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A Turbulent Affair

The role of TFC in environmental analysis

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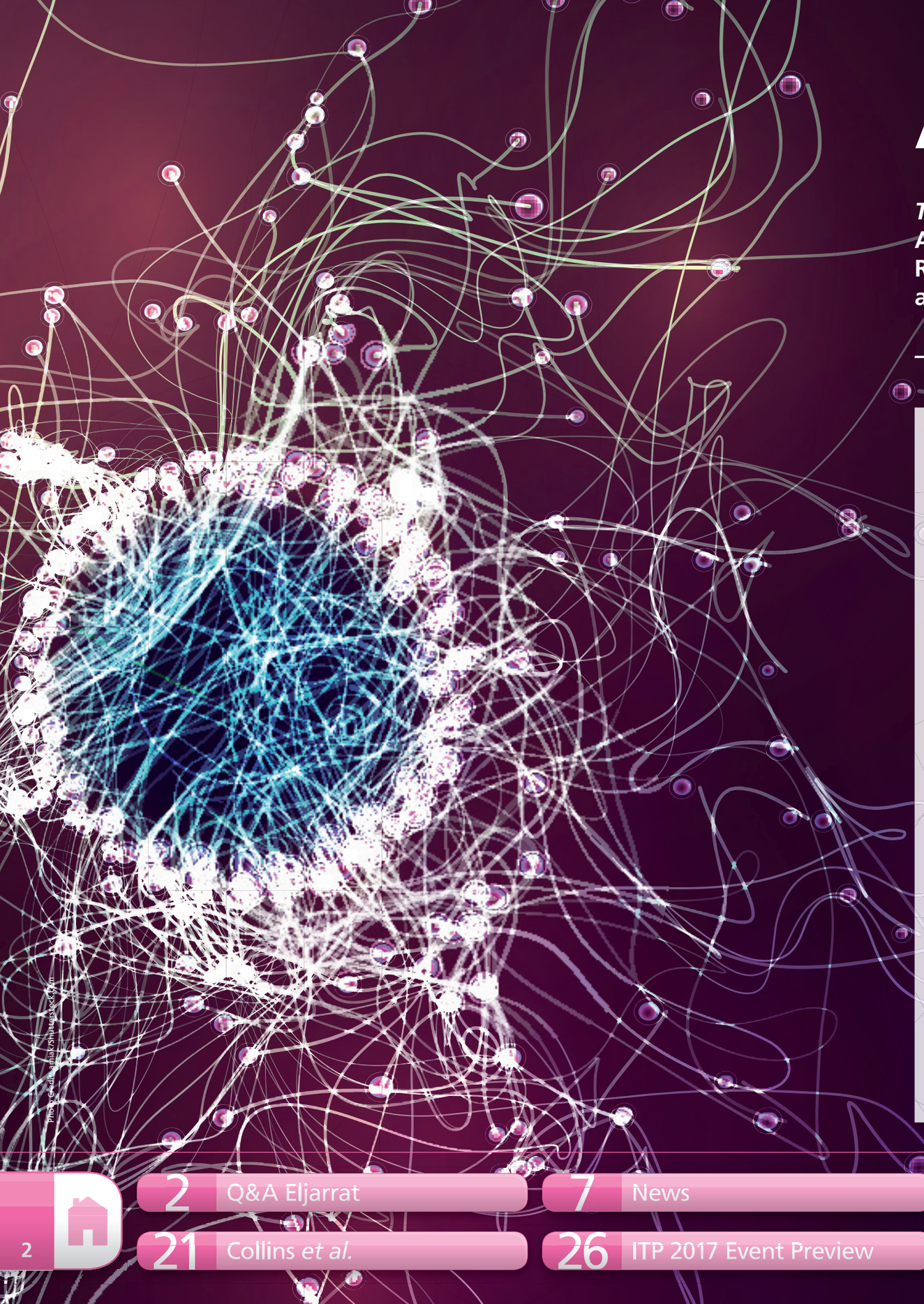

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A Turbulent Affair

The Column spoke to Ethel Eljarrat of the Institute of Environmental Assessment and Water Research (IDÆA), Spanish Council of Scientific Research (CSIC), in Barcelona, Spain, about her work in environmental analyses using turbulent flow chromatography (TFC).

—Interview by Kate Mosford

Q. What is turbulent flow chromatography (TFC)?

A: Turbulent flow chromatography (TFC) was developed in the late 1990s, and combines "size exclusion" and traditional stationary phase column chemistry to separate macromolecules, such as proteins, from smaller molecules and analytes of interest. TFC is used for on-line sample cleanup of biological matrices in liquid chromatography–mass spectrometry (LC–MS) applications. The determination of contaminants in biological fluids or tissues has been a challenging task for a very long time. This was due to the complexity of the biological matrices, which required timeconsuming sample preparation steps. The recent implementation of on-line extraction (including TFC) has allowed fast sample cleanup and partly removed the bottleneck associated with sample preparation. TFC methods are based on the direct injection of biological extracts without previous cleanup onto a column packed with large particles.

After the sample is injected onto a column that utilizes TFC, the high flow rate (1.5–5.0 mL/min) generates turbulent flow conditions inside the column. The small analyte molecules are retained via diffusion into the particle pores, while the proteinaceous material is washed to waste. Once the compounds of interest are extracted from the biological matrix, they are eluted from this column onto the analytical column.

Q. In 2016, your group published a study analyzing organophosphorus flame retardants (OPFRs) in environmental matrices using TFC (1). What led your group to begin this study?

A: Our group has been studying the environmental impact of halogenated flame retardants (HFRs) for more than 15 years. However, in recent years, the scientific community has focused their attention on evaluating the environmental impact produced by another family of flame retardants (FRs), the organophosphorus FRs (OPFRs). Previous studies showed high levels of contamination in environmental and biotic samples, often with even higher levels than those found for HFRs—some of which have already been banned at international scale (1,2). Moreover, some of these compounds are neurotoxic, endocrine disruptors, and carcinogenic (3,4).

Taking this into account, appropriate analytical methods for OPFR determination and quantification are needed. Unlike halogenated FRs, which are analyzed by gas chromatography (GC), OPFRs are determined by liquid chromatography (LC). Our group previously investigated the simultaneous determination of 16 OPFRs in fish by LC–MS/MS (5). However, off-line extraction and cleanup steps must be performed, once more making the sample preparation step the bottleneck of the analytical process.

TFC has been previously optimized and applied for the determination of other contaminants, such as pesticides, perfluoroalkyls, or veterinary drugs (6,7). Therefore, our group decided to develop an analytical method based on TFC to determine OPFRs in environmental and biological matrices, reducing the sample preparation steps and minimizing the time of analysis.

Q. What were the main analytical challenges you encountered and how did you overcome them?

A: One of the main challenges encountered in this study was the physico-chemical differences between selected analytes, with molecular weight ranging from 266 to 452, and log K_{OW} ranging between 1.71 and 9.49. With the use of turbulent flow, large molecules (for example, proteins and lipids) pass through the purification column, while the smaller molecules are retained on the porous cavities of the column packing. Thus, clean extracts without interfering compounds are achieved. In our tests, two OPFRs, *tris*(2-ethylhexyl)phosphate (TEHP) and trihexyl phosphate (THP), which are the larger molecules, presented the lowest recoveries. Thus, a second purification column was added to retain these compounds and avoid their loss with the turbulent flow. With the addition of a C18 column, it was possible to obtain good recoveries for all the selected analytes.

A further critical point is the transfer time when purification and analytical columns were connected. This time had to be reduced to the maximum, 120 s, to obtain the optimum conditions where the target compounds had been transferred onto the analytical column and sample matrix remained in the purification column that utilized TFC.

Another important aspect that should be noted is the complexity of the analyzed samples. In the case of environmental samples (for example, sediments), this is not so critical, and no problems were found when selecting the extraction method. We chose to use pressurized liquid extraction (PLE), which is a fast and automatic extraction method. However, for biological samples with many ingredients and a high fat content, the selection of appropriate sample extraction is a crucial step for the correct development of the method. In this case, the best option was an ultrasound extraction because it is a mild extraction method, allowing a lower number of interfering compounds in the obtained extracts.

Q. Are OPFRs becoming a bigger issue within the environment?

A: As mentioned before, OPFRs constitute another family of chemical compounds widely used and applied as flame retardants. They are also used as plasticizers, and the plastic industry is one of the largest industries worldwide. Unfortunately, even though plastics have made our lives easier, the contamination produced by them is undeniable. This is produced not only for the material itself, but also by the chemicals used in its manufacturing. To give stability to these polymers, some chemicals known as *plasticizers* are added into the mixture. Both applications, as flame retardants and as plasticizers, make these substances into chemical compounds produced on a large industrial scale.

A recent published review (8) has highlighted the concentration levels usually found in different environmental compartments, such as air, water, sediment, and soil. There are several articles where the presence of OPFRs is confirmed in fish from different world locations (1,2,9–11). These works showed high concentrations of OPFRs in comparison to the average levels found for PBDEs, the most widely used HFRs, and included on the list of Stockholm Convention, which regulates the elimination of persistent organic pollutants (POPs).

Q. What can TFC coupled to LC–MS/MS offer over other techniques?

A: TFC coupled to LC–MS/MS offers a simple and easy method of analysis of organic pollutants in complex matrices. By performing only one extraction step, we are able to quickly and easily determine the contaminants of interest in biological matrices. The obtained sensitivity and selectivity are higher than those obtained by classical off-line methodologies.

Q. Can this technology or method be used in any other applications?

A: Based on the good results obtained in our study, we want to optimize analytical methods for OPFR determinations in other complex matrices, such as human fluids (plasma, urine). We also plan to focus on the use of TFC for the determination of other emerging contaminants of interest, such as pharmaceuticals and personal care products.

Q. What results did you find?

A: A fast on-line analytical method based on TFC in combination with MS/MS has been developed obtaining acceptable recoveries (between 47% and 112% for sediment, and between 47% and 98% for fish) with very low relative standard deviations (always below 8.8% for sediment, and below 16% for fish). Moreover, limits of detection (LODs) and limits of quantification (LOQs) are similar or even lower than those reported in other works using off-line methodologies (1). LODs ranged between 0.02 and 1.25 ng/g dry weight (dw) and between 0.19 ng/g and 19.3 ng/g lipid weight (lw) for sediments and fish samples, respectively. The applicability of the developed methodology was demonstrated by the analysis of real samples, corresponding to river sediments as well as river and marine fish samples. OPFRs were detected in all samples, with values up to 549 ng/g dw, 15.8 ng/g lw, and 646 ng/g lw for sediment, marine fish, and river fish, respectively. As expected, lower contamination was found in the marine environment because of the water dilution.

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Ethel Eljarrat Esebag obtained her Ph.D. in chemistry at the University of Barcelona in 1999. Since July 2008 she has been a scientific researcher at the Institute of Environmental Assessment and Water Research (IDÆA), Spanish Council of Scientific Research (CSIC), in Barcelona, Spain. She has published more than 140 research papers (H index = 35), and she is editor of two books (*Brominated Flame Retardants (BFRs)* and *Emerging Organic Contaminants in Sludges*). Her research area includes the development of analytical methodologies for the study of the presence and environmental behaviour of organic persistent pollutants, as well as of emerging contaminants, such as halogenated and organophosphate flame retardants, and pyrethroid insecticides, in different environmental compartments and biota samples. She has worked across different studies related to pollution in aquatic and terrestrial ecosystems and in some of these studies she combined and related the study of the environmental levels with the effects on the aquatic organisms.

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News

Shigeru Terabe Receives Arnold O. Beckman Medal

Shigeru Terabe has received the Arnold O. Beckman Medal and Award for Outstanding Scientific Achievements in the Field of Electro-Drive Separation Techniques. The Sciex sponsored award was presented to Terabe as part of a Special Award Plenary Session at the 33rd International Symposium on Microscale Bioseparations (MSB 2017).

A driving force in understanding the fundamentals of capillary electrophoresis, Terabe was also involved in the introduction of micellar electrokinetic chromatography and its myriad of applications.

"This annual medal and award sponsored by Sciex recognizes the achievements of those that have made a momentous impact on capillary electrophoresis," said Jeff Chapman, Sr. Director of Sciex. "Professor Terabe's invention of micellar electrokinetic chromatography has inspired many scientists."

"I am humbled to be the recipient of the prestigious Arnold O. Beckman award, a notable honour that has recognized distinguished colleagues that have impacted diverse scientific and technical disciplines," said Shigeru Terabe, Professor Emeritus of the University of Hyogo in Kamigori, Hyogo, Japan. "Dr. Beckman's achievements have inspired me and I would like to express my gratitude to MSB for this esteemed award."

Phenomenex Announces New Californian GC Column Facility

Phenomenex Inc. (Torrance, California, USA) has announced the opening of a new manufacturing and development facility dedicated to the company's gas chromatography columns. The 15,000-square-foot facility located in Sacramento, California, USA, supports twice the production capacity, reportedly improving Phenomenex's logistics and delivery speeds.

"Detailed and exhaustive planning went into creating an efficient production floor, using lean principles to maximize the use of space while minimizing the movement of people and materials," said Emmet Welch, senior product development manager for Phenomenex.

"We have also included a centralized piping system that reduces the cost and movement of process gasses. With advanced, automated workflows, this new facility will be capable of supporting significant growth in Phenomenex GC manufacturing and new product development for many years," continued Welch.

Commenting on the décor of the new facility, Phenomenex founder Fasha Mahjoor praised the "vibrant colours and pleasing architectural spaces."

"Our people are the reason for our success, and it's our goal and responsibility to give them an environment that inspires teamwork and camaraderie and promotes their health and wellbeing," said Mahjoor.

For more information, please visit **www.phenomenex.com**

Photo Credit: Pheno unmenex Inc.

Eurofins Purhase Nab Labs Group

Eurofins Scientific (Luxembourg) has announced the acquisition of Nab Labs Group (Jyväskylä, Finland), one of the largest independent environment testing laboratories in Finland.

Providing a comprehensive range of environmental research and testing services across Finland, Nab Labs has a strong record working with industrial process analytics and forestry sectors.

Reportedly generating annual revenues in excess of €9 million, the acquisition of Nab Labs further consolidates Eurofins position in the environmental testing market of Finland, following the recent acquisitions of Ramboll Labs and Ahma.

"The acquisition of Nab Labs is another illustration of Eurofins' commitment to consolidating the Group's leading footprint in the markets where it operates," said Gilles Martin, Eurofins CEO.

"Together with the recently acquired Ahma and Ramboll networks of laboratories in the country, we look forward to supporting these laboratories' respective operations and further developing their capabilities to continue providing customers in Finland with the highest level of analytical service," said Martin.

For more information, please visit **www.eurofins.com**

New Evidence of Bitumen Trade Found Using GC–MS

Researchers investigating fragments of black organic matter found scattered at the 7th century ship-burial at Sutton Hoo, Suffolk, UK, have discovered the material is not what it was originally identified as (1).

First excavated in 1939, and the subject of continued archaeological research ever since, the ship-burial at Sutton Hoo is one of the most significant discoveries ever made in Britain. The richly furnished burial site contained many significant artefacts including the famed Sutton Hoo helmet and shield, as well as significant objects from throughout the known world of the time.

Also identified during the excavation were several tarry-looking materials, including two groups of fragments located near the head and foot of the coffin. These were initially identified as manganese oxide, however, a later study in the 1970s used solubility tests and paper chromatography to overturn the initial conclusion, instead identifying the material as "Stockholm Tar", which is often used as a waterproofing agent and timber preservative. This conclusion had stood ever since with the tarry-looking lumps residing at the British Museum in London.

As part of a wider research project a reinvestigation of the Sutton Hoo tars was undertaken using Fourier-transform infrared spectrometry (FTIR), gas chromatography–mass spectrometry (GC–MS), and elemental analysis-isotope ratio mass spectrometry (EA-IRMS). The surface morphology of the fragments was also examined by optical microscopy and reflectance transformation imaging (RTI).

The research concluded that the residues had been erroneously identified again and were in fact bitumen. The existence of bitumen within the burial mound also required re-evaluation because its significance has been both misunderstood and understated.

A possible prestige item of the time, the origin of the bitumen is of importance because sources within the British Isles would have been located outside the East Anglian kingdom to which the burial mound is attributed. However, composition and molecular ratio analysis of the bitumen revealed a source from much further afield with the bitumen being similar to the Middle Eastern bitumen of the Dead Sea family. This represents an archeologically significant find as other goods identified within the burial assemblage have a possible Syrian origin. This adds further evidence to the extent of trading routes at the time, and despite the original form of the bitumen being undiscernible, also represents the first evidence of bitumen being traded from the Middle East to the British Isles. — L.B.

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LCGC TV Highlights

***LCGC* TV: 3D LC: Is It Practical?**
Space-based and time-based separations are often a preferred approach. Peter Schoenmakers from the University of Amsterdam discusses the complexities of 3D LC.
Watch Here>>

***LCGC* TV: Optimizing Gradients for 2D LC**
Paola Dugo from the University of Messina discusses her strategies for optimizing the gradient in the second dimension of a comprehensive 2D LC reversed-phase separation.
Watch Here>>

Peaks of the Week

The *LCGC* Blog: Time Interval Deconvolution as an Alternate Strategy to Peak Integration Using Gas Chromatography–Vacuum Ultraviolet Spectroscopy—Precise and accurate quantitative analysis based on chromatographic measurements has historically relied very heavily on careful peak integration. Seasoned analysts know that while automated algorithms exist in modern chromatography software, it is a best practice to manually check that the integration points—the points at the beginning and end of a peak, between which the peak will be integrated to obtain a peak area—are appropriately specified. **Read Here>>**

UHPLC, Part 1: Perspectives and Instrumental Features—This instalment highlights historical perspectives on the development of ultrahigh-pressure liquid chromatography (UHPLC) into a modern high performance liquid chromatography (HPLC) platform and describes the important instrumental features common to most commercial equipment. **Read Here>>**

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Improving Foot and Mouth Vaccine Stability Using HPSEC—Researchers from the Chinese Academy of Science in Beijing, China, have used high-performance size-exclusion chromatography (HPSEC) and differential scanning calorimetry (DSC) to study the stabilization of inactivated foot and mouth disease virus. **Read Here>>**

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News In Brief

A direct large volume injection (DI-LVI) high performance liquid chromatography–tandem mass spectrometry (HPLC–MS/MS) method has been developed and validated for the quantitative determination of 16 systemic insecticides and their main plant metabolites. The team from Oregon State University tested commercial red and white wines made from grapes grown in major wine-producing regions both nationally and internationally. The study highlights the importance of determining both parent and metabolite forms of systemic insecticides in the finished product.
DOI.org/10.1016/j.chroma.2017.05.019

Shimadzu Scientific (Sydney, Australia) has announced an Australasia distribution partnership with Biotage (Uppsala, Sweden). The two companies will work together to offer sample preparation and organic chemistry equipment and consumables to their customers in the region.
www.shimadzu.com.au
www.biotage.com

Priorclave (London, UK) has announced the appointment of Barbra Wells as President and CEO of its American operation. Barbra joined the North American organization three years ago, predominantly in a marketing role, and has been key in driving the growth of the company in the USA. The company has confirmed that the USA continues to be a strong market for them.
www.priorclave.co.uk

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Chromatographers: What's Your Problem?

How do you deal with problems that arise in the laboratory?

We all get into trouble in the laboratory. Whether it's problems with sample preparation, instrument operation, data processing, or the separation itself, we all need help from time to time.

The skill is knowing where to go for help, and I guess this is what I have seen changing most during my career. In my own early years in the industry there was an expert to whom you could turn and they tended to specialize in a particular technique: high performance liquid chromatography (HPLC), gas chromatography (GC), spectroscopy. They had honed their knowledge and skills through trial and error, learning from their predecessors, working with instrument manufacturers, and networking through conferences, meetings, and exhibitions. Whilst some of those in-house experts still exist, they are a much rarer breed these days.

Manufacturers were also better structured in the "early days" to provide post-sales support, with engineers, application specialists, trainers, and technical support people all employed to make your new (and not so new) purchases work like you wanted them to. Of course, all post-sales support is a cost of sale to manufacturers, and in these modern times where the instruments are designed to work "out of the box" and are somewhat commoditized, they can afford to reduce these costs because advanced equipment should be "plug and play". Should be!

Of course, the biggest resource of them all, the internet, was non-existent when I started in chromatography and only in its infancy when I was really getting my teeth into the technique. There are now manufacturer-specific resource pages, application notes, chat rooms, forums, journals, business publications, and pretty much everything you can imagine in terms of support for the laboratory worker.

So why do I encounter so many people with problems, who are unsure as to the

cause let alone the cure? Why do I see so many people struggling with methods that are, to put it politely, suboptimal? Why are there so many instances where data quality is at best below par and at worst indefensible?

Let's look at some recent statements that I've heard and see if we can draw some conclusions:

- "We were recommended to try (solution X) by a colleague in the organics lab."
- "We found this suggestion on-line and thought we'd give it try."
- "This application note was written for matrix X and we thought it would work for our matrix."
- "We didn't know what to try so we found an article on-line which suggested that parameter Y can be used to minimize variability."
- "We've tried everything that we can, so posted the problem on (forum X) and someone suggested that we try (solution Y)."

Can you spot a common theme? Whilst all of the above sources may be of some use, they are not qualified in terms of their validity. One does not know what experience or expertise has gone into making these recommendations. I see this use of unqualified resources very often and my feeling is that their use is increasing.

So what's so bad about this if it provides a solution to the problem? My main concern is that even if the "fix" is successful, it leaves you wondering how and why the fix worked. Is this important?

Well, ask yourself the following question? Would you go ahead and try to fix a problem with your TV, washing machine, or car on the recommendation of a piece of information from the web or someone who claims to know about cars? For some of you the answer may be yes, in which case you are probably mechanically or electronically minded with some underlying skills or experience. For the vast majority of readers, I suspect that the answer is no.

This poses two questions: How did we get into the situation where self-help is often the only help available and what can we do the next time we encounter a problem?

I've already eluded to the answer to the first question. Instruments are designed to run, trouble-free, for a host of applications without an issue. You don't need a manual to show you how to use a vacuum cleaner and analytical equipment is viewed in much the same light in modern times. "Plug and play" for the digital native generation. A tool which is a means to an end.

Most manufacturers will produce a number of application notes that indicate

the analytical column and instrument settings for "indicative" separations over a diverse range of application areas, and of course you will get the instrument manual and sometimes, if you are lucky, an instructional DVD or post-installation training from the vendor's engineer. After that, you're pretty much on your own.

What if you know little about the theory of chromatography, the chemistry of the analytes and chromatography column, or the settings that affect the various aspects of the separation? If you think this doesn't happen in the modern analytical laboratory, I can absolutely assure you that it happens in many laboratories around the world and I've seen it with my own eyes.

Isn't that what standard operating procedures (SOPs) and standard methods are for? Yes, but I also see many poor quality methods in use, where the main instrument parameters may be clearly stated, but the less obvious parameters may be completely omitted from the document or there is an assumption that there are certain "generic" parameters set and left well alone.

Further, even if all the standard method parameters are correctly set, what happens if there is an issue with the chromatography or the data quality doesn't meet the method requirements? Where do you go for help?

This leads us on to the answer to the second question and takes us into the murky world of the modern-day analytical laboratory. As I have pointed out numerous times within this column (1–3), the problem lies with the changing industry paradigm as we move from chromatography as a complex measurement science and research tool, through to a tool used by a wide variety of end users to support their research aims. We are effectively in a state of flux as both the end user and vendor communities move from a highly complex technology that requires a deep understanding to operate at even a fundamental level, to an everyday "plug and play" tool. Our employers are firmly wedged between a rock and a hard place in this paradigm: wishing to increase analytical throughput often via the purchase of more instruments, being told by the vendors that the instrumentation is very robust and easy to operate, and wanting to pay a commensurate rate to operate all this extra equipment.

This is all very well as long as nothing goes wrong or there's no need to improve analysis or develop new methods!

Where do you therefore go for help when faced with the unknown? Many

companies have had the wisdom to retain an expert chromatographer on staff, so that is an obvious place to go for help. However, and it pains me to say this, there is a big difference between a chromatography expert and someone who has used chromatography for a period of time. The latter are increasingly being taken for the former, and this is dangerous for both the employer and employee.

Manufacturers do still offer a reasonable level of post-sales support, and of course their expertise is (or at least should be) qualified. However, it is rare that the vendor will want to get embroiled in solving more deep-rooted problems with your separations. They may even take the view that your business should have the level of knowledge and expertise to work through the problem—you shouldn't be "struggling with the basics", as they might see it!

Of course, the internet may give some guidance as to how your problems may be solved. There are certainly a lot of good teaching sites these days as well as the host of manufacturer and journal literature where one might find credible help. But please make sure it's just that—credible.

Finally, there are many chromatographers who used to be employed by large organizations but who are now part of smaller organizations or who may even have made the brave step to set up as a consultant. Whilst your own organizations may provide support and expertise, if you do ever find yourself in a bind where internal expertise can't provide a solution, I would urge you to seek out some of these folks for some sound and credible advice. A short amount of time with these folks can often solve a very intractable problem and save you a lot of heartache and your business a lot of time and money.

They say the best tradesmen, such as plumbers, come from recommendations from friends or acquaintances. Why should it be any different for experts in a different type of plumbing?

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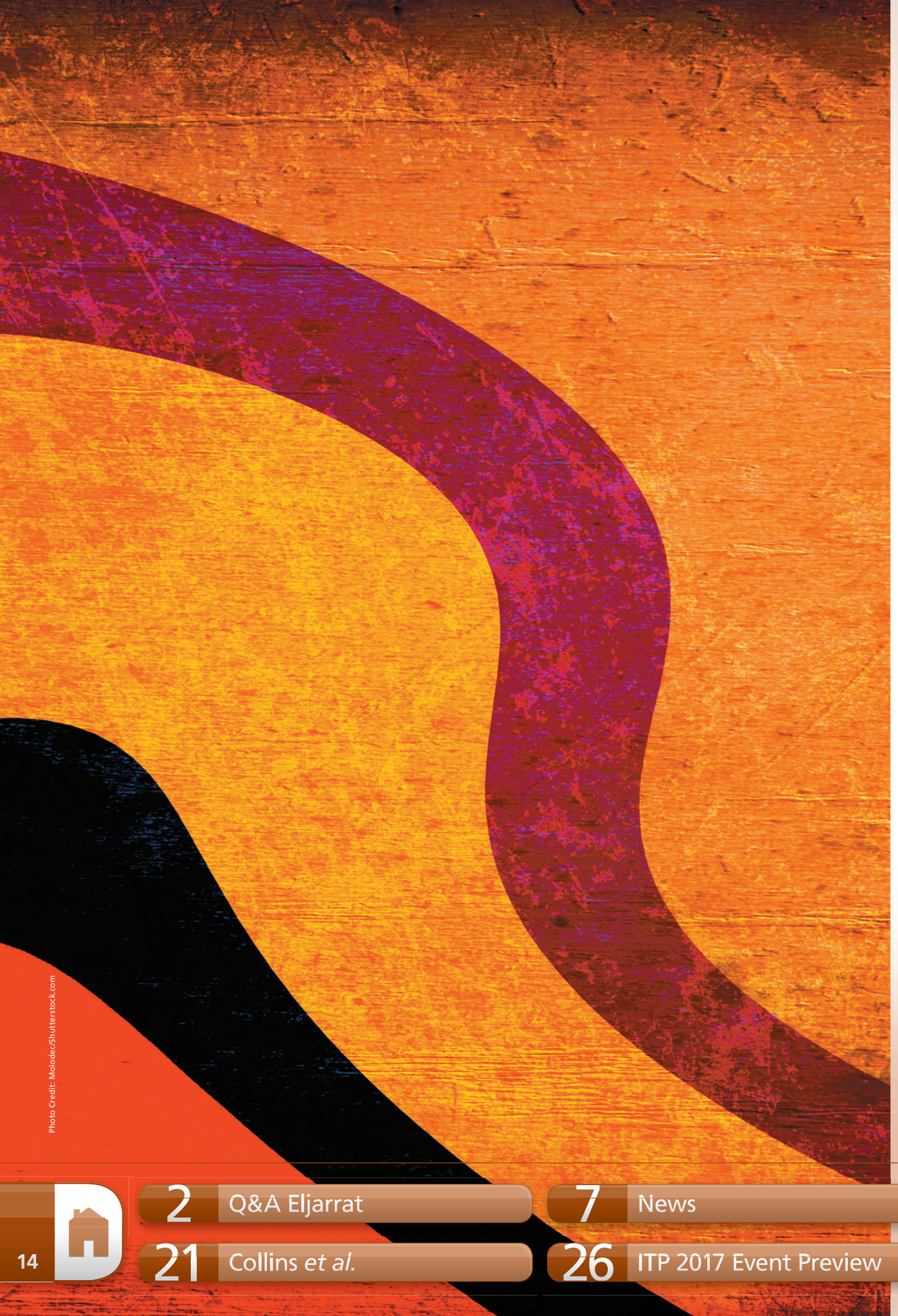

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A Guide to Modern Comprehensive Two-Dimensional Gas Chromatography

Laura McGregor and David Barden, SepSolve Analytical, Peterborough, UK

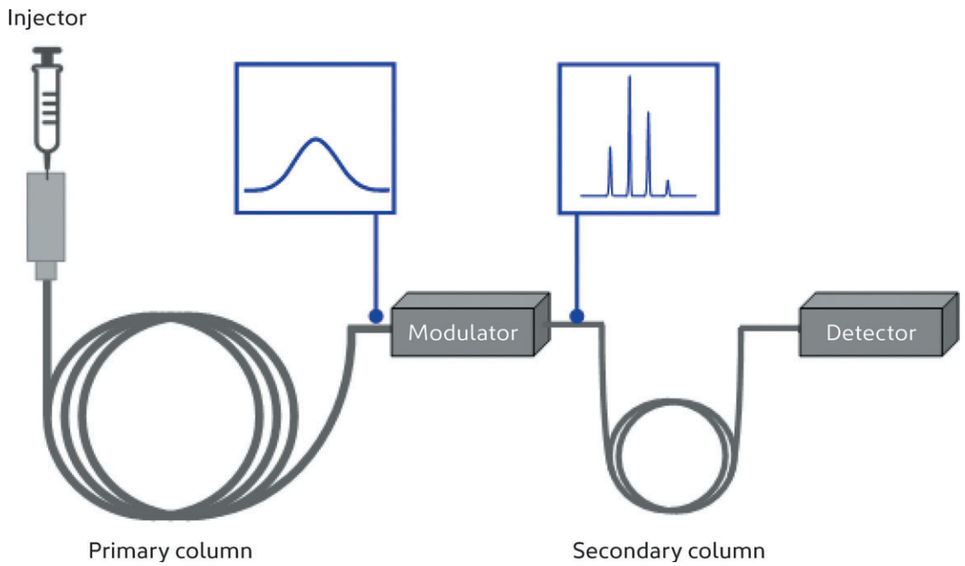
This article provides a short overview of the theory and practice of the rapidly developing field of two-dimensional gas chromatography (GC×GC). Included in the discussion are a summary of the detectors used, an assessment of the options available for modulating the first-column eluate, and some recent developments in methodologies for interpreting the results.

Comprehensive two-dimensional gas chromatography (GC×GC) is a high-performance analytical technique with an increased separation capacity that enhances the analysis of complex samples, such as petrochemicals, fragrances, and environmental extracts (1).

GC×GC involves coupling two columns with different stationary phases, to allow separation of a mixture based on two different separation mechanisms (Figure 1). The sample is therefore separated in two dimensions (2). This provides GC×GC with the capacity to resolve an order of magnitude more compounds than traditional gas chromatography (3).

As with a conventional GC system, the sample is introduced (by a range of mechanisms, such as headspace [HS], thermal desorption [TD], solid-phase microextraction [SPME], or liquid injection) into a heated port and swept through the column by a carrier gas. The first dimension (^{1}D) typically consists of a long (20–30 m) nonpolar capillary column, while the second dimension (^{2}D) employs a shorter (1–5 m) polar column; and this is categorized as normal-phase GC×GC. However, reversing the column polarity has been shown to provide better group type separation in certain cases (4). Configuring the column set in such a way is known as reversed-phase (or inverse-phase) GC×GC.

Figure 1: Schematic of a GC×GC system.



GC×GC provides the ability to separate out previously unresolved coelutions found in many complex mixtures. When applied to samples, such as petrochemicals or environmental extracts, commonly used fractionation processes that are applied before the analysis can be reduced or eliminated (5). A complex sample can be injected as a single extract without involving time-consuming fractionation processes. This gives fast screening of the entire sample, allowing many classes of organic contaminants to be monitored simultaneously.

Detectors

GC×GC has been coupled with a range of detectors, but because of the narrow peak widths generated in the secondary column, a detector with a data acquisition rate of 30–200 Hz is often used (6). A popular detector used with GC×GC is the flame ionization detection (FID). FID is an affordable and rugged detector well-suited for quantitative analysis of hydrocarbons because the response is directly proportional to the number of carbons present in the analyte molecules. However, confident identification can be difficult because retention times (^1t_R

and ^2t_R) must be used to characterize the components. Instead, coupling to a mass spectrometer provides an additional level of information on the sample composition by allowing identification of specific peaks based on chemical structure.

In a literature review by Seeley and Seeley (7), the majority (67%) of published works were obtained using time-of-flight mass spectrometry (TOF-MS), with the use of FID and single quadrupole MS also being significant (16% and 11%, respectively). However, a number of papers have also been published using selective detectors, such as sulfur chemiluminescence detection (SCD) and electron capture detection (ECD), as well as isotope ratio MS, tandem mass spectrometry (MS/MS), and, most recently, vacuum ultraviolet (VUV) spectroscopy (8).

Modulation

The most critical part of the GC×GC system is the modulation device. Peaks eluting from the first column are sampled and re-injected as narrow chromatographic bands into the secondary column where they are further separated (2). Separations within the secondary column are fast—normally under 10 s in length. To preserve the separation achieved in the primary column, it is recommended that each peak eluting from the primary column is sampled three or four times (9).

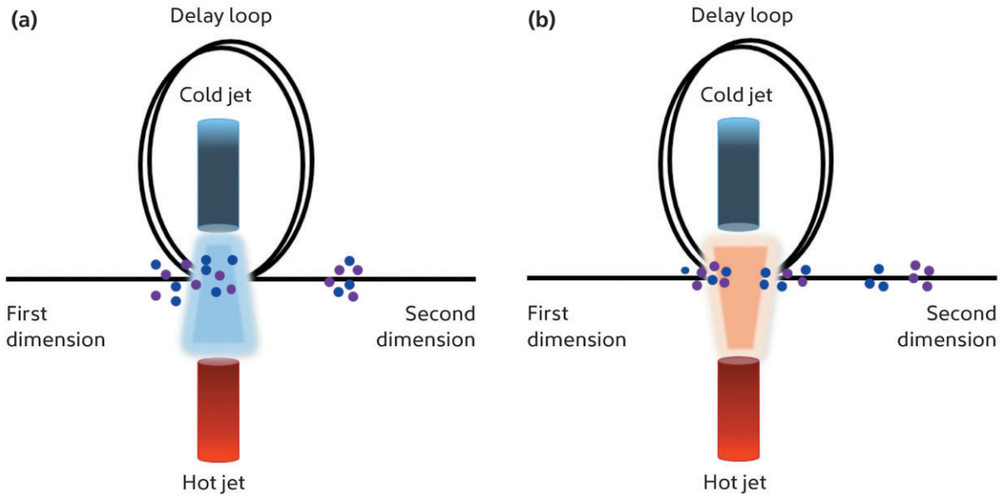
This process of focusing primary column effluent into narrow bandwidths results in improved signal-to-noise ratios for the analyte peaks, generally providing a 10-fold improvement in sensitivity with respect to 1D GC. Ineffective modulation results in broad, tailing peaks in the second dimension, which limits peak capacity.

The two main types of commercially available modulator, thermal and flow devices, are described in the following sections.

Thermal Modulation: Thermal modulators use broad temperature differentials (by way of hot and cold jets) to retain or desorb analytes eluting out of the primary column (10). These devices typically use two-stage operation. In the first stage, the cold jet traps and focuses the eluate at the head of the secondary column (Figure 2[a]). The hot jet then desorbs the analytes from the stationary phase (Figure 2[b]), and they continue on to the next cooling stage of the modulation process. Commercial devices use either a quad jet approach (where there are two pairs of jets to trap or desorb the analytes on two different sections of the column) or a delay loop (where the column circles back between the hot or cold jets). Both of these approaches ensure that there are two attempts to focus the analytes.

This process allows the primary column eluate to be focused into narrow injection

Figure 2: Schematic of a thermal (delay loop) modulator. (a) The cold jet traps the first-column eluate, and then (b) a pulse of hot air arrives from the hot jet, deflecting the cold jet, and forcing analytes onto the second dimension for further separation.



bands, which increases secondary column resolution and therefore peak capacity. Currently, thermal modulators are the most widely used in GC×GC (11). The main drawback of thermal modulation is that volatile components cannot be trapped by the cold jet, even when liquid cryogen is used to cool it. Typically, thermal modulators using liquid cryogen can modulate C_4 and above, while those relying on a chiller unit to cool the jets may only be able to modulate C_8 and above.

Flow Modulation: Flow modulators use precise control of carrier and auxiliary gas flows to fill and flush a sampling channel or loop (12). In the first generation of flow modulators, called *forward fill/flush*, any over-filling of the sample loop flowed directly on to the second dimension, causing poor peak shape and reduced peak capacity. In addition, breakthrough of analytes from the primary column to the secondary column frequently occurred during the flushing stage.

To overcome this, *reverse fill/flush* dynamics have been developed to improve peak shape and limit the baseline rise between modulations by directing any overfill to a bleed line (13). The sample loop is filled in the forward direction from the first column (Figure 3[a]), and then rapidly flushed in the reverse direction onto the second column (Figure 3[b]). The total modulation period (P_M) is the time taken for the fill and flush modes to complete.

A key benefit of flow modulation is that it does not suffer from the same volatility restrictions associated with thermal modulation, which enables volatiles from C_1 to be efficiently modulated, so expanding the range of applications that can be tackled by GC×GC. There is also an obvious cost benefit because no liquid cryogen or chiller unit is required.

Additionally, flow modulators are known to exhibit excellent repeatability, because of the precise control of flow in each dimension. Thermal modulators, on the other hand, may show retention time fluctuations as a result of small variations in column position between the jets, or variation of cryogen flow to the cold jets, making it more difficult to compare large sample batches.

A potential difficulty with flow modulators is that they require a high flow rate in the second dimension to compress the primary column eluate, making it challenging to achieve direct coupling to mass spectrometric detectors (14). To overcome this, the flow is typically split after the secondary column to two detectors. When using a second detector with different capabilities, this offers the additional advantage of capturing two complementary datasets in a single run. For example, robust quantitation can be achieved using FID, while confident identification can be performed by TOF-MS.

However, it has recently been demonstrated that optimization of GC×GC parameters can allow flow rates compatible with mass

Figure 3: Schematic of a flow modulator using reverse fill or flush dynamics: (a) fill mode and (b) flush mode.

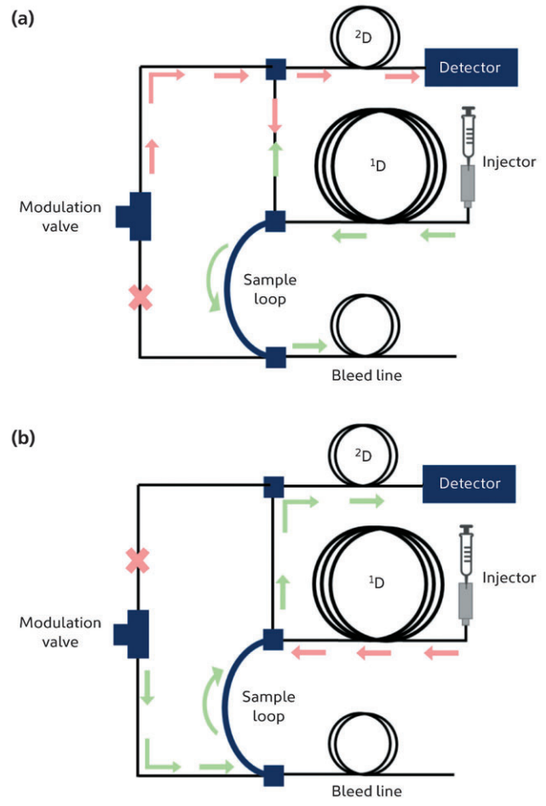
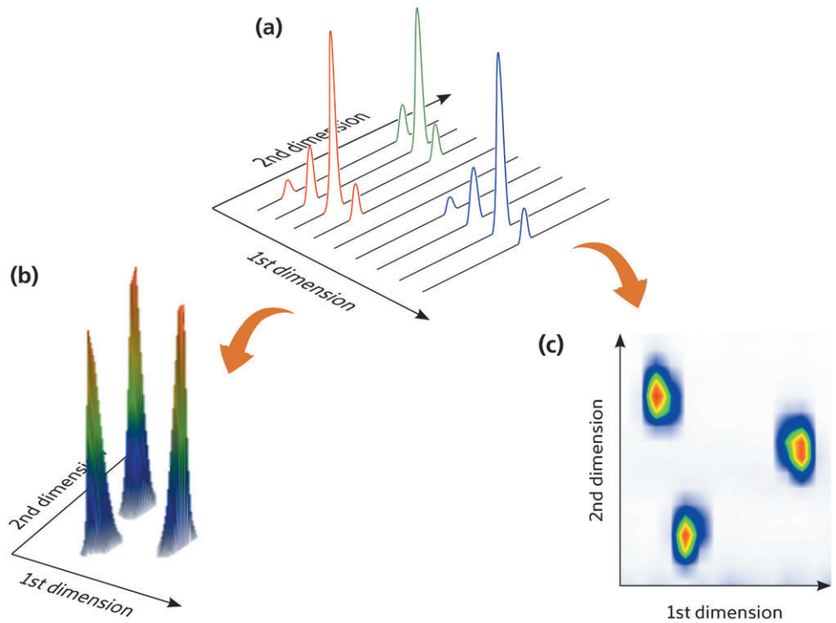


Figure 4: In GC×GC, the eluate from the first-dimension column is split into fractions that are individually fed into a much faster eluting second-dimension column. The resulting chromatograms (a) are "stacked" to form surface plots (b), which in turn can be viewed "from above" as colour (contour) plots (c).

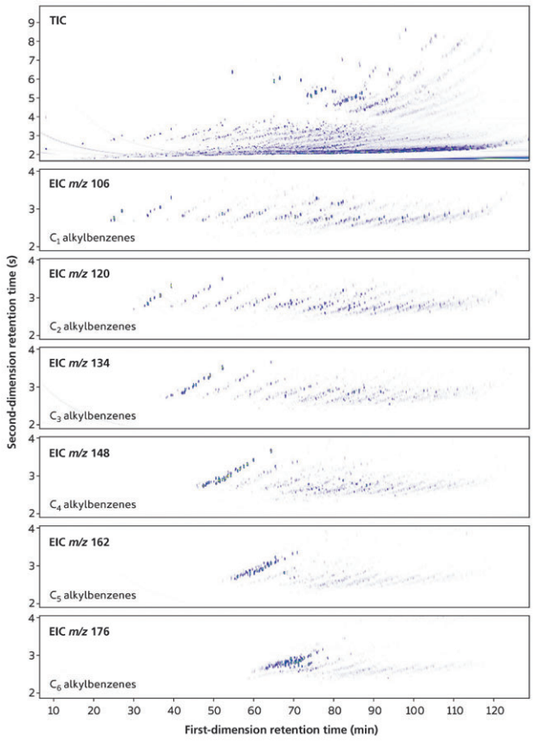


spectrometers (~4 mL/min) to be achieved and avoid the need for splitting (15). If required, this means that the entire flow can be directed to the mass spectrometer to avoid compromising sensitivity.

Visualization of Results

The modulated, linear detector output from GC×GC can be represented as a three-dimensional landscape (known as a surface plot) by stacking the fast secondary separations side by side (Figure 4). The results can be evaluated using this type of chart, but it is typically simpler to compare samples using two-dimensional colour (or contour) plots. In a colour plot, the *x*-axis represents the retention time in the primary column (^1t_R), the *y*-axis represents the retention time in the second dimension (^2t_R), and the colour gradient represents the intensity of the peak, whereas in a 3D surface plot the additional *z*-axis represents the peak intensity.

Figure 5: The "roof-tiling" phenomenon in a GC×GC colour plot of diesel.



A colour plot can therefore be thought of as a bird's-eye view of the surface plot.

Structured Ordering

An advantage of GC×GC chromatograms is the structured ordering or "roof-tiling" effect (Figure 5). Compounds from the same chemical class typically elute together in bands, allowing fast, tentative identification of the major components present in the mixture.

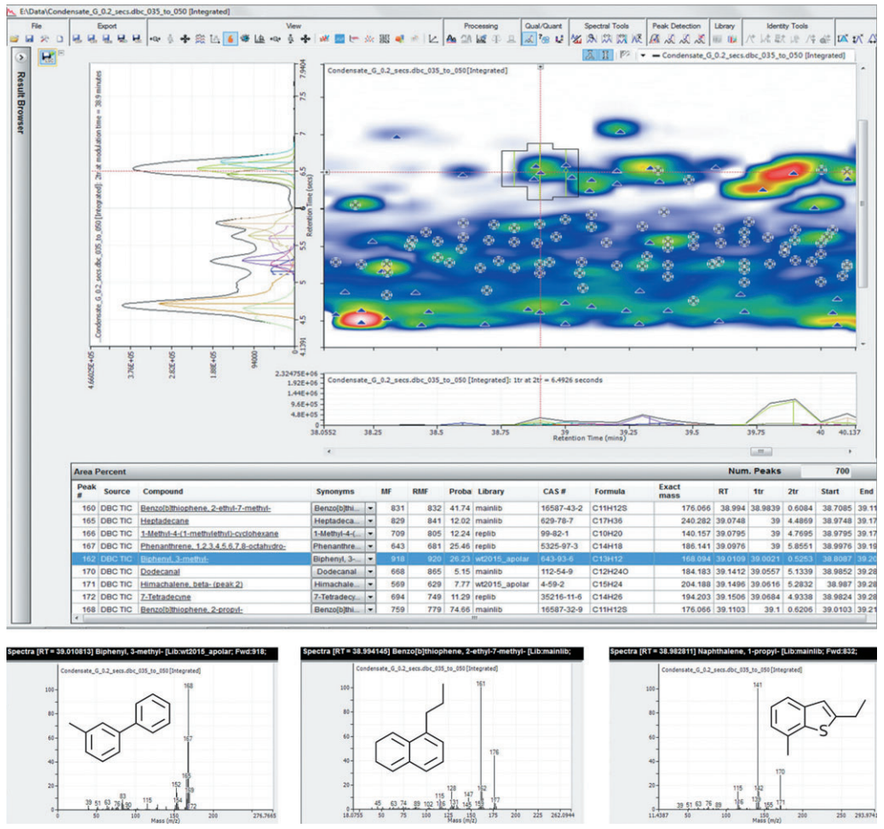
In contrast, when a complex mixture is analyzed by 1D GC, it is difficult to make assumptions about the chemical structure of eluates based solely on their retention times, because they are only separated based on a single chemical property. For example, compounds from many different chemical classes may have similar boiling points, so this alone would not allow classification of different chemical families. However, if these components are further separated based on polarity, as in normal-phase GC×GC, classification of chemical families is easier because of the chemical similarities measured by two distinct properties.

This type of structure allows characteristic patterns to emerge, enabling experienced analysts to quickly identify the main chemical classes within a complex mixture.

Software for GC×GC

GC×GC data is acquired by a detector in a linear (1D) format, so specialist software is required to "fold" the data (based on the known modulation time) to view colour and surface plots (16). There are now a number of commercially available software packages for GC×GC data processing—some are specific to a particular instrument, while others are capable of processing third-party data files from a range of instrumentation.

Figure 6: Deconvolution applied to GC×GC–TOF-MS of a petrochemical sample. The spectra shown are derived from three coeluting compounds (boxed area).



When analyzing the most complex samples, it is often the case that even two dimensions of separation are not sufficient to achieve full analyte separation. In such cases, deconvolution can play a major role. Figure 6 shows the deconvolution of three peak profiles from a single TIC peak in a petrochemical sample.

Conclusions

GC×GC technology has progressed significantly in the past 10 years, with

advances in modulation and software making the technique more applicable to routine applications. The technology is already established in a number of diverse fields, including petrochemical, environmental, and fragrance analysis, and is likely to provide further insights into challenging samples for years to come. At the current time, areas of growing interest include breath profiling for disease diagnosis, and aroma profiling in the food and beverage industries.

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Rapid Identification of Illicit Drug Substances Using Thermal Desorption Coupled with a Portable Toroidal Trap GC–MS System

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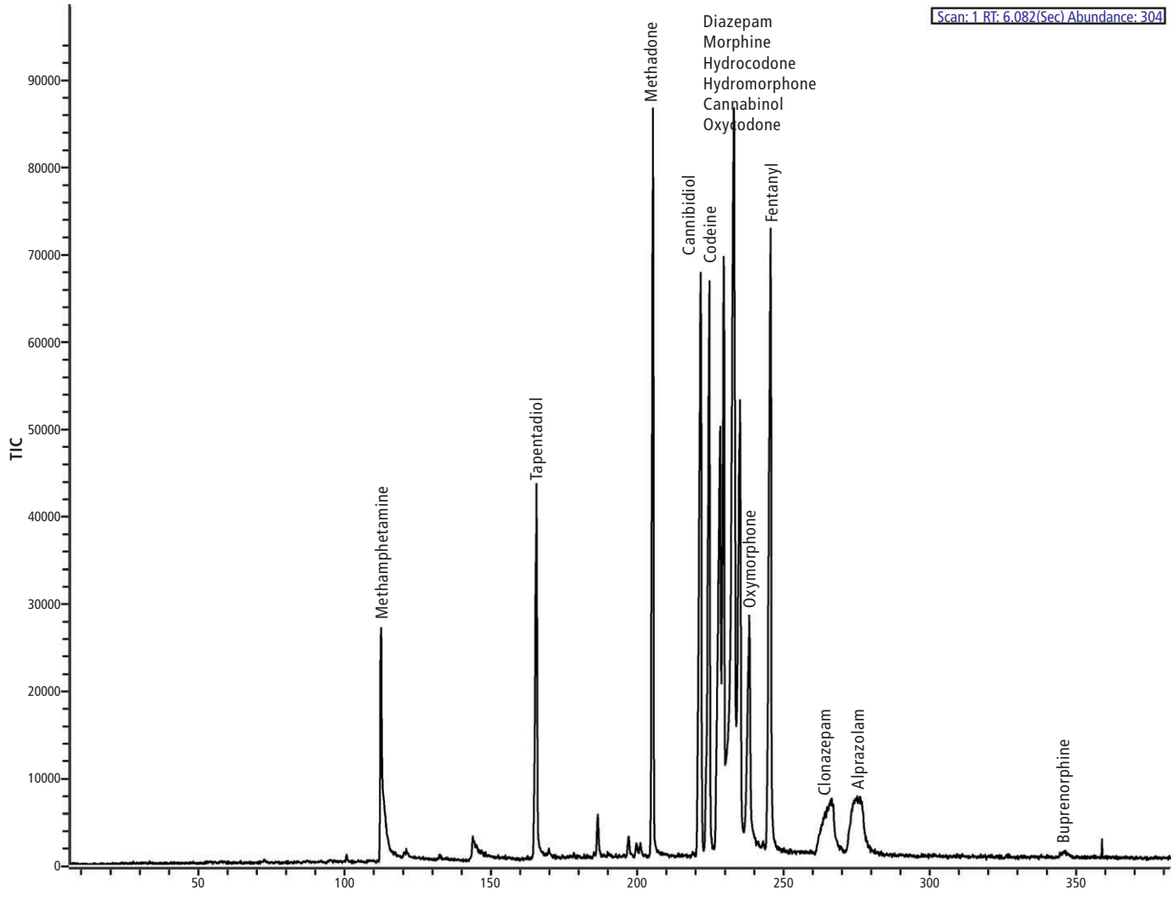
Recent developments in the miniaturization of gas chromatography–mass spectrometry (GC–MS) instrumentation are making the technique available for field-based investigations, offering a simple, onsite identification of drug substances. This article describes the identification of sixteen drugs compounds in less than 10 min using portable gas chromatograph-toroidal ion trap mass spectrometry combined with a coiled-wire-filament (CWF) sampling injector to provide an effective tool for the rapid analysis of illicit drug substances.

The misuse of drug substances has created an urgent need to rapidly determine the type of chemical compounds present in the field. Gas chromatography–mass spectrometry (GC–MS) has routinely been used by scientists in the laboratory to characterize the presence of pain medication compounds. Unfortunately, the confirmation process can be extremely slow and time-consuming because the sample must be taken back to the laboratory to perform the analysis. As a result of this limitation, a rapid, on-site solution to the

Figure 1: A chromatogram of all 16 drugs investigated in this study.



identification and characterization of these compounds is desirable. This study will describe an approach for performing this analysis, using a compact, portable GC–MS system for use by nonspecialist operators in the field.

Methods

The portable GC–MS technology used in this investigation is described in the open literature (1,2). When used in conjunction with an extensive NIST database and custom-built target compound libraries, unknown peaks

Figure 2: Chromatogram showing the coelution of hydrocodone–morphine and cannabinol–hydromorphone.

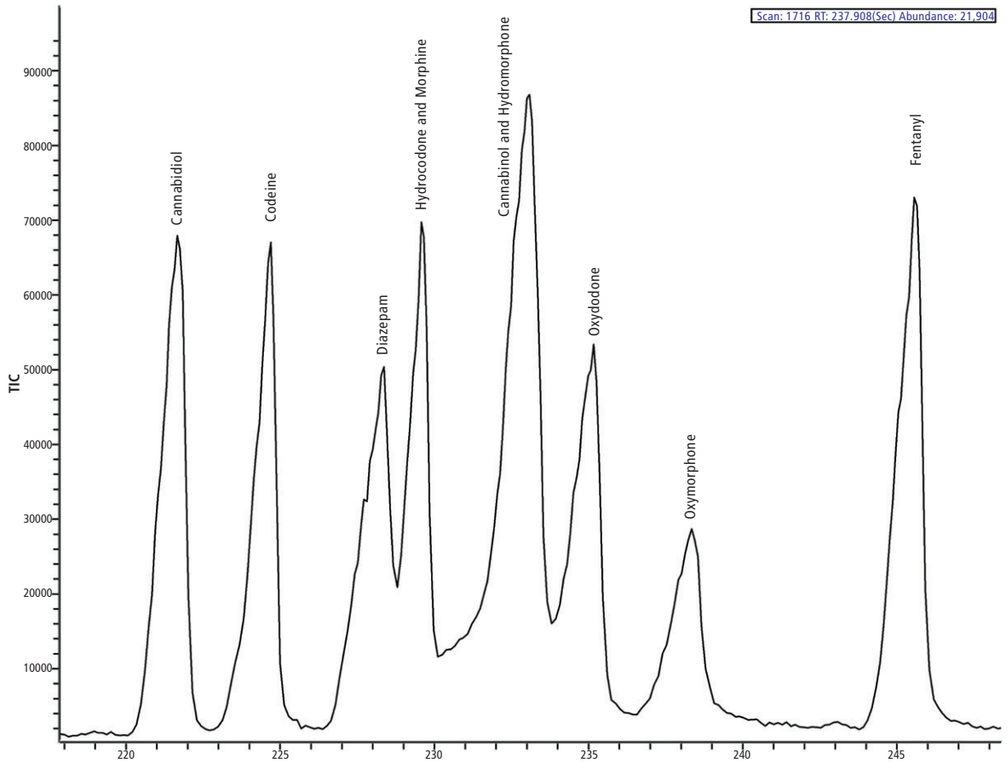
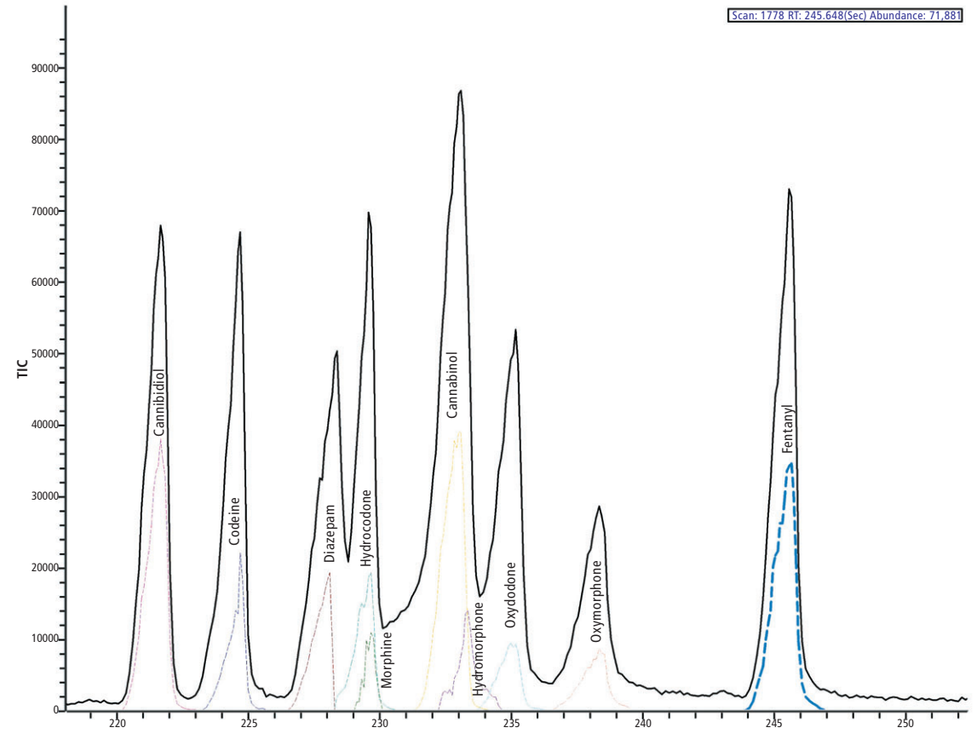


Figure 3: Deconvolution and identification of the two pairs of drugs shown in Figure 2 can be achieved using the on-board algorithms.



can be positively identified by nonexperienced personnel in the field (3,4).

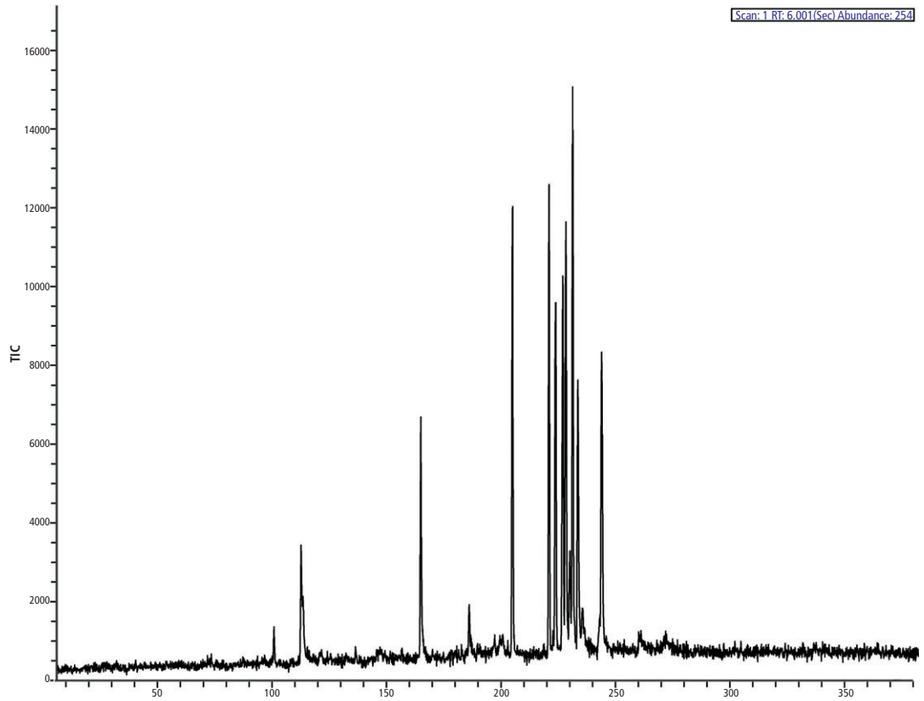
Sample Collection: For different sample types, a small, battery-operated, sampling accessory (SPS-3, PerkinElmer Inc.) was available (5), which requires the use of a needle trap (NT) to transfer the analytes to the GC–MS system. Based on the samples being studied, the choice of sampling strategies include:

- Heated headspace (HS) for volatile compounds in solid and liquid samples
- Purge and trap (P&T) for volatiles in liquid (aqueous) samples
- Thermal desorption (TD) for volatiles using a conventional TD tube
- Internal standard (IS) addition module
- The needle trap can also be used independently to sample gases without the sampling module
- Solid-phase microextraction (SPME) can be used for gas and liquid samples
- A coiled wire filament (CWF) is available for semi-volatiles dissolved in solvent samples.

For this study, a CWF sampling accessory was used. A method for the separation and identification of sixteen painkilling drugs using this approach was performed.

Sample Preparation and Analysis: Methanol solutions (1.0 mg/mL) of standard drug substances (methamphetamine, tapentadiol, methadone, cannabidiol, hydrocodone, morphine, hydromorphone, codeine, diazepam, oxycodone, cannabinol, oxymorphone, fentanyl, clonazepam, alprazolam, and buprenorphine) were used (Cerilliant) for the investigation. A single mixture of all sixteen drugs was prepared (60 µg/mL). Prior to injection, and to eliminate backflash of the methanol within the injector of the gas chromatography toroidal mass

Figure 4: Chromatogram of all 16 drugs when dipping the CWF into a drug mixture (60-µg/mL of each drug).



spectrometer (GC–TMS) system, the sample was first added to a novel deactivated stainless steel CWF (PerkinElmer) with subsequent evaporation of methanol at room temperature and pressure under a fan (1–5 min). Upon introduction of the CWF into the injector of the GC–TMS system, the compounds were thermally desorbed.

The sample was applied to the CWF by the following two methods:

- Direct application of a measured volume using a 10-µL syringe
- Dipping the coil

Direct application using a syringe allowed for known and variable (0.5–10 µL) sample volumes to be applied consistently to the CWF. Larger volumes (5–10 µL) required extended drying time (3–5 min). Dipping resulted in approximately 0.5 µL of sample coating the coil; drying time was consistently 1 min. Once dry, the CWF containing the sample was introduced into the injection port of the GC–TMS system and the coil exposed for 20 s. The gas chromatographic separation conditions and mass spectrometer

parameters used for the analysis are detailed below.

Gas Chromatographic Separation Conditions: Sample delivery: coiled wire filament injection; injection type: splitless; split injection times: 50:1 split on: 20 s, 50:1 split off: 60 s; injector temperature: 275 °C; column: 5 m × 0.1 mm, 4-µm MXT-5 low-polarity phase diphenyl dimethyl polysiloxane (Restek); initial temperature and hold time: 40 °C for 20 s; temperature ramp rate: 1.25 °C/s; final temperature and hold time: 300 °C for 147 s; total analysis time: 375 s.

Mass Spectrometer Operating Conditions: Mass spectrometer: Toroidal Ion Trap (PerkinElmer); ionization source: electron impact; MS operating temperature: 160 °C; mass range: 45–500 amu; resolution: <0.5 *m/z* at 300 amu; MS scan rate: 10–15 scans/s; detector: Electron Multiplier (DeTech).

A custom user-defined compound library was created with individual drug standards to determine drug retention times and mass fragmentation patterns. The library was subsequently used to identify the mixture of sixteen target analytes employing an on-board deconvolution algorithm for the drugs that coeluted.

Results and Discussion

Figure 1 shows a chromatogram of all sixteen drugs when direct application of 5 µL of a drug mixture (60-µg/mL of each drug) was applied to the CWF prior to injection. Twelve of the sixteen drugs had unique retention times and 10 could be separated with baseline resolution.

Although two pairs of drugs (hydrocodone–morphine and cannabinol–hydromorphone) coelute (Figure 2), their mass spectra are significantly different, which can then be deconvoluted and identified using the on-board algorithm, as shown in Figure 3. Under ideal conditions, the whole identification and confirmation process requires approximately 8–10 min.

All sixteen drugs were simultaneously identified with a direct application of 5 µL (~300 ng/drug) to the CWF, as seen in Figure 1. However, this was not possible with dipping the coil in the sample. This is exemplified in Figure 4, which shows a chromatogram of all sixteen drugs when dipping the CWF into a drug mixture (60 µg/mL of each drug).

After separation and deconvolution, only 10–12 of the sixteen drugs could be identified. With this lower amount of sample (~30 ng/drug), signal intensity for fentanyl (~7000), and many other compounds, is approximately 1/10th the signal intensity in Figure 1 (70,000). In fact, the later eluting compounds (clonazepam, alprazolam, and buprenorphine) cannot be seen or identified. In addition, at least one of the lower-intensity compounds (morphine, hydromorphone, and oxymorphone) is unidentified after separation and deconvolution. On the other hand, the dipping technique is much simpler and requires less time, but also requires approximately 10× the concentration of drugs for an equivalent signal. As previously mentioned, it is estimated that ~0.5 µL of the sample is collected on the CWF during dipping.

Conclusion

This study has shown that the preparation, separation, and identification of sixteen common drug substances is possible in the field using a combination of a coiled wire filament sampling device coupled with a gas chromatograph–toroidal ion trap mass spectrometer in under 10 min. This portable, field-based approach has opened up new possibilities for on-site analysis using nonexperienced operators, without having to take samples back to the laboratory for confirmation of the chemical compounds.

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Ramon Soto Alvarez, Ashley Thornock, and Serena Michalsky are undergraduate students at Brigham Young University, Idaho, USA, who are gaining research experience investigating separation science techniques. Ramon and Ashley are chemistry majors, and Serena is a biochemistry major, who plans to attend medical school.

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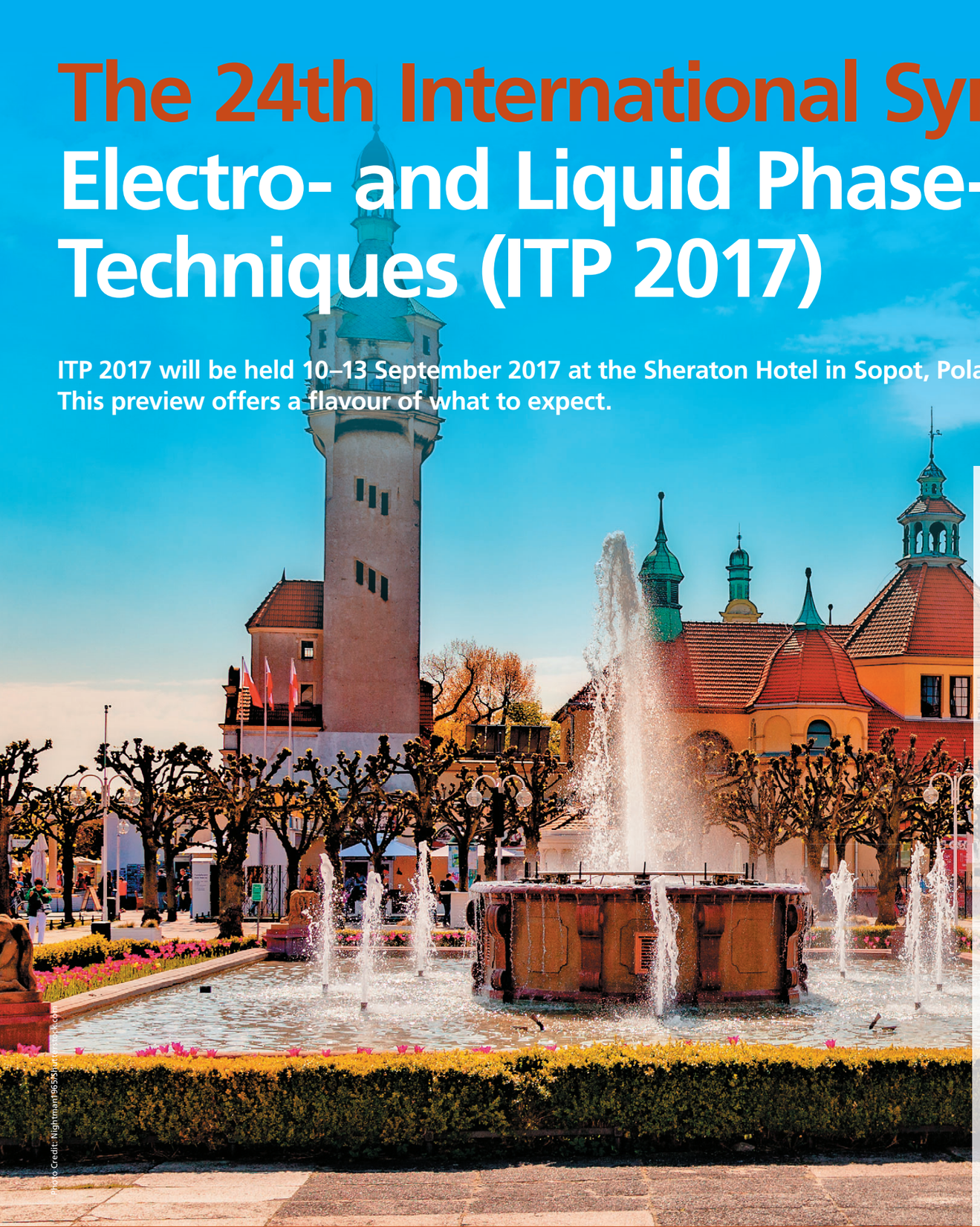
The 24th International Symposium on Electro- and Liquid Phase- Separation Techniques (ITP 2017)

ITP 2017 will be held 10–13 September 2017 at the Sheraton Hotel in Sopot, Poland. This preview offers a flavour of what to expect.



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One of the most recognized international symposium series, the **24th International Symposium on Electro- and Liquid Phase- Separation Techniques (ITP 2017)** addresses the latest discoveries, developments, and production in all areas of electro- and liquid phase-separation techniques in multiple disciplines.

The organizers are pleased to announce that **ITP 2017** will be held in **Sopot, Poland,** at the **Sheraton Hotel, 10–13 September 2017**.

The 24th ITP will continue the tradition of the series by updating attendees on the advances in different separation techniques and their applications in various areas with an emphasis on pharmaceutical and environmental analysis.

The dynamic 2017 programme will again highlight a wide range of technologies that use electro- and liquid phase- separations, such as capillary and microchip electrophoresis, electrokinetic chromatography and electrochromatography, two-dimensional electrophoresis, high performance liquid chromatography (HPLC), ultrahigh-pressure liquid chromatography (UHPLC), and micro- and nano-scale HPLC.

One of the major aims of the symposium is to provide a forum for high-level scientific exchange between analytically oriented scientists from the whole world in a friendly atmosphere. Sopot, with its special atmosphere, can offer excellent opportunities for scientific, cultural, and social experiences in a unique seaside setting.

ITP 2017 will also be running three stimulating and highly informative workshops by recognized experts in their field, which will be run in parallel on Sunday 10 September from 9:00 to 13:00. These workshops include:

Workshop 1: Fundamentals of Sample Preparation and Recent Developments in Microextraction Technologies – Where "Big" Fails "Small" Can Do More—Barbara Bojko, Nicolaus Copernicus University, Poland, and Janusz Pawliszyn, University of Waterloo, Canada;
Workshop 2: Challenges in Analytical Development: The Need in Orthogonal Chromatographic Methods for a Small Molecule Project in Innovative Drug Development—Vladimir Ioffe, Teva Pharmaceutical Industries, Ltd., Israel;
Workshop 3: Principles and Applications of Modern CE-MS—G.W. Somsen, Vrije Universiteit Amsterdam, The Netherlands.

Furthermore, participants are invited to submit manuscripts based on presentations at the **ITP 2017** meeting for possible publication in the *Journal of Chromatography A*, with the intention of publishing a virtual special issue (VSI) dedicated to the meeting. This will eliminate possible delays in publication for contributors to the special issue, making the conference special issue more complete and accessible than it has ever been.

Additionally, this year **ITP 2017** will be held in parallel with the 11th Polish Conference on Chromatography, the national meeting organized every 3 years.

Oral and poster presentations will be organized during the conference and the organizers encourage interested presenters to submit their abstracts as soon as possible.

More details on the topics, invited speakers, registration, abstract submission, conference fees, and important dates can be found on the conference website.

The organizers look forward to seeing you in Sopot!

Prof. Michał Markuszewski
Chairman

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Website: **www.itp2017.com**

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Website: http://www.chromacademy.com/gc-training.html

The GC and GC–MS Clinic
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University of Nottingham,
Nottingham, UK
Website: http://www.anthias.co.uk/training-courses/GC-clinic

GC Theory and Methods
6 September 2017
The Open University,
Milton Keynes, UK
Website: http://www.anthias.co.uk/training-courses/handson-GC-theory-methods

HPLC/LC–MS

The Theory of HPLC
On-line training from
CHROMacademy
Website: http://www.chromacademy.com/hplc-training.html

Fundamental LC–MS
On-line training from
CHROMacademy
Website: http://www.chromacademy.com/mass-spec-training.html

HPLC Troubleshooter
On-line training from
CHROMacademy
Website: http://www.chromacademy.com/hplc_troubleshooting.html

HPLC and UHPLC Troubleshooting
12 October 2017
Manchester, UK
Website: www.hichrom.com

SAMPLE PREPARATION

Overview of Solid-Phase Extraction
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MISCELLANEOUS

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Introduction to IR Spectroscopy
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GPC/SEC: Theory and Praxis
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Website: http://www.pss-polymer.com

Big Molecules–Big Challenges II (SEC, HIC, Affinity, updated for 2017)
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Website: www.hichrom.com

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Website: www.ISPPP.org

19–22 September 2017

23rd International Symposium on Separation Sciences (ISSS 2017)

Vienna University of Technology, Vienna, Austria

E-mail: info@isss2017.at

Website: www.isss2017.at

9–12 October 2017

17th International Nutrition & Diagnostics Conference

Hotel Duo, Prague, Czech Republic

E-mail: info@indc.cz

Website: www.indc.cz

7–10 November 2017

8th International Symposium on Recent Advances in Food Analysis (RAFA 2017)

Prague, Czech Republic

E-mail: rafa2017@vscht.cz

Website: www.rafa2017.eu

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