axis and Signal (mAU) for *y*-axis.

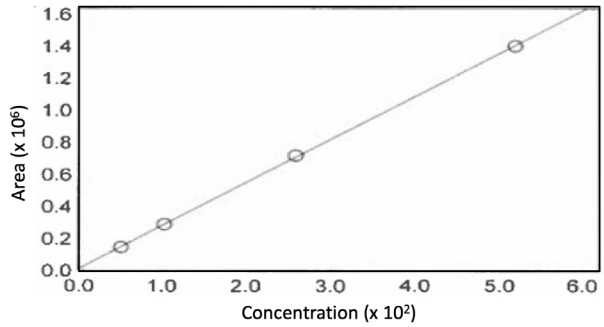

FIGURE 3: Typical calibration curve using the HPLC conditions of the multi-analyte method for the Spirotetramat standard. Axis labels are Concentration ($\times 10^2$) for *x*-axis and Area ($\times 10^6$) for *y*-axis.

TABLE III: Formulated products examined by HPLC conditions of the multi-analyte method

No.	Active Substance	Formulation Type*	λ (nm)	Unit	Declared Content	FAO Tolerances (±)
1	Acetamiprid	ME	260	g/kg	0.05	0.008
2	Amidosulfuron	WG	240	g/kg	75	2.5
3	Asulam	SL	260	g/L	400	20
4	Atrazine	SL	280	g/L	4	0.6
5	Benzovindiflupyr	EC	260	g/L	75	7.5
6	Bifenthrin	ME	260	g/L	0.02	0.003
7	Bixafen	EC	260	g/L	60	6
8	Carfentrazone-et	WG	260	g/kg	33.3	1.67
9	Chloridazon	WP	260	g/kg	650	25
10	Chloroantriniliprole	FS	280	g/kg	50	25
11	Chlorothalonil	SC	260	g/L	500	25
12	Chlorotoluron	SC	240	g/L	250	15
13	Cloquintocet-mexyl	EC	340	g/L	25	2.5
14	Cymoxanil	WG	260	g/kg	500	25
15	Daminozide	WG	220	g/kg	85	8.5
16	Difenacoum	RB	260	g/kg	0.005	0.0013
17	Diflufenican	SC	280	g/kg	200	12
18	Dimethomorph	WP	260	g/kg	500	25
19	Epoxiconazole	SC	260	g/L	50	5
20	Flazasulfuron	WG	260	g/kg	250	12.5
21	Flonicamid	WG	260	g/kg	500	25
22	Florasulam	SC	260	g/L	50	5
23	Fluazinam	EC	260	g/L	400	20
24	Flufenacet	SC	245	g/L	400	20
25	Flumioxazin	SC	280	g/L	300	15
26	Fluopicolide	SC	260	g/kg	5.53	0.55
27	Fluopyram	EC	260	g/L	65	6.5
28	Fluoxastrobin	EC	260	g/L	75	7.5
29	Flurtamone	SC	280	g/L	120	7.2
30	Fluxapyroxad	EC	260	g/L	62.5	6.25

formulation type being analyzed. The procedures are different for liquid and solid formulations and for technical material. Once the samples are prepared, the active substances are analyzed by either high performance or ultrahigh-pressure liquid chromatography with either UV or diode-array detection (HPLC-UV, HPLC-DAD, UHPLC-UV, or UHPLC-DAD, respectively), depending on the instrumentation available.

Liquid or Suspension Formulations

Thoroughly shake the sample container to homogenize the sample before use. Weigh in duplicate (to the nearest 0.1 mg) sufficient sample to contain 9 to 11 mg of the active substance into a volumetric flask (100 mL). For suspension formulations add 5 mL of water to disperse. Subsequently, add 70 mL of acetonitrile. If it is necessary, sonicate for dissolution of active ingredient. Allow the solution to cool to ambient temperature and fill to the mark with acetonitrile (Solutions S_A and S_B). This gives an active ingredient concentration of approximately 100 mg/L. For UHPLC, filter samples through 0.22 µm filter.

Solid Formulations

For solid formulations, dispersion with 10% water prior to solvent addition is recommended. Weigh 15–20 g of the sample into a mortar and pestle or grinder/pulverizer and homogenize thoroughly. Subsample by weighing in duplicate, to the nearest 0.1 mg, sufficient sample to contain 9 to 11 mg of the active substance into a 100 mL volumetric flask. Add 70 mL of the internal standard solution. If it is necessary, sonicate for dissolution of active ingredient. Allow the solution to cool to ambient temperature, fill to the mark with acetonitrile, and mix well (Solutions S_A and S_B). This gives a sample concentration of approximately 100 mg/L. For UHPLC, filter samples through a 0.22 µm filter.

Mobile Phase Preparation

Mobile Phase A, Aqueous

Prepare by pipetting phosphoric or formic acid into a 2000 mL volumetric flask containing 1500 mL of water, add water to the 2000 mL mark, and thoroughly mix. pH should between 2.0 and 2.2.

31	Halauxifen-methyl	XX	260	g/L	6.3	0.9
32	Imidacloprid	SL	260	g/L	0.125	0.0188
33	Iodosulfuron-Me-Na	SC	240	g/L	0.3	0.05
34	Iprodione	SC	240	g/L	256	12.8
35	Isoproturon	SC	260	g/L	500	25
36	Isoxaflutole	WG	260	g/kg	100	10
37	Linuron	SC	260	g/L	450	22.5
38	Mesosulfuron-Me	WG	240	g/kg	0.9	0.14
39	Mesotrione	SC	260	g/L	70	7
40	Metamitron	SC	260	g/L	700	70
41	Metazachlor	SC	260	g/L	375	18.8
42	Metconazole	EC	260	g/L	60	6
43	Methomyl	SC	240	g/kg	20	1.2
44	Metsulfuron-methyl	WG	260	g/kg	68	10
45	Myclobutanil	ME	260	g/L	0.075	0.0113
46	Nicosulfuron	WG	260	g/kg	750	25
47	Oxadiazon	SC	260	g/L	4.8	0.72
48	Oxamyl	GR	240	g/kg	5	0.5
49	Pendimethalin	SC	260	g/L	455	22.8
50	Penoxsulam	WG	280	g/kg	0.04	0.01
51	Penthiopyrad	SC	260	g/L	100	10
52	Picloram	SL	260	g/L	67	6.7
53	Pinoxaden	EC	260	g/L	100	10
54	Propaquizafop	EC	340	g/L	100	10
55	Proquinazid	EC	260	g/L	200	12
56	Prothioconazole	EC	260	g/L	200	12
57	Pyraclostrobin	SC	260	g/L	133	8
58	Pyraflufen-ethyl	SL	260	g/L	0.33	0.05
59	Pyridate	WP	260	g/kg	45	2.25
60	Pyrimethanil	SC	260	g/L	400	20
61	Pyroxsulam	WG	310	g/kg	7.1	0.71
62	Quizalofop-p-tefuryl	EC	240	g/L	40	4
63	Tebuthiuron	WG	260	g/kg	20	1.2
64	Tepraloxydim	EC	260	g/L	50	5
65	Thiacloprid	SC	240	g/L	40.4	2.02
66	Thifensulfuron-methyl	WG	260	g/kg	682	25
67	Tribenuron-methyl	SG	260	g/kg	40	2
68	Trifloxystrobin	SC	260	g/L	16	1.6
69	Triflusulfuron-methyl	WG	260	g/kg	500	25
70	Trinexapac-ethyl	EC	260	g/L	250	12.5
71	Triticonazole	ME	260	g/L	7.5	1.1

Source: *Formulation Types: EC – Emulsifiable Concentrate, SL – Soluble Liquids, ME – Micro Encapsulate, WG – Wettable Granule, SC – Soluble Concentrate, WP – Wettable Powder, SG – Soluble Granule, XX – Unknown

Mobile Phase B

Acetonitrile.

HPLC Chromatographic Conditions

Column: HPLC column, Kinetex C18, 150 mm × 4.6 mm i.d. × 2.6 µm or equivalent
Flow rate: 1.0 mL/min
Injection volume: 0.5 µL
Detector wavelength: 220–340 nm. The optimum wavelength can be established by analyzing the analytical standard on PDA detector prior to analysis if enforcement method is not available for reference.
Run time: 18 min
Initial mobile phase concentration: A = 65%: 0.1% pH 2.0–2.2; o-phosphoric or formic acid-adjusted water; B = 35%: Acetonitrile
HPLC mobile phase gradient: *see Table I.*
Column temperature: 25 °C

UHPLC Chromatographic Conditions

Column: Kinetex C18, 150 mm × 2.1 mm (i.d.) × 2.6 µm or equivalent, and precolumn Phenomenex C18, 2.1 mm i.d. or equivalent.
Flow rate: 0.4 mL/min
Injection volume: 0.5 µL
Detector wavelength: 210–330 nm
The optimum wavelength can be established by analyzing the analytical standard on a DAD detector prior to analysis if registration method is not available for reference.
Run Time: 8.5 min
Initial mobile phase concentration: A = 65%: 0.1% pH 2.0–2.2; o-phosphoric or formic acid-adjusted water; B = 35%: Acetonitrile
UHPLC mobile phase gradient: *see Table II.*
Column temperature: 25 °C

Equilibration of the System

For HPLC

Pump sufficient mobile phase through the column to equilibrate the system approximately 20 to 30 min at the recommended flow rate of 1.0 mL/min. Inject 5µl portions of the standard solution until the response obtained from two consecutive injections deviates by less than 1.0 %, preferably less than 0.5%.

For UHPLC

Pump sufficient mobile phase through the column to equilibrate the system approximately 20 to 30 min at the recommended flow rate of 0.4 mL/min. Inject 0.5 µl portion of the

TABLE IV: Formulated products examined by UHPLC conditions of the multi-analyte method

No.	Active Substance	Formulation Type*	λ (nm)	Unit	Declared Content	FAO Tolerances (±)
1	Acetamiprid	SP	240	g/kg	200	12
	Acetamiprid	AL	240	g/L	0.05	0.0075
2	Azoxystrobin	SC	220	g/L	120	7.2
3	Clomazone	CS	210	g/L	360	21.6
4	Deltamethrin	OD	210	g/L	10	1.5
5	Difenoconazole	FS	210	g/L	25	3.75
6	Diflufenican	SC	230	g/L	40	4
7	Dimethomorph	WP	240	g/kg	60	6
8	Epoxiconazole	SC	210	g/L	125	7.5
9	Fenhexamid	SC	240	g/L	500	25
10	Fipronil	WG	220	g/kg	0.143	0.036
11	Fludioxonil	FS	210	g/L	25	3.75
12	Flufenacet	SC	210	g/L	80	8
13	Flurprimidol	EC	240	g/kg	130	7.8
14	Giberelin GA4/GA7	SL	210	g/L	10	1.5
15	Chlortoluron	SC	240	g/L	280	14
16	Metamitron	SC	220	g/L	700	25
17	Pendimethalin	CS	240	g/L	455	22.75
18	Pethoxamid	EC	240	g/L	600	25
19	Prothioconazole	EC	254	g/L	250	15
20	Quizalofop-p-ethyl	EC	330	g/L	50	5
21	Pyraclostrobin	SE	260	g/L	85	8.5
22	Sedaxane	FS	220	g/L	25	3.75
23	Silthiofam	FS	210	g/L	125	7,5
24	S-metolachlor	EC	220	g/L	960	25
25	Spinetoram	SC	254	g/kg	117	7
26	Tebuconazole	WG	220	g/kg	500	25
	Tebuconazole	SC	220	g/L	200	12
27	Tefluthrin	GR	220	g/kg	15	3.75
28	Terbuthylazine	SE	220	g/L	187.5	11.22
29	Thiacloprid	OD	210	g/L	100	10
30	Thiophanate-methyl	GCCB	260	g/kg	23	5,75
31	Triclopyr	AL	295	g/kg	0.7	0,105
32	Trifloxystrobin	WG	220	g/kg	250	15
33	Triflusulfuron-methyl	OD	240	g/L	150	9
34	Trinexapac-ethyl	EC	240	g/kg	50	5
35	Triticonazole	FS	260	g/L	25	3.75

Source: *Formulation Types: CS – Capsule Suspension, EC – Emulsifiable Concentrate, EW – Concentrated aqueous emulsion, FS – Flowable Concentrate, GCC – Ground Corn Cob, is an effective carrier for various pesticides., GR – Granules, ME – Micro Encapsulate, OD – Oil Dispersion, SE – Suspoemulsion (Combination of SC and EW), SC – Soluble Concentrate, SG – Soluble Granule, SL – Soluble Liquids, WG – Wettable Granule, WP – Wettable Powder, XX – Unknown

standard solution until the response obtained from two consecutive injections deviates by less than 1.0% for retention times and by less than 2.0% for peak areas.

Determination of the Species of Interest

For HPLC

Inject in duplicate 5 µL portions of each sample solution, bracketing them by injections of the calibration solutions as follows; calibration solution C_A, sample solution $S1_A$, sample solution $S1_B$, calibration solution C_B, sample solution $S2_A$, sample solution $S2_B$, and so on. Measure the relevant peak areas.

For UHPLC

Inject in duplicate 0.5 µL portions of each sample solution, bracketing them by injections of the calibration solutions as follows; calibration solution C_A, sample solution $S1_A$, sample solution $S1_B$, calibration solution C_B, sample solution $S2_A$, sample solution $S2_B$, and so on. Measure the relevant peak areas.

Calculations

Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating the active substance contents of the bracketed sample injections as shown below.

Individual response factors for C_A and C_B:

$$fi = \frac{s \times P}{Hs} \quad [1]$$

Mean response factor for C_A and C_B:

$$f = \frac{fiA + fiB}{2} \quad [2]$$

Content of active substance wt % active substance =

$$\frac{f \times Hw}{w} \text{x100} \quad [3]$$

where:

fi = individual response factor

f = mean response factor

Hs = peak area of active substance in the calibration solution

Hw = average peak area of the active substance from the two injections of the sample solution

s = mass of the active substance working standard in the calibration solution (mg)

w = mass of sample taken (mg)

P = purity of the active substance working standard (g/kg)

TABLE V: A comparison of linearity and accuracy equivalency data for eight active ingredients by individual GLP enforcement and the multi-analyte method

Compound	Method Type	Linearity			Accuracy		
		Correlation Coefficient	Y-Intercept	Slope	Average Recovery (%)	RSD	Passed Modified Horwitz
Bifenthrin	GLP Enforcement	0.9998	-25.66	1478	99.53	1.14	Yes
	Multi-analyte	1.000	-9.98	1550	100.32	0.87	Yes
Cyantraniliprole	GLP Enforcement	1.0000	0.055	719.8	100.18	0.85	Yes
	Multi-analyte	0.9999	0.021	719.7	97.99	0.50	Yes
Indoxacarb	GLP Enforcement	0.9995	0.010	502.5	98.41	0.80	Yes
	Multi-analyte	0.9997	0.008	419.6	97.45	1.28	Yes
Flutriafol	GLP Enforcement	0.9991	-23.65	1095	98.94	0.45	Yes
	Multi-analyte	0.9999	82.87	1089	99.02	0.33	Yes
Triflusulfuron	GLP Enforcement	0.9999	0.001	95.8	99.12	0.52	Yes
	Multi-analyte	0.9999	0.006	97.11	99.25	0.45	Yes
Azimsulfuron	GLP Enforcement	0.9993	-0.014	2.64	100.17	0.23	Yes
	Multi-analyte	0.9992	-0.004	2.63	100.16	0.37	Yes
Chlorsulfuron	GLP Enforcement	0.9999	0.014	633.6	98.61	0.48	Yes
	Multi-analyte	0.9997	0.043	613.6	98.51	0.61	Yes
						n = 6	

Results and Discussion

The conditions that have been outlined above for both HPLC and UHPLC have been used to analyze the compounds listed in Table III and Table IV, respectively. Given in these tables are the formulation types of the samples where the method was used successfully, the UV maximum wavelength determined during our experiments, and the declared content including the units of the specific material.

Figures 1 and 2 show example chromatograms for two representative compounds analyzed by the HPLC and UHPLC methods, respectively.

Validation of Technical Materials Using the Multi-Analyte Method)

Acceptable linearity of the active ingredient for all the products outlined in Table III and Table IV was demonstrated by using a characterized analytical standard of the same AI using a three-point calibration curve with concentrations at the product concentration and two further points at +50% and -50%. Figure 3 shows a typical calibration curve. Repeatability was calculated via dual preparation and dual injection of each sample. Seven individual active ingredients were used to study the difference in results for Santé et Consommateurs (Directorate General Health and Consumers, European Commission, or SANCO) guideline validation parameters (3) using both the registration method for the technical material and the multi-analyte method described herein. The validation includes linearity, precision, accuracy, and specificity or selectivity. The active ingredients examined during this comparison were chlorantraniliprole, azimsulfuron, bifenthrin, chlorsulfuron, cyantraniliprole, flutriafol, indoxacarb, and triflusulfuron.

To prove equivalency, a seven-point calibration curve was determined for all seven active ingredients. As can be seen in Table V, the correlation coefficient for each active using both the registration method and the multi-analyte method was 0.99 or greater.

Both method and instrument precision were tested in these experiments. Eight individual weighings of one sample of each active ingredient were measured to determine the method precision. One sample injected eight individual times defined instrument precision. Results of this can also be found in Table V.

Accuracy equivalency was established by preparing duplicate samples at three separate concentrations. These concentrations were set at 75, 100, and 125% of the GLP sample solution concentration in the enforcement method. Average weight percent results, which ranged from 98.5 to 100.3 with relative standard deviations (RSD) for n=6 ranging from 0.23 to 1.28, all passed the modified Horowitz limit. The data can be found in Table VI.

TABLE VI: A comparison of precision equivalency data for eight active ingredients by individual GLP enforcement and the multi-analyte method

Compound		Linearity			Precision		
	Method Type	Correlation Coefficient	Y-Intercept	Slope	Average Weight (%)	RSD Method	RSD Instrument
Bifenthrin	GLP Enforcement	0.9998	-25.66	1478	100	1.18	0.14
	Multi-analyte	1.000	-9.98	1550	99.45	0.45	0.09
Cyantraniliprole	GLP Enforcement	1.0000	0.055	719.8	98.11	0.86	0.19
	Multi-analyte	0.9999	0.021	719.7	98.67	1.17	0.22
Indoxacarb	GLP Enforcement	0.9995	0.010	502.5	98.59	1.05	0.07
	Multi-analyte	0.9997	0.008	419.6	98.20	1.08	0.05
Flutriafol	GLP Enforcement	0.9991	-23.65	1095	100.97	0.41	0.71
	Multi-analyte	0.9999	82.87	1089	99.77	0.27	0.23
Triflusulfuron	GLP Enforcement	0.9999	0.001	95.8	99.23	0.21	0.06
	Multi-analyte	0.9999	0.006	97.11	99.25	0.28	0.04
Azimsulfuron	GLP Enforcement	0.9993	-0.014	2.64	100.97	0.53	0.06
	Multi-analyte	0.9992	-0.004	2.63	101.19	0.52	0.17
Chlorsulfuron	GLP Enforcement	0.9999	0.014	633.6	97.32	0.69	0.20
	Multi-analyte	0.9997	0.043	613.6	97.74	0.60	0.11
						n = 8	n = 8

A qualitative analysis for potential impurity interferences for all samples was conducted to determine method specificity. Ideally, an impurity exhibits no interference, and acceptable interference is less than 3% of the overall sample composition. No interferences were seen in either method for the seven active ingredients.

Conclusion

Although the multi-analyte method is not the registered method, it does provide a very useful screening tool for laboratories that see a variety of agricultural product samples. It eliminates the need for a wide variety of columns and can accurately measure the active ingredient content in the formulated product, thus determining whether the material meets the registered manufacturer's specifications.

The validation comparison for eight active ingredients using the multi-analyte and enforcement methods diligently shows the equivalence of the method for linearity, accuracy, and precision. It needs to be continually tested for specificity with each active ingredient and shows great promise in meeting GLP statistical requirements.

The multi-analyte method is evergreen in terms of its future use, and the addition of actives and products are possible if the following criteria are met:

- An unequivocal identity confirmation; preferably mass spectrometry
- Injection repeatability data for standards – RSD should be ≤ 1%
- Linearity should be demonstrated between 50 and 150 mg/kg
- The repeatability for sample analysis should be less than 5%—typically three subsamples of the product and two injections of each sub-sample.

A proficiency test or collaborative trail data would be beneficial to support the use of this methodology for any additional agricultural compounds.

Acknowledgment

The authors would like to express their appreciation to the staff in their laboratories involved in this work.

This article has additional supplemental information only available online. **Scan code for link.**

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VIEWPOINTS

ExTech 2023, Celebrating 25 Years of Innovation in Extraction Technologies

Emanuela Gionfriddo

The 25th anniversary of ExTech, the International Symposium for Advances in Extraction Technologies, was held on the beautiful island of Tenerife, Spain, from July 18–21, 2023. The honorary chairman of the event was Janusz Pawliszyn, who founded ExTech in 1999, at the University of Waterloo, in Ontario, Canada.

ExTech is undoubtedly the largest conference dedicated to extraction technology, a field within separation chemistry that has seen remarkable growth over the past three decades. It brings together researchers working on pre-chromatographic separations, that save chromatographic systems and instrumentation from the "garbage in-garbage out" effect while enhancing instrumental performance through clean-up and pre-concentration. This year, we celebrated this symposium's important anniversary through an exquisitely organized event that gathered 222 participants from 25 countries with a total of 3 plenary lectures, 21 keynote lectures, 62 oral communications and 174 posters.

The symposium opened with a plenary lecture by Pawliszyn and Stig Pedersen-Bjergaard that updated the audience on the latest advances in the microextraction field and provided important insights on the future of the field. The two plenary speakers specifically highlighted the importance of incorporating the fundamentals of extraction processes into graduate and undergraduate textbooks, to train the next generation of scientists and unify teaching strategies for this important and ever-growing field of separation chemistry. The plenary concluded with an important message: "When the next generation of scientists enters the regulatory affairs, the analytical manufacturers, and the analytical laboratories, they will have a very strong sustainability commitment, and will for sure facilitate the Green Paradigm Shift. SPME and LPME (respectively, solid and liquid phase microextraction) and many other ideas discussed at ExTech 2023 will be their initial scientific platform. Therefore, what we do now will be very important in 10–15 years!"

The symposium also dedicated eight oral sessions to young investigators, providing 29 scientists under the age of 35 an opportunity to present their work alongside leading scientists from around the world. This initiative aimed to encourage young scientists' participation, fostering interactions with, and learning from, experienced researchers. More than half of the 174 poster communications at the conference were presented by young scientists. On this topic, symposium chairwoman Veronica Pino commented, "Young researchers are presenting very important innovations in the field, and this is something that I want to highlight because we wanted to encourage presentations by younger researchers to show what they are doing and what they can offer to our community. At the same time, these young scientists have an opportunity to closely interact with seniors and this environment is fantastic for their development and to gain confidence in what they are doing."

Symposium chairman Javier Hernández-Borges also commented on the aftermath of ExTech 2023, saying, "We, the organizing committee of ExTech, are proud of the overall attendance of the symposium, especially considering about half of the attendees are PhD students. This testifies that research in the extraction technology field is very active and vibrant, and we are truly impressed with the quality of the oral and poster communications delivered by these young scientists."

By the end of ExTech, Hernández-Borges delivered a powerful take-home message for those young scientists: "Continue to attend conferences like ExTech to network with your community (especially with senior scientists in the field) and keep updated with the most recent trends in the field. Do your research with curiosity, professionalism, and ethics. Ethics are nowadays one of the most important aspects of academic research to produce high-quality and reliable results. Believe in yourself and be proud of the great work you all are doing. Be open to new challenges and be bold when thinking about new solutions for chemical analysis, this is how we achieve scientific progress."

I bet by the end of this article, you will want to know where the next ExTech will be organized and join this vibrant community of separation scientists. Well… I have good news! Elena Stashenko of the Industrial University of Santander in Bucaramanga, Santander, Colombia and Milton Rosero-Moreano of University of Caldas in Manizales, Caldas, Colombia announced that the 26th iteration of ExTech will be organized next year in Bucaramanga, Colombia. I am sure we will be in for another excellent meeting and innovative discussions on extraction technologies. Stay tuned for more updates!

Emanuela Gionfriddo is an Associate Professor of Chemistry at the Department of Chemistry and Biochemistry of The University of Toledo, Ohio. Direct correspondence to: emanuela.gionfriddo@utoledo.edu

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Antec Scientific

SenCell™ for Detection
Proprietary Adjustable Spacer Technology (AST) for highest sensitivity (US Patent 9,310,330).

µ-PrepCell™ for Reactions
Redox reactions upfront MS: disulfide bond reduction in proteins, mimicking drug metabolism, drug stability testing/degradation.

SweetSep™ Colums
The new benchmark for superior separation of all classes of carbohydrates using HPAEC with PAD or MS detection.

Company Description

For over 33 years, Antec Scientific has been the world's leading supplier of analytical instrumentation based on electrochemistry (EC). Antec's line of instruments include electrochemical detectors (ECD), analyzers built on HPLC-ECD, and electrochemical reactors for use with MS (EC–MS) and synthesis.

Electrochemical Detectors for HPLC
The DECADE™ line of ECDs (Elite™ and Lite™) have become the benchmark in detection, and can be used with any third party (U)HPLC system. Different flow cells are available to cover a broad range of applications from the analysis of antibiotics to sugars or pharmaceuticals.

Dedicated Analyzers
The ALEXYS™ Analyzers based on HPLC with ECD are turnkey instruments built for highest performance and to ensure ease-in-use. Guaranteed applications have been developed with the ALEXYS Carbohydrate, Antibiotics, and Neurotransmitter Analyzers. Other application areas are in clinical/diagnostics, food/beverage, environmental, and drugs/pharmaceuticals analysis.

Electrochemical Reactors for MS and Synthesis
With the introduction of the ROXY™ Exceed EC system, REDOX reactions can be monitored directly by mass spectrometry (MS). The on-line coupling of electrochemical reactors with MS (EC–MS) has found many application areas from proteomics, (reduction of disulfide bonds), drug metabolism, and drug stability testing, to environmental degradation. Another emerging application of Antec's flow and batch reactors is in Flow Chemistry for rapid electrochemical synthesis of products.

ALEXYS Carbohydrate Analyzer

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The Netherlands

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YEAR FOUNDED
1990, since 2010 in USA

Antec® Scientific

CDS Analytical

Company Description

CDS Analytical is an ISO 9001:2015 certified manufacturer of thermal sample preparation instrumentation for 55 years. CDS offers a complete suite of diverse front-end GC equipment including pyrolyzers for Py-GC-MS, purge & trap concentrator for EPA waste and drinking water test methods (8260, 524, 624), thermal desorption systems for TD-GC-MS to meet the requirements from today's most demanding analytical customers. CDS Analytical has acquired the rights from 3M™ to manufacture licensed Empore™ solid-phase extraction products, extending the reach into laboratory consumables to provide solutions for Environmental, Life Science, and Biopharma customers. CDS Analytical also distributes various lab equipment including chillers and rotary evaporators for chemistry labs.

Chief Chromatographic Techniques Supported

- Pyrolysis-GC-MS
- Purge and Trap-GC-MS
- Thermal Desorption-GC-MS
- Solid-Phase Extraction

Markets Served

CDS Analytical products have been serving the following markets as: Polymer, Rubber, Environmental, Aerospace, Automobile, Energy, Food and Flavor, Forensics, Paper & Ink, Pharmaceuticals, Semiconductor, Textiles, Tobacco, Life Science, and Clinical Diagnosis.

Major Products/Services

Pyroprobe™ with DISC technology offers easy sample loading, unparalleled reproducibility, and superior resolution. The 7550S 72-position Thermal Desorption autosampler supports the Peltier electric cooling to handle C2 to C44 level VOCs with 10,000:1 Sample Split and quantitative Sample Saver. The 8500A 110-position automated purge and trap concentrator is the newly launched 3rd gen Purge and Trap platform to maximize efficiency for EPA 8260, 524, and 624 methods. The Empore™ SPE 47 mm and 90 mm disks, 1 ml, 3ml, and 6 ml cartridges ,96-well plate, Ez-disk and Stagetip adopts a proprietary membrane technology from 3M to offer the fastest flow rate with the smallest dead volume among all Solid Phase Extraction filtration products. The water chiller, rotary evaporator and digestion blocks are reliable and cost effective lab equipment to help the sample preparation work.

Facility

CDS Analytical's facility is located in Oxford, Pennsylvania. CDS products passed various safety regulations, including CE, METlab and NRTL. The Oxford plant has been ISO 9001:2015 certified and has GMP-compliant ISO clean room capability. CDS Analytical is bringing its production compliance to the next level..

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WEB SITE
www.cdsanalytical.com

YEAR FOUNDED
1969

CDS Analytical, Inc.

CEM Corporation

Company Description

CEM Corporation is a leading global company specializing in scientific solutions for critical laboratory applications. We design and manufacture systems for analytical laboratories, bioscience applications, life sciences, and processing plants worldwide. Our product portfolio includes innovative instrumentation for sample preparation for elemental or chromatographic analysis, chemical synthesis, peptide synthesis, and other applications.

Chief Chromatographic Techniques Supported

- LC
- LC–MS
- GC
- GC–MS

Markets Served

CEM instrumentation is used by chemists, scientists, and technicians in private industry, as well as leading universities, analytical laboratories, and research facilities around the world. We support numerous markets, including the pharmaceutical, chemical, environmental, applied materials, food, and petroleum industries, among others.

Major Products/Services

Good analysis starts with sample preparation. The CEM suite of products is designed to bring speed, simplicity, automation, and accuracy to sample prep for chromatographic analysis.

EDGE PFAS™ and MARS 6 PFAS™ are designed specifically for PFAS extraction. With no background contamination, simple operation, and the flexibility of either sequential automated extraction, reducing the need for SPE, or high throughput extractions, PFAS preparation has never been easier.

The Discover Prep™ is an automated microwave sample prep workhorse for applications such as hydrolysis and extraction of samples like plastics/polymers, pharmaceutical/nutraceuticals, consumer products, and more.

Extractions that typically take hours are complete in minutes using a closed system that is safer for laboratory staff.

The MARS 6™ Extraction system can be used for the same applications as the Discover Prep but in a high-throughput platform that allows for processing of up to 40 samples per batch, still in a fraction of the time of traditional extraction methods.

The EDGE® is a fully automated extraction system that performs solvent addition, extraction, filtration, and system wash in a fraction of the time of other techniques. The EDGE supports extraction of foods, cannabis, soils, and more.

Facility

CEM's global headquarters, research laboratories, and manufacturing facilities are located in Matthews, North Carolina. The company has subsidiaries in the United Kingdom, Ireland, Germany, Italy, France, Japan, and Singapore, as well as more than 50 distributors worldwide.

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NUMBER OF EMPLOYEES
USA: 260
Elsewhere: 90

YEAR FOUNDED
1978

Hamilton Company

Chromatography Products for HPLC and GC

Company Description

Hamilton Company is an industry leader in the design and manufacture of liquid handling, process analytics, robotics, and automated storage solutions. For 60 years, Hamilton has been satisfying customer needs by combining quality materials with skilled workmanship to ensure the highest level of performance. Hamilton's lifelong commitment to precision and quality has earned a global ISO 9001 Certification.

Founded on the technology of analytical Microliter™ and Gastight® syringes, Hamilton has a broad offering of laboratory products, including manual and semi-automated precision fluid measuring instruments, chromatography products, process sensors, laboratory electrodes, pipettes, and more.

Chief Chromatographic Techniques Supported

- HPLC
- Polymeric HPLC columns: reversed phase, anion exchange, cation exchange, and ion exclusion
- Silica HPLC columns
- GC septa
- Chromatography syringes (HPLC and GC)

Markets Served

- Analytical
- Pharmaceutical
- Biotechnology
- Industrial
- Clinical
- Environmental

Major Products/Services

HPLC Columns: We manufacture 18 types of polymeric HPLC columns for reversed phase, anion exchange, cation exchange, and ion exclusion separations, providing a wide range of retention characteristics and performance benefits. Our polymer-based HPLC columns provide maximum inertness and pH stability (0–14) with the pressure stability of silica-based columns. With our Polymeric Reversed Phase (PRP™) HPLC columns and resins, the sample dictates the necessary separation conditions, not the limitations of the column. We can also customize HPLC columns to meet a desired application need. Each product is manufactured to achieve the highest level of accuracy.

Chromatography Syringes: We offer the most complete selection of syringes on the market for use in various applications including GC and HPLC (autosamplers and manual injection), thin layer chromatography (TLC), liquid handling, and life sciences. Crafting exceptional syringes is an evolving science, which is why we are dedicated to the continuous research and development of this product line. We constantly enrich our entire syringe offering by either improving existing models or introducing new ones. Using customers' needs and feedback as a guide, we innovate in ways that maximize flexibility, performance, and value.

Microlab® 600 Diluter/Dispenser: The Microlab 600 is a highly precise syringe pump with a touchscreen interface designed to quickly and easily dilute and dispense fluids, while increasing throughput and reducing cost and wasted buffer. This positive displacement system provides better than 99% accuracy, independent of a liquid's viscosity, vapor pressure, and temperature. The inert fluid path minimizes sample carryover and is compatible with harsh chemicals.

Facility

We are a global enterprise with headquarters in Reno, NV; Hopkinton, MA; Giarmata, Romania; and Bonaduz, Switzerland, and offices worldwide.

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YEAR FOUNDED
1953

HAMILTON®

KIN-TEK Analytical, Inc.

Gas Calibration Equipment

KIN-TEK Analytical, Inc.
504 Laurel St
La Marque, TX 77568

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WEBSITE
www.kin-tek.com

YEAR FOUNDED
1970

Company Description

KIN-TEK Analytical, Inc. (KIN-TEK) is a preferred provider of devices and instrumentation for creating trace concentration calibration gas standards and complex gas mixtures.
KIN-TEK revolutionized permeation tube technology and further developed it to produce Trace Source™ Disposable and Refillable Permeation Tubes. KIN-TEK's Trace Source™ technology is employed in KIN-TEK's Gas Standard Generators to provide accurate, NIST-traceable calibration standards. KIN-TEK's products include a range of stand-alone gas standard generators and generator systems, gas mixers, and permeation devices to fit almost any application that relies on the delivery of an accurate trace gas concentration.

Chief Chromatographic Techniques Supported

- Calibration gases
- Gas standards
- GC calibration
- GC/MS calibration
- Trace gas standards
- Permeation tubes
- Permeation tube supplies

Markets Served

KIN-TEK delivers trace gas calibration product solutions and services worldwide to solve customer problems in the laboratory, field (portable), and process industries. Industries served include Analyzer Manufacturers, Aerospace and Aviation, Environmental, Food & Beverage, Petrochemical, Pharmaceutical, Refineries, Semiconductor manufacturers, R&D, Universities, and many more!

Major Products/Services

Gas generator modules can be operated as stand-alone calibrators or combined into a Gas Standard Generator System.

FlexMixer™ Gas Blender (New!)
Multi-Gas Blending/Diluting System.

FlexStream™ Gas Standard Generator
Automated expandable modular permeation system.

491Flex™ Gas Standard Generator
Manually operated expandable modular permeation system.

EcoFlex™ Gas Standard Generator
Manually operated stand-alone permeation module.

Span Pac™ H_2O Trace Moisture Generator
Manually operated trace moisture permeation system.

Span Pac™ I Industrial Gas Standard Generator Industrial permeation system for on-line calibration of process GCs.

CO395 Flex™ Certification Oven
Stand-alone permeation tube oven.

Trace Source™ Disposable and Refillable Permeation Tubes >550 certified standards.

Facility

Our corporate headquarters is in La Marque, Texas. All manufacturing, certifications, and operations take place at this facility.

KIN-TEK
The Calibration Specialists

LabVantage Solutions, Inc.

Company Description

LabVantage Solutions is the recognized leader in enterprise laboratory software and services, as well as advanced analytics. More than 1500 global customer sites across industries rely on LabVantage's highly configurable, 100% web browser-based Laboratory Information Management System (LIMS) platform to innovate faster in the R&D cycle, improve manufactured product quality, comply with regulations, increase cybersecurity, and leverage AI. As a technology and digital transformation leader, LabVantage offers configurable Software-as-a-Service (SaaS), cloud-hosting, or on-premise implementations. Investments in analytics bring the benefits of AI to customers, while global services provide consistent, knowledgeable deployments, validation, migration, and professional and managed services.

Chief Chromatographic Techniques Supported

LabVantage supports all analytical techniques, including all chromatographic techniques, by interfacing to any commercial chromatography data system (CDS).

Markets Served

- Life Sciences
- Pharmaceutical/ Biotechnology
- Medical Device
- Biobanking
- Food & Beverage
- Consumer Goods
- Oil, Gas, and Energy
- Genetics & Diagnostics
- Public Health
- Healthcare
- Forensics

Major Products/Services

LabVantage's integrated laboratory informatics platform includes the most technologically and architecturally modern Laboratory Information Management System (LIMS) available, with optional embedded Electronic Lab Notebook (ELN), Laboratory Execution System (LES), and Scientific Data Management System (SDMS). LabVantage Analytics, an advanced analytics solution seamlessly integrated to offer AI, ML, and more through the platform, enhances analysis and insights from a greater range of data: LIMS, business, and external sources. The complete LabVantage platform is entirely configurable without the need for any coding. It features modern technology and architecture that enable remote, compliant user-access from any device; its zero-footprint means no client programs or plugins to install or validate; it can be installed on-premise, via the cloud, or SaaS (with standard or enterprise options). Purpose-built industry accelerators of its LIMS are pre-configured for ease of use and faster deployment in pharma, biobanking, food & beverage, oil & gas, and more.

Facilities

LabVantage customers are supported locally from nearly two dozen global offices with support and service contracts, as well as training.

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NUMBER OF EMPLOYEES
Approximately 500

YEAR FOUNDED
1983

LabVantage®

LECO Corporation

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WEBSITE
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YEAR FOUNDED
1936

Company Description

For over 85 years, industries around the world have trusted LECO to deliver technologically advanced products and solutions. Today's technologies for separation science reflect LECO's commitment to understanding your laboratory's challenges and providing solutions that can investigate highly complex samples while streamlining your analysis. LECO instruments offer unparalleled separation, accuracy, resolving power, deconvolution, and speed to increase your laboratory's productivity using GC–TOFMS, GCxGC, GCxGC–TOFMS, and high-resolution GC– and GCxGC–TOFMS. For more information, please visit them on the web at www.leco.com.

Chief Chromatographic Techniques Supported

- GC–TOFMS
- High Resolution GC–TOFMS
- GCxGC–FID/ECD
- GCxGC–TOFMS
- High Resolution GCxGC–TOFMS

Markets Served

LECO Separation Science instruments are used in a variety of applications, helping users to uncover unidentified compounds in their samples. Markets include metabolomics, consumer product safety, food, flavor, fragrance, environmental, and energy and fuels analysis.

Major Products/Services

An ideal choice for laboratories investigating highly complex samples, LECO products feature exclusive ChromaTOF® brand software with all the tools needed to easily locate, identify, and quantify both target and unknown analytes, dramatically reducing overall analysis times and increasing productivity.

Products include:

- Pegasus® BT GC–TOFMS: The tried-and-trusted reliability and durability of our Pegasus brand in a convenient benchtop unit
- Pegasus BT 4D GCxGC–TOFMS: Four dimensions of analytical resolution offer a more complete analysis in a benchtop instrument
- Pegasus GC-HRT+: GC–MS with industry-leading resolution (up to 50,000) and mass accuracy for high-information content analysis
- Pegasus GC-HRT+ 4D: Combining the highest performance GCxGC and TOFMS on the market with High Resolution Deconvolution®(HRD®)

Facility

LECO's global headquarters in St. Joseph, Michigan include the Elizabeth S. Warren Technical Centre, a 28,000 square-foot facility exclusively dedicated to the research and development of innovative instrumentation and equipment for LECO's separation science line. LECO has more than 25 subsidiaries worldwide, plus additional international distributors.

Markes International

Markes International Ltd.
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Western Ave.,
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Markes International, Inc.
2355 Gold Meadow Way
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Markes Instruments (Shanghai) Co., Ltd
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NUMBER OF EMPLOYEES
200

YEAR FOUNDED
1997

Company Description

Markes International—an industry leader in extraction and enrichment (sample preparation) technology for trace organic analysis—manufactures a range of instrumentation and software that enhances the analytical capability and productivity of GC–MS systems.

Markes' technologies enable analysts to discover more about their samples, and to deliver higher throughput for both research and routine applications.

Chief Chromatographic Techniques Supported

- Thermal desorption (TD) as a sample concentration and introduction technique for GC
- Automated sample preparation and concentration (headspace, headspace-trap, SPME, SPME-trap, and high capacity sorbent extraction)
- Time-of-flight mass spectrometry (TOF-MS) for GC and GC×GC (through Markes' sister company, SepSolve Analytical)
- GC–MS data reprocessing software (through Markes' sister company, SepSolve Analytical)

Markets Served

- Defense/Homeland security
- Environmental
- Food and drink
- Forensic and toxicology
- Breath analysis
- Petrochemical

Markes' global customer base includes major industry, government agencies, academia, and the contract service laboratory sector.

Major Products/Services

Markes is globally recognized for its innovation, high-quality products, unrivaled technical expertise, and high level of customer service within the field of separation science. As a global technology leader of thermal desorption and other sample preparation for GC, Markes has introduced many highly successful products and technologies to the laboratory, enabling analysts to discover more and deliver more.

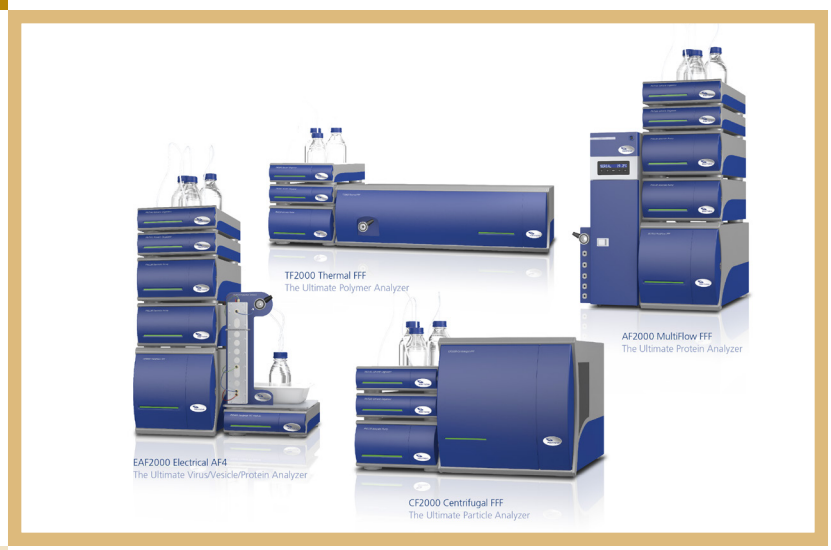
- A range of analytical thermal desorption systems for tube, online, and canister sampling (products include: UNITY-xr, TD100-xr, TT24-7 and CIA *Advantage*-xr)
- Centri: A breakthrough in automated sample extraction and enrichment for GC–MS
- Micro-Chamber/Thermal Extractor for fast sampling of emissions from products and materials
- TC-20 sorbent tube conditioner
- Wide range of supplies and consumables for sample preparation (extraction and enrichment)

Facility

Markes International's factory, technical center, and headquarters are located near Cardiff, UK. The company also has technical centers in Germany, USA, and China. It also supports a global distributor network. Markes and its sister company, SepSolve Analytical Ltd, are companies of the Schauenburg Analytics Ltd group.

MARKES international

Postnova Analytics Inc.



Company Description

Field-Flow Fractionation (FFF) is a separation technology that separates macromolecules or particles that are dissolved or dispersed in a suitable solvent in an open separation channel. Postnova is the exclusive manufacturer offering the whole range of FFF systems including Flow FFF, Thermal FFF, Centrifugal FFF, and SPLITT FFF. FFF provides fast, gentle, and high resolution separations of any particulate matter from 1 nm up to 100 µm in any liquid media in various matrices. Particularly in the fields of nanotechnology, nanomedicine and biotechnology, but also with increasingly complex questions in the area of food, polymer and environmental sciences, FFF technology has become a reliable alternative to established chromatographic separation methods like GPC/SEC or HPLC. Because of its broad application range and its compatibility with detection systems known from liquid chromatography, such as Multi-Angle Light Scattering (MALS), Dynamic Light Scattering (DLS), Raman Microscopy, Refractive Index and ICP-MS.

Separation Techniques Provided

- Field-Flow Fractionation (FFF)
- Asymmetrical Flow FFF (AF4)
- Electrical Asymmetrical Flow FFF (EAF4)
- Thermal FFF
- Centrifugal FFF
- Multi-Angle Light Scattering (MALS)

Facilities

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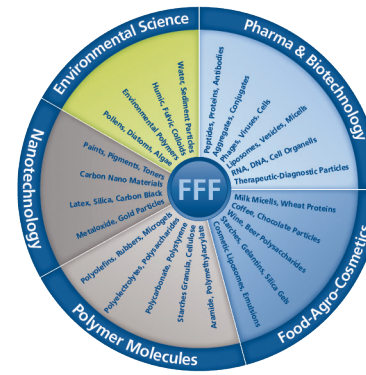

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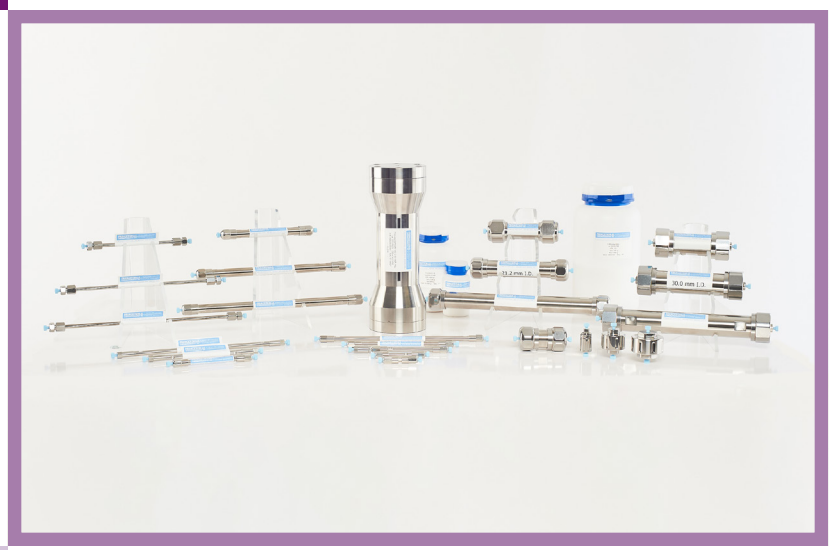
POSTNOVA

Princeton Chromatography Inc.



Company Description

With over 25 years of experience, Princeton Chromatography Inc. delivers premium chromatography products backed by unmatched technical support. From SFC to HPLC, analytical to preparative, all columns are subjected to rigid quality standards. We are one of the earliest developers of novel commercial SFC phases, and continue to lead in this area. We also provide bulk chromatographic media, column packing services, and consulting.

Chief Chromatographic Techniques Supported

- UHPLC
- HPLC
- SFC
- Analytical
- Preparative

Markets Served

Globally providing chromatographic columns and media to the following markets: pharmaceutical, bioscience, cannabinoids, education, and drug discovery.

Major Products/Services

Princeton Chromatography Inc. offers a wide range of stationary phases to support analytical, prep, and ultrahigh-pressure liquid chromatography workflows. With new, innovative phases being added periodically, we continue to expand our catalog to meet your evolving HPLC and SFC needs. All phases are available in a range of particle sizes, column diameters and lengths, from 2.0 mm i.d. to 50.0 mm i.d. Popular phases include C18, 2-Ethylpyridine, Diol, and Cyano. In addition to our standard phases, we also specialize in custom phases as well as column packing services, and bulk chromatographic material.

Facility

Headquarters, production, and testing laboratories are located in Cranbury, NJ.

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NUMBER OF EMPLOYEES
5

YEAR FOUNDED
1994

PRINCETON CHROMATOGRAPHY INC

PRINCETON CHROMATOGRAPHY INC

Over 25 Years of Quality HPLC and SFC Columns

Offering a wide range of phases and dimensions for both analytical and prep

- Expansive catalog of HPLC, UHPLC and SFC columns
- Specialty stationary phase design and manufacturing
- Superior bulk material for large-scale applications
- Custom column packing and OEM services
- Personalized support

Visit our website for more information.

www.pci-hplc.com | 609.860.1803

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WEBSITE
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NUMBER OF EMPLOYEES
500+

YEAR FOUNDED
1985

Company Description

For over 30 years, Restek has been a leader in developing technologies and manufacturing products for gas and liquid chromatography (GC and LC), including columns, reference standards, sample preparation materials, accessories, and more. We have decades of hands-on, practical experience in chemistry, chromatography, and engineering, and our reputation for going the extra mile with Plus 1 customer service and top-performing products is well known throughout the chromatography community. Restek is proud to assist analysts around the world with monitoring the quality and safety of air, water, soil, food, pharmaceuticals, and petroleum. We proactively offer integrated solutions—products, applications, and assistance—perfectly matched to your needs, regardless of your industry. www.restek.com

Chief Chromatographic Techniques Supported

- UHPLC
- HPLC
- LC–MS
- GC
- GC–MS
- GC×GC

Markets Served

- Air monitoring
- Chemical
- Clinical
- Environmental
- Food safety
- Forensic
- Industrial hygiene
- Petrochemical
- Pharmaceutical

Major Products/Services

Plus 1 Service in everything we do. Living this corporate core value every day ensures we will surpass your expectations every time you contact us! Our customer service team will suggest time- and money-saving options, and is dedicated to getting your products to you fast. Our technical service chemists can help you from set-up to method development. Visit our website, where you can interact with our chemists' blogs and explore an extensive library of technical publications, chromatograms, product documentation, step-by-step guides, interactive calculators, animations, and educational material.

Restek's commitment to continuous innovation in chromatography sets us apart from our competitors. We introduce and stock hundreds of new products every year, designed by chromatographers for chromatographers.

- Exceptional columns for UHPLC, HPLC, LC–MS, GC, GC–MS, and GC×GC
- Innovative accessories, instrument replacement parts, and consumables
- Air monitoring canisters and sampling supplies
- Sample preparation products
- Reference standards: stock and custom-prepared formulations
- Thousands of innovative products, hundreds of chromatograms

Facilities

Restek opened for business in 1985 in a small business incubator in central Pennsylvania. Today, more than 500 employee-owners work, play, and celebrate milestones in a state-of-the-art 140,000-square-foot facility in Pennsylvania, and in our regional offices in China, England, France, Germany, Italy, Spain, and Japan.

Sciencix

Facility

- 28,000 square foot facility
- 132+ combined years of experience in reverse engineering, with an emphasis on quality
- Serving customers in 100+ countries
- High stock levels, same-day shipping on most orders
- Customer-driven pipeline for new product development

Company Description

Sciencix, Inc. was founded in 1985 and serves customers in over 100 countries as a trusted ISO 9001:2015 certified supplier of HPLC & Mass Spec repair parts and PM kits proven comparable to OEMs in fit, form, and function. With same-day shipping and up to 30% less cost than OEMs, customers can rely on Sciencix for efficient and easy ordering.

Products & Services

- Repair parts for HPLC and Mass Spec equipment, deuterium lamps, piston seals, check valves, rotor seals, needles, capillary electrodes, fittings, and tubing
- Custom PM kits with only the components you need
- Designs and manufactures custom "off the main menu" parts
- Free technical support and consult on Sciencix parts from team of engineers with 132+ years of experience
- Techniques Supported: HPLC, UHPLC, DAD, VWD, Mass Spec

Major Markets Served

- Pharmaceutical
- Environmental
- Bioanalytical Chemistry
- Forensic & Toxicology
- HPLC & Mass Spec Service Companies
- Academia
- Food & Beverage
- Energy & Fuels

Sciencix
Research & Engineering Lab Headquarters
14261 W. Burnsville Pkwy
Burnsville, MN 55306

Customer Relations & Marketing Headquarters
110 Brady Court, Cary, NC 27511

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WEBSITE
www.sciencix.com

NUMBER OF FACILITIES: 2

NUMBER OF EMPLOYEES
27

YEAR FOUNDED
1985

Sciencix

Scion Instruments

Company Description

Built on the history of Varian in GC and GC–MS, Scion Instruments was acquired by the Techcomp group in 2014. Scion Instruments is committed to continuing the 50+ year legacy of products, service, and innovation. We design, develop, supply, and support GC, GC–MS, LC, Headspace, and CompassCDS (chromatography data system) product lines. Scion Instruments maintains a global infrastructure to support sales and service not only for Scion Instruments customers but also for users of legacy Varian and Bruker systems. Our gas and liquid chromatography solutions help you boost productivity and generate data confidently.

Chief Chromatographic Techniques Supported

- Gas Chromatography (GC)
- GC Mass Spectrometry (GCMS)
- High-Performance Liquid Chromatography (HPLC)
- Static and Dynamic Headspace
- Autosamplers for GC, GC–MS, and HPLC
- Chromatography Data System (CDS)
- Preventative Maintenance and Instrument Repair
- Laboratory Accessories
  - GC Columns
  - HPLC Columns
  - GC Gas Filters
  - GC Inlet Liners and Septa
  - Syringes
  - Vials
  - Balances
  - Temperature Control Units

Markets Served

- Petrochemical and Refineries
- Pharmaceutical
- Food & Beverage
- Flavors & Fragrance
- Chemical
- Contract Laboratories
- Environmental
- Forensic
- Academic
- Cannabis

Major Products/Services

- **Scion 8500 GC** and **8300 GC** offer versatility and superior performance for any application. Detectors: FID, TCD, ECD, PFPD, NPD, PDHID, MS
- **Scion 8700 SQ** and **8900 TQ** are designed for today's fast-paced labs. Both have a small footprint without compromising on quality.
- **Scion LC6000** aims for confidence in results through outstanding lifetime performance and superior gradient precision.
- Scion's **Versa** and **HT3** provide static and dynamic headspace solutions for any laboratory.
- **Scion 8400Pro** and **8410Pro** offer dual injector access with one autosampler.
- **CompassCDS** is our industry-proven, powerful, and operator-friendly chromatography data system software solution.
- Preventative Maintenance and Instrument Repair

Facility

Scion Instruments prides itself on global sales and service infrastructure with the US office located in Fulton, MD. Our instruments are designed at our R&D facility in Livingston, Scotland and manufactured at our headquarters in Goes, The Netherlands.

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SCION INSTRUMENTS
A Techcomp Company

SepSolve Analytical Ltd.

Company Description

SepSolve Analytical provides analytical platforms for separation scientists, including equipment for automated sample introduction, advanced GC separation, state-of-the-art mass spectrometry, and powerful data analysis.

With many years of experience in the field and access to a range of leading equipment suppliers, SepSolve is very well placed to advise on the most difficult challenges in analytical science, helping analysts to discover more and deliver more—in everything from environmental monitoring and biomarker discovery to petrochemical analysis, food aroma profiling, and more.

SepSolve Analytical Ltd, and its sister company Markes International, are part of the Schauenburg Analytics Ltd group of companies.

Chief Chromatographic Techniques Supported

- GC and GC×GC
- TOF-MS
- Software for GC/GCxGC-MS
- Sample preparation (extraction and enrichment): Thermal desorption, SPME and SPME–trap, High-capacity sorptive extraction, Headspace and Headspace–trap

Markets Served

- Biomarker discovery
- Food and drink
- Petrochemical
- Fragrance
- Environmental
- Tobacco and e-cigarettes
- Cannabis

Major Products/Services

The wide range of products and techniques offered by SepSolve includes the company's own INSIGHT-Flow and INSIGHT-Thermal GC×GC modulators, ChromSpace® and ChromCompare+ software for GC and GC×GC, and BenchTOF range of time-of-flight mass spectrometers with groundbreaking simultaneous hard- and soft-ionization technology—Tandem Ionisation®. SepSolve also offers sample preparation equipment, robotic autosamplers and thermal desorbers from leading global suppliers.

Facility

SepSolve has offices and demonstration laboratories in Peterborough, UK, and Waterloo, Canada, and works closely with partners to support customers worldwide, with facilities in countries including the United States, Germany, and China.

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YEAR FOUNDED
2016

SepSolve Analytical

Shimadzu Scientific Instruments

Company Description

Shimadzu Scientific Instruments (SSI) is the North American subsidiary of Shimadzu Corporation's Analytical and Measuring Division. Headquartered in Columbia, Maryland, SSI offers a comprehensive portfolio of robust, precision-engineered platforms. From teaching environments and QA/QC to innovative R&D projects, customers can count on the stability, experience, and support only Shimadzu offers.

Chief Chromatographic Techniques Supported

- Analytical HPLC, UHPLC
- Prep HPLC, SFC
- Inert UHPLC
- SFE–SFC
- Ion Chromatography
- LC–MS/MS
- Q-TOF LC–MS
- Multiplex LC–MS
- GC
- GC–MS/MS

Markets Served

Shimadzu's product line flexibility enables chromatographers in any environment to select the instrument best suited to their application. Shimadzu instruments are found in a wide range of laboratories, including pharma/biopharma, environmental, food and beverages, petrochemical, life sciences, and clinical. Shimadzu provides free technical support for the life of the instruments, and encourages customer alliances to further product development.

Major Products/Services

Shimadzu offers a wide range of advanced UHPLC/HPLC, GC, and mass spectrometry systems and components. Key systems include:

- Nexera 40 series UHPLC—Offering the most advanced performance features available, the 40 series enables smart, efficient workflows and delivers excellent data quality.
- Nexera SFC/Prep SFC—User-focused automation and streamlined software simplify processes, enhance accuracy and increase productivity
- Single-quad LCMS-2050—Compact, robust system provides a complete package of easy-to-use high-level performance
- Triple-quad LC–MS/MS—Outstanding speed, sensitivity, and robust operation for maximum uptime and the utmost in data quality
- Nexis GC-2030—Offers a modern approach to a classic chromatographic technique, delivering exceptional performance and faster ROI.
- * Brevis GC-2050—Best-in-class analytical performance and scalability in an ultra-compact design.
- GC–MS/GC–MS/MS Systems—With Smart Technologies, single- and triple-quad systems enable new possibilities in sensitivity, durability, stability, and reliability.

Facilities

Shimadzu's US headquarters includes a customer service and training center, a solution center to showcase technologies, and an innovation center for promoting collaborative projects with customers. Shimadzu's regional facilities, strategically located around the US, provide customers with local sales, service, and technical support.

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WEBSITE
www.ssi.shimadzu.com

NUMBER OF EMPLOYEES
US: 605
Worldwide: 13,800

YEAR FOUNDED
Shimadzu Scientific Instruments: 1975
Shimadzu Corporation: 1875

SHIMADZU
Excellence in Science

SHIMADZU
Excellence in Science

Brevis™ GC-2050

Reshaping Gas Chromatography in a Compact, Robust Design

Discover Shimadzu's new **Brevis GC-2050 gas chromatograph**, which delivers best-in-class analytical performance and scalability in an ultra-compact design, and reduces power consumption by 30% compared to conventional models. Despite the 35% footprint decrease, the Brevis GC-2050 can be configured with a variety of detectors and sample introduction techniques, including dual-line configurations, using industry-standard columns and consumables.

The Brevis GC-2050 offers:

- **B**ig impact, compact footprint
- **R**emote display monitoring and control
- **E**nhanced analytical performance
- **V**ersatile configurations
- **I**ntelligence automated
- **S**implified operation and maintenance

Learn more about Shimadzu's Brevis GC-2050. Visit us online at

www.ssi.shimadzu.com

Shimadzu Scientific Instruments Inc., 7102 Riverwood Dr., Columbia, MD 21046, USA

Shodex – Resonac America, Inc.

Company Description

Shodex HPLC columns have been manufactured by Showa Denko K.K. in Japan since 1973. Now under Resonac America, Inc. our columns are designed to provide superior separation and purification of complex mixtures, making them a valuable tool for researchers in a wide range of industries. Our strength in polymer synthesis led to the production of many unique and durable polymer-based columns. The golden-mechanical fish is a symbol of fast and smooth liquid chromatography analysis.

Chief Chromatographic Techniques Supported

Chief Chromatographic Techniques Supported HPLC column selection, method development, and trouble shooting, as well as providing educational seminars.

Markets Served in North and South America

- BioPharma
- Applied sciences
- Life sciences
- Analytical chemistry
- Environmental sciences
- Food industry

Major Products/Services

Shodex carries thousands of different HPLC columns with various separation modes. We are best known for high quality size exclusion and saccharide analysis columns, as well as unique polymer-based reversed and HILIC phase columns. With additional selections of normal phase, ion exchange, ion chromatography, high temperature, and multimode columns, we are confident we can provide the most suitable column for customers' needs.

Facilities

Resonac America, Inc., based in New York, New York, provides technical and sales support for Shodex HPLC columns for customers and distributors in North and South America. Our stock point in New York allows for fast delivery in the United States. Working with our laboratories in New York City and Japan, we support customers' analytical needs.

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YEAR FOUNDED
1973

Shodex™
CAPTURE THE ESSENCE

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NUMBER OF EMPLOYEES
65

WEBSITE
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YEAR FOUNDED
2009

Company Description

SilcoTek® Corporation is the world's leading provider of flow path deactivations and coatings. Our patented CVD coating technologies improve analytical reliability, increase system uptime, and boost your bottom line. SilcoTek's coatings are used in a large, diverse variety of industries and applications where analytical accuracy and superior performance are critical. Whether for industry-leading chemical compatibility, corrosion resistance, bio-inertness, or hydrophobicity, SilcoTek coatings will expand the material limits of your instruments and products.

Chief Chromatographic Techniques Supported

- Sulfur analysis
- VOC analysis
- Environmental
- Thermal desorption
- Fenceline monitoring
- CEMS
- Flare subpart JA
- Stack monitoring
- Protein analysis
- NOx, SOx testing
- GC
- LC

Markets Served

SilcoTek coating technologies are utilized in diverse applications and industry segments worldwide. Since 1987, customers have relied on SilcoTek coatings to improve the performance of their products and increase revenue in the following markets and applications:

- Process
- Analytical
- Bio/Pharma
- Oil & Gas
- Refining
- Petrochemical
- Semiconductor
- Corrosion
- Coking/Fouling
- Automotive
- Aerospace

Major Products/Services

Major Products/Services: SilcoTek offers custom silicon coatings to OEMs and labs worldwide. Send in your stainless steel, alloy, glass, or ceramic part and we'll apply our signature CVD coatings. Our coating options include:

- SilcoNert®: The most inert coating available.
- Dursan®: An inert, high durability coating. Ideal for lab and field analysis.
- Silcolloy®: A high purity corrosion resistant coating.
- SilcoKlean®: Prevent carbon coking and fouling in automotive and aerospace.
- SilcoGuard®: A low outgassing, high purity coating, ideal for research and semiconductor applications.
- Dursox®: A high purity corrosion-resistant coating.
- Siltride®: SilcoTek's most protective and versatile silicon nitride CVD coating technology.

Facility

SilcoTek applies coatings to customer-supplied products from their newly renovated 70,000 square foot facility state of the art facility in Bellefonte, Pennsylvania. Want to speak to an expert about your application?
Email TechService@SilcoTek.com
or call +1 (814) 353-1778.

To find a sales representative nearest to you, please visit
www.silcotek.com/international-reps

Syft Technologies

Company Description

Syft is the world leader in real-time, direct injection mass spectrometry with more than 20 years of SIFT-MS expertise. Syft instruments support a broad range of industries worldwide including pharma and CDMOs, environmental protection, consumer goods, food, flavor and fragrance, semiconductor manufacturing and many more. Continually developed and proven in high stakes commercial environments, you can be assured of operational robustness, speed and support. Syft has offices throughout the world offering 24/7 service and support including those in New Zealand, Korea, Taiwan, Singapore, Germany and the U.S.

Chief Chromatographic Technique Supported

- Mass spectrometry

Markets Served

- Pharma/CDMO
- Environmental
- Consumer Product Safety
- Food, Flavors, & Fragrances
- Semiconductor
- Automotive
- Petrochemcial
- Food Packaging
- Border Security
- Indoor Air Safety
- Container Safety

Major Products/Services

- Syft Tracer
- Syft Tracer Pharm11
- Voice200*Infinity*
- ContainerSure

Facilities

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NUMBER OF EMPLOYEES
>150 worldwide

YEAR FOUNDED
2002

Tosoh Bioscience LLC

Company Description

Tosoh Bioscience LLC is a major supplier of chromatography products worldwide, particularly to the pharmaceutical, biotechnology, and polymer industries. The company is a division of Tosoh Corporation, a global chemical company with headquarters and manufacturing facilities in Japan. Tosoh's portfolio of products encompasses all common modes of liquid chromatography.

Chief Chromatographic Techniques Supported

- Size exclusion
- Reversed phase, normal phase (hydrophilic interaction)
- Ion exchange
- Hydrophobic interaction
- Affinity
- Mixed-mode

Markets Served

Located in King of Prussia, Pennsylvania, Tosoh Bioscience LLC provides sales and service to pharmaceutical and biotechnology customers in North and South America.

Major Products/Services

Tosoh Bioscience LLC offers a comprehensive line of TOYOPEARL®, TSKgel®, and Ca^{++}Pure-HA™ bulk resins for process chromatography. A variety of screening tools, including SkillPak™ pre-packed columns are available as an additional tool for convenient scale-up. In addition, bulk media volumes of <1 L are offered for process development.

Analytical U/HPLC columns containing TSKgel media are designed for the analysis of proteins, peptides, other biopolymers, low molecular weight compounds, and organic soluble polymers. The packings in the columns are either silica-based or polymer-based material, in particle sizes ranging from 2 µm to 30 µm.

The EcoSEC Elite™ GPC System and the EcoSEC™ High Temperature GPC System are dedicated GPC instruments developed specifically for polymer analysis. These all-in-one systems deliver top performance, reliability, and superior results.

The LenS™3 multi-angle light scattering detector allows direct measurement of molecular weight and provides best-in-class sensitivity. The new LenS3 MALS-V is the only all-in-one MALS-Viscometry detector, offering a simple and compact solution for triple detection measurements.

The Octave™ BIO and Octave PRO MCC Systems bring the power of continuous chromatography to our product portfolio.

Facilities

Tosoh Bioscience LLC has US offices in King of Prussia, PA, and Madison, WI; manufacturing operations in Madison, WI, and Grove City, OH; and supply chain operations in Grove City, OH. Asia is served by Tosoh Corporation in Tokyo, Japan, Shanghai, China, and Singapore. Products manufactured by Tosoh Corporation, Japan.

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WEBSITE
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NUMBER OF EMPLOYEES
130

YEAR FOUNDED
1987

TOSOH BIOSCIENCE
TOSOH

UCT, Inc.

Company Description

UCT is at the forefront of sample preparation technology and a leader in chromatography consumables. The company's wide range of highly reproducible silica and polymeric SPE sorbents provides scientists a consistent extraction technique. UCT is in a unique position within the sample prep and chromatographic sorbent industry; we are one of the few manufacturers of chromatographic silane materials.

This expertise provides us with greater control and extensive knowledge of the chemical processes involved in producing high quality bonded phases. UCT's commitment to ensuring customer satisfaction is accomplished by delivering on our promises: top quality, reproducible chromatographic and sample prep products, and unmatched technical support. The company's sample prep product lines include SPE cartridges and well plates, bulk sorbents, QuEChERS tubes, derivatizing reagents, hydrolyzing enzymes, premeasured buffer salts, and extraction manifolds. Chromatography products include HPLC columns and GC liners.

Chief Techniques Supported

- Solid-phase extraction
- QuEChERS
- HPLC

Markets Served

- Clinical
- Environmental
- Food safety
- Forensic
- Pharmaceutical
- Veterinary

Major Products/Services

- CLEAN SCREEN® SPE columns
- Refine® Ultra Filtration Columns & Well Plates
- CLEAN UP® SPE columns
- STYRE SCREEN® SPE columns
- XtrackT® SPE columns
- ENVIRO-CLEAN® SPE cartridges
- ENVIRO-CLEAN® universal cartridges
- Fusion® Ag+
- Chlorofiltr® Sorbents
- QuEChERS
- Quick QuEChERS
- SpinFiltr™
- LipiFiltr®
- Selectra® U/HPLC columns
- SelectraCore core-shell columns
- SELECTRASORB™ bulk sorbents
- SELECTRA-SIL® derivatizing reagents
- SELECTRAZYME® & ABALONASE™ β-glucuronidase enzyme
- SELECT pH buffer pouches
- Positive pressure manifolds
- SPeVAP® Multi-Function Solvent Evaporator
- Vacuum manifolds
- MicroPrep™ 96-Well Microelution Plate

Facility

UCT is headquartered in Bristol, Pennsylvania. Manufacturing and distribution sites are in Lewistown, Pennsylvania, and Wexford, Ireland. UCT is represented worldwide by more than 50 partners and distributors.

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NUMBER OF EMPLOYEES
>100

YEAR FOUNDED
1986

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NUMBER OF EMPLOYEES
US: 365
Europe: 135

YEAR FOUNDED
1968

Company Description

Founded in 1968, VICI is an international group of companies. Our products can be found in nearly every national lab, military facility, and university research department around the globe. For 50 years VICI has driven the field of chromatography with valves, fittings, detectors, and instruments for precision analytical, biomedical, and biocompatible applications. We also customize components and complete systems to improve chromatographic analyses.

Chief Chromatographic Techniques Supported

- High Pressure Liquid Chromatography
- Liquid Chromatography
- Gas Chromatography
- Flow Injection Analysis
- Supercritical Fluid Chromatography
- Gas Permeation Chromatography
- Process Gas Chromatography

Major Products/Services

VICI produces a complete line of valves for analytical chemistry. We specialize in zero dead volume fittings and filters for the separation sciences and are the original creators of the pulse discharge detector (PDD). Our miniPDD consumes less than one-fifth the amount of helium required by the PDDs used on complete GC systems. It has similar sensitivity to the original PDD with a slightly lower dynamic range.

Our VICI Metronics division produces PEEK, FEP, PFA, and ETFE tubing, ambient temperature gas purifiers, ValcoBond GC capillary columns, and Dynacal gas calibration standards. VICI Precision Sampling manufactures analytical quality syringes, sampling probes, as well as standard and custom formed stainless and special alloy tubing. VICI AG International produces some Valco products as well as the Jour line of HPLC fittings and lab safety products. VICI DBS builds our new line of laboratory hydrogen, nitrogen, and zero air gas generators.

Our newest products include the C82 line of UHPLC valves, fittings that permit direct connection of 360 µm tubing, the Universal valve actuator, and the C52 platform integrated HPLC injectors and selectors.

Facility

With over 150 state-of-the-art CNC machines, our instruments and components are manufactured to exacting standards at our facilities in Houston, USA, and Schenkon, Switzerland. Our other manufacturing facilities include VICI Metronics in Washington, VICI Precision Sampling in Louisiana, and VICI DBS located in Italy. VICI Canada serves our neighbor to the north from its offices in Brockville, Ontario.

John Wiley & Sons

Wiley Science Solutions – Spectroscopy Databases & Software

Company Description

Wiley Science Solutions is home to high-quality, comprehensive spectral databases and powerful software for spectral analysis and identification, and data management.

About Wiley

Wiley is a knowledge company and a global leader in research, publishing, and knowledge solutions. Dedicated to the creation and application of knowledge, Wiley serves the world's researchers, learners, innovators, and leaders, helping them achieve their goals and solve the world's most important challenges. For more than two centuries, Wiley has been delivering on its timeless mission to unlock human potential. Visit us at Wiley.com.

Chief Spectroscopic Techniques Supported

- MS
- IR
- Raman
- NMR
- UV-vis

Markets Served

- Metabolomics
- Forensics and Toxicology
- Polymers and Materials
- Pharmaceutical and Biotech
- Environmental
- Food and Cosmetics
- Quality Assurance, and many more

Major Products/Services

Spectroscopy Software & Spectral Libraries

Wiley is a leader in spectral data with IR, MS, NMR, Raman, and UV-vis spectra that are essential to analytical laboratory workflows for the interpretation, identification, verification, and classification of spectra. Consistently evolving to increase coverage for meeting today's research demands, these trusted databases cover a wide range of applications including polymer/materials, environmental, forensics/toxicology, pharmaceutical, biotech, automotive/aerospace, food/cosmetics, and more. At the helm of our collections are the renowned Wiley Registry, KnowItAll Spectral Libraries, Sadtler Libraries, and many other important databases used in spectral analysis.

Learn more: https://sciencesolutions.wiley.com/spectral-databases/

Wiley's KnowItAll software offers integrated solutions to analyze and manage multiple types of spectral and chemical data in multiple file and instrument formats. KnowItAll's integrated toolsets eliminate the need for multiple software packages and increase overall lab efficiency. Combined with Wiley's comprehensive reference databases, KnowItAll Software provides an unparalleled solution for spectral analysis. So, whether you use one or more analytical techniques, KnowItAll has the right solution for your laboratory.

Learn more: https://sciencesolutions.wiley.com/knowitall-analytical-edition-software/

John Wiley & Sons
111 River Street, Hoboken, NJ, 07030

WEB SITE
sciencesolutions.wiley.com

MAIN WEB SITE
www.wiley.com

YEAR FOUNDED
1807

WILEY

Take spectral analysis to new heights with KnowItAll 2024

Powerful software. Quality data. Results you can rely on.

KnowItAll offers solutions to **identify, analyze, and manage** analytical data. Combined with Wiley's comprehensive, high-quality spectral reference libraries, it provides an unparalleled solution for **fast, reliable spectral analysis**.

To empower researchers, KnowItAll 2024 revamps manual GC-MS processing to provide users greater control of component separation and identification. These improvements, combined with the completely automated data analysis in the previous release, make KnowItAll an even more reliable, efficient, powerful, and accurate tool for GC-MS analysis.

Supports multiple techniques and instrument vendor formats.

WILEY

www.knowitall.com/lcgc

Quantitative Analysis of Microplastics in Shellfish using Pyrolysis-GC-MS

Author: Shilei Liu, Karen Sam

Five hundred million tons of plastic was manufactured in 2020 and production keeps ramping upwards. Over 50% of that plastic winds up as environmental pollution. Once in the environment, the plastic degrades into particles smaller than 5 mm in size particles, otherwise known as microplastics. Through the food chain, microplastic enters plants and animals. Consequently, when we eat, microplastics, along with attached additives, enter our bodies, resulting strong concerns and demands to quantify microplastics in the environment.

Many analytical techniques are capable of quantifying microplastics. Py-GC-MS can be an effective solution to detect multiple microplastics in a single analysis under 40 minutes, as opposed to days by optical counted-based methods. In this application note, a Py-GC-MS method combining with sample prep by cryo-milling is used to quantify microplastics in Shellfish on a CDS Pyroprobe, which is a pulsed resistive heating pyrolyzer without 2nd pyrolysis even at the slowest flow rate, due to the minimum temperature gradient between sample and heating element during pyrolysis. This design feature enables the instrument to quantify complex microplastic samples at any GC/MS split ratio.

Experimental

All solvents were pre-filtered on a 0.45 µm PTFE membrane. Four types of shellfish were purchased, de-shelled, and 1 gram of each sample type were transferred to individual 30 mL glass flasks with glass stoppers for alkaline digestion. After digestion, the sample was filtered onto glass fiber filters under vacuum. Filters containing particles from alkaline digestion were then ground with a CDS liquid nitrogen cryo-mill (P/N 6204-3023). Each filter was placed into the 5 mL grinding vessel of the cryo-mill with a 9.6 mm grinding ball and capped. Next, each vessel was immersed in liquid nitrogen for 5 min, then removed and ground in the mill for 40s at a 65Hz vibration frequency for a total of 8 repetitions. A 2 mg ground subsample was added to a pre-cleaned DISC tube for pyrolysis.

For the standard calibration, 1 mg each of Polyethylene (PE), polymethyl methacrylate (PMMA), polyethylene terephthalate (PET), polyvinyl chloride (PVC), polystyrene (PS) and polypropylene (PP), were weighed and added to cryo-mill vessels with 0.15 g of diatomaceous earth and ground to obtain a 6.67 µg/mg concentration of microplastics as a stock standard. Then 0.66 mg, 0.97 mg, 1.48 mg, 1.97 mg and 2.44 mg of stock standard were placed in pre-cleaned DISC tubes and analyzed for a total of 5 calibration levels.

Table I: 6 Chosen Indicator Peaks for 6 microplastics

Microplastic	Indicator Compound	RT Quant Ion
PMMA	Methyl Methacrylate	3.82 100
PS	Styrene	6.92 104
PP	2,4-Dimethylhept-1-ene	5.99 70
PVC	Naphthalene	10.25 128
PE	1-Undecene	5.99 55
PET	Biphenyl	11.09 154

Table II: Calibration Curve Linearity

Polymer	R^2
PMMA	0.999
PS	0.992
PP	0.970
PVC	0.982
PE	0.985
PET	0.987

Table III: RSD Cryo-grinding Replicates.

	Area Counts				
Polymer	1	2	3	4	RSD
PMMA	930897	968001	887338	970507	4.15%
PS	207426	214896	203695	213600	2.51%
PP	208176	224057	204323	222725	4.67%
PVC	242205	246945	241199	257526	3.02%
PE	87323	83657	87368	89454	2.77%
PET	723135	686987	667420	695292	3.34%

Results

To distinguish microplastics from each other, indicator compounds were chosen to both identify and quantify each microplastic (Table 1). Calibration was performed by plotting polymer weight against quant ion area counts. Each of the six microplastic standards presented a linear calibration with an R^2>0.97 (Table 2). A replicate study on the 13.13 µg level provided RSDs around or under 4% (Table 3). The replicate chromatograms are overlayed in Figure 1.

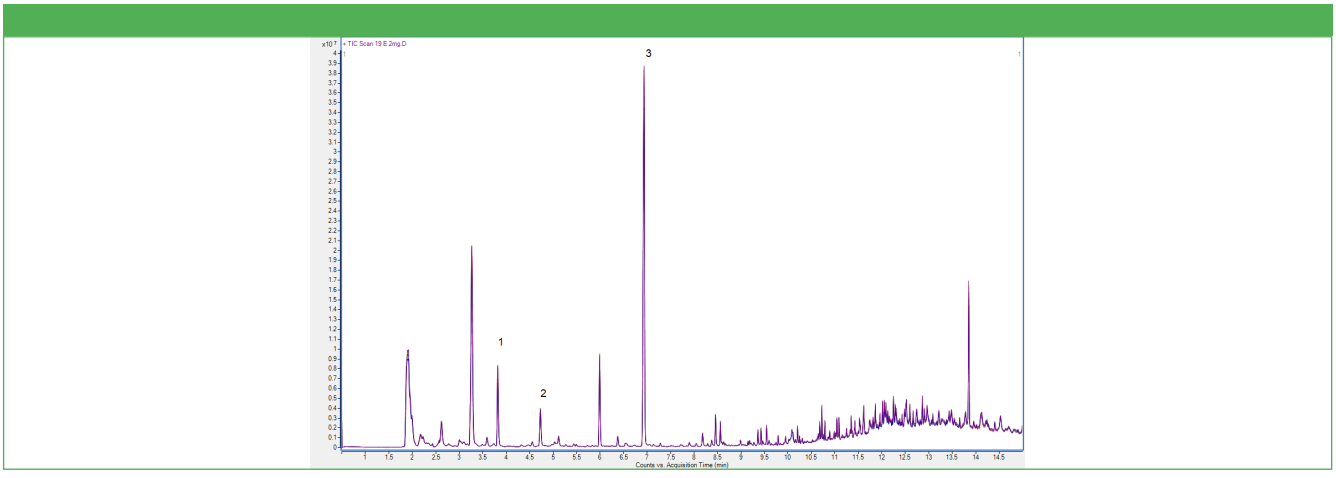

Figure 1: Overlay of 4 replicate standards. Peak Identifications 1: Methyl methacrylate, 2: 2,4-Dimethylhept-1-ene, 3: Styrene.

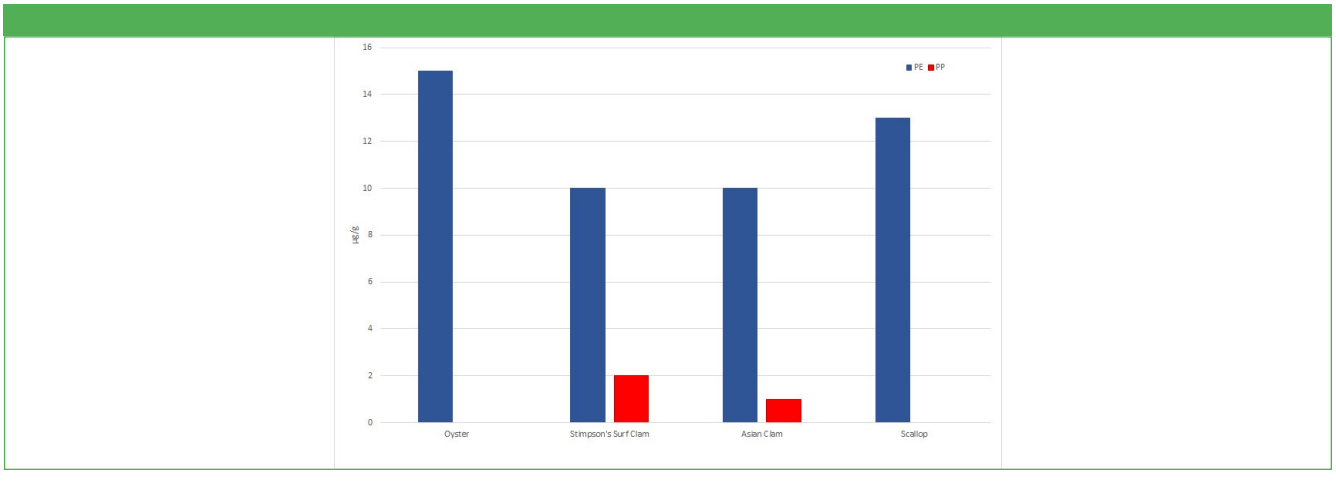

Figure 2: Shellfish sample test results.

Quantification of the microplastics in the shellfish was performed by applying the linear equations generated from each calibration plot. It was found that the shellfish contained PE and PP, exceeding 80% of the total microplastic amount, with ranges from 1 µg/g to 15 µg/g (Figure 2).

Conclusion

Quantification of microplastics in shellfish was accomplished by sample preparation involving a solid diluent and cryo-milling, followed by Py-GC-MS.

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Shodex LB-800 HQ Column Series for SEC-MALS Analysis

Introduction

Multi-angle light scattering (MALS) detection has become an indispensable tool for large molecule analyses such as protein and polymer characterization. MALS detectors determine absolute molar mass and conformation of macromolecules, comparable over a wide range of molecular weights. Measurements are made without reference to molar mass standards, column calibration, or molecular conformation.

The coupling of MALS with high-performance size exclusion chromatography (SEC) has provided a unique and attractive technique for obtaining absolute molecular weight information and molecular size information about macromolecular systems including both natural and synthetic materials.

Shodex™ OHpak™ LB-800 HQ series is a size exclusion chromatography column compatible with aqueous solvents as well as organic solvents such as N,N-dimethylformamide (DMF). The column series is suitable for the separation and molecular weight distribution analysis of high molecular weight polymers, proteins, and/or oligomers. Further improvement of the Shodex SB column series for SEC-MALS detection for superior base-line noise performance has been achieved with the LB column series.

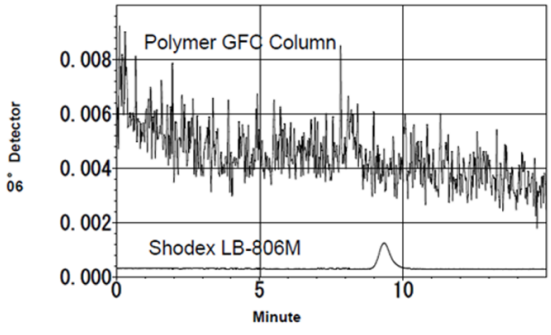
Shodex™ has successfully developed the OHpak™ LB-800 columns which are suitable for MALS detection. The LB column series is comparable to the SB column series; however, extra washing is performed on the stationary phase to reduce column shedding. The LB series are well-suited to provide signal at low concentration with low molecular weight standards.

Experimental conditions: A Shodex™ OHpak™ LB-806M column (8 mm I.D. x 300 mm, 13 µm) was used with a MALS detector. The method conditions were isocratic, using 0.1M $NaNO_3$ with a flow rate of 1.0 mL/min, and the column temperature at 30°C.

Results: ShodexTM has successfully developed the ShodexTM OHpakTM LB-800 columns, which are suitable with MALS detection for SEC analysis. In comparison to the Polymer GFC column, the ShodexTM OHpakTM LB-806M provides low S/N using the MALS detector, and therefore, better chromatography capabilities for different MW Pullulan standards (Figure 1).

Conclusion: The ShodexTM OHpakTM LB-800 column series is well-suited to provide signals at low concentration with low molecular weight standards. ShodexTM OHpakTM LB-803 HQ coupled with MALS detection has very low baseline noise and improved chromatography.

Improvement of Baseline Noise



Comparison of Signal to Noise Ratio (S/N)

0.5% Pullulan	Polymer GFC	LB-806M
Mw 1420	-	17
Mw 5300	-	96
Mw 12000	-	189
Mw 20800	4	422
Mw 46700	6	567
Mw 95400	13	780
Mw 194000	20	1375

Figure 1: The chromatogram compares the performance of a Polymer GFC column to the Shodex™ OHpak™ LB-806M column to detect 0.5% Pullulan (MW of 12,000 Da) using a MALS detector. The table shows the Signal to noise ratio comparison of a Polymer GFC column to the Shodex™ OHpak™ LB-806M column.

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Characterization of a Novel Antibody Drug Conjugate Mimic by Size Exclusion and Hydrophobic Interaction Chromatography

Empowered antibodies, such as antibody drug conjugates (ADCs), continue to be investigated as biotherapeutic drug candidates. ADCs combine the tumor specificity and targeting capability of mAbs with the cytotoxicity of potent small molecule drugs into hybrid molecules that are promising anticancer therapeutics. These molecules are comprised of three components: a monoclonal antibody, a stable linker, and a cytotoxic small molecule drug. For the cysteine-linked ADC mimic used in this study, a dansyl fluorophore (~668 Da) is covalently bonded to an IgG_1 mAb (150 kDa) via an LC-SMCC crosslinker (Figure 1). This procedure results in a mixture of drug-loaded antibody species with 0 to 8 drugs (Figure 2).

ADC conjugation plays a role in both drug efficacy as well as clearance, and it must be well understood during drug development. Size exclusion chromatography (SEC) and hydrophobic interaction chromatography (HIC) are two commonly employed techniques used to characterize the drug-to-antibody ratio (DAR) under native, physiological conditions. In this application note, the ADC mimic was analyzed by size exclusion chromatography/mass spectrometry (SEC/MS) using a TSKgel® SuperSW3000 column and by HIC using a TSKgel Butyl-NPR (nonporous resin) column. Coupling these chromatographic techniques allowed elucidation and verification of the DAR profile for this model biomolecule.

Experimental HPLC Conditions

SEC/MS Conditions

Column:	TSKgel SuperSW3000, 4 µm, 2 mm ID × 30 cm
Mobile phase:	100 mmol/L ammonium acetate, pH 7.0
Gradient:	isocratic
Flow rate:	0.07 mL/min
Detection:	ESI-MS
Temperature:	35 °C
Injection vol.:	1.0 µL
Samples:	ADC mimic, 100 µg/mL (MilliporeSigma™), 100 mmol/L ammonium acetate, pH 7.0
MS mode:	Scanning, m/z 1000-8000

HIC/UV Conditions

Column:	TSKgel Butyl-NPR, 2.5 µm, 4.6 mm ID × 10 cm
Mobile phase:	A. 50 mmol/L potassium phosphate, 1.5 mol/L ammonium sulfate, pH 7.0 plus 5% (v/v) isopropyl alcohol B. 50 mmol/L potassium phosphate, pH 7.0 plus 20% (v/v) isopropyl alcohol
Gradient:	0% B to 100% B in 50 min
Flow rate:	1.0 mL/min
Detection:	UV @ 215 nm
Temperature:	35 °C
Injection vol.:	5.0 µL
Samples:	ADC mimic, 100 µg/mL (MilliporeSigma),

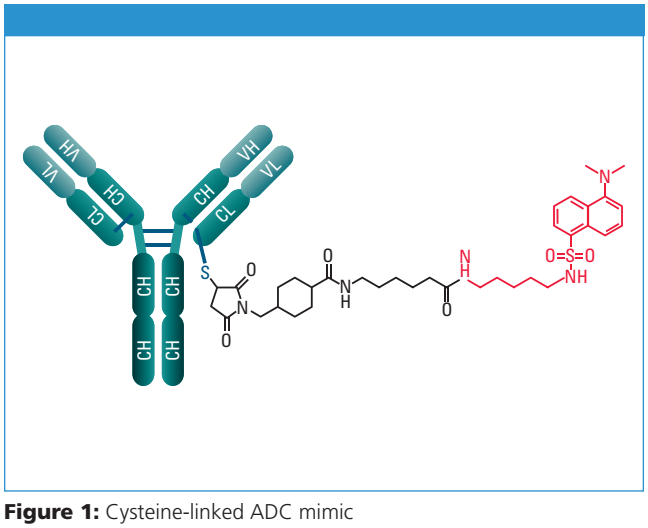

Figure 1: Cysteine-linked ADC mimic

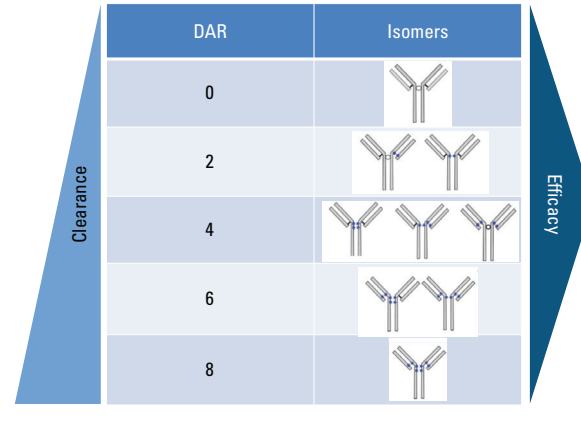

Figure 2: Heterogeneity of cysteine-conjugated ADCs

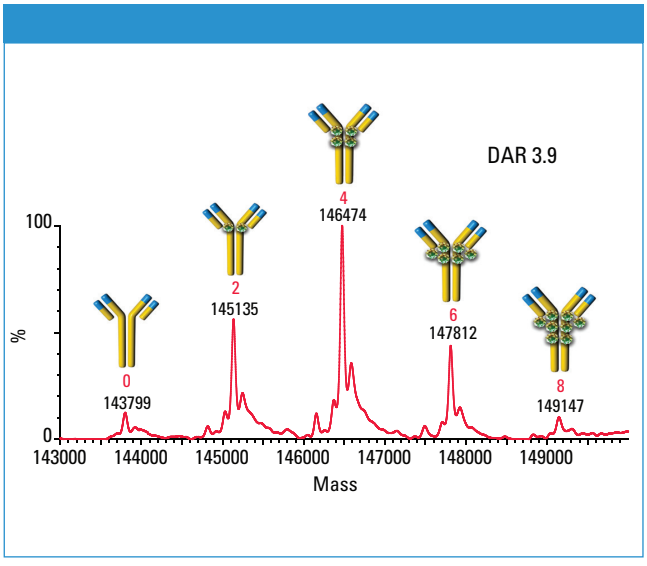

Figure 3: Native SEC/MS spectrum of the ADC mimic

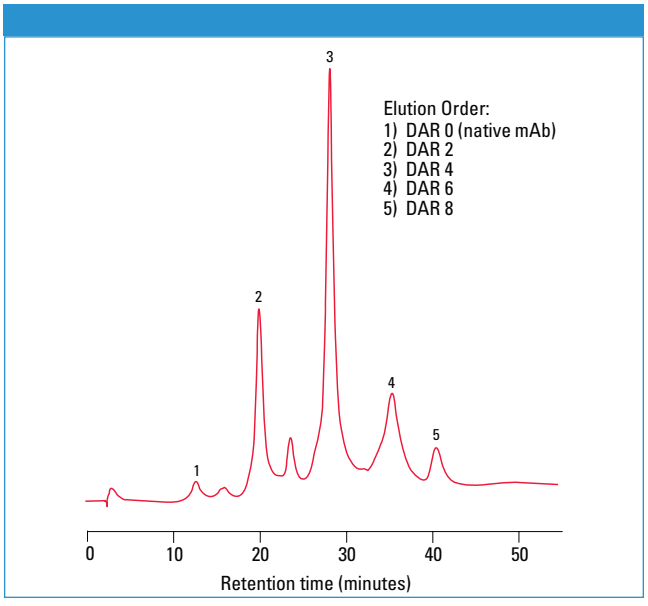

Figure 4: HIC/UV analysis of native ADC mimic

Results and Discussion

The ADC mimic was injected onto a TSKgel SuperSW3000 SEC column coupled to a mass spectrometer in order to examine the DAR profile. Figure 3 shows the deconvoluted mass spectrum of the ADC mimic. Main peaks can be seen at m/z 143,799; 145,135; 146,474; 147,812; and 149,147. The difference in molecular weight between each main peak is 1336 Da, corresponding to the molecular weight of two dansyl fluorophore molecules. The average DAR was found to be 3.9.

The DAR profile was then confirmed by HIC using a TSKgel Butyl-NPR column and UV detection. As more of the drug is conjugated to the mAb vehicle, the ADC becomes more hydrophobic and is retained longer by the HIC stationary phase, allowing resolution of the different drug loaded species. Figure 4 shows the DAR profile of the ADC mimic. The chromatogram shows well resolved peaks ranging from a DAR of 0 to 8.

Conclusions

SEC/MS and HIC/UV can be effectively used to characterize the DAR profile of ADCs. The mobile phase ensured a non-denaturing, MS compatible condition that was successfully used alongside the TSKgel SuperSW3000 SEC column to elucidate the molecular weight of the ADC species present in the drug mimic by high resolution ESI-MS detection; SEC/MS analysis indicated that the average DAR was 3.9. HIC/UV using a TSKgel Butyl-NPR column further confirmed the DAR profile by probing the hydrophobic character of the various antibody-payload combinations present in the sample. An average DAR of 3.9 was verified via HIC/UV analysis.

Data Contributed by MilliporeSigma:

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OTHER COLUMNS, LOW-PRESSURE

Hamilton Company
800-648-5950
SiliCycle Inc.

REVERSED-PHASE

Hamilton Company
800-648-5950
SiliCycle Inc.
Sorbent Technologies, Inc.
Waters Corporation
YMC Europe
+49 20644270

RIGID SIZE-EXCLUSION

Waters Corporation

COLUMNS, MICROBORE (1-2 MM I.D.)

CAPILLARY

Greyhound Chromatography and Allied Chemicals Ltd
LC Services Ltd

CUSTOM

Hamilton Company

GEL PERMEATION (SIZE-EXCLUSION)

Tosoh Bioscience GmbH
+49 6155-7043700
Waters Corporation
YMC Europe
+49 20644270

ION-EXCHANGE

Hamilton Company
PolyLC Inc.
YMC Europe
+49 20644270

ION-EXCLUSION

Hamilton Company
800-648-5950

NORMAL-PHASE

Develosil
HILICON AB
Teknokroma Analítica S.A.
YMC Europe
+49 20644270

OTHER COLUMNS, MICROBORE (1-2 MM I.D.)

Hamilton Company
HILICON AB

REVERSED-PHASE

Develosil
Hamilton Company
800-648-5950
Phenomenex, Inc.
Regis Technologies, Inc.
Teknokroma Analítica S.A.
YMC Europe
+49 20644270

ULTRAHIGH-PRESSURE LC (UHPLC)

Develosil
HILICON AB
YMC Europe
+49 20644270

COLUMNS, SPECIALTY

AMINO ACIDS

Develosil
HILICON AB
Regis Technologies, Inc.
Teknokroma Analítica S.A.
Welch Materials, Inc.

BASIC COMPOUNDS

Develosil
Teknokroma Analítica S.A.
Welch Materials, Inc.
YMC Europe
+49 20644270

BIOPOLYMERS

Develosil
Hamilton Company
800-648-5950
PSS GmbH - Perfect Separation Solutions

CARBOHYDRATES

Hamilton Company
800-648-5950
HILICON AB
Teknokroma Analítica S.A.
Welch Materials, Inc.

MONOCLONAL ANTIBODIES

Develosil
Hamilton Company
800-648-5950
Novilytic
PSS GmbH - Perfect Separation Solutions
Tosoh Bioscience GmbH
+49 6155-7043700
YMC Europe
+49 20644270

NUCLEOSIDES AND NUCLEOTIDES

Develosil
Hamilton Company
800-648-5950
HILICON AB
Welch Materials, Inc.
YMC Europe
+49 20644270

ORGANIC ACIDS

Develosil
Hamilton Company
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HILICON AB
Welch Materials, Inc.

OTHER COLUMNS, SPECIALTY

Hamilton Company
800-648-5950
Regis Technologies, Inc.

PEPTIDES

Develosil
Hamilton Company
800-648-5950
HILICON AB
Phenomenex, Inc.
SiliCycle Inc.

Teknokroma Analítica S.A.
Welch Materials, Inc.

POLYNUCLEAR AROMATIC HYDROCARBONS
Hamilton Company
800-648-5950
Teknokroma Analítica S.A.

POLYSACCHARIDES
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PSS GmbH - Perfect Separation Solutions
YMC Europe
+49 20644270

PROTEINS
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Hamilton Company
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Phenomenex, Inc.
SiliCycle Inc.
Teknokroma Analítica S.A.
YMC Europe
+49 20644270

TRICYCLIC ANTIDEPRESSANTS
Develosil
Teknokroma Analítica S.A.

DETECTOR ACCESSORIES

FLOW CELLS
ECOM spol. s r.o.
+42 0221511310
LC Services Ltd
Waters Corporation

LAMPS
HPLC Direct Ltd
LC Services Ltd

OTHER ACCESSORIES
LC Services Ltd

DETECTORS

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KNAUER Wissenschaftliche Geräte GmbH
Verulam Scientific Ltd

CHARGED AEROSOL
Karin_Aspen

CHIRAL
KNAUER Wissenschaftliche Geräte GmbH
PDR-Separations

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Shimadzu Scientific Instruments
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Verulam Scientific Ltd
Waters Corporation
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FLAME IONIZATION
PerkinElmer Chromatography Solutions

FLUORESCENCE
Karin_Aspen
KNAUER Wissenschaftliche Geräte GmbH
PerkinElmer Chromatography Solutions
Postnova Analytics
+49 81919856880
Shimadzu Scientific Instruments
(800) 477-1227
Verulam Scientific Ltd

INFRARED
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Polymer Char

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KNAUER Wissenschaftliche Geräte GmbH
Postnova Analytics
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PSS GmbH - Perfect Separation Solutions
Tosoh Bioscience GmbH
+49 6155-7043700
Verulam Scientific Ltd
Waters Corporation
Wyatt Technology

MASS SPECTROMETRIC
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McKinley Scientific
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Shimadzu Scientific Instruments
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PH-METERING
Verulam Scientific Ltd

RADIOACTIVITY
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Verulam Scientific Ltd

REFRACTIVE INDEX
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KNAUER Wissenschaftliche Geräte GmbH
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Postnova Analytics
+49 81919856880
Shimadzu Scientific Instruments
(800) 477-1227
Verulam Scientific Ltd
Waters Corporation
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UV–VIS, FIXED-WAVELENGTH OR FILTER
D-Star Instruments, Inc.
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+42 0221511310
LC Services Ltd
Postnova Analytics
+49 81919856880
Shimadzu Scientific Instruments
(800) 477-1227
Verulam Scientific Ltd

UV–VIS, PHOTODIODE-ARRAY

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KNAUER Wissenschaftliche Geräte GmbH
PerkinElmer Chromatography Solutions
Postnova Analytics
+49 81919856880
Shimadzu Scientific Instruments
(800) 477-1227
Verulam Scientific Ltd
Waters Corporation

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+49 81919856880
Verulam Scientific Ltd

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PerkinElmer Chromatography Solutions
Verulam Scientific Ltd
Welch Materials, Inc.

HPLC

Advanced Materials Technology
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Berthold Technologies GmbH & Co.KG
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D-Star Instruments, Inc.
Dr. Maisch HPLC GmbH
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GL Sciences BV
KCA Laboratories
KNAUER Wissenschaftliche Geräte GmbH
Ladybug Scientific LLC
PDR-Separations
PerkinElmer Chromatography Solutions
Sciencix
(952) 895-8292
Shimadzu Scientific Instruments
(800) 477-1227
SunChrom GmbH
Waters Corporation

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COUNTERCURRENT CHROMATOGRAPHY ACCESSORIES

CC Biotech LLC
DWK Life Sciences GmbH

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Postnova Analytics
+49 81919856880
VICI Valco Instruments Co. Inc.
713-688-9345.

FITTINGS FOR LOW-PRESSURE LC

Postnova Analytics
+49 81919856880
VICI Valco Instruments Co. Inc.
713-688-9345

FLASH CHROMATOGRAPHY ACCESSORIES

DWK Life Sciences GmbH

FRITS

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Postnova Analytics
+49 81919856880
VICI Valco Instruments Co. Inc.
713-688-9345

MIXING TEES

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VICI Valco Instruments Co. Inc.
713-688-9345

SOLVENT RESERVOIRS AND ACCESSORIES

DWK Life Sciences GmbH

SYRINGES

Greyhound Chromatography and Allied Chemicals Ltd
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+49 81919856880
VICI Valco Instruments Co. Inc.
713-688-9345

TUBE CONNECTORS

HILICON AB
Postnova Analytics
+49 81919856880
VICI Valco Instruments Co. Inc.
713-688-9345

TUBING

Greyhound Chromatography and Allied Chemicals Ltd
HILICON AB
Postnova Analytics
+49 81919856880

PUMPS AND ACCESSORIES

DEBUBBLERS

PerkinElmer Chromatography Solutions

GRADIENT

ECOM spol. s r.o.
+42 0221511310
PerkinElmer Chromatography Solutions
Verulam Scientific Ltd

ISOCRATIC

ECOM spol. s r.o.
+42 0221511310
Eldex Corporation
PerkinElmer Chromatography Solutions
Postnova Analytics
+49 81919856880
Verulam Scientific Ltd

LOW-FLOW-RATE

ECOM spol. s r.o.
+42 0221511310
Eldex Corporation
GL Sciences BV

METERING

Eldex Corporation

OTHER PUMPS AND ACCESSORIES

GenTech Scientific LLC
VICI Valco Instruments Co. Inc.
713-688-9345

PERISTALTIC

Postnova Analytics
+49 81919856880

PISTON

Eldex Corporation
PerkinElmer Chromatography Solutions
Postnova Analytics
+49 81919856880
Verulam Scientific Ltd

PULSE-DAMPENING DEVICES

Eldex Corporation
PerkinElmer Chromatography Solutions
Verulam Scientific Ltd

PUMP SEALS AND SPARE PARTS

Bal Seal Engineering
Eldex Corporation
HPLC Direct Ltd
PerkinElmer Chromatography Solutions
Postnova Analytics
+49 81919856880
Quantum Analytics
Verulam Scientific Ltd

SYRINGE

PerkinElmer Chromatography Solutions
Postnova Analytics
+49 81919856880

TITANIUM OR NONMETALLIC

PerkinElmer Chromatography Solutions
Verulam Scientific Ltd

SYSTEM COMPONENTS

CDS Analytical
Phone 1(800) 541-6593
E-mail info@cdsanalytical.com
www.cdsanalytical.com

AUTOSAMPLERS

Axcend
GenTech Scientific LLC
KNAUER Wissenschaftliche Geräte GmbH
LC Services Ltd
Postnova Analytics
+49 81919856880
Quantum Analytics
Shimadzu Scientific Instruments
(800) 477-1227
Verulam Scientific Ltd

COLUMN SELECTION AND SWITCHING SYSTEMS

KNAUER Wissenschaftliche Geräte GmbH
PDR-Separations
Verulam Scientific Ltd
VICI Valco Instruments Co. Inc.
713-688-9345

DEGASSING EQUIPMENT

ECOM spol. s r.o.
+42 0221511310
KNAUER Wissenschaftliche Geräte GmbH
Postnova Analytics
+49 81919856880
Verulam Scientific Ltd

FLOW CONTROLS AND FLOW PROGRAMMERS

Hoffer Flow Controls Inc.
VICI Valco Instruments Co. Inc.
713-688-9345

FLOWMETERS, LIQUID

Hoffer Flow Controls Inc.

FRACTION COLLECTORS, AUTOMATED

ECOM spol. s r.o.
+42 0221511310
Gilson, Inc.
KNAUER Wissenschaftliche Geräte GmbH
PDR-Separations
Postnova Analytics
Shimadzu Scientific Instruments
(800) 477-1227
Verulam Scientific Ltd

FRACTION COLLECTORS, DROP COUNTERS FOR

Postnova Analytics
+49 81919856880

FRACTION COLLECTORS, REFRIGERATED

Postnova Analytics
+49 81919856880
Verulam Scientific Ltd

GRADIENT MIXERS

PDR-Separations

GRADIENT PROGRAMMERS

PDR-Separations

INTERFACES, LC-MS

Waters Corporation

LEAK DETECTORS, SOLVENT

Verulam Scientific Ltd

NITROGEN GENERATORS, LC-MS

GenTech Scientific LLC
McKinley Scientific

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Eldex Corporation

PRESSURE REGULATORS

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713-688-9345

RECONDITIONED EQUIPMENT

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Quantum Analytics

SAMPLE INJECTORS, MANUAL

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Postnova Analytics
+49 81919856880
VICI Valco Instruments Co. Inc.
713-688-9345

SAMPLE INJECTORS, MOTORIZED

KNAUER Wissenschaftliche Geräte GmbH
PDR-Separations
Postnova Analytics
+49 81919856880
VICI Valco Instruments Co. Inc.
713-688-9345

TEMPERATURE CONTROLLERS

VICI Valco Instruments Co. Inc.
713-688-9345

VALVES, MANUAL

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+42 0221511310
KNAUER Wissenschaftliche Geräte GmbH
Postnova Analytics
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VICI Valco Instruments Co. Inc.
713-688-9345

VALVES, MOTORIZED

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Postnova Analytics
+49 81919856880
VICI Valco Instruments Co. Inc.
713-688-9345

VALVES, PROPORTIONAL

Postnova Analytics
+49 81919856880

SYSTEMS

AMINO ACID
908 Devices
PerkinElmer Chromatography Solutions
Shimadzu Scientific Instruments (800) 477-1227
Verulam Scientific Ltd

COUNTERCURRENT
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CC Biotech LLC
PDR-Separations

FLASH CHROMATOGRAPHY
Advion Interchim Scientific
ECOM spol. s r.o. +42 0221511310
Verulam Scientific Ltd

GRADIENT
D-Star Instruments, Inc.
ECOM spol. s r.o. +42 0221511310
GIBNIK Analytical Solutions SL
KNAUER Wissenschaftliche Geräte GmbH
PDR-Separations
PerkinElmer Chromatography Solutions
Shimadzu Scientific Instruments (800) 477-1227
Verulam Scientific Ltd
Waters Corporation

HIGH-PRESSURE (CLOSED-COLUMN)
Axcend
ECOM spol. s r.o. +42 0221511310
KNAUER Wissenschaftliche Geräte GmbH
PDR-Separations
PolyLC Inc.
Verulam Scientific Ltd
Welch Materials, Inc.

HIGH-TEMPERATURE
ECOM spol. s r.o. +42 0221511310
LNEYA
Shimadzu Scientific Instruments (800) 477-1227
Tosoh Bioscience GmbH +49 6155-7043700

ION
GIBNIK Analytical Solutions SL
KNAUER Wissenschaftliche Geräte GmbH
Shimadzu Scientific Instruments (800) 477-1227
Verulam Scientific Ltd

ISOCRATIC
D-Star Instruments, Inc.
ECOM spol. s r.o. +42 0221511310
GenTech Scientific LLC
KNAUER Wissenschaftliche Geräte GmbH
PDR-Separations
Shimadzu Scientific Instruments (800) 477-1227
Verulam Scientific Ltd

LC-MS (COMPLETE SYSTEMS)
908 Devices
Advion Interchim Scientific
Axcend
Conquer Scientific LLC
GenTech Scientific LLC
GIBNIK Analytical Solutions SL
Gilson, Inc.
Jaytee Biosciences Ltd
KNAUER Wissenschaftliche Geräte GmbH
McKinley Scientific
PerkinElmer Chromatography Solutions
Polymer Char
Postnova Analytics +49 81919856880
Quantum Analytics
Shimadzu Scientific Instruments (800) 477-1227
Waters Corporation

LOW-PRESSURE (OPEN-COLUMN)
PerkinElmer Chromatography Solutions

MICRO LC
Axcend
GenTech Scientific LLC
Shimadzu Scientific Instruments (800) 477-1227
Verulam Scientific Ltd

MULTIDIMENSIONAL SYSTEMS
GIBNIK ANALYTICAL SOLUTIONS SL
PSS GmbH - Perfect Separation Solutions
Shimadzu Scientific Instruments (800) 477-1227
Waters Corporation

ON-LINE ANALYZERS
Postnova Analytics +49 81919856880
Verulam Scientific Ltd

OTHER COMPLETE SYSTEMS
PolyLC Inc.
Polymer Char
Postnova Analytics +49 81919856880

PARTITION CHROMATOGRAPHY EQUIPMENT
AECS-QuikPrep Ltd
GIBNIK ANALYTICAL SOLUTIONS SL
Gilson, Inc.

PREPARATIVE
AECS-QuikPrep Ltd
D-Star Instruments, Inc.
ECOM spol. s r.o. +42 0221511310
GIBNIK Analytical Solutions SL
KNAUER Wissenschaftliche Geräte GmbH
PDR-Separations
Shimadzu Scientific Instruments (800) 477-1227
Tosoh Bioscience GmbH +49 6155-7043700
Verulam Scientific Ltd
Welch Materials, Inc.

PROCESS
AECS-QuikPrep Ltd
ECOM spol. s r.o. +42 0221511310
Tosoh Bioscience GmbH +49 6155-7043700

SIMULATED MOVING BED
AECS-QuikPrep Ltd
KNAUER Wissenschaftliche Geräte GmbH
Verulam Scientific Ltd

SIZE-EXCLUSION
KNAUER Wissenschaftliche Geräte GmbH
Polymer Char
Postnova Analytics +49 81919856880
PSS GmbH - Perfect Separation Solutions
Shimadzu Scientific Instruments (800) 477-1227
Tosoh Bioscience GmbH +49 6155-7043700
Verulam Scientific Ltd
Waters Corporation
Wyatt Technology

ULTRAHIGH-PRESSURE LC (UHPLC)
Advion Interchim Scientific
Axcend
GenTech Scientific LLC
GIBNIK Analytical Solutions SL
Jaytee Biosciences Ltd
KNAUER Wissenschaftliche Geräte GmbH
PerkinElmer Chromatography Solutions
Shimadzu Scientific Instruments (800) 477-1227
Verulam Scientific Ltd
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GAS CHROMATOGRAPHY

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SUPPORTS, DIATOMACEOUS-EARTH
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SUPPORTS, OTHER
Porvair Sciences

COLUMNS

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VICI Valco Instruments Co. Inc. 713-688-9345

CAPILLARY (OPEN-TUBULAR)
Ladybug Scientific LLC
PerkinElmer Chromatography Solutions
VICI Valco Instruments Co. Inc. 713-688-9345

CAPILLARY, CHIRAL
VICI Valco Instruments Co. Inc. 713-688-9345

CAPILLARY, ENVIRONMENTAL
GL Sciences BV
PerkinElmer Chromatography Solutions
Phenomenex, Inc.
Sorbent Technologies-SorbTech
Teknokroma Analítica S.A.
VICI Valco Instruments Co. Inc. 713-688-9345

CAPILLARY, HIGH-TEMPERATURE
Frontier Laboratories Europe
LECO Corporation 269-985-5768
PerkinElmer Chromatography Solutions
Phenomenex, Inc.
Teknokroma Analítica S.A.
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CUSTOM-MADE
Frontier Laboratories Europe
Gilson
Ladybug Scientific LLC
Phenomenex, Inc.
Teknokroma Analítica S.A.
VICI Valco Instruments Co. Inc. 713-688-9345

OTHER COLUMNS
VICI Valco Instruments Co. Inc. 713-688-9345

PACKED, ANALYTICAL
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PerkinElmer Chromatography Solutions
Porvair Sciences
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PACKED, CAPILLARY (MICROPACKED)
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Teknokroma Analítica S.A.
Welch Materials, Inc.

PACKED, PREPARATIVE
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VICI Valco Instruments Co. Inc. 713-688-9345

FLAME IONIZATION
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Shimadzu Scientific Instruments (800) 477-1227
VICI Valco Instruments Co. Inc. 713-688-9345

FLAME PHOTOMETRIC
PerkinElmer Chromatography
Shimadzu Scientific Instruments
(800) 477-1227

FLUORESCENCE (MERCURY SPECIFIC)
PerkinElmer Chromatography Solutions

HELIUM IONIZATION
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713-688-9345

ION MOBILITY SPECTROMETERS
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(800) 477-1227
Syft Technologies
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LECO Corporation
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PerkinElmer Chromatography Solutions
Syft Technologies

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PerkinElmer Chromatography Solutions

OTHER DETECTORS, DETECTOR ACCESSORIES
DETector Engineering & Technology
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713-688-9345

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Shimadzu Scientific Instruments
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VICI Valco Instruments Co. Inc.
713-688-9345

THERMIONIC (N–P SELECTIVE)
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VICI Valco Instruments Co. Inc.
713-688-9345

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Phenomenex, Inc.
Teknokroma Analítica S.A.
VICI Valco Instruments Co. Inc.
713-688-9345

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VICI Valco Instruments Co. Inc.
713-688-9345

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Teknokroma Analítica S.A.

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713-688-9345

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Teknokroma Analítica S.A.

LEAK DETECTORS
GL Sciences BV

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Hamilton Company
800-648-5950

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Nitrogen & Hydrogen Generators - Compressors
PEAK Scientific

PUMPS
Gilson
LC Services Ltd

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713-688-9345

SEPTA
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Phenomenex, Inc.
Teknokroma Analítica S.A.

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713-688-9345

TUBING
Gilson

VALVES
VICI Valco Instruments Co. Inc.
713-688-9345

ZERO-AIR GENERATORS
Jaytee Biosciences Ltd
LC Services Ltd
PEAK Scientific

SYSTEM COMPONENTS

AUTOSAMPLERS
Frontier Laboratories Europe
Gilson
Greyhound Chromatography and Allied Chemicals Ltd
LC Services Ltd
LECO Corporation
269-985-5768

COLUMN OVENS
Gilson

CRYOGENIC COOLING ACCESSORIES
Frontier Laboratories Europe
Gilson

FLOW CONTROLLERS
Frontier Laboratories Europe
Gilson
Hoffer Flow Controls Inc.
VICI Valco Instruments Co. Inc.
713-688-9345

FLOW PROGRAMMERS
Gilson

FRACTION COLLECTORS AND ACCESSORIES
Gilson
Structurals

GAS-SAMPLING VALVES
Gilson
VICI Valco Instruments Co. Inc.
713-688-9345

HEADSPACE ANALYZERS
CDS Analytical
Phone 1(800) 541-6593
E-mail info@cdsanalytical.com
www.cdsanalytical.com

LC Services Ltd
Markes International Ltd
SepSolve Analytical Ltd

INTERFACES, GC–MS
LECO Corporation
269-985-5768

PURGE-AND-TRAP DEVICES
CDS Analytical
Phone 1(800) 541-6593
E-mail info@cdsanalytical.com
www.cdsanalytical.com

PYROLYZERS
CDS Analytical
Phone 1(800) 541-6593
E-mail info@cdsanalytical.com
www.cdsanalytical.com

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GL Sciences BV
Pyromation, Inc.

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Jaytee Biosciences Ltd

SAMPLE-INJECTION SYSTEMS
908 Devices
Frontier Laboratories Europe
GL Sciences BV
LECO Corporation
269-985-5768
VICI Valco Instruments Co. Inc.
713-688-9345

TEMPERATURE CONTROLLERS/PROGRAMMERS
VICI Valco Instruments Co. Inc.
713-688-9345

THERMAL-DESORPTION DEVICES
CDS Analytical
Phone 1(800) 541-6593
E-mail info@cdsanalytical.com
www.cdsanalytical.com
See ads on pages xxx, xxx

Frontier Laboratories Europe
GL Sciences BV
LECO Corporation
269-985-5768
Markes International GmbH
Markes International Inc.
Markes International Ltd
SepSolve Analytical Ltd

SYSTEMS

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Conquer Scientific LLC
GenTech Scientific LLC
GIBNIK Analytical Solutions SL
Jaytee Biosciences Ltd
PerkinElmer Chromatography Solutions
Shimadzu Scientific Instruments
(800) 477-1227
Syft Technologies
VICI Valco Instruments Co. Inc.
713-688-9345

GAS ANALYZERS
PerkinElmer Chromatography Solutions
Syft Technologies
VICI Valco Instruments Co. Inc.
713-688-9345

GC–FTIR (COMPLETE SYSTEMS)
PerkinElmer Chromatography Solutions

GC–MS (COMPLETE SYSTEMS)
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GenTech Scientific LLC
GIBNIK Analytical Solutions SL
Greyhound Chromatography and Allied Chemicals Ltd
LECO Corporation
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PerkinElmer Chromatography Solutions
Shimadzu Scientific Instruments
(800) 477-1227

MULTIDIMENSIONAL
GIBNIK Analytical Solutions SL
LECO Corporation
269-985-5768
SepSolve Analytical Ltd
Shimadzu Scientific Instruments
(800) 477-1227

ON-LINE ANALYZERS
Syft Technologies

OTHER SYSTEMS
Gilson
VICI Valco Instruments Co. Inc.
713-688-9345

PORTABLE
Kindwell Inc.
PerkinElmer Chromatography Solutions
Syft Technologies

PREPARATIVE
Biotage AB, Sweden
Gilson

PROCESS
Syft Technologies
VICI Valco Instruments Co. Inc.
713-688-9345
Quantum Analytics

SUPERCRITICAL FLUID CHROMATOGRAPHY

Products and Services for Chromatography

COLUMNS

CAPILLARY (OPEN-TUBULAR)
Gilson

CUSTOM-MADE
Dr. Maisch HPLC GmbH
Gilson
HILICON AB

PACKED
Gilson
HILICON AB
Regis Technologies, Inc.
SiliCycle Inc.
YMC Europe
+49 20644270

SPECIALTY (CHIRAL, LIQUID-CRYSTAL, ETC.)
Gilson
Regis Technologies, Inc.
YMC Europe
+49 20644270

DETECTORS, DETECTOR ACCESSORIES

FLAME IONIZATION
GenTech Scientific LLC

FLOW CELLS FOR UV-VIS DETECTORS
ECOM spol. s r.o.
+42 0221511310
Gilson

MASS SPECTROMETER
GenTech Scientific LLC
Gilson
Waters Corporation

THERMIONIC IONIZATION
DETector Engineering & Technology

UV–VIS, FIXED-WAVELENGTH
ECOM spol. s r.o.
+42 0221511310
Gilson

UV–VIS, PHOTODIODE-ARRAY
ECOM spol. s r.o.
GenTech Scientific LLC
Waters Corporation

UV–VIS, VARIABLE-WAVELENGTH
ECOM spol. s r.o.
+42 0221511310
GenTech Scientific LLC
Gilson

SFC ACCESSORIES

FERRULES
VICI Valco Instruments Co. Inc.
713-688-9345

FITTINGS
VICI Valco Instruments Co. Inc.

PUMPS
Gilson

SAMPLE SPLITTERS
FRITSCH GmbH • Milling and Sizing

SAMPLE-INJECTION VALVES
VICI Valco Instruments Co. Inc.
713-688-9345

STATIONARY PHASES
HILICON AB

SYSTEMS

CAPILLARY COLUMN
Gilson
Shimadzu Scientific Instruments
(800) 477-1227

OTHER SYSTEMS
Gilson

PACKED COLUMN
Gilson

PREPARATIVE
ECOM spol. s r.o.
+42 0221511310
Gilson
Shimadzu Scientific Instruments
(800) 477-1227

SFC–MS (COMPLETE SYSTEMS)
Shimadzu Scientific Instruments
(800) 477-1227
Waters Corporation

THIN LAYER CHROMATOGRAPHY

Products and Services for Chromatography

EQUIPMENT, SUPPLIES

APPLICATORS
Miles Scientific

COATING EQUIPMENT
Miles Scientific

COATINGS
Miles Scientific
SiliCycle Inc.

DESICCATORS
DWK Life Sciences GmbH
Miles Scientific

DEVELOPING CHAMBERS
DWK Life Sciences GmbH
Miles Scientific
SiliCycle Inc.

DIPPING CHAMBERS
DWK Life Sciences GmbH
Miles Scientific

DRYING RACKS
Miles Scientific

HPTLC ACCESSORIES
Miles Scientific

HPTLC EQUIPMENT
Miles Scientific

PHOTOGRAPHY DEVICES OR SYSTEMS
Miles Scientific

PLATES
Gilson
Miles Scientific
Porvair Sciences
SiliCycle Inc.
Sorbent Technologies, Inc.

PREPARATIVE EQUIPMENT AND ACCESSORIES
Gilson
Miles Scientific
Porvair Sciences

SCRAPING DEVICES
Miles Scientific

SORBENTS
BRADY Europe, Middle East & Africa
Miles Scientific
SiliCycle Inc.
Sorbent Technologies, Inc.

SPRAYING EQUIPMENT
Miles Scientific

UV LAMPS FOR TLC
Miles Scientific

VIEWING CABINETS
Miles Scientific

VISUALIZATION DEVICES
Miles Scientific

ZONE COLLECTORS
Miles Scientific

ELECTROPHORESIS

Products and Services for Chromatography

CAPILLARY ELECTROPHORESIS

COMPLETE SYSTEMS
908 Devices

GEL ELECTROPHORESIS

ACCESSORY EQUIPMENT
Hamilton Company
800-648-5950

REAGENTS, SOLVENTS, STANDARDS

Products and Services for Chromatography

REAGENTS,

AMINO ACIDS
Verulam Scientific Ltd

AMPHOLYTES
Acrolab

BIOCHEMICAL
Acrolab
Biovenic
Cayman Chemical Company

BIOLOGICAL-GRADE
Acrolab

BUFFERS
Acrolab
PreOmics GmbH

CHIRAL
Acrolab
Regis Technologies, Inc.

DERIVATIZING AGENTS
Acrolab
Regis Technologies, Inc.

Verulam Scientific Ltd

DYES
Acrolab

ENZYMES
Acrolab
Cayman Chemical Company

GENERAL LABORATORY CHEMICALS
Acrolab
Emerald Scientific
Greyhound Chromatography and Allied Chemicals Ltd
HPLC Direct Ltd

ION CHROMATOGRAPHY BUFFERS
Acrolab
Regis Technologies, Inc.

ION-PAIR REAGENTS
HPLC Direct Ltd
Regis Technologies, Inc.

PEPTIDE SYNTHESIS REAGENTS
SiliCycle Inc.

PROTEINS
Acrolab
Cayman Chemical Company

SOLVENTS
Acrolab
Emerald Scientific
Greyhound Chromatography and Allied Chemicals Ltd
Regis Technologies, Inc.

VISUALIZATION REAGENTS
Cayman Chemical Company

WATER, HIGH-PURITY
Acrolab
Emerald Scientific

STANDARDS

CLINICAL
Acrolab
Cayman Chemical Company
Chiron AS
ChromaDex Reference Standards and Services
LGC
Verulam Scientific Ltd

ENVIRONMENTAL
Acrolab
Cayman Chemical Company
Chiron AS
ChromaDex Reference Standards and Services
Greyhound Chromatography and Allied Chemicals Ltd
LGC
Phenomenex, Inc.

ION CHROMATOGRAPHY
Acrolab
ChromaDex Reference Standards and Services
LGC

OTHER STANDARDS
Acrolab
Cayman Chemical Company
Chiron AS
ChromaDex Reference Standards and Services
Emerald Scientific
Jaytee Biosciences Ltd
LGC

PEPTIDE-PROTEIN
Acrolab
ChromaDex Reference Standards and Services
LGC

PESTICIDE AND HERBICIDE
Acrolab
Cayman Chemical Company
Chiron AS
ChromaDex Reference Standards and Services
Greyhound Chromatography and Allied Chemicals Ltd
LGC

PHARMACEUTICAL
Acrolab
Cayman Chemical Company
Chiron AS
ChromaDex Reference Standards and Services
Emerald Scientific
Greyhound Chromatography and Allied Chemicals Ltd
IJEST
LGC
Roots Analysis

POLYMERS
Acrolab
ChromaDex Reference Standards and Services
LGC
Polymer Char
Waters Corporation

PRIMARY
Acrolab
Cayman Chemical Company
ChromaDex Reference Standards and Services
LGC

RADIOCHEMICAL
ChromaDex Reference Standards and Services
LGC

SPECTROCHEMICAL
ChromaDex Reference Standards and Services
LGC

DATA HANDLING, CALIBRATION

Products and Services for Chromatography

HARDWARE

ANALYTICAL SYSTEMS AND CONTROLLERS FOR GC
Servicios Integral en Cromatografía - SISCHROM

ANALYTICAL SYSTEMS AND CONTROLLERS FOR LC
ECOM spol. s r.o.
+42 0221511310
Verulam Scientific Ltd
Waters Corporation

AUTOMATED WORKSTATIONS, IBM- COMPATIBLE
Biotage AB, Sweden

CALIBRATION SYSTEMS, LC
Waters Corporation

DATA ACQUISITION AND DATA HANDLING
ACD/Labs (Advanced Chemistry Development)
Solutions for LC/GC-MS data and applications
Ultraview Corporation
Verulam Scientific Ltd
Waters Corporation

DATA CONVERTORS, ANALOG-TO-DIGITAL
Verulam Scientific Ltd
Ultraview Corporation

INSTRUMENT–COMPUTER INTERFACES
Verulam Scientific Ltd

PROCESS CONTROL
Hoffer Flow Controls Inc.

SOFTWARE

BARCODE SYSTEMS/ SOFTWARE
Autoscribe Informatics LIMS
BRADY Europe, Middle East & Africa

CALIBRATION
Infometrix, Inc.
Jaytee Biosciences Ltd
Solutions for LC/GC-MS data and applications
Waters Corporation

CE PREDICTION AND MODELING
S-Matrix Corporation

CHEMOMETRICS
Infometrix, Inc.
Markes International GmbH
Markes International Inc.
Solutions for LC/GC-MS data and applications
Wiley Science Solutions

CUSTOM, FOR CHROMATOGRAPHY
Infometrix, Inc.
LECO Corporation
269-985-5768
Solutions for LC/GC-MS data and applications
Waters Corporation

DATA ACQUISITION
Autoscribe Informatics LIMS
BRADY Europe, Middle East & Africa
SepSolve Analytical Ltd
Solutions for LC/GC-MS data and applications
Waters Corporation

DATA ANALYSIS AND REPORTING
ACD/Labs (Advanced Chemistry Development)
Autoscribe Informatics LIMS
CD ComputaBio
Infometrix, Inc.
Markes International GmbH
Markes International Inc.
Northwest Analytics Inc
S-Matrix Corporation
SepSolve Analytical Ltd
Shimadzu Scientific Instruments
(800) 477-1227
Solutions for LC/GC-MS data and applications
Waters Corporation
Wiley Science Solutions

DATA CONVERTORS, ANALOG-TO-DIGITAL
Ultraview Corporation

EXPERT SYSTEMS
Infometrix, Inc.
S-Matrix Corporation
SepSolve Analytical Ltd
Solutions for LC/GC-MS data and applications

GC METHOD DEVELOPMENT
ACD/Labs (Advanced Chemistry Development)
Jaytee Biosciences Ltd
LECO Corporation
269-985-5768
S-Matrix Corporation
Solutions for LC/GC-MS data and applications

GRAPHICS OR MANUSCRIPT PREPARATION
ACD/Labs (Advanced Chemistry Development)
Solutions for LC/GC-MS data and applications

INSTRUMENT CONTROL
ACD/Labs (Advanced Chemistry Development)
Hoffer Flow Controls Inc.
KNAUER Wissenschaftliche Geräte GmbH
SepSolve Analytical Ltd
Waters Corporation

LABORATORY INFORMATION MANAGEMENT SYSTEMS
ACD/Labs (Advanced Chemistry Development)
Autoscribe Informatics LIMS
BRADY Europe, Middle East & Africa
Waters Corporation
Wiley Science Solutions

LC METHOD DEVELOPMENT
ACD/Labs (Advanced Chemistry Development)
Jaytee Biosciences Ltd
S-Matrix Corporation
Solutions for LC/GC-MS data and applications
Waters Corporation

PRINTERS
BRADY Europe, Middle East & Africa

ROBOTICS SYSTEM CONTROL
Autoscribe Informatics LIMS

SFC METHOD DEVELOPMENT
S-Matrix Corporation

SIZE EXCLUSION
PSS GmbH - Perfect Separation Solutions
S-Matrix Corporation
Waters Corporation

STATISTICAL ANALYSIS
Infometrix, Inc.
LECO Corporation
269-985-5768
Markes International GmbH
Northwest Analytics Inc
S-Matrix Corporation
SepSolve Analytical Ltd
Solutions for LC/GC-MS data and applications

CHROMATOGRAPHY SERVICES

Products and Services for Chromatography

CHROMATOGRAPHY SERVICES

ANALYSES, ENVIRONMENTAL
GERSTEL
Cayman Chemical Company
Syft Technologies
Utility Testing Laboratory

ANALYSES, FORENSIC
Cayman Chemical Company
GERSTEL
Solutions for LC/GC-MS data and applications

ANALYSES, GC, GC–FTIR
Cayman Chemical Company

ANALYSES, GC, GC–MS
Cayman Chemical Company
GenTech Scientific LLC
GERSTEL
LC Services Ltd
LECO Corporation
269-985-5768
Solutions for LC/GC-MS data and applications
Syft Technologies
Utility Testing Laboratory

ANALYSES, LC, LC–MS
Advanced Materials Technology
Axcend
Cayman Chemical Company
Fortis Technologies Ltd
GenTech Scientific LLC
GERSTEL
Gilson
LC Services Ltd
McKinley Scientific
Postnova Analytics
SiliCycle Inc.
Solutions for LC/GC-MS data and applications
Waters Corporation

ANALYSES, SEC
PSS GmbH - Perfect Separation Solutions
Waters Corporation

ANALYSES, SFC, SFC–MS
Waters Corporation

ANALYSES, SPE
Creative Biogene
Gilson
SiliCycle Inc.

CHROMATOGRAPHY DATA SYSTEM VALIDATION
Waters Corporation

COLUMN REPACKING, LC
Dr. Maisch HPLC GmbH
Fortis Technologies Ltd
Regis Technologies, Inc.
SiliCycle Inc.

CONSULTING, ENVIRONMENTAL
Infometrix, Inc.

CONSULTING, GC
Anthias Consulting Ltd
Infometrix, Inc.

CONSULTING, GENERAL
Anthias Consulting Ltd
Quantitative in silico analytical chemistry

CONSULTING, LC
Anthias Consulting Ltd
Fortis Technologies Ltd
Infometrix, Inc.
KNAUER Wissenschaftliche Geräte GmbH
PDR-Separations
SiliCycle Inc.

CONSULTING, SPE
Anthias Consulting Ltd
SiliCycle Inc.

CONTRACT DEVELOPMENT
S-Matrix Corporation
SiliCycle Inc.

EQUIPMENT QUALIFICATION OR VALIDATION
Jaytee Biosciences Ltd

LEASING OR RENTING, GC EQUIPMENT
Quantum Analytics
Syft Technologies
Gilson
McKinley Scientific
Quantum Analytics

METHOD DEVELOPMENT, CE
Advanced Materials Technology

METHOD DEVELOPMENT, GC, GC–MS
Anthias Consulting Ltd
Cayman Chemical Company
Jaytee Biosciences Ltd
LECO Corporation
269-985-5768
Regis Technologies, Inc.
Solutions for LC/GC-MS data and applications

METHOD DEVELOPMENT, LC, LC-MS
Anthias Consulting Ltd
Cayman Chemical Company
Fortis Technologies Ltd
Jaytee Biosciences Ltd
KNAUER Wissenschaftliche Geräte GmbH
PDR-Separations
Regis Technologies, Inc.
S-Matrix Corporation
SiliCycle Inc.
Solutions for LC/GC-MS data and applications
Waters Corporation
YMC Europe
+49 20644270

METHOD DEVELOPMENT, SEC
PSS GmbH - Perfect Separation Solutions
Waters Corporation

METHOD DEVELOPMENT, SFC, SFC–MS
Regis Technologies, Inc.

METHOD DEVELOPMENT, SPE
Anthias Consulting Ltd

OTHER GC-RELATED SERVICES
Infometrix, Inc.
KCA Laboratories
LECO Corporation
269-985-5768
Solutions for LC/GC-MS data and applications

OTHER LC-RELATED SERVICES
ChromaDex Reference Standards and Services
KCA Laboratories
Solutions for LC/GC-MS data and applications

PREPARATIVE GC
Solutions for LC/GC-MS data and applications

PREPARATIVE LC
Advion Interchim Scientific
Dr. Maisch HPLC GmbH
Eldex Corporation
Gilson
PDR-Separations
SiliCycle Inc.
Solutions for LC/GC-MS data and applications

PREPARATIVE SFC
Eldex Corporation

PROCESS LC
Eldex Corporation
SiliCycle Inc.
Solutions for LC/GC-MS data and applications

REPAIR AND MAINTENANCE, GC EQUIPMENT
Jaytee Biosciences Ltd
LC Services Ltd
Eldex Corporation
Jaytee Biosciences Ltd
LC Services Ltd

REPAIR AND MAINTENANCE, LC EQUIPMENT
LC Services Ltd
McKinley Scientific
Verulam Scientific Ltd

STABILITY TESTING
Regis Technologies, Inc.
Solutions for LC/GC-MS data and applications

THIN-LAYER CHROMATOGRAPHY
Advion Interchim Scientific
Gilson
SiliCycle Inc.

TRAINING PROGRAMS FOR DATA SYSTEMS
Waters Corporation

TRAINING PROGRAMS FOR GC
Anthias Consulting Ltd
Jaytee Biosciences Ltd
LCGC
Quantum Analytics

TRAINING PROGRAMS FOR LC
Anthias Consulting Ltd
Fortis Technologies Ltd
Jaytee Biosciences Ltd
KNAUER Wissenschaftliche Geräte GmbH
LCGC
YMC Europe
+49 20644270

TRAINING PROGRAMS FOR SEC
Anthias Consulting Ltd
PSS GmbH - Perfect Separation Solutions
Waters Corporation

TRAINING PROGRAMS FOR SPE
Anthias Consulting Ltd

WATER ANALYSIS
GERSTEL

SAMPLE PREPARATION AND HANDLING

Products and Services for Chromatography

FILTERS, MEMBRANE

CELLULOSE
Cytiva
Teknokroma Analítica S.A.

ION-EXCHANGE
Phenomenex, Inc.

NYLON
Cytiva
HPLC Direct Ltd
Phenomenex, Inc.
SILEX Chromatography
Teknokroma Analítica S.A.
Welch Materials, Inc.

OTHER FILTERS, MEMBRANE
Cytiva
Porvair Sciences
PreOmics GmbH
Teknokroma Analítica S.A.
Welch Materials, Inc.

POLYPROPYLENE
Cytiva
Porvair Sciences
Teknokroma Analítica S.A.
Welch Materials, Inc.

POLYSULFONE
Cytiva
Teknokroma Analítica S.A.

PTFE
Cytiva
HPLC Direct Ltd
Phenomenex, Inc.
Porvair Sciences
Teknokroma Analítica S.A.
Welch Materials, Inc.

PVDF
Cytiva
HPLC Direct Ltd
Phenomenex, Inc.
Teknokroma Analítica S.A.
Welch Materials, Inc.

FILTERS, OTHER

BACTERIOLOGICAL
Cytiva

CAPSULE
Cytiva

CENTRIFUGAL
Porvair Sciences
PreOmics GmbH

FILTER HOLDERS
Cytiva

GAS-PURIFICATION
Cytiva

GLASS-FIBER
Cytiva
Welch Materials, Inc.

LAB ENVIRONMENT
Cytiva

OTHER FILTERS
Cytiva

PAPER
Cytiva

PUMP-INLET
Cytiva
Verulam Scientific Ltd

STAINLESS STEEL IN-LINE
VICI Valco Instruments Co. Inc.

SYRINGE
Cytiva
ePrep Automated Sample Preparation
Sorbent Technologies, Inc.
Welch Materials, Inc.

PRESSURIZED FLUID AND EQUIPMENT

Markes International Ltd
Phone +44 (0)1443 230935
E-mail enquiries@markes.com
www.markes.com

MULTIPLE SAMPLE SYSTEMS
ePrep Automated Sample Preparation
GERSTEL

SINGLE SAMPLE SYSTEMS
Verulam Scientific Ltd

SYSTEMS, AUTOMATED
ePrep Automated Sample Preparation
GERSTEL
Verulam Scientific Ltd

SAMPLE HANDLING

2-IN-1 KITS
LC Services Ltd

96-POSITION BLOCK SYSTEMS
Gilson

AUTOMATED SAMPLE-HANDLING EQUIPMENT
Biotage AB, Sweden
ePrep Automated Sample Preparation
ePrep chromatography sample preparation
Gilson, Inc.
Hamilton Company
800-648-5950
KNAUER Wissenschaftliche Geräte GmbH
LECO Corporation
269-985-5768

Markes International Ltd
Phone +44 (0)1443 230935
E-mail enquiries@markes.com
www.markes.com

Polymer Char
Porvair Sciences
PreOmics GmbH
Quantum Analytics

HEADSPACE VIALS & CLOSURES
DWK Life Sciences GmbH
Greyhound Chromatography and Allied Chemicals Ltd
HPLC Direct Ltd

Markes International Ltd
Phone +44 (0)1443 230935
E-mail enquiries@markes.com
www.markes.com

Ningbo Excellent New Materials Co Ltd
Porvair Sciences
Quantum Analytics
Teknokroma Analítica S.A.
Welch Materials, Inc.

MICRO-INSERTS
DWK Life Sciences GmbH

Markes International Ltd
Phone +44 (0)1443 230935
E-mail enquiries@markes.com
www.markes.com

Teknokroma Analítica S.A.
Welch Materials, Inc.

OTHER SAMPLE-HANDLING ACCESSORIES
Biotage AB, Sweden
Gilson
Hamilton Company
800-648-5950
Markes International Ltd
VICI Valco Instruments Co. Inc.

SAMPLE-STORAGE EQUIPMENT

Markes International Ltd
Phone +44 (0)1443 230935
E-mail enquiries@markes.com
www.markes.com

SEALS
Bal Seal Engineering
DWK Life Sciences GmbH
Porvair Sciences

ULTRABOND CLOSURES

Markes International Ltd
Phone +44 (0)1443 230935
E-mail enquiries@markes.com
www.markes.com

VIAL RACKS
DWK Life Sciences GmbH
ECOM spol. s r.o.
+42 0221511310
Gilson
Porvair Sciences
Welch Materials, Inc.

VIALS AND ACCESSORIES
DWK Life Sciences GmbH
Gilson
HPLC Direct Ltd
Markes International Ltd
Ningbo Excellent New Materials Co Ltd
Teknokroma Analítica S.A.
Welch Materials, Inc.

SAMPLE PREPARATION

AUTOMATED GAS-SAMPLE COLLECTORS
Acrolab
Markes International GmbH
Markes International Inc.

AUTOMATED SOLID-PHASE EXTRACTION EQUIPMENT
Acrolab
Biotage AB, Sweden
ePrep Automated Sample Preparation
ePrep chromatography sample preparation
GIBNIK Analytical Solutions SL
Gilson, Inc.
Markes International GmbH
SILEX Chromatography
Verulam Scientific Ltd

CONCENTRATORS OR EVAPORATORS
Acrolab
Biotage AB, Sweden
Markes International Ltd
Porvair Sciences
SILEX Chromatography
Verulam Scientific Ltd

CRIMPERS & DECAPPERS
DWK Life Sciences GmbH
HPLC Direct Ltd
Teknokroma Analítica S.A.

EXTRACTORS
Acrolab
Gilson

MAGNETIC BEAD SEPARATION
Acrolab
Cytiva
Gilson
Phenomenex, Inc.
PreOmics GmbH

MANIFOLDS FOR SOLID-PHASE EXTRACTION
Acrolab
Biotage AB, Sweden
ePrep Automated Sample Preparation
Porvair Sciences
SiliCycle Inc.
Welch Materials, Inc.

MICROWAVE SYSTEMS
Biotage AB, Sweden

ROBOTIC EQUIPMENT
ePrep Automated Sample Preparation
ePrep chromatography sample preparation
GIBNIK Analytical Solutions SL
Porvair Sciences
PreOmics GmbH
SepSolve Analytical Ltd

SAMPLE-COLLECTION DEVICES FOR AIR MONITORING
GL Sciences BV
Markes International GmbH
Markes International Inc.
Markes International Ltd
Quantum Analytics

SOLID-PHASE EXTRACTION COLUMNS, DISKS, AND PACKINGS
Biotage AB, Sweden
ePrep Automated Sample Preparation
Gilson
GL Sciences BV
HILICON AB
HPLC Direct Ltd
Phenomenex, Inc.
Porvair Sciences
SiliCycle Inc.
Sorbent Technologies-SorbTech
Teknokroma Analítica S.A.
Welch Materials, Inc.

SOLID-PHASE MICROEXTRACTION EQUIPMENT
ePrep Automated Sample Preparation
ePrep chromatography sample preparation
Gilson
Markes International GmbH
Markes International Inc.
Markes International Ltd
Phenomenex, Inc.
SepSolve Analytical Ltd

SORBENTS FOR SAMPLE PREPARATION
FRITSCH GmbH • Milling and Sizing
HILICON AB
Markes International Inc.
Markes International Ltd
Phenomenex, Inc.
Porvair Sciences
SILEX Chromatography
SiliCycle Inc.
Sorbent Technologies-SorbTech

GENERAL SCIENTIFIC EQUIPMENT AND ACCESSORIES

Products and Services for Chromatography

ANALYZERS, SENSORS, AND TESTING INSTRUMENTS

BIOSENSORS
908 Devices

CONDUCTIVITY METERS
Karin_Aspen

ELECTRONIC NOSE INSTRUMENTS
LECO Corporation
269-985-5768
Verder Scientific Inc

ELEMENTAL ANALYZERS
LECO Corporation
Verder Scientific Inc

FLOW-INJECTION ANALYSIS EQUIPMENT
VICI Valco Instruments Co. Inc.
713-688-9345

ION-SELECTIVE ELECTRODES
VICI Valco Instruments Co. Inc.
713-688-9345

MICROPLATE READERS
Berthold Technologies GmbH & Co.KG
Emerald Scientific

MOISTURE ANALYZERS
Emerald Scientific
FRITSCH GmbH • Milling and Sizing
LECO Corporation
269-985-5768
Verder Scientific Inc

PARTICLE SIZE ANALYZERS
Emerald Scientific
FRITSCH GmbH • Milling and Sizing
LECO Corporation
269-985-5768
Verder Scientific Inc

PETROLEUM TESTING
Emerald Scientific
FRITSCH GmbH • Milling and Sizing
LECO Corporation
269-985-5768
Verder Scientific Inc

PH METERS
Karin_Aspen

PYROLYSIS EQUIPMENT
Eldex Corporation
LECO Corporation
269-985-5768
Verder Scientific Inc

REFRACTOMETERS
Wyatt Technology

RTDS/ACCESSORIES
Pyromation, Inc.

SENSORS
Pyromation, Inc.

THERMOCOUPLERS/ ACCESSORIES
Karin_Aspen
Pyromation, Inc.

THERMOMETERS
Pyromation, Inc.

UV-VIS, SPECTROPHOTOMETERS
Berthold Technologies GmbH & Co.KG
International Crystal Laboratories
LC Services Ltd

VISCOMETERS
Polymer Char
Verder Scientific Inc
Wyatt Technology

LABORATORY EQUIPMENT

BALANCES
Acrolab
Emerald Scientific
Greyhound Chromatography and Allied Chemicals Ltd
SILEX Chromatography

BATHS
Acrolab
Gilson, Inc.
LNEYA

CENTRIFUGES
Acrolab
Chem Science INC
Emerald Scientific
Gilson, Inc.
LNEYA

CRUCIBLES
Chem Science INC

DATA RECORDERS
Ultraview Corporation

DISSOLUTION TESTERS
Acrolab

DISTILLATION SYSTEMS
Acrolab
Eldex Corporation
LNEYA

FITTINGS/FLANGES
Hamilton Company
800-648-5950
VICI Valco Instruments Co. Inc.
713-688-9345

FLUID HANDLING EQUIPMENT
Eldex Corporation
FRITSCH GmbH • Milling and Sizing
Hamilton Company
800-648-5950
Hoffer Flow Controls Inc.
LNEYA

FREEZE DRYERS
Acrolab
Emerald Scientific

GLASSWARE
Acrolab
Chem Science INC
DWK Life Sciences GmbH

HEATERS/COOLERS
LNEYA

HOMOGENIZERS
Acrolab
PreOmics GmbH

IMAGING EQUIPMENT/ ACCESSORIES
BRADY Europe, Middle East & Africa
Verder Scientific Inc

MICROSCOPES/ACCESSORIES
Verder Scientific Inc

OVENS
Verder Scientific Inc

PIPETTES
Acrolab
BRAND GMBH + CO KG
DWK Life Sciences GmbH
Gilson, Inc.
Greyhound Chromatography and Allied Chemicals Ltd
Hamilton Company
800-648-5950

PLASTICWARE
Acrolab
Chem Science INC
DWK Life Sciences GmbH

PUMPS
Eldex Corporation
Gilson, Inc.
Verder Scientific Inc

REFRIGERATORS/FREEZERS
Acrolab
LNEYA
Marvel Refrigeration
SILEX Chromatography

SHAKERS/STIRRERS
Acrolab

SHAKERS/STIRRERS
Verder Scientific Inc

SYRINGES
Gilson, Inc.
Greyhound Chromatography and Allied Chemicals Ltd
International Crystal Laboratories
VICI Valco Instruments Co. Inc.
713-688-9345

TEMPERATURE/PRESSURE CONTROLLERS
BRADY Europe, Middle East & Africa
LNEYA
VICI Valco Instruments Co. Inc.
713-688-9345

TUBING
VICI Valco Instruments Co. Inc.
713-688-9345

ULTRASONIC EQUIPMENT
Acrolab

VACUUM EQUIPMENT
Acrolab
Biotage AB, Sweden

VALVES
GL Sciences BV
VICI Valco Instruments Co. Inc.
713-688-9345

WATER PURIFICATION SYSTEMS
Acrolab
ELGA Labwater
SILEX Chromatography

LABORATORY FURNISHINGS

CELL CULTURE EQUIPMENT
Gilson, Inc.
Emerald Scientific

ENCLOSURES
Verder Scientific Inc

FUME HOODS
Emerald Scientific

FURNITURE
Emerald Scientific
IAC Industries
Verder Scientific Inc

GLASSWARE WASHERS
Emerald Scientific

LIGHTING
IAC Industries

PROTECTIVE CLOTHING/ EYEWEAR
Emerald Scientific

SAFETY EQUIPMENT/ SIGNAGE
BRADY Europe, Middle East & Africa

SHELVING/STORAGE
Emerald Scientific
IAC Industries

WORKBENCHES
Emerald Scientific
IAC Industries

MANUFACTURERS DIRECTORY

908 DEVICES
645 Summer Street,
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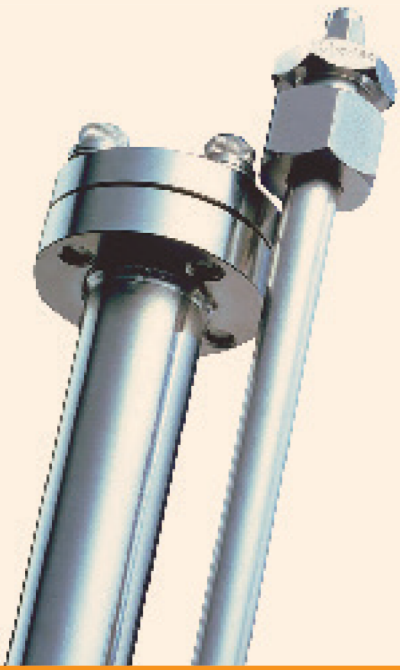
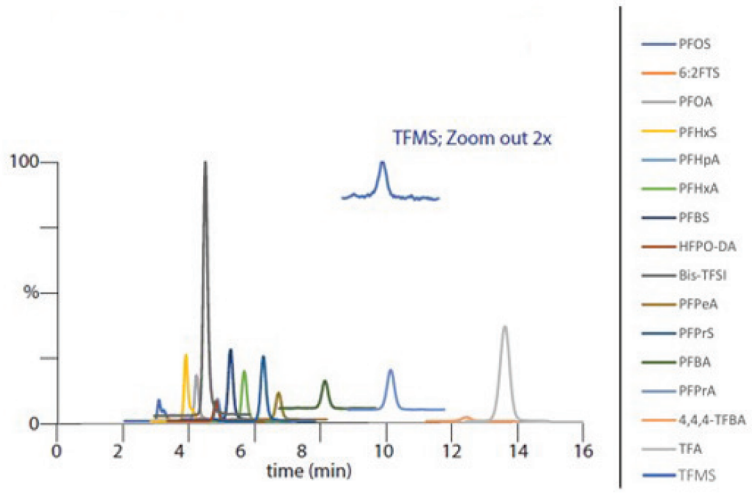


Analyisis of Ultra-Short PFAS



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