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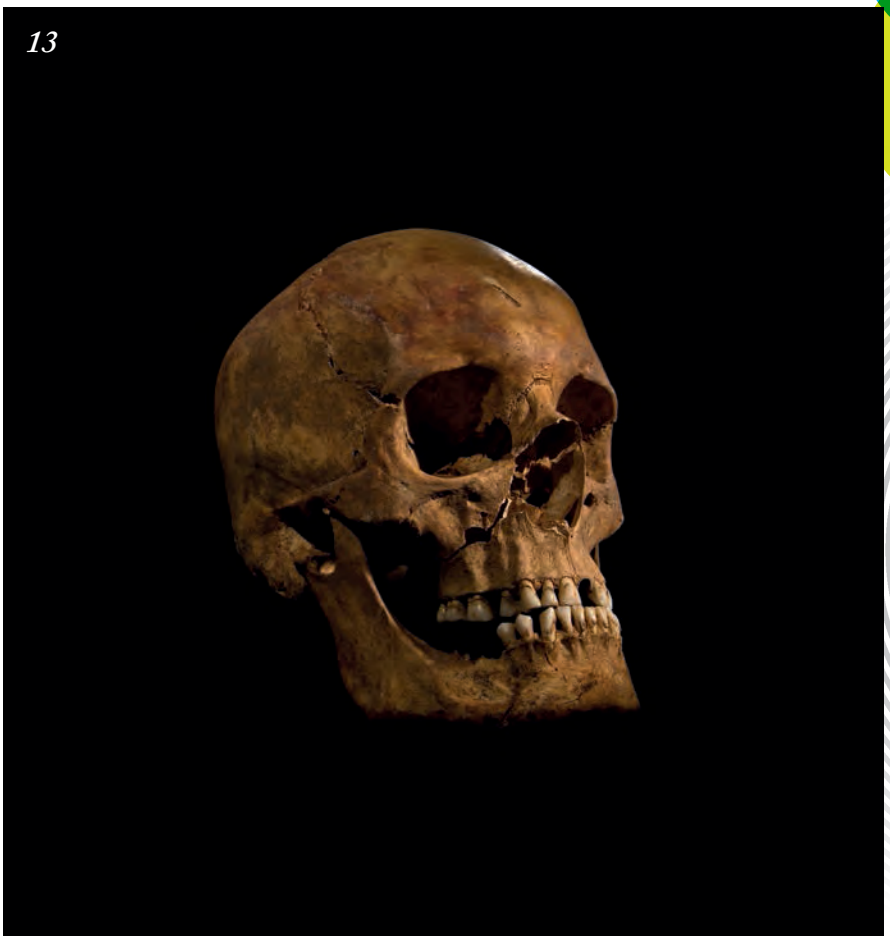

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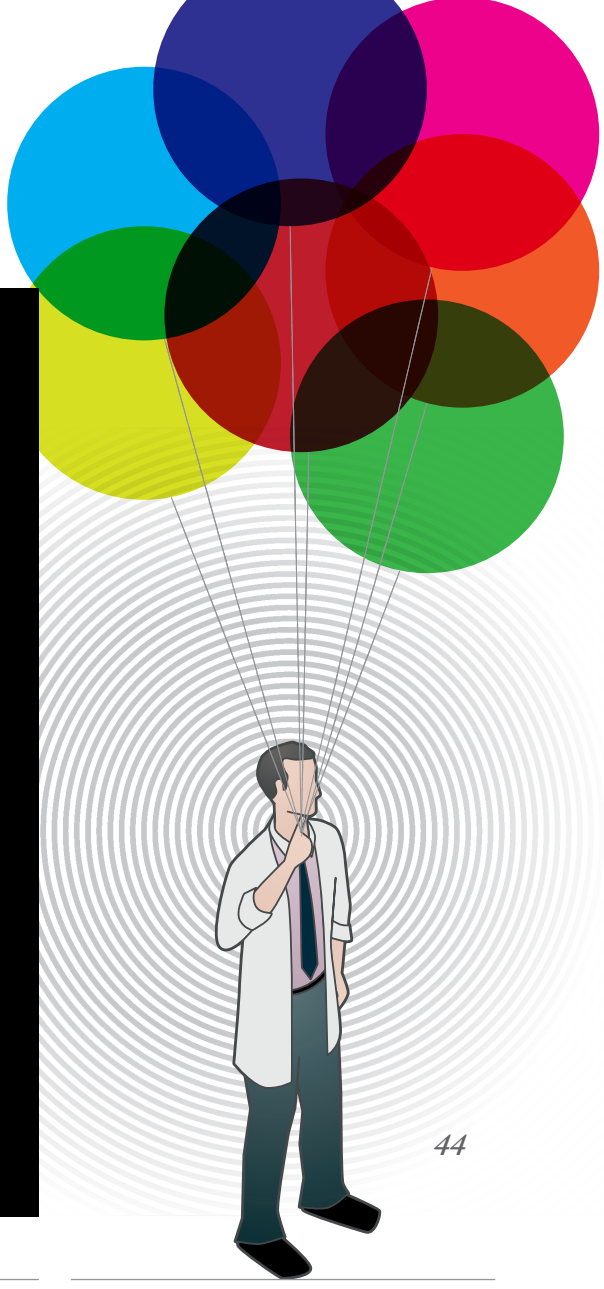
University of Leicester



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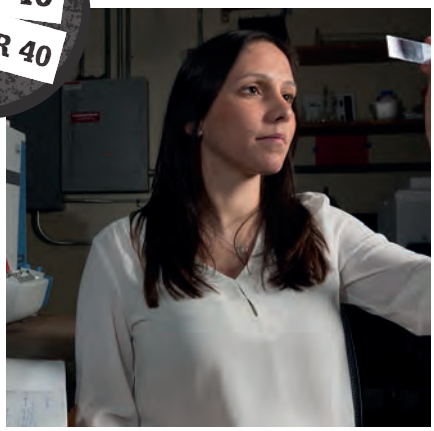
On The Cover

Pastiche of the album art from 1977's iconic "Nevermind the Bollocks, Here's the Sex Pistols".

Upfront

In My View



TOP 40 UNDER 40

Features

Departments

Sitting Down With

the Analytical Scientist

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A Level Playing Field?

Last year, we berated ourselves for the disappointing show of women in our Top 100 Power List (a measly eight percent). So, how did we do in our Top 40 Under 40?

For the 2014 Power List, we wanted to take a break from the Top 100 most influential analytical scientists and instead focus on "the next generation" (the Top 100 will be back in 2015). The Top 40 Under 40 has a pleasant ring to it (and if it's good enough for Fortune, it's good enough for us – though the timing is purely coincidental). Throughout the process, one question was burning in our minds – a legacy from our inaugural Power List – will women be better represented?

Thankfully, we can answer that question with a sigh of relief. In fact, a refreshing 32.5 percent of the 2014 Power List are women. And, while it's some way off the 'ideal' ratio of 50:50, it does perhaps reflect the current realities of women in science. In any case, we've come a long way from the days when Apryll Stalcup (see Sitting Down With, page 50) was the only female in the class at graduate school: "I grew up in the USA in an era when women didn't really do science. I wasn't aware that it was even an option for me," she notes.

As far as our list goes, it appears as though society is moving roughly in the right direction – I'd be interested to hear your thoughts. Certainly, the nominees for the Top 40 Under 40 were overwhelmingly positive about their chosen career; if given the chance to go back and change their path, would they do so? A resounding "no!" was the response.

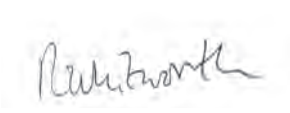
That's not to say that inequality doesn't exist in science. It does. American astrophysicist Neil deGrasse Tyson (head of New York City's Hayden Planetarium) tackled the question of "women in science" in an amusing but very astute way back in 2009 (1): "I've never been female…" he begins, "but I have been black my whole life." He goes on to say that despite wanting to be an astrophysicist since the age of nine, it was "hands down, the path of most resistance through the forces of society".

"Don't you want to be an athlete?" teachers would ask. The story echoes Stalcup's. How did Tyson get to where he is today? Simple: because his interest in the universe was so vast, and because he was so absolutely driven that he pushed through all the obstacles placed in his path – just like Stalcup. But how many great scientists get lost along the way – pushed out by outmoded expectations?

Rebuking previous answers to the question of women (or other minorities) in science, Tyson concludes, "Before we start talking about genetic differences, [we've] got to come up with a system where there's equal opportunity…"

To conclude – never mind the backlash – enjoy The Power List Top 40 Under 40 issue!

Rich Whitworth
Editor



Reference

1. *Neil deGrasse Tyson at a New York Academy of Sciences, Center for Inquiry conference: "Secular Society and its Enemies." tas.txp.to/1014/tyson (for the full panel discussion, visit tas.txp.to/1014/tyson2).*

Contributors:

Anne Aubry

Anne Aubry never anticipated, when she made the decision to study Pharmaceutical Sciences at the age of 17, that the breadth of subjects taught in pharmacy school – from statistics and deontology to biology and medicinal chemistry – would give her such a great foundation. "It served me well throughout my career in the pharmaceutical industry. In graduate school, I specialized in analytical chemistry because I loved solving problems and because of the access it gave me to a range of other sciences and scientists who all had a great story to tell." Anne is no longer in the lab, but she still loves discussing new challenges and thinking of ways to solve them.

On page 16, Anne turns her attention to the art and science of analysis.

Kim H. Esbensen and Claas Wagner

Although originally trained as a geologist/geochemist, it took 30 years before Kim Esbensen actually started to work in a bona fide geoscience institution. "In the meantime, I toured the engineering world extensively, establishing two research groups dealing with PAT and chemometrics in the process." He first found the real love of his life, scientifically speaking, some 15 years ago, with the Theory of Sampling. The field of representative sampling has occupied his career ever since: "The complexities of heterogeneity is fascinating. But unfortunately its impact is often missing from analysis, education, and in the industries involved." He is specifically interested in the interaction between materials heterogeneity, representative sampling and augmented measurement uncertainty

Originally trained as an economist, Claas Wagner realized that his real interests were with environmental and energy related topics. Sustainable resource management, emission reduction procedures and energy efficiency issues share common ground: decisions need to be based on valid data. This led to Claas' PhD on representative sampling and data analysis for quality monitoring in large-scale combustion plants. Currently, Claas combines his fields of interest as a consultant for various industries, providing quality assurance approaches. Throughout all of his work reigns representative sampling.

Kim and Claas dig into representative sampling on page 30.

Martin Gilar

Martin Gilar is a principal investigator in a core research group at Waters Corporation, where he has worked since 1998. He has more than 20 years of experience in the separation sciences, including chromatography, electrophoresis, and mass spectrometry. "My research interests lie in the analysis of biopolymers and 2D-LC. I received my PhD in analytical chemistry from the Institute of Chemical Technology in Prague in 1996, and spent my postdoc years developing separation methods for antisense oligonucleotides and fraction collectors for DNA molecules."

Martin has over 40 peer-reviewed papers to his name and stands up for other industrial researchers on page 17.

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Antipodean Analytical Adventure

Inspired by the individuals on The Power List? Jump-start your post-graduate career at a new Australian center focused on portable analysis

The University of Tasmania's Emily Hilder features in this year's Power List – she also appeared on the 2013 Power List of the Top 100 most influential analytical scientists. Here, Emily shares information on an ambitious new collaborative project – and outlines who she's looking to work with.

What?

The ARC Training Centre for Portable Analytical Separation Technologies (ASTech) is a recently established center funded under the Australian Research Council's (ARC) Industrial Transformation Research Program (ITRP) and is a partnership between the University of Tasmania and Trajan Scientific and Medical.

Why?

We will explore science and technologies that can lead to portable analytical separation systems for point-of-sample analysis of complex samples in food, environmental, and clinical applications – ultimately bringing 'the lab' to the sample. ASTech is also about research training, in particular coaching the next generation of industry-ready researchers.

Where?

ASTech is primarily located at the University of Tasmania (Hobart, Australia), also home to the Australian Centre for Research on Separation Science (ACROSS, www.utas.edu.au/across). Research will also be undertaken at Trajan's site in Australia (Ringwood, Victoria), and with Trajan and its partners internationally.

Who?

We are looking for candidates with a background in science or engineering who have an interest in analytical science and are motivated to see the results of their research lead to new commercial product developments. There are positions for ten PhD candidates and three postdoctoral fellows. All candidates will be working in close collaboration with industry, including at least a one-year industry placement.

When?

Prospective candidates should be available to start by February 28, 2015. Applications for postdoctoral fellows will close on November 4, 2014. We will begin assessing applications for PhD candidates from 31 October 2014, but don't treat this as a deadline; we will accept applications for PhD candidates until all positions are filled.

How?

Visit www.astech.org.au for details or email: emily.hilder@utas.edu.au

Bloodstain Spectrums

How to put instant blood identification and age estimation into the hands of forensic investigators

A group of UK researchers were overwhelmed (and "ecstatic") when their work using visible wavelength hyperspectral imaging to identify, and estimate the age of, blood stains was highlighted in Parliament as a "great example of scientific research that could have a significant impact on crime scene investigation". Meez Islam and his team at Teesside University are hoping to create a portable hyperspectral device that can be used on location at crime scenes and in forensic labs (1).

"Techniques already exist for identifying blood stains, but accurately estimating the age of the stain is one of the 'holy grails' of forensic science and no validated method exists to achieve this," says Islam. "A number of elaborate techniques have been tried previously, but the method we're investigating is extremely simple; it's based on the fact that fresh blood is bright red and older stains are darker. We look at this spectroscopic change in more detail to estimate its age."

Specifically, Islam says that the red color of haemoglobin is caused by the absorption of light around 415 nm (the Soret band absorption), which is sharper than that for other red substances. Statistical correlation can be used to compare the spectrum of a suspect bloodstain against a reference spectrum.

Hyperspectral imaging was originally developed for satellite imaging, and Islam claims his team is among the first to use the technology in the forensics industry. The project is also Islam's first foray into forensics, which came about by chance when he was speaking with a biochemist colleague (Liam O'Hare) and a crime scene science colleague (Peter Beveridge) from the university's forensic science department. "We asked him what kind of technology he would like for a crime scene. He said it would be great to immediately identify various substances, like blood, explosives, bodily fluids, and so on. In my naivety I said, 'But surely you can do all that already with spectroscopy?'"

Apparently not. So Islam and his colleagues decided to build something. At first he had big ambitions involving a robot that scans the whole room with spectrometers… "But of course that was a little too grandiose to begin with," he says. "We scaled the whole project back and decided just to focus on blood. Having said that, the technique does have the potential to go beyond blood; it could be applied to other bodily fluids or drugs, and we plan to try this in the future."

Going back to blood stains, the team understands much of the science and knows what is needed to convert the prototype lab instrument into a portable, robust and reliable commercial device. To that end, Islam and his colleagues have established a spin-out company called Chemicam to try and bring the technology to the market. However, there's still work to be done: "Age estimation has so far only been performed under controlled conditions. To get it to work at real life crime scenes, we'll need to take into account environmental variables," says Islam, "and we think we know how to model the spectroscopic change for this. In the end, we want one instrument that both detects a blood stain and also estimates its age."

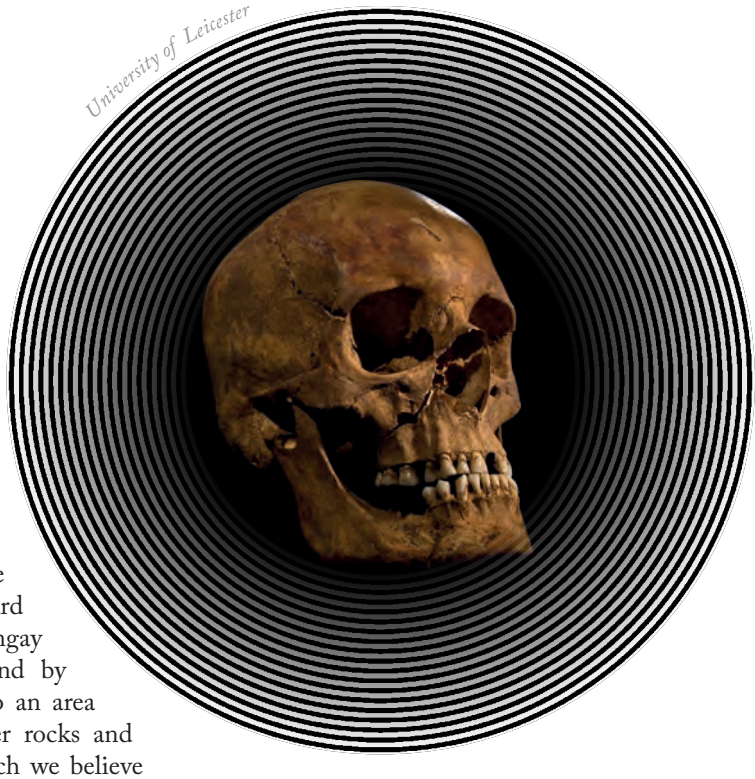
It was the applied nature of the project that caught the eye of UK politician Ian Swales, who is hopeful it will one day be used by police forces around the country. Islam concludes, "If this problem can be solved, then it could have a massive impact on the criminal justice system." *SS*

Reference

1. *Meez Islam et al., "The Detection and Age Estimation of Bloodstains at Crime Scenes Using Visible Wavelength Hyperspectral Imaging", Evidence Technol. Mag. 12 (1), 12–14 (2014).*

Reconstructing the King's Tale

Multi-isotope analysis uncovers the life story of King Richard III – and, for the first time, links wine intake to oxygen isotope composition



Avid readers of The Analytical Scientist will know that we've been following the story of King Richard III for a while. From the identification of the remains found in Greyfriars Friary Church in Leicester using DNA analysis to the use of radiocarbon dating to shed light on his eating habits. The researchers – based at the British Geological Survey and the University of Leicester – have recently published the results of the full multi-isotope analysis (1). Angela Lamb, an isotope geochemist with the British Geological Survey and lead author of the paper, tells us more.

What are the latest findings?

The early isotope details came from data generated from accelerator mass spectrometry radiocarbon dating of the rib bone of Richard III, which gives an average picture of the last few years of his life. We wanted to analyze several different locations on the skeleton so that we could piece together a more detailed life history.

We sequentially analyzed bioapatite and collagen from a second premolar tooth with its root intact, so that we could reconstruct Richard's childhood and early adolescence. This is possible because oxygen and strontium isotopes are fixed in enamel biogenic phosphate at the time of tooth formation, and once fixed will not change during life. Bone, however, regenerates through life, which means that the isotopes reflect average conditions over time. As bone remodels at different rates, we chose to sample the femur (which averages conditions over the last 10-15 years of life), and the rib (which remodels faster and represents the last few years of life). By combining the data from these three locations, we were able to reconstruct Richard's changing diet and location.

Our analyses of the tooth showed that Richard had moved from Fotheringay Castle in eastern England by the time he was seven to an area with higher rainfall, older rocks and with a changed diet, which we believe is relative to his place of birth in Northamptonshire. From the femur, we could tell that Richard moved back to eastern England when he was a young adult, and had a diet that matched the highest aristocracy.

How were you able to gain such specific details?

Some parts of Richard's life are well documented, including his birthplace and whereabouts in the latter part of his life, so we were able to compare our results to these facts. There is a good match, which was very encouraging and gave us the confidence to extrapolate the data to the periods we know less about. Isotope analysis can't tell you exactly where he was living, but by combining oxygen and strontium analysis we can suggest potential areas of the country. Similarly, diet-based isotope reconstructions can inform us about the amount of protein consumed and the types of protein, such as freshwater or marine fish.

His rib oxygen isotope value is too high for someone living in eastern England, where he was known to be during the last few years of his life. Given that the carbon and nitrogen isotopes in his rib bone also suggest that he was eating more luxury foods, such as wildfowl and freshwater fish, we suggested that the high value could represent an increase in wine consumption too.

Are further analyses planned?

We are not planning any further analysis on Richard III at the moment, but it has raised some questions we'd like to explore further. To our knowledge, this is the first example where the intake of wine has been suggested as having an impact on the oxygen isotope composition of an individual. Thus we'd like to follow this up with more work. We also hope this paves the way for more detailed multi-isotope and multi-tissue reconstructions in the future.

Read more about the analyses of King Richard III:
Winter of Content (tas.txp.to/1014/Winter)
A Meal Fit for a King (tas.txp.to/1014/Meal)

Reference

1. *Angela Lamb et al., "Multi-Isotope Analysis Demonstrates Significant Lifestyle Changes in King Richard III", Journal of Archaeological Science 50, 559-565 (2014).*

Non-invasive Prostate Cancer Screening

Can SERS accurately detect the early stages of the most common male cancer?

The US Preventative Services Task Force now recommends against the use of prostate specific antigen (PSA)-based screening because of the potential for over diagnosis. Hoping to fill the gap, researchers in China have combined surface-enhanced Raman scattering (SERS) with support vector machine (SVM) algorithms to develop a non-invasive diagnostic test for prostate cancer (1).

The technique developed by Shaoxin Li and his team at Guangdong Medical College involves mixing the subject's blood serum with silver nanoparticles ahead of SERS. The resultant spectrum is then processed using an SVM classifier model. Other research teams have experimented with SERS in similar applications, but the signals of prostate cancer have typically been too subtle to accurately detect.

The SVM classifier models developed by Li's team were able to distinguish between the SERS spectra of the 68 cancer patients and 93 healthy volunteers involved in the study with an accuracy of 98.1 percent, compared with 91.3 percent for principle component analysis methods, which the researchers used to assess the performance of the SVM algorithms.

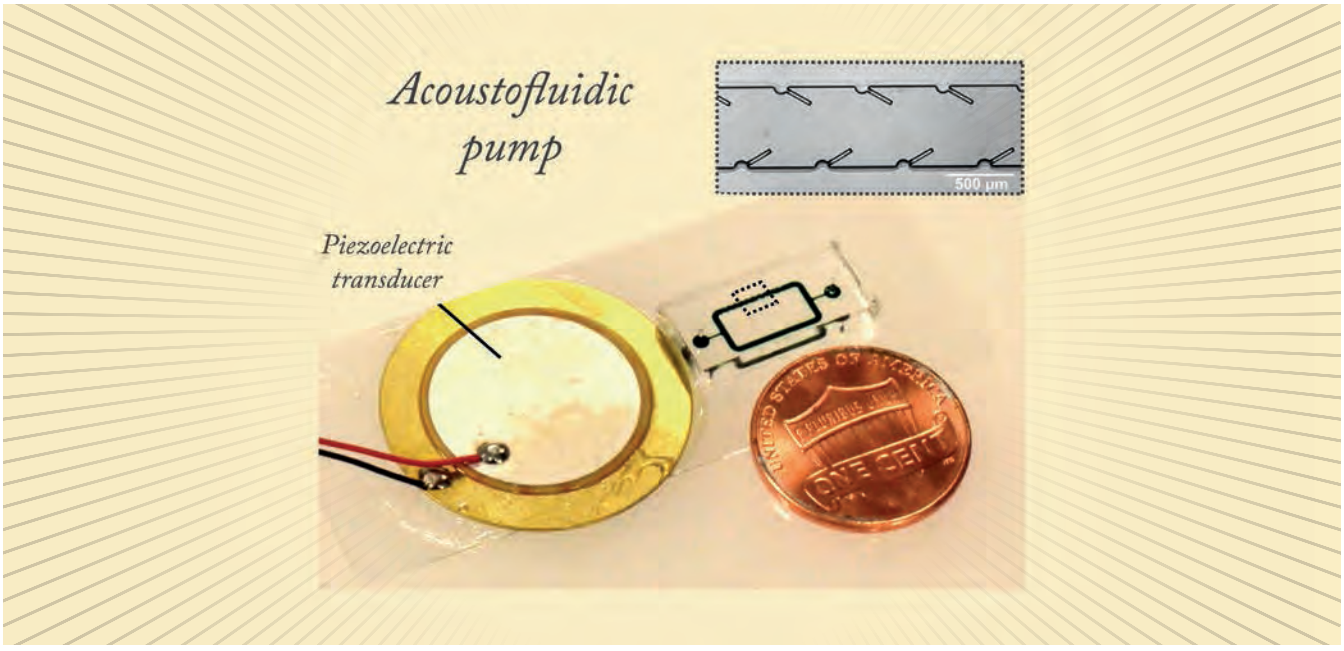
"The fundamental idea of SVM involves separating classes with the optimal hyperplane, which maximizes the margin of separation between the hyperplane and the closest data points on both sides. SVM has been successfully applied in various applications including face recognition, text categorization and gene selection, and could also be applied to other spectroscopic studies," says Li. "This study demonstrates that label-free serum SERS analysis technique combined with SVM diagnostic algorithm has great potential for noninvasive prostate cancer screening."

The technique only requires 10 µl of serum to perform the measurement, and can be used conveniently and repeatedly for high-risk patients. Li says it's also a very simple process. "Perhaps one of the main challenges of the work was adopting appropriate SERS-active nanoparticles, which is important because the reproducibility of the Raman scattering signals is related to their homogeneity, stability, biocompatibility and enhanced capability," explains Li. "To build the classifier models for SVM, we collected serum samples with a rich diversity."

The team has not yet performed direct comparisons with other screening methods. The next goal of the work will be to develop a study plan for SERS and SVM to explore other cancer types and cancer staging. *SS*

Reference

1. *Shaoxin Li et al., "Noninvasive Prostate Cancer Screening Based on Serum SERS and SVM", Applied Physics Letters, 105 (9), DOI: 10.1063/1.4892667 (2014).*



Biomimetic Micropump

Could a new acoustic micropump inspired by the legs of swimmers open doors for microfluidics?

The potential of microfluidics and lab-on-a-chip technology is clear, but real-world applications have been slow to emerge. A lack of effective and inexpensive micropump options has been one challenge slowing the pace of development. Now, researchers from Penn State University believe they have the answer: an acoustofluidic pump, powered by a piezoelectric transducer (1). The pump uses acoustic waves to deliver fluids and, because of its low power consumption, it could be easily integrated into cell phone-based point-of-care diagnostic systems.

"Swimmers were our inspiration," says Tony Huang who led the team. "When we swim, we have to keep our legs kicking, which exerts a force on the water. The water, simultaneously, exerts a force back, making our body swim forward. This is the motion of Newton's third law. In this case, if we keep our upper body stationary, while consistently kicking, there must be a net force exerted on water to push the flow backward. We started to think about whether we could use previously proposed sharp-edge oscillation to realize fluid propulsion motion."

Indeed, the pump works by acoustically oscillating solid, tilted sharp-edge structures that are constructed onto the sidewall of the microfluidic channel. Upon oscillation, these structures generate acoustic streaming effects, which in turn generate net forces in the direction that the sharp-edge structured oriented. While the idea is relatively simple and the fabrication is relatively easy, the sharp-edge structures are constructed in a silicon mold using deep reaction ion etching, which could be a challenge in terms of mass production.

However, Huang believes the effort is worthwhile as the technology offers several advantages over other micropumps. "Active, on-demand control means that the pump works whenever we want it to," says Huang. "It can also generate a range of pumping flow rates, from nanoliters to microliters per minute; and the performance can be adjusted by changing the numbers of sharp-edge structures and the voltages applied to the piezoelectric transducer. Perhaps most special of all, it can achieve various kinds of flow operations by programming the input signals to the piezoelectric transducers; the pulsed pumping flow, for instance, can be used to mimic cardiac flow in microfluidic chip to perform cardiovascular-related studies."

Reference

1. *Po-Hsun Huang et al., "A Reliable and Programmable Acoustofluidic Pump Powered by Oscillating Sharp-Edge Structures", Lab-on-a Chip, DOI: 10.1039/C4LC00806E (2014).*

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In My View

In this opinion section, experts from across the world share a single strongly-held view or key idea.

Submissions are welcome. Articles should be short, focused, personal and passionate, and may deal with any aspect of analytical science. They can be up to 600 words in length and written in the first person.

Contact the editors at edit@texerepublishing.com

The Art of Analysis, Reprised

"During human progress, every science is evolved out of its corresponding art," wrote Herbert Spencer in 1861. So, is analytical chemistry truly as much an art form as a science?

By Anne Francoise Aubry

In the career pages of the American Chemical Society, analytical chemistry is described as the "art and science of determining what matter is and how much of it exists". Of the six disciplines of chemistry, it is the only one to be described with these words, the other five being defined as "the study of" [a particular type of molecule or material]. "Art" can be perceived as a derogatory term in the context of science but it should not be. Art represents savoir-faire and experience – the clever ways learned over time in the practice of one's craft. Art precedes science; and it is in attempting to explain experimental results that theories are being developed. Yet, this may be symptomatic of the image of analytical sciences in the scientific community at large.

I think the root of the problem is that analytical chemistry has gone from mostly solution chemistry to almost exclusively instrumental analysis. Many scientists in all fields now practice analytical chemistry. Some are so good at it that they become full-time analytical chemists. Instrumental analysis has made it possible for non-specialists to purchase and use the sophisticated analytical instruments of the modern analytical laboratory, leading to the impression that analytical chemistry is just an enabler of other sciences. If everyone practices analytical chemistry, what makes an analytical scientist? Just like a skilled craftsman produces a more perfect object than a neophyte, using the same tool, a trained analytical chemist is able to get more from their instrument – more sensitivity, or higher resolution, or better precision. The art and science of analysis is also about choosing the right technique to solve a particular problem – and how to combine techniques in new ways to achieve the desired result.

Many years ago, while working on a new formulation of a drug in development, we found that one excipient was eluting as a "blob" in the chromatographic impurity test, obliterating the drug peak and all impurities and degradation products. I will never forget the look of disbelief on the face of the formulator (who thought himself pretty knowledgeable in chromatography), when I showed him a clean chromatogram the following week; the excipient was nicely tucked out of the way and the impurity profile clearly visible. I had theorized what the root of the problem was and how to solve it (the science) and used a new chromatographic column that I thought would fix the

"Many great inventions have been made possible by an advance in the analytical sciences."

problem (the art). And indeed it did.

This is but one simple example of what goes on every day in analytical laboratories around the world. Many great inventions have been made possible by an advance in the analytical sciences. One only has to remember the discovery of the double helix structure of DNA, made evident by the picture of a "fuzzy X" on Rosalind Franklin's X-ray diffraction image. Further technical development allowed scientists, several decades later, to photograph DNA fibers directly using transmission electron microscopy (1). The value of analytical research is only fully realized once it has been applied to a real-life problem. Analytical chemists love these challenges that test the limits of their art, lead to new discoveries, and ultimately advance analytical and other sciences.

Anne Francoise Aubry is the 2014 president of the Eastern Analytical Symposium and Exposition in Somerset, NJ, USA. The 2014 symposium will be held on November 17–19. The theme: "The Art and Science of Analysis." www.eas.org

Reference

1. *F. Gentile et al., "Direct Imaging of DNA Fibers: The Visage of Double Helix", Nano Lett., 12 (12), 6453–6458 (2012).*

Recognizing Research in Industry

There are more researchers working in industry than in academic facilities – and, in many ways, they are shaping the future more profoundly than their academic colleagues. It's time to push them into the limelight.

By Martin Gilar, principal investigator, Waters Corporation, Milford, MA, USA.

Industrial scientists develop therapeutics, mobile phones and analytical instruments, yet they remain mostly unknown to their colleagues and the general public. It's a paradox with a simple explanation: they are not motivated to publish their research. Their funding does not depend on grants; their salary or promotion is not based on citation factors; and, of course, they do not intend to apply for tenure. In reality, many industrial researchers are positively discouraged from discussing discoveries in public because companies do not want to tip off competitors.

Yes, industrial researchers apply for patents and sometimes present their data at scientific conferences, but very few choose to fight corporate guidelines and reviewers to publish their discoveries in peer-reviewed journals. The late Uwe Neue was a rare breed among researchers. Although he spent his entire career in the industry, he wrote numerous papers that earned him the respect of both industrial and academic colleagues.

When I joined Waters Corporation, Uwe was directing an applied technology group. As a young researcher, I often sought the advice of more seasoned colleagues on various aspects of chromatography. I quickly found out that Uwe was approachable and knowledgeable. The topic of my interest did not matter, as he was familiar with seemingly any field of separation science. Thanks to his knowledge of the literature and years of experience, his advice guided me in many projects. He was not always right – just most of the time. But that was more that one can hope for in a research environment, where the solutions to problems are, by definition, unknown.

Very sadly, Uwe died in 2010 after succumbing to a serious disease. The remaining unanswered questions I must solve alone – though perhaps with the help of Uwe's papers (still being posthumously published). At Waters, we also deliberated over another question: how do we honor Uwe's legacy? Many of you will know that Waters decided to sponsor an award dedicated to him; the Uwe D. Neue Award in Separation Science is now given annually at the HPLC symposium series. In the spirit of Uwe Neue, the award recognizes an industrial scientist, preferably 15-20 or more years after receiving his or her doctoral degree, who has made a significant contribution to the field of separation science and continues to advance it.

The first award was presented at HPLC 2013 in Amsterdam to Jack Kirkland; the second to Gerard Rozing at HPLC 2014 in New Orleans. Who will be the recipient of the 2015 award in Geneva? In many ways, that is up to you…

Nominations are open until November 30, 2014. To recognize a contribution of an outstanding industrial researcher, include a primary letter from the original nominator and a supporting document from a second person. For more information, go to www.waters.com. Please send nominations to Martin_Gilar@waters.com.

Late to the Flipping Party

Christopher Harrison described the flipped classroom as an inevitable evolution in teaching. And he's right – but why has it taken so long for university lecturers to catch on?

By Olaf de Groot, senior consultant, Stichting APS, Utrecht Area, The Netherlands.

As an expert in implementing technology in education, I've known for quite some time that using lecture videos positively shifts the way teachers organize their lessons – in a direction that reflects the way students like to learn. Which is why it strikes me as odd that a lecturer – whose job it is to engage students – has only just discovered the use of lecture videos. I'm not pointing the finger at Harrison in particular. In fact, I applaud him for going out on a limb and trying it!

As a former primary school teacher, I know that the use of technology is not taught at 'teacher school' – at least not extensively – and the same is true for university lecturers. I believe that the desire to incorporate technology into your primary process in the classroom has to be in your DNA. Maybe this is the reason that most teachers and lecturers don't use lecture videos routinely.

Of course, the need to carefully choose the right pedagogical approach never changes; a video isn't always appropriate, especially for subjects that are better taught face–to-face. However, as University of Washington principal biology lecturer Scott Freeman states, "We've got to stop killing student performance and interest in science by lecturing and instead help them think like scientists," (1) – which is exactly my point. Using all of your time giving lectures is a waste of your students' time as well as your own. Freeman's statement is not baseless. He is lead author of a paper that compares lecturing with active learning by looking at 225 studies of undergraduate education across science, technology, engineering and mathematics (2).

For me, it's clear that we must provide active learning to our students. And I think that you will agree that an instructional video or pre-recorded lecture isn't the complete solution. What it gives you is more time with your students – and that's the key.

It's true that not all teachers have the skills and vision of Steven Spielberg, so the desire to create a "perfect" video can be a big first hurdle. But as Jonathan Bergman – one of the first teachers to call the use of video "flipping the classroom" – pointed out: less-than-perfect videos actually work better than masterpieces in many cases (3). Indeed, according to Donald Clark, poor quality video is rarely a problem when it comes to learning and retention; bad audio, on the other hand can be crippling (4). In other words, you just need decent equipment.

Besides quality audio, there are some other "rules" to a good video:

i) It cannot be too long – not longer than six minutes according to research done by Philip Guo (5).
ii) Video with accompanying text is a no-no. And never put the script up at the same time as the video; it overloads working memory and damages learning. Richard Mayer suggests that both a visual and a narrative description lead to measurably lower retention (6).
iii) Aim for YouTube not an Oscar. Students prefer a more informal, personal and – above all – enthusiastic performance from teachers. Hesitations, a relaxed style, and even corrected errors are perfectly acceptable.

You don't need to be a complete whiz kid. You just need to master one tool to record and edit your videos well. No doubt, this will take time – but so did becoming a teacher and learning how to create good tests…

The greatest challenge is to rethink your class time. How can you promote active learning for your students? In many ways, that should always be the primary objective of a teacher. I don't have all the answers, but I do know that creating a broad personal learning network (for example, following the blogs of colleagues, teachers, and though leaders) and keeping track of new insights on learning and technology can help. Perhaps most importantly, you need to discover what motivates and excites your students. It could be a rough ride, but an interesting journey. Isn't teaching always like a rollercoaster anyway?

There is no single route to the best flipped classroom; it has to fit you as a teacher. One thing is clear: you cannot continue as you have in years past. So, come on – follow Harrison's good example!

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A Recipe for Disaster?

Incorrect labeling of foods – sometimes a result of inappropriate or incorrect analysis – can cause needless suffering for people with food allergies. We analytical scientists must step up and change the landscape of food allergen analysis.

By Bert Pöpping

It was apparently the philosopher Lucretius – who lived more than 2,100 years ago – who said, "What is food to one, is to others bitter poison." Certainly, it would be a significant challenge to establish if Lucretius had a sound understanding of allergens back then, but his quote is as true today as it was millennia ago. In fact, the current big issue with food allergies is not so much with labeled ingredients, but those ingredients that enter into the food inadvertently or through fraudulent activity.

Food manufacturers are typically responsible for a range of products with many different recipes, which means that allergens can inadvertently enter products that, according to their ingredients list, do not contain allergenic components. After all, cleaning of product lines is not always 100 percent efficient. You may have seen the label: "produced in a facility that processes X, Y and Z." In other cases, a more expensive ingredient might be replaced – without an eye for quality, safety, or correct labeling – by a cheaper one. The Food Allergen Resource and Research Programme (FARRP) shared one incident where pine nuts in a pesto were replaced by peanuts – it's a recipe for disaster.

Clearly, when final products are not labeled as containing allergens, they may consequently cause serious harm. The only way to detect (and potentially quantify) the presence of undeclared allergens is by using analytical tools, which can be grouped into three main areas:

"Kit and equipment manufacturers often proudly bamboozle us with a myriad of data that demonstrate the supposed superiority of their assay."

i) DNA-based methods; for example, polymerase chain reaction (PCR)
ii) Immunological methods; for example, enzyme-linked immuno sorbent assay (ELISA) and lateral flow devices (LFD)
iii) Mass spectrometric assays.

Although all of these techniques tend to work well with unprocessed or lightly processed materials, such as flour, it is more difficult to obtain accurate results with many processed materials. Why? Because the allergens either degrade or react with the matrix and cannot be recovered by extraction. Unfortunately, these allergens may still trigger reactions.

For many processed products, significantly different results can be obtained even within a group of methods. In PCR, target sequences may be single or multi copy, and the polymerase may be more or less efficient. In ELISA, antibodies – and subsequent specificity – can differ, as can extraction methods. Efficient extraction is essential when it comes to processed foods potentially containing egg and/or milk., but all too often assays fail to detect even high quantities of allergens. It is here that mass spectrometric techniques appear to have a significant advantage. Because they work with small, digested peptide fragments, allergens can be detected even in highly processed samples.

For analytical scientists using any of the methods, it is important to look very closely at the validation data – that is to say, what has been validated and how. Is it highly or lightly processed food? Were materials spiked or are the samples incurred? This information allows the analyst to make the best possible choice from the set of available methods; some methods only work with (or have been validated for) raw materials, while others may not be applicable because there is little or no target analyte (for example, aiming to detect chicken DNA in egg white).

Kit and equipment manufacturers often proudly bamboozle us with a myriad of data that demonstrate the supposed superiority of their assay. Unfortunately, in some cases, quantity of data is mistaken for quality. Any data provided should be objectively scrutinized, taking known issues of any of the applicable methods into account.

By now, there are already several European technical specifications (TSs) available for food allergen

detection, some of which are based on proprietary assays. While TSs are useful in the absence of any other standardized method, it is much more desirable to develop method performance criteria (MPCs). MPCs are typically derived from a validation study of one or two methods in the particular field (for example, allergen analysis), and include parameters like sensitivity, selectivity, and robustness. However, once the MPCs have been deducted from the validation study, they are no longer tied to those methods, becoming stand-alone criteria. This has the significant advantage that any new method that meets those MPCs can be used. Such an approach drives the development of new, potentially better methods without the need to use older (potentially outdated) standards or technical specifications.

"For analytical scientists using any of the methods, it is important to look very closely at the validation data."

In fact, the approach has already been in place for some contaminant and residue methods, where European regulations set MPCs (European Commission Decision 2002/657/EC).

In the field of allergen analysis, there have been very encouraging new method developments in the last few years, especially in mass spectrometry, which raises my hopes for a robust and accurate methodology in the not too distant future. This will benefit food manufacturers and, ultimately, the allergic consumer.

Current existing methods are certainly not a recipe for disaster, but it is important for analysts and food manufacturers to understand the limitations of existing methodologies. Both parties should take suitable measures, using appropriate information on test certificates and appropriate product labeling, to prevent adverse health effects caused by inadvertent consumption.

NEVER MIND THE BACKLASH

HERE'S THE

Power List

In 2013, we compiled a list of the Top 100 most influential analytical scientists – an endeavor that received a significant amount of attention and, it's fair to say, caused more than a little controversy. Our 2014 Top 40 Under 40 list almost halves the average age of the celebrated individuals, with many of them born in the same year as the iconic Sex Pistols cover – 1977. Though we admit that no list of this type can be definitive, it does clearly show that the future of our field is very bright indeed. And so, without further ado, we present (in alphabetical order) the analytical movers and shakers under 40, who prove what can be achieved with determination, passion – and inspiration.

Jordi Arbiol

ICREA Research Professor, Group of Advanced Electron Nanoscopy (GAeN), Institut de Ciencia de Materials de Barcelona (ICMAB-CSIC), Bellaterra, Catalonia, Spain.

Calling: "I've always dreamed of being a scientist – exploring the limits of the Universe. Now, I explore the limits of matter at the atomic scale."
Hope: "Based on transmission electron microscopy, I hope to be able to map and distinguish isotopes at atomic scale or map phonons and photons in a 3D reconstruction."

Fernando Benavente

Associate Professor, Department of Analytical Chemistry, Faculty of Chemistry, University of Barcelona, Spain.

Emphasis: "I am focused on improving sample preparation, separation and characterization methods for the analysis of peptides, proteins and glycoproteins in complex samples."
Prediction: "High performance microscale and nanoscale separation techniques will be game changers, because we will be able to solve all sensitivity and reproducibility issues."

Michael Breadmore

Professor, Australian Centre for Research on Separation Science (ACROSS), School of Physical Science, University of Tasmania, Hobart, Tasmania, Australia.

Emphasis: "Simplifying sample preparation to make micro total analytical systems (μTAS) through the use of electrokinetic and hydrodynamic phenomena."
Hope: "To see widespread uptake of electrophoresis in miniaturized analytical devices."
Prediction: "Additive manufacturing will completely change what we can do in microfluidics."

Jared Anderson

Professor, Department of Chemistry and Biochemistry, The University of Toledo, OH, USA.

Calling: "I always viewed a career as a chemistry professor to be the ultimate dream job! You are able to combine passions for both research and teaching while also training new generations of analytical scientists."
Emphasis: "I am interested in developing sample preparation and separation methods using ionic liquids, polymeric ionic liquids, and magnetic ionic liquids."
Triumph: "The graduate students and other young scientists who have been trained in my laboratory constitute my biggest professional achievement."
Hope: "First, to develop bioanalytical sample preparation methods focused on overcoming challenges currently encountered within the life sciences and the pharmaceutical industry. Second, to improve our understanding of ionic liquids in separation science."
Advice: "Always believe in yourself and your ideas. Surround yourself with other motivated scientists (students, collaborators, colleagues) with whom you can share ideas and discuss science."

Ken Broeckhoven

Assistant Professor, Department of Chemical Engineering, Vrije Universiteit Brussel (VUB), Brussels, Belgium.

Respect: "Uwe Neue for his contributions to chromatography and his ability to provide critical, but constructive feedback. And Gert Desmet, who instilled in me a love for the field and taught me so much regarding the theory of chromatography."
Advice: "Never forget the fundamentals! If your results appear odd, investigate; the results you don't expect are often the most interesting."

Murthy Chavali

Professor and Research Coordinator, Department of Chemistry, Vignans Foundation for Science Technology and Research, Guntur, AP, India.

Calling: "My interest in nano chemistry was inspired by the discovery of Fullerenes in 1985. Since then, I have slowly moved myself into the nano world."
Emphasis: "Analytical chemistry, nanochemistry, nanomaterials, nanosensors, nanoelectronics, nanoantennas."
Respect: "Jack Kirkland and Joseph Wang."

Livia Eberlin

Postdoctoral Scholar, Department of Chemistry, Stanford University, CA, USA.

Calling: "I am passionate about research at the interface of chemistry and medicine. Through the development of novel mass spectrometry techniques, I believe cancer diagnosis and treatment will be significantly improved."
Triumph: "I received the 2012 ACS Nobel Laureate Signature Award for Graduate Education, given to the best doctoral thesis in Chemistry."
Respect: "Graham Cooks and Richard N. Zare."

Bernd Bodenmiller

Assistant Professor, Institute of Molecular Life Sciences, University of Zurich, Switzerland.

Calling: "I was always fascinated by how cells sense and process information, but also by bleeding edge analytical methods. So I decided to develop the latter to study the former."
Regret: "No. I would do all of it again. I feel very lucky that everything worked out as it did."
Emphasis: "We develop mass cytometry based and computational methods to better understand tumor biology, especially how cell-to-cell communication drives cancer metastasis."
Triumph: "The development of imaging mass cytometry. This technology allows simultaneous visualization of 120 biomarkers in (tumor) tissues with subcellular resolution."
Hope: "I hope that single cell analysis methods in general will improve our understanding of human disease and will ultimately improve patient classification, treatment and care."
Prediction: "Combining live cell analysis with comprehensive measurements of transcriptome, metabolome and signaling networks could change how we study biological systems."
Advice: "Try to find a topic you are really, really excited about, then everything else will happen automatically."

James Edwards

Assistant Professor, Department of Chemistry, Saint Louis University, St Louis, MO, USA.

Calling: "I've always loved solving challenging problems. Therefore, analytical chemistry was a natural fit for me. Developing metabolomic technologies for investigating diabetic complications holds a personal interest as I have juvenile -onset diabetes."
Predicton: "Portable mass spectrometry use will be expanded to surgery rooms and onsite forensic/pharmaceutical/food analyses."

Michael Fogwill

Senior Research Chemist, Waters Corporation, Milford, MA, USA.

Calling: "I have a passion for science, an innate drive to understand, and I enjoy creating and building things."
Prediction: "The paradigm shift toward operator ease-of-use when developing future analytical instrumentation will, for example, move medical testing from the laboratory to the exam room."
Advice: "Throughout your career, there will be failures – so embrace them, learn from your mistakes, and move forward. Remember to appreciate and enjoy the process and not to simply focus on the end result."

Helen Gika

Lecturer, Department of Chemical Engineering, Aristotle University, Thessaloniki, Greece.

Calling: "Disease biomarker discovery saves lives and improves the quality of life of millions. I choose to work with the most fragile newborn babies, which have limited means to communicate."
Respect: "Alexander Makarov and Janusz Pawliszyn"
Prediction: "Miniaturization in LC-MS, though it may need more than five years to mature…"

Szabolcs Fekete

Scientific Collaborator, Analytical Pharmaceutical Chemistry, School of Pharmaceutical Sciences, University of Geneva, Switzerland.

Emphasis: "Analysis of therapeutic proteins, fundamental research in LC, SFC, column technology and method development."
Respect: "Pat Sandra and J. Calvin Giddings."
Prediction: "The pressure capability of modern SFC systems will be significantly extended and extra-column volumes should drastically be reduced, pushing this technique into a new level."

Isabelle Francois

UPC²/SFC & Strategic Separation Technologies Business Development Manager for Europe and India, Waters Corporation, Saint-Quentin, France.

Calling: "My interest in science and continuous 'need to understand' inspired me to commence my PhD research. The next step was to utilize that knowledge in real-life applications for a commercial organization rather than to focus on fundamentals in an academic environment."
Advice: "Dream big, work hard and never give up."

David Giljohann

Chief Executive Officer, AuraSense Therapeutics LLC, Skokie, IL, USA.

Emphasis: "We are developing biofunctionalized nanoparticles for gene regulation and detection. Our "Smart-Flares" are now being sold commercially by Merck-Millipore."
Advice: "Commercializing technology is a rush. There is tremendous feeling of accomplishment to see a project turn into a product you can buy."

Rachel Gomes

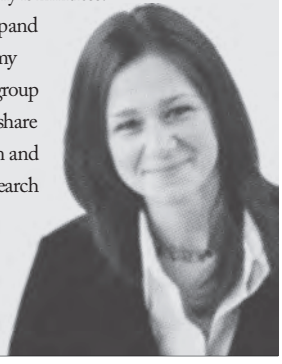
Assistant Professor, Chemical and Environmental Engineering, Manufacturing and Process Technologies Research Division, University of Nottingham, UK.

Calling: "Curiosity chose my career. Driven by 1990s news articles about chemicals in water feminizing fish. Now, I not only explore solutions to societal issues, but also share my curiosity through teaching."
Prediction: "Advances in analytical equipment and utilization for trace analysis of chemical metabolites to inform on wastewater process understanding."

Deirdre Cabooter

Assistant Professor, Pharmaceutical Analysis, Department of Pharmaceutical and Pharmacological Sciences, University of Leuven, Belgium.

Calling: "I've been hooked on the diversity, challenges and possibilities of research since my master's degree and have wanted to pursue a career in research ever since."
Triumph: "Being a research professor at KU Leuven: a prestigious position with reduced teaching load that allows me to focus on research."
Respect: Gert Desmet is my mentor, a former promoter and a friend, who introduced me into the world of chromatography with all his enthusiasm. He continues to inspire me. And Pat Sandra who further aroused my interest in chromatography. His knowledge of chromatography is limitless."
Hopes: "To expand and establish my own research group wherein I can share my enthusiasm and passion for research with young researchers."



John Hanrahan

Chief Technical Officer and Co-founder, Glantreo Ltd, Cork City, Ireland.

Triumph: "In science: co-developing the Titan Range of HPLC columns. In business: starting and growing an R&D focused company."
Respect: "J. J. Kirkland (Founder and Owner of AMT Wilmington DE) and Fasha Mahjoor (CEO of Phenomenex)."
Prediction: "Chromatography columns packed/manufactured with something other than small spherical porous silica particles!"

Margaret Hardy

Postdoctoral Research Fellow, Division of Chemistry and Structural Biology, Institute for Molecular Bioscience, The University of Queensland, Brisbane, Australia.

Emphasis: "My research is focused on the discovery and characterization of new 'green' insecticides from the venom of Australian spiders. These compounds are specific for insects, and may have novel mechanisms."
Advice: "First, recognize that innovation is central to good science. Second, cultivate other 'soft' skills that can help with funding applications."

James Harynuk

Associate Professor, Department of Chemistry, University of Alberta, Edmonton, Canada.

Calling: "Chemistry is fascinating – everything is chemistry, right? Why analytical and specifically GC×GC? You can't beat the fun of building tools to probe chemical systems. The more complex the better."
Triumph: "My awesome kids. Followed closely by our contributions to GC and GC×GC modeling."
Respect: "Easy. Pat Sandra and Milton Lee."

Sebastiaan Eeltink

Associate Professor, Department of Chemical Engineering, Vrije Universiteit Brussel, Brussels, Belgium.

Calling: "I was lucky to meet the right people during my studies who provided me not only good advice but also gave me the opportunity to conduct a PhD study and post-doctoral research."
Emphasis: "My research aims at the design, development, and characterization of novel materials (including nanostructured monoliths and coatings in capillaries and microfluidic chips) and separation strategies for liquid chromatography."
Respect: James Jorgenson and Peter Schoenmakers
Hope: "To establish a dynamic research team and try to break new ground."
Prediction: "Spatial 3D chromatography: this is a new concept in which components are separated in the space domain with each peak being characterized by its X, Y, Z coordinates in the separation body. The technology potentially provides unparalleled performance in a short analysis time."
Advice: "Try to set-up good collaborations and share knowledge."

Christy Haynes

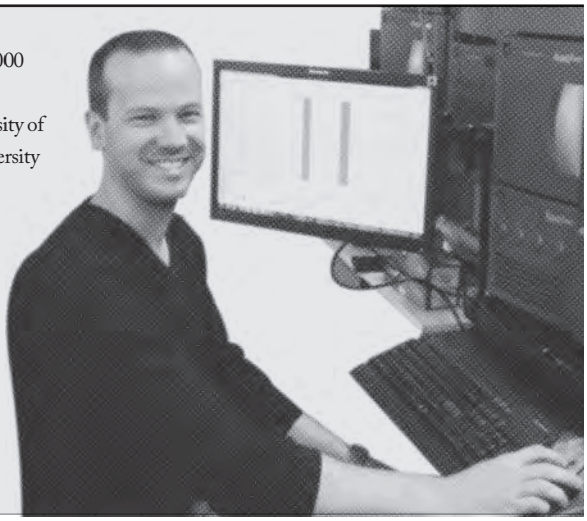
Professor, Department of Chemistry, University of Minnesota, Twin Cities, MN, USA.

Emphasis: "I'm excited about creating safe, sustainable nanomaterials and pushing the limits of analytical technology to understand critical biological processes."
Prediction: "Real-time, high spatial resolution in situ chemical analysis of nanoparticles in complex matrices."
Advice: "Work with people who challenge you and love science; be gracious in sharing credit. Focus on solving scientific problems that you care deeply about."

Davy Guillarme

Senior Lecturer, School of Pharmaceutical Sciences, University of Geneva/University of Lausanne, Geneva, Switzerland.

Calling: "I became interested in chromatography thanks to my highly motivated supervisor of master thesis and PhD in Lyon, France."
Emphasis: "The development of chromatographic techniques (including LC, UHPLC, SFC, HILIC) and their hyphenation with mass spectrometry for pharmaceutical analysis."
Triumph: "The development of a calculator for method transfer from HPLC to UHPLC that has been downloaded more than 11,000 times to date."
Respect: "Jean-Luc Veuthey (University of Geneva) and Sabine Heinisch (University of Lyon)."
Hope: "I would really like to expand my knowledge on the analytical characterization of biopharmaceuticals (monoclonal antibodies) as this is an important trend in the pharmaceutical industry."



Ingeborg Iping Petterson

Research Fellow, School of Physics, University of Exeter, Devon, UK.

Calling: "I was inspired by my parents who were both scientists, and by an enthusiastic professor who had a very engaging and interactive teaching style."
Emphasis: "Applied Raman spectroscopy in general – and my current research focuses on biomedical applications of this technique."
Prediction: "3D printers enabling the cheap construction of disposable, portable, analytical instruments."

Alexander Leitner

Senior Scientist/Researcher, Institute of Molecular Systems Biology, Department of Biology, ETH Zurich, Switzerland.

Emphasis: "The development of advanced mass spectrometric methods for studying protein structure, function and interactions; predominantly using chemical cross-linking."
Advice: "Do not expect your career path to be a straight line – and don't be afraid of interdisciplinary projects!"

Lísa Miroslav

Research Worker, Department of Analytical Chemistry, University of Pardubice, Pardubice, Czech Republic.

Triumph: "Development of several (U)HPLC/MS or SFC/MS methods for comprehensive characterization of the lipidome in biological samples."
Respect: "Pat Sandra and Alexander Makarov."
Prediction: "Supercritical fluid chromatography will play an important role in analytical science."

Emily Hilder

Professor of Chemistry; Director of ARC Training Centre for Portable Analytical Separation Technologies, School of Physical Sciences, University of Tasmania, Hobart, Australia.

Emphasis: "I'm driven by how we can use new materials to improve analytical systems and ultimately enable more portable separation science applications."
Advice: "Be opportunistic and don't be afraid to take risks. Trust your own ideas. Many breakthroughs come from young scientists."

Sergio Nanita

Principal Investigator, DuPont Crop Protection, Stine-Haskell Research Center, Newark, Delaware, USA.

Triumph: "Establishing academic-quality analytical research in industry that produced the infinite dilution method and faster pesticide residue analysis."
Respect: "My most influential mentor – R. Graham Cooks, and Jonathan Sweedler."
Prediction: "The coupling of smartphones and unmanned aerial vehicles to analytical chips and instrumentation… Sampling and analysis by anyone!"

Lucie Novakova

Associate Professor, Department of Analytical Chemistry, Faculty of Pharmacy, Charles University, Hradec Kralove, Czech Republic.

Respect: "Davy Guillarme and David V. McCalley."
Hope: "Getting rid of matrix effects and doing sample preparation quickly is my 'science-fiction' hope. Becoming an expert in the field of HRMS and ion mobility MS might be more realistic."
Prediction: "SFC will be a game-changer; we're already seeing new instrumental platforms and stationary phases."

Paola Picotti

Assistant Professor, Institute of Biochemistry, ETH Zurich, Zurich, Switzerland.

Triumph: "The development of a method to analyze protein structural changes directly in biological samples and on a large scale."
Hope: "To advance knowledge on protein misfolding and support the identification of therapeutic strategies for diseases."
Advice: "Follow only what interests you, entertains you, and tickles your curiosity. Expose yourself to different mindsets and scientific backgrounds."

Georgia Purcaro

Associate Professor, University of Udine, Udine, Italy.

Emphasis: "I am currently working on optimization of advanced chromatographic-mass spectrometric techniques to assess quality and authenticity in food."
Triumph: "The 2010 Leslie Ettre award, for the most innovative presentation made by a young scientist in the chromatography field."
Respect: "Luigi Mondello and Pat Sandra."

Herbert Oberacher

Associate Professor, Bioanalytical Mass Spectrometry Group, Institute of Legal Medicine and Core Facility Metabolomics, Innsbruck Medical University, Innsbruck, Austria.

Calling: "As an undergraduate, I had inspiring teachers who awakened my interest in analytical chemistry. After finishing my PhD, I had several offers and went for an academic career."
Emphasis: "With my group, I am developing workflows for the qualitative and quantitative analysis of (bio)organic molecules that find immediate application in life sciences"
Triumph: "The development of the first reliable, robust, and universal tandem mass spectral library dedicated for small molecular identification."
Respect: "Two "local heroes": Fritz Pregl won the Nobel Prize in Chemistry in 1923 for making important contributions to quantitative organic microanalysis, one of which was the improvement of the combustion train technique for elemental analysis. Klaus Biemann is a professor emeritus of chemistry at the Massachusetts Institute of Technology. Biemann's work was centered on structural analysis in organic and biochemistry. He has been called as one of the 'fathers of organic mass spectrometry.'"
Hope: "I hope that I will stay curious and excited. As I am bubbling over with ideas, I hope that I will get the chance to put them into practice."
Prediction: "I do not expect that one groundbreaking technology will appear within the next five years that will completely change bioanalytical chemistry as a whole. Nevertheless, I expect that miniaturized, transportable high-resolution mass spectrometers could make a significant contribution to onsite analytics (for example, forensics, food, and environment)."
Advice: "Develop a career plan. Work hard. Be open-minded. Take risks and opportunities."

Brandon Ruotolo

Assistant Professor, Department of Chemistry, University of Michigan, MI, USA.

Calling: "I've been in love with analytical chemistry since my seventh class in Earth Science. I got my first taste of spectrophotometry (rock samples from a field trip) and was hooked!"
Triumph: "Our work with collision induced unfolding, which is enabling rapid acquisition of biotheraputic structure and protein-ligand binding information from mixtures."

Koen Sandra

R&D Director Life Sciences and Metablys, Research Institute for Chromatography, Kortrijk, Belgium.

Emphasis: "My main focus nowadays is on applying chromatography and mass spectrometry for full characterization of proteins, lipids and metabolites."
Triumph: "Starting up a successful life science business delivering high level R&D in (bio)pharma and diagnostics."
Prediction: "I have high hopes for the technologies of MS based protein quantification, MS imaging, ion mobility MS and 2D-LC."

Kevin Schug

Associate Professor & Shimadzu Distinguished Professor of Analytical Chemistry, The University of Texas at Arlington, USA.

Triumph: "Facilitating the acquisition of $26.5M in instrumentation and support to establish a unique industrial partnership and world-class analytical research and teaching facilities at UTA."
Emphasis: "Developing ultra-trace methods for determination of bioactive compounds in biological fluids, and assessing the potential impact of industrial processes on the environment."

Caroline West

Associate Professor, Institute of Organic and Analytical Chemistry, University of Orléans/ CNRS, Orléans, France.

Calling: "As a student, I was interested in forensics – a job that had the attraction of mystery to me. I only knew that analytical chemistry was one way to get there."
Regret: "Probably not. I took the opportunities that offered themselves."
Emphasis: "Chromatography with carbon dioxide-based mobile phases (SFC) is still keeping me very busy. There is so much more to learn!"
Hope: "I like exploring the frontiers, particularly those that I am being told are impassable. Thus the polarity limits of SFC, especially towards biomolecules, are of interest to me."
Advice: "First: hard work! Second: forget about copy & paste – find your own way."

Alok Sharma

Principal Scientist Head, Analytical Development Lab, Lupin Limited, Pune, India.

Triumph: "Being honored as a US Pharmacopoeia expert panel member for therapeutic proteins (2011-2015)."
Hope: "To gain more understanding about molecular function through structural and conformational tools; development of surrogate assays."
Advice: "Be passionate about that which is not obvious. Stick to the basics."

Dwight Stoll

Associate Professor, Gustavus Adolphus College, Department of Chemistry, St. Peter, MN, USA.

Calling: "I got into analytical chemistry, specifically separation science, because I observed that progress in other disciplines is more hindered by the limitations of existing tools than it is by the imagination of researchers."
Respect: "J. Calvin Giddings."
Emphasis: "The development of non-comprehensive approaches to multi-dimensional separations; for example, selective comprehensive 2D-LC."

André de Villiers

Associate Professor, Department of Chemistry and Polymer Science, Stellenbosch University, Stellenbosch, South Africa.

Calling: "My career path was determined by a 30 min discussion with Pat Sandra and Henk Lauer in 1999, which not only informed my post-grad studies, but also my current research…"
Emphasis: "The development and application of improved chromatographic and mass spectrometric methods for the analysis of complex samples, especially natural products."

Yu-Liang Yang

Assistant Research Fellow, Agricultural Biotechnology Research Center, Academia Sinica, Taipei, Taiwan.

Calling: "Forcing myself to wander, following my interests."
Emphasis: "Microbial interactions, development of new biological control agents."
Prediction: "Any breakthrough technology and analysis platform of next generation sequencing is likely to have a huge impact."

Bo Zhang

Associate Professor, Department of Chemistry, University of Washington, Seattle, USA.

Emphasis: "Fluorescence-enabled electrochemical microscopy for imaging neurons and single nanoparticle electrocatalysis."
Triumph: "The development and use of fluorescence-enabled electrochemical microscopy (FEEM)."
Respect: "Allen Bard and George Whitesides."
Advice: "Think outside the box and tackle hard problems."

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Why We Need the Theory of Sampling

Without representative sampling, measurement uncertainty is compromised. Here, we present the current Theory of Sampling versus Measurement Uncertainty debate. The verdict? *Nolo contendere!*

By Kim H. Esbensen and Claas Wagner

The purpose of sampling is to extract a representative amount of material from a 'lot' – the 'sampling target'. It is clear that sampling must and can only be optimized before analysis. In a recent paper, we show how non-representative sampling processes will always result in an invalid aliquot for measurement uncertainty (MU) characterization (1).

A specific sampling process can either be representative – or not. If sampling is not representative, we have only undefined, mass-reduced lumps of material without provenance (called 'specimens' in the theory of sampling) that are not actually worth analyzing. Only representative aliquots reduce the MU of the full sampling-and-analysis process to its desired minimum; and it is only such MU estimates that are valid. Sampling 'correctness' (which we define later) and representativity are essential elements of the sampling process.

The Theory of Sampling – TOS – has been established over the last 60 years as the only theoretical framework that:

i) deals comprehensively with sampling
ii) defines representativity
iii) defines material heterogeneity
iv) furthers all practical approaches needed in achieving the required representative test portion.

The starting point of every measurement process is the primary lot. All lots are characterized by significant material heterogeneity – a concept only fully acknowledged and defined by TOS – where it is crucially subdivided into constitutional heterogeneity and distributional heterogeneity (spatial heterogeneity). The heterogeneity concept (and its many manifestations) are introduced and discussed in complete detail in the pertinent literature (4-12). The full pathway from 'lot-to-analytical-aliquot' is complex (and in some aspects counter-intuitive because of specific manifestations of heterogeneity) and is subject to many types of uncertainty contributions in addition to analysis.

Unfortunately, the GUM (2) and EURACHEM/CITAC (3)

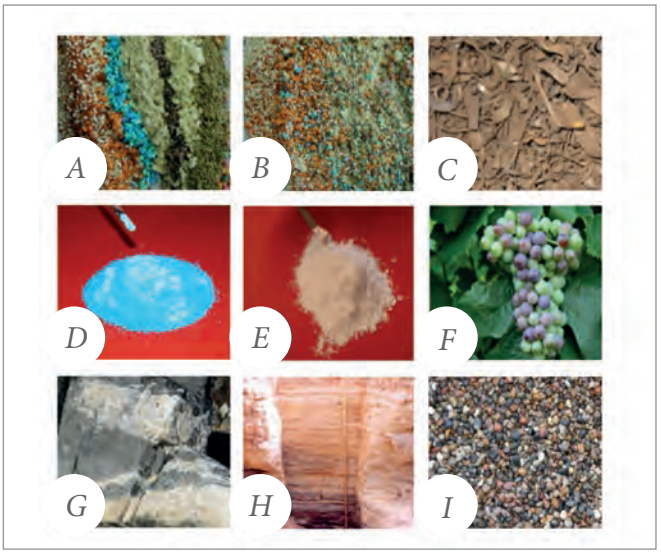


Figure 1. The almost infinite manifestations of heterogeneity in science, technology and industry. Materials and lots sometimes appear 'uniform' to the naked eye (E), but visual homogeneity does not guarantee that chemical characteristics are also distributed randomly. Heterogeneity can be structured (A, G, H), but is often highly irregular (B, C, D, I).

guides focus only on estimating the total MU in a completely passive mode. TOS, on the other hand, focuses on the conceptual and practical active steps needed to minimize all sampling contributions to MU. The main thrust of our argument is that if the test portion is not representative – in other words, if all sampling error effects have not been reduced or eliminated where possible – all MU estimates are compromised. But this does not have to be the case – TOS to the fore!

The impact of heterogeneity

Proper understanding of the phenomenon of heterogeneity, its influence on sampling correctness and, most importantly, how heterogeneity can be counteracted in the sampling process requires a certain level of understanding. Here, we present a bare minimum of TOS tenets to give an appreciation of the deficiencies inherent in current MU approaches.

Before defining these concepts theoretically, Figure 1 illustrates selected examples of the many appearances of heterogeneity. In reality, heterogeneity exhibits almost infinite manifestations – a point that appears rather hopeless at first.

Heterogeneity plays out its role on all scales from constituent particles to full lot scale, and can only be fully understood by considering both constitutional and distributional aspects and characteristics. Irregular and segregated heterogeneity, for example stratification, layering, or significant individual grain irregularity, cannot be expected to follow any standard statistical distribution. Figure 1D shows a strongly heterogeneous spatial distribution – all grains with diameters above the average have been dyed blue, while all spherules with diameters below-average remain white. In this example, heterogeneity is partly physical and partly compositional because the spherules actually carry different grades/contents of the analyte in question. Compositional heterogeneity is also clear in Figure 1F: which grapes are ripe and ready for harvesting and which are not? These examples are only meant to illustrate the serious issues surrounding securing representative primary samples; there are many other examples in the literature.

Already, it should seem obvious that the notion of modeling every manifestation of heterogeneity within the fixed concepts of systematic and random variability is too simple to cover the almost infinite variations of the real world's lot and material heterogeneity. This fact is argued and illustrated in full detail in the TOS literature. Previously, it has been argued that it is obvious that grab sampling will (always) fail in this context. Composite sampling is the only way forward (18).

For well-mixed materials (those that appear 'homogenous' to the naked eye, for example, Figure 1E), notions of simple random sampling have often been thought to lend support to the statistical assumption of systematic and random variability components; however, these comprise only a very minor proportion of materials with special characteristics, so clearly cannot be used to justify the same approach for significantly heterogeneous materials.

Theoretical analysis of the phenomenon of heterogeneity shows that the total heterogeneity of all types of lot material must be discriminated as two complements, the constitutional heterogeneity (CH) and distributional heterogeneity (DH). CH depends on the chemical and/or physical differences between individual 'constituent units' in the lot (particles, grains, kernels), generically termed 'fragments'. Each fragment can exhibit any analyte concentration between 0 and 100 percent. When a lot (L) is sampled using single-increment procedures – grab sampling – CHL manifests itself in the form of a fundamental sampling non-representativity. The effect of this fundamental sampling error (FSE) – which is unavoidable using grab sampling – is the most fundamental tenet of TOS. CHL increases when the compositional difference between fragments increases; CHL can only be reduced by comminution, typically crushing.

DHL, on the other hand, reflects the irregular spatial distribution of the constituents at all scales between the sampling tool volume and the entire lot. DHL is caused by the inherent tendency of particles to cluster and segregate locally (grouping) as well as more pervasively throughout the lot (segregation, layering) – or a combination of the two, as exemplified in the bewildering diversity of material heterogeneity in science, nature, technology and industry. DHL can only be reduced by mixing and/or by

TOS Versus MU in Brief

Current Measurement Uncertainty (MU) approaches do not take sufficient account of all sources affecting the measurement process, in particular the impact of sampling errors. All pre-analysis sampling steps (from primary sample extraction to laboratory mass reduction and handling – sub-sampling, splitting and sample preparation procedures – to the final analytical test portion extraction) play an important, often dominating role in the total uncertainty budget, which, if not included, critically affects the validity of measurement uncertainty estimates.

Most sampling errors are not included in the current MU framework, including incorrect sampling errors (ISEs), which are only defined in the theory of sampling (TOS). If ISEs are not appropriately reduced, or fully eliminated, all measurement uncertainty estimates are subject to uncontrollable and inestimable sampling bias, which is not a similar to the statistical bias because it is not constant. The sampling bias cannot, therefore, be subjected to conventional bias-correction. TOS describes why all sources of sampling bias must be competently eliminated – or sufficiently reduced (in a fully documentable way) – to make MU estimates valid. TOS provides all the theoretical and practical countermeasures required for the task.

TOS must be involved before traditional MU to provide a representative analytical aliquot; otherwise, a given MU estimate does not comply with its own metrological intentions.

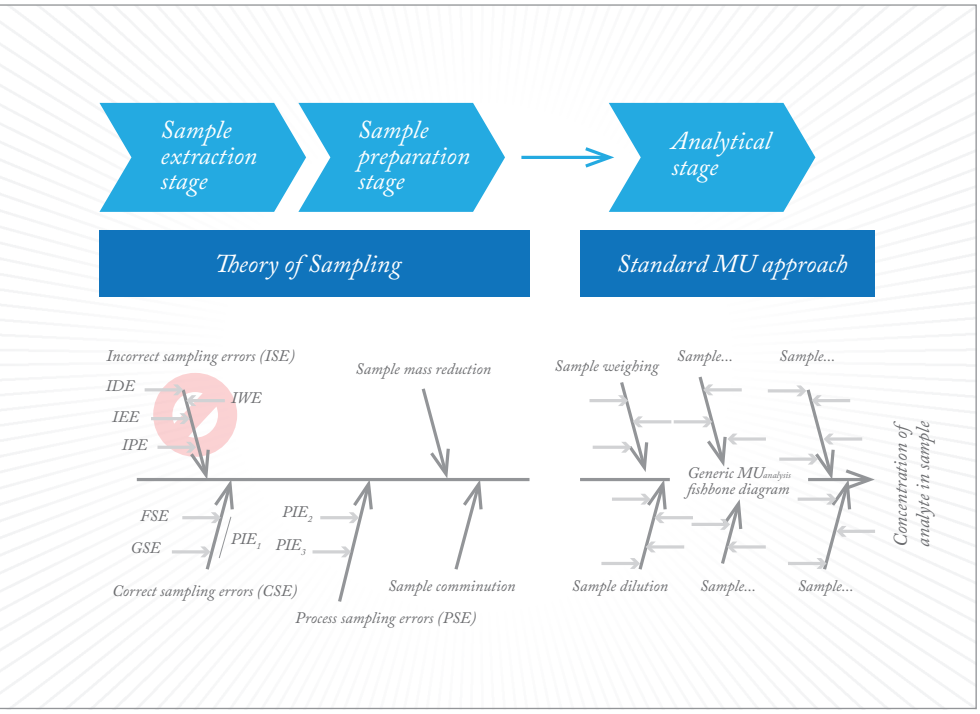
informed use of composite sampling with a tool that allows a high number of increments (4, 8, 11-13).

It is rarely possible to carry out forceful mixing of an entire primary lot. Therefore, if sampling lots have a high DHL, there is a very high likelihood for significant primary sampling errors: FSE + a grouping and segregation error (GSE). That is, of course, unless you pay close attention to the full complement of TOS principles.

The good news is that once you have acknowledged the principles of TOS, they are applicable to all stages and operations from the lot scale to the laboratory – representative sampling is scale-invariant. Lots come in all forms, shapes and sizes spanning the whole gamut of at least eight orders of magnitude, from microgram aliquots to million ton industrial or natural system lots.

It is critical to note that DHL is not a permanent, fixed property of the lot; GSE effects cannot be reliably estimated, as the spatial heterogeneity varies in both space and time as lots are manipulated, transported, on- and off-loaded, and so on. DHL can be changed intentionally (reduced) by forceful mixing, but can also be altered unintentionally, for example, by transportation, material handling or even by laboratory bench agitation.

An essential insight from TOS is that it is futile to estimate DHL based on assumptions of constancy. TOS instead focuses on the necessary practical counteracting measures that will reduce GSE as much as possible (the goal is full elimination)



Acronym Antidote

CH:	constitutional heterogeneity
DH:	distributional heterogeneity (spatial heterogeneity)
FSE:	fundamental sampling error
GSE:	grouping and segregation error
GUM:	guide to the expression of uncertainty in measurement
ISE:	incorrect sampling errors
L:	lot
MU:	measurement uncertainty
TAE:	total analytical error
TSE:	total sampling error
TOS:	theory of sampling

Figure 2: Proposed TOS/MU integration using an augmented fishbone flow path diagram with three components: sampling, sample preparation and analytical measurement errors. The figure shows integration of TOS principal sampling uncertainty sources in an augmented MU framework. The standard MUanalysis fishbone diagram is shown on the right. TOS is charged with delivering a representative analytical aliquot, including all sampling aspects of sample preparation (crushing, mass-reduction, weighing a.o.) with a view to eliminating incorrect sampling errors (1).

as an integral part of the sampling and sub-sampling process. In reality, it is rarely possible to completely eliminate GSE effects but they can always be brought under sufficient quantitative control (15).

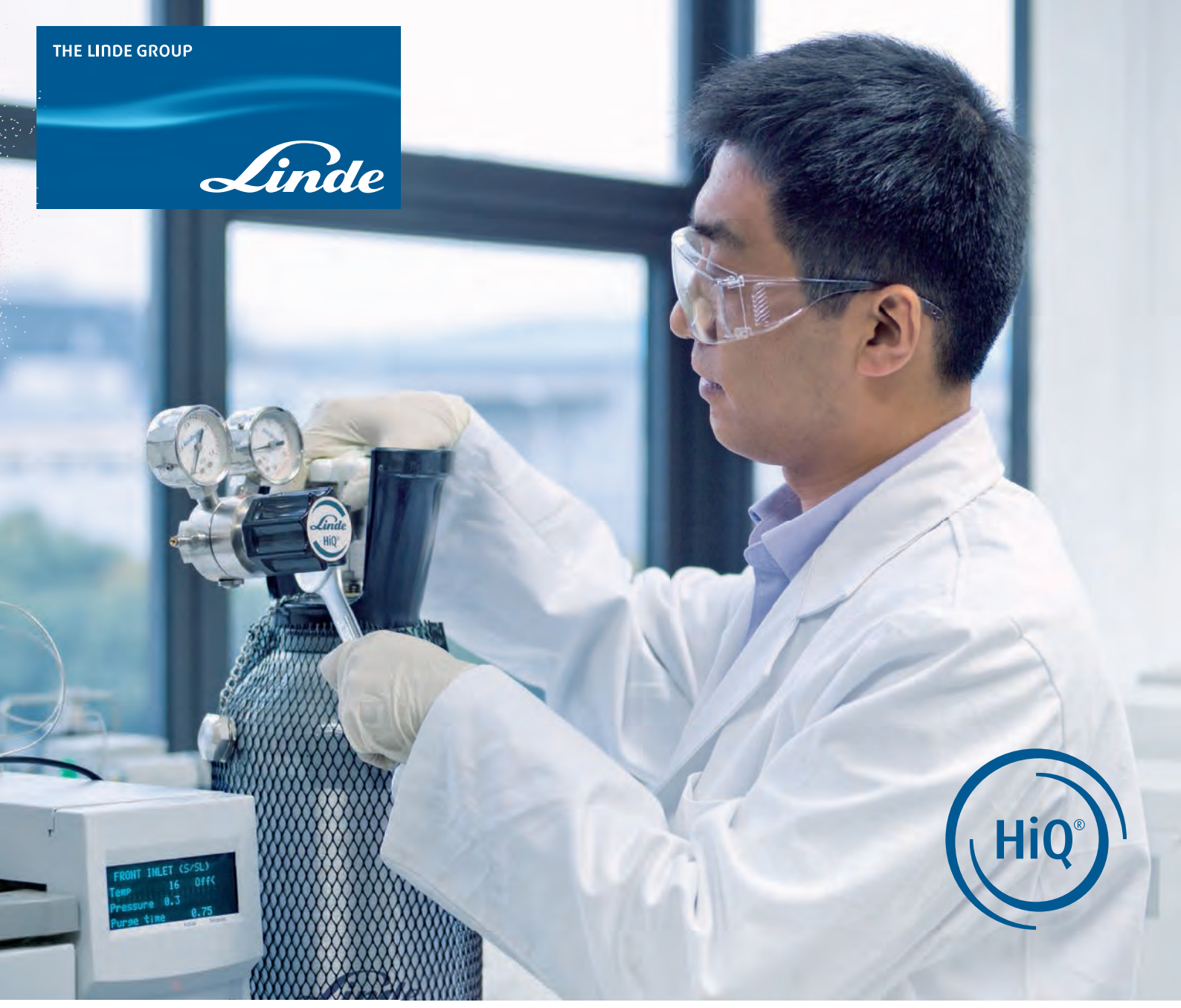
Most materials in science, technology and industry are demonstrably not composed of many identical units. Instead the DHL irregularity is overwhelming (the illustrations in Figure 1 only show the tip of the iceberg). Lot heterogeneity (CHL + DHL), especially DHL, is simply too irregular and erratic to be accounted for by traditional statistical approaches that rely on systematic/random variability. In fact, this issue constitutes the primary difference between TOS and MU.

Representative sampling in practice

The focus of TOS is not on 'the sample' but exclusively on the sampling process that produces the sample – a subtle distinction with very important consequences. Without specific qualification of the sampling process, it is not possible to determine whether a particular sample is representative or not. Loosely referring to 'representative samples' without fully described, understandable and documented lot provenance and sampling processes is an exercise in futility. We repeat: a sampling process is either representative or it is not representative; there is no declination possible of this adjective.

The primary requirement in this context is sampling correctness, which means elimination of all bias-generating errors, termed 'incorrect sampling errors' (ISE) in TOS. After meeting this requirement (using only correct sampling procedures and equipment), the main thrust in TOS is to ensure an equal likelihood for all increments of the lot to be selected and extracted as part of an increment. This is called the 'Fundamental Sampling Principle' (FSP), without which all chances of representativity are lost. FSP underlies all other matters in TOS.

A unified approach for valid estimation of the sum of all sampling errors (TSE) and all analytical errors (TAE) was recently presented in the form of a new international standard, 'DS 3077 Representative Sampling - HORIZONTAL standard' (15). This standard analyzes the general sampling scenario comprehensively, especially how heterogeneity interacts with the sampling process and what to do about it.



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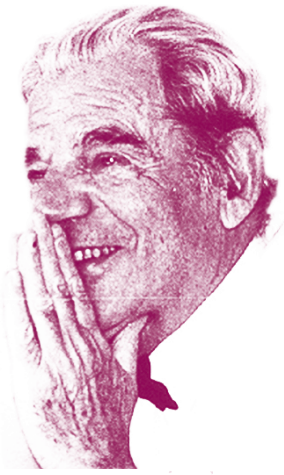
Pierre Gy and the History of TOS

Pierre Gy was born in Paris in 1924. He obtained a Masters degree in chemical engineering (1956), a PhD in physics (1960) and another PhD in mathematical statistics in 1975. Pierre has been awarded four major international scientific organization awards, including two gold medals from the Société de l'industrie minérale, and the Lavoisier Medal.

A special issue of "Chemometrics and Intelligent Laboratory Systems" was dedicated to the scholar's distinguished achievements in science and technology: "50 years of the Theory of Sampling (TOS)" (7), which includes a tribute to Pierre Gy that summarizes his scientific career; a three-paper series: "Sampling of particulate materials I-III"; as well as "50 years of sampling theory – a personal history"; and a listing of his complete scientific oeuvre – the latter five comprise the last professionally published papers from the pen of Pierre himself. In it, he outlines the complex history and the reason behind the development of TOS in a fascinating web of personal, scientific and industrial stories.

The special issue also forms the proceedings of the First World Conference on Sampling and Blending (WCSB1), which was dedicated to the 50-year anniversary.



A call for integration

We have shown that MU is not a fully comprehensive, universal, nor guaranteed approach to estimate a valid total measurement uncertainty if it does not include all relevant sampling effects. Around 60 years of theoretical development and application of TOS practice have shown that sampling, sample handling and sample preparation processes are associated with significantly larger uncertainty components, TSE, than the analysis (measurement) itself, TAE, typically multiplying total analytical error (TAE) by 10-50 times, dependent on the specific lot heterogeneity and sampling process in question. Occasional, very special deviations from this scenario cannot be generalized.

While GUM focuses on TAE only, the EURACHEM guide does point out some of the potential sampling uncertainty sources, but does not provide sampling operators with the necessary means to take appropriate actions. Only TOS specifies which types of errors can – and should – be eliminated (incorrect sampling errors, ISE) and which cannot, but which must instead be minimized ('correct sampling errors', CSE); our critique (1) crucially shows how!

Strikingly, the MU literature does not allow for sufficient understanding of the heterogeneity concept (CHL and DHL) or acknowledges the necessary practical sampling competence, both of which are essential for representative sampling. Incorrect sampling errors are non-existent in the MU framework, and the grouping and segregation error is only considered to an incomplete extent, leaving the TAE and the fundamental sampling error as the only main sources of measurement uncertainty here.

Also, the critically important sampling bias is only considered to a limited extent and only based on assumptions of statistical constancy. However, the main feature of the sampling bias is its very violation of constancy, which follows directly from a realistic understanding of heterogeneity. The only scientifically acceptable way to deal with the sampling bias is to eliminate it, as has been the main tenet of TOS since its inception in 1950 by its founder Pierre Gy (see sidebar). Here lies the major distinction between TOS and MU: TOS states that the sampling bias is a reflection of the incorrect sampling error effects interacting with a specific heterogeneity, whereas MU limits itself to acknowledging a statistical (constant) bias resulting from systematic effects attributable to protocols or people only (1).

MU is a top-down approach that is dependent upon an assumed framework of random and constant systematic effects (wrong), so individual uncertainty sources, such as GSE and ISE, are not subject to separate identification, concern, estimation, nor appropriate action (elimination/reduction). Indeed, the full measure of uncertainty sources connected to sampling, TSE, are almost completely disregarded in the MU approach. It is simply assumed that the analytical sample, which ends up as the test portion, has been extracted and mass reduced in a representative fashion. But if this assumption does not hold up, the uncertainty estimate of the analyte concentration is invalid; it will unavoidably be too small by an unknown, but significant (and variable) degree. The very different perspectives offered by MU and TOS are in

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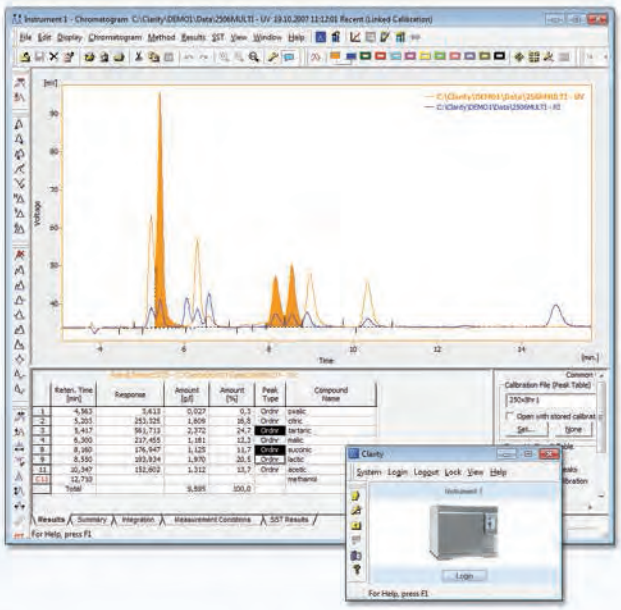

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serious need of clarification and reconciliation.

To that end, we call for an integration of TOS with the MU approach, illustrated by Figure 2, which shows three interconnected fishbone diagrams representing sampling, sample preparation and the standard complement of measurement uncertainty sources. Note that the intermediary complement represents the specific sample preparation error types, vividly illustrated and commented upon in a recent article in The Analytical Scientist (16).

To prevent underestimation of active uncertainty sources, we must integrate the effects of all three components (sample extraction stage, sample preparation stage and analytical stage), which can actually be done in a seamless fashion; there is no need to change the current framework for MUanalysis only to acknowledge the critical role played by TOS, which is to say, the framework for MUsampling.. These sampling and sample preparation branches should be implemented in every MU framework, as sampling uncertainty contributions logically must be dealt with ahead of the traditional measurement uncertainties. In this scheme, TOS delivers a valid, representative analytical aliquot (the horizontal arrow in Figure 2) as a basis for the now valid MUanalysis estimation.

An end to the debate?

Hopefully, we have explained the critical deficiencies in MU and have shown that TOS should be introduced as an essential first part in the complete measurement process framework, taking charge and responsibility of all sampling issues at all scales – along the entire lot-to-aliquot process. We want to see a much-needed reconciliation between two frameworks that have, for too long, been the subject of quite some antagonism. Indeed, the 'debate' between the TOS and MU communities has at times been unnecessarily hard, but we may add that such hostilities have been unilateral (always directed towards TOS). A 'representative (!) example of this quite unnecessary confrontation can be appreciated in the appendix of a recent doctoral thesis (17).

Squabbling aside, we hope that our efforts are seen as a call for constructive integration of TOS and MU, and look forward to comments from the wider community.

Kim H. Esbensen is a research professor at the Geological Survey of Denmark and Greenland, Copenhagen, Denmark, and Claas Wagner is an energy and environmental consultant and specialist in feed and fuel QA/QC.

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Our Unholy Alliance

Science and business: tenacious partners in a shaky marriage or eternally bound non-identical twins?

By Lee DesRosiers

Technology collides with human behavior. That's where the action is – either phenomenon alone is prosaic, which is why biotechnology and its motley relatives are so fascinating and – let's face it – the only game in town. Of course, the ancient art of brewing was the first really useful biotech application in commerce. But since the advent of beer, the business of science has really only accelerated in the last 40 years or so…

A pharmaceutical company, in conjunction with its partners, manipulates large clinical trial data to deliberately mislead the authorities. It's a misguided attempt to gain approval for a drug that cannot succeed – "cooking the books" in accountant vernacular. Directors of institutes in Tokyo and the National Institutes of Health commit suicide in response to one of their staff attempting fraud.

Are these events related? Do commercial concerns and academic goals drive otherwise discerning adults over ethical boundaries? Certainly. But every day, all day, all over the world, scientific and commercial transactions of all kinds occur ethically and fairly (subjective though those terms are). Both the business and scientific communities are remarkably hard on unethical behavior. And rightly so, but let us not pretend that we don't understand the pressures.

Why don't you get a job?

I grew up with biotechnology – that is to say, I grew up as biotechnology grew up. In the late 70s, when biotech started to "work" (showing potential in actual applications), I got a job. I moved from a protracted academic "lifestyle" to employment in the lab. To be more specific, I was hired to further develop a single celled photosynthetic bacterial protein source for developing countries. It was a morally unassailable position that I didn't hesitate to boast about to my reprobate college friends – all for a whopping $10,500 Canadian a year (twice the "$100 a week" my father would have described as a good salary

15 years earlier). Fifteen years later, in the intoxicating world of senior management in the life sciences, that salary seems like a rounding error…

My first "real" job was classic "micro", and a couple of grants later, it would be early "molecular." I was a player (albeit a peripheral one) amongst the fighter jet pilots of biotech: the gene jockeys. This was still in a totally academic environment, of course.

We redistilled our own phenol, worked daily with acrylamide, hydrazine, DFSO, ethidium bromide, P_{32} and I_{125}. Toxic, carcinogenic, radioactive, corrosive, explosive: all part of the macho arrogance of molecular biology – the only field that mattered back then.

I learned to sequence DNA, to do restriction fragment length polymorphism analysis, to clone DNA in the days when these processes were opaque, inscrutable, finicky, dangerous, involved, and laborious. It was a time when restriction enzymes were still spoken of with awe. And, at maybe 200 bases a day, it would have taken me a tad over 41,000 years to sequence the human genome…

We chewed and spat out inscrutable jargon, those who couldn't follow were doomed to be passengers in the future we were creating. We spoke of the elegance of our experiments. We scoffed at immunology, micro, plant biology but especially medicine. Why struggle to save aging overweight humans? It was their own fault and it would never happen to us. We understood aging and mortality but assumed we were exempt. Business might as well have been astrology.

We would ask ourselves: who but the most intellectually challenged – the most lacking in resolve and imagination – would ever consider any type of corporate affiliation? To "prostitute oneself" was the standard analogy. Yes, the impression among us at the time was that business people were amoral, insincere and unintelligent. It was acceptable for a scientist to simply not show up to an agreed upon rendezvous with one of the ubiquitous sales representatives that bravely sought our attention. We pitied them. We mocked them. They didn't warrant our respect. And we were far too intelligent to be sold to.

The sales people we met had been apparently forced, due to a lack of intelligence, to be in the questionable, dark world of business. Mysteriously, we thought our pursuits purer, although there was certainly no mystery about the source of our funding: taxes from the very companies and people who were beneath us.

Isn't it time you moved out?

When biotech "moved out of the house" into commerce in the 80s, I was caught up in the wave and went from being a molecular biology research assistant to a product manager for a biochemical company, selling to the same people I had previously worked with. My ex-colleagues recoiled in horror.

It turned out to be a subtle, judgmental world – one of shifting loyalties and difficult decisions. Constant dilemmas involved two positive alternatives or two negative alternatives. It was as disorienting as a concussion.

I had to learn to dress. In general, business people know how to dress; academics don't, as it is too banal an issue to consider. I showed up on my first day in brown suede shoes and a blue suit that I had been obliged to purchase for my father's funeral two years earlier. At the end of that seemingly relentless first day, my boss advised me to "lose the shoes".

Business people (ideally) had emotional intelligence: they looked you in the eye, they shook your hand firmly. Scientists at the time did not

make eye contact, if they could avoid it, and were unfamiliar with any physical contact – or so it seemed from their dead fish handshakes.

The affable back-slapping business mentality was sneered at and looked down upon by science people while business people saw scientists as socially inept, stylistically incompetent introverts who lived as perennial school children in clutter and relative poverty, just to avoid the responsibility of adulthood.

This clash of culture became very apparent to me when I returned to the lab, visiting as a product manager with the local sales representative. I was particularly struck by the disorder, squalor and generally unhealthy feel to the lab. And then there was the petty possessiveness; even pens had nametags on them and people were proud to have a phone to call their own.

When I first got the job, my new company called me in my old lab to ask if I preferred the bookcase or the credenza for my office. Office? I was stunned, I didn't even know what a credenza was. And I'm still not entirely sure.

Emotional intelligence, while in short supply everywhere, is required to excel in business but, until more recently, relatively underemphasized in science, where a gruff, irritable reclusive attitude was seen as part of the aura. Intelligence – in its brute direct form – is "nice to have" in business, but essential in science. Business is more about resilience, intestinal fortitude, looking people in the eye, reading the situation, thinking on your feet, actually liking people, having the maturity and the security to let others excel and surpass you. Admittedly, these often turn out to be only partially achievable ideals.

Scientists, despite their occasional bravado, were timid and conservative in their approach. Business required courage and a thick skin. The closest a scientist might come would be during a thesis defense. Scientists thought applying for a grant constituted pressure. The pressure in sales, marketing, management is monumental by comparison – and is in full bloom daily. In graduate school, 65 percent is a pass. In business, 95 percent of forecast can still be a disaster.

In the intervening decades, science has moved into business and vice versa and these effects have been lessened. There was a brief period when there seemed to be no end to biotech, when an idea was enough to start a company. Scientists dreamed of the apparently endless money the naïve business world would lavish on them – money that had been previously allotted unfairly to undeserving corporate drones.

"Scientists dreamed of the apparently endless money the naïve business world would lavish on them – money that had been previously allotted unfairly to undeserving corporate drones."

But we've all grown considerably since then. Realistic, achievable collaborations too numerous to mention have been successful. There has consequently been considerable cross pollination: business 'models' cannot seem to stop trying to apply scientific models to business scenarios and fields like pharmacoeconomics have bloomed. More and more often it is the science of business and the business of science.

Academic approaches to business and commercial mentalities in science notwithstanding, the drive to bring purer applicable research into medical, diagnostic and biotech carries its own limitation.

A commercial concern will acquire a research effort or a researcher, instantly removing them from the market the business is most interested in and inundating them with the very corporate culture the company is trying to enlighten. Small start-ups with aggressive, innovative approaches are swallowed by large Pharma who are looking to get closer to the real market. The corporate culture is methodically forced downward until the academic connections and approach fade under a results driven regime.

Is this on the test, professor?

But all is not lost. The marriage of business and science changes color and texture. Progress continues. Wounds heal.

Now, I teach management (management "science", actually) to young graduate students, many of them in high-tech or medical fields. I try to broaden their view and strengthen their hold on the future. The young talent drawn to business and science are no longer so clearly delineated. They display an admirable open mindedness; they want to know both worlds as one.

Big money doesn't necessarily ruin everything – and science is big money now. It might yet be the biggest. The two worlds alike will always be driven by the same hopelessly addictive allure. Promise.

Besides, we will always have beer.

Lee DesRosiers is a lecturer at McGill University in Montreal, Canada.

Rise Above the Risk: Effective GC Solutions to Optimize Helium Usage

Helium Shortage Affects Laboratory Productivity

The helium supply chain crisis has negative implications on research and laboratory operations world-wide. Despite the willingness of the GC and GC-MS laboratories to pay anywhere from $350 (on average) to a more costly $1,500 per cylinder (~3.5 m^3) for ultra-high purity (UHP) helium, rationing and delayed deliveries still cause difficulty in production planning and uncertainty in instrument productive uptime. Although the GC and GC-MS segment consumes less than one percent of the global helium supply usage per year, the shortages and delivery interruptions have wide-spread consequences for many industries utilizing varied analytical techniques.

Barriers to the Adoption of Renewable Gas Options

The severity of the helium crisis is evident when acknowledging that the helium, itself, is a non-renewable resource that is not in extreme abundance. Helium prices have tripled or quadrupled in some areas, and delivery uncertainty has not convinced the majority of GC-MS laboratories to switch to hydrogen – a readily available and renewable alternative carrier gas. For an effective carrier gas switch, methods need to be re-developed or translated and re-validated, the QA/QC criteria require adjustment, and problems caused by the reactive hydrogen gas in the MS ion source need to be addressed. In addition, for some regulated industries, helium carrier gas is still stipulated in the GC or GC/MS methods to which they must adhere.

Solutions to Mitigate Helium Supply Constraints

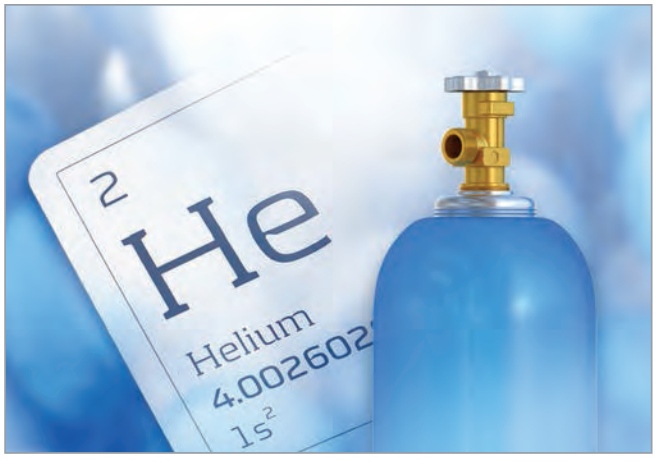
A revolutionary invention provides an effective solution to limit the effects of the helium crisis in the GC and GC-MS industry. The **Thermo Scientific™ Helium Saver technology**, available as an instant connect module that a user can easily place into the GC, allows for a ***single cylinder of helium to last up to 14 years*** under certain conditions – or for the lifetime of most GC-MS instruments.

The use of helium is optimized by this innovative GC inlet, which is supplied with two gases: helium for the analytical column flow, and nitrogen for septum purge, split flow, and sample vaporization and introduction, thus eliminating the helium waste typical in a standard GC inlet. To keep things simple, the split/splitless injector remains exactly the same, and the helium carrier gas flow through the analytical column remains the same at the specified flow rate.

Maintain Methods while Conserving Costs and Supplies

This technology enables you to continue your analyses without helium supply concerns or extensive supply costs. Save precious non-renewable noble helium during the analytical run, as well as when your instrument is idle. Since all of your analytical conditions remain the same, the retention time does not change. Keep your methods intact, validation-effective, and regulation-compliant. Significant savings can be realized in helium supply throughout the lifetime of your GC or GC-MS instrument.

- Maintain your Methods
- Prolong your Supply
- Save your Budget



Find out how to conserve helium during your analyses and extend helium cylinder lifetime at thermoscientific.com/heliumsaver

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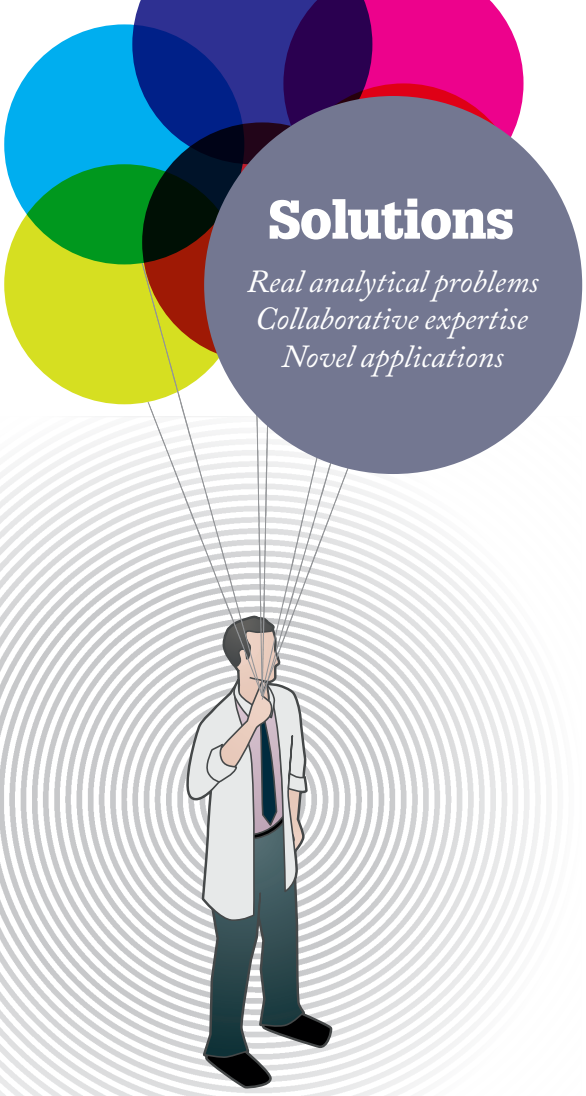

The 'Noble' Undertaking of Helium Conservation

Is it possible for a cylinder of helium to last years, rather than months? When using a novel split-splitless injection port for gas chromatographs, the simple answer is "yes." Here, I present the more technical answer.

By Ed McCauley

The Problem

Shortages and supply chain disruptions have recently reduced the availability and increased the price of helium. Certainly, switching to hydrogen is a viable option for some applications, but sometimes that is not possible or desirable. How can we make more efficient use of helium in gas chromatography?

Background

Back in my chemistry set days, I was fascinated with helium balloons, lighter-than-air craft, and things that went "boom". I would spend hours collecting zinc from dead batteries and occasionally "borrow" a bit of muriatic acid from a neighbor's pool house. Flaming dirigibles were only a bottle, a balloon, and a cigarette lighter away...

Things have changed considerably since the 'good ol' days', but the chemistry of helium and hydrogen has remained the same. One is remarkably inert, while the other is a bit more gregarious. Despite their differences, helium and hydrogen are the only viable carrier gases for GC-MS instrumentation today.

In light of the recent helium shortage/supply-chain disruptions, a lot of attention has focused on switching from helium to hydrogen as a carrier gas for GC-MS work. The advent of low-cost bench-top hydrogen generators makes this switch appear attractive. Such units can produce remarkably pure hydrogen and remove the need to deal with high pressure cylinders in the laboratory. For fast GC or in fields where methods are not highly regulated, hydrogen may be the best choice. Indeed, hydrogen systems can deliver GC peaks that are both faster and slimmer than those of their helium counterparts.

However, though hydrogen does indeed offer the 'sexiest' chromatography, it is not a panacea. This is particularly true when a mass spectrometer is on the receiving end. Hydrogen has poorer pumping speed, lower sensitivity in electron ionization (EI) by a factor of about three; forms a reactive plasma in the ion source that can alter spectra; requires changing standardized method retention times and response factors; can react with analytes or halogenated solvents in the hot zone (particularly with active sites)… and it has the potential to go "boom". (In fairness, hydrogen generators can be safe, particularly when a hydrogen sensor is placed in the GC oven proper.)

Traditional methods of reducing helium consumption include lowering the amount of gas that is split, switching to a standby gas, such as nitrogen, when the instrument is idle, or "simply" switching to a different carrier gas, such as hydrogen.

Reducing the split flow can cause elevated baselines or premature fouling of the head of the column, since the outgassing

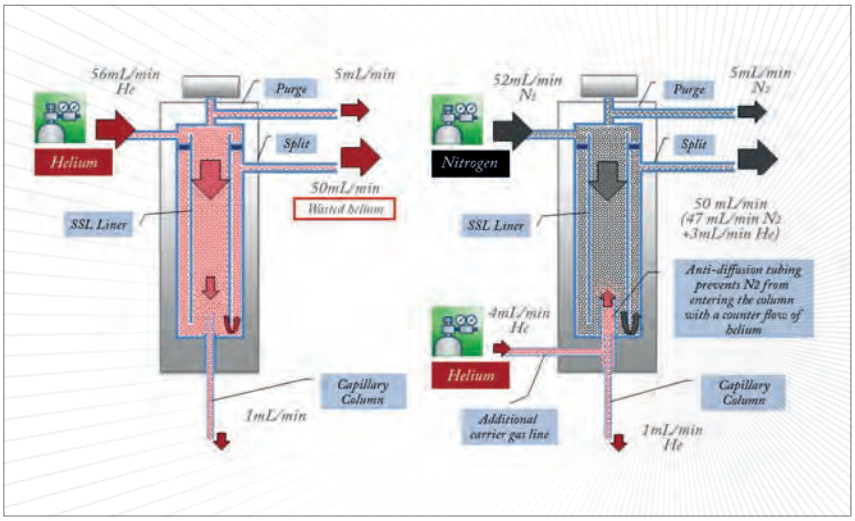

Figure 1. Conventional split/splitless (SSL) injection on left compared with Helium Saver SSL on right.

of matrix residuals is not as effectively diluted. Switching to a standby gas when the instrument is idle is a solution, particularly if end users never intend to use their instrument. If they do, there is a protracted waiting period on start up due to nitrogen saturation of the carbon filter upstream of the split/splitless (SSL) injection port. There is a far superior way to conserve helium: decoupling the purging process from the analytical gas requirement.

The Solution

The value of decoupling the purging gas from the gas used during analysis is readily apparent in Figure 1,where helium is consumed in an SSL-type injector. About 1 ml/min is used for the analytical separation (column flow), 50 ml/min is used for the split flow, and 5 ml/min is used for the septum purge. (Mileage may vary). So, 55 times as much gas is used to purge matrix residuals from the liner, and silicone goo from the septum, than is used for the chromatographic process.

I first became interested in the wasted gas at the split and septum purge vents in 2009 and devised a device that harvests the wasted gas, compresses and purifies it then re-introduces it into the supply stream. This solution was quite complicated. Even at the time, I thought there simply must be something more elegant.

A few months later, I found myself on the telephone with Paolo Magni, a chromatography expert and colleague of mine in Milan, Italy. We were talking about oxygen diffusion through graphite ferrules using a split/splitless injector in backflush mode (who hasn't had that conversation?). In this mode of operation, the entire injector flow is routed, during backflush, to a tee piece that is inserted between the analytical column and a large bore pre-column. One of the downsides of this arrangement was that since the total flow is imposed, the split flow setpoint affects the retention times due to the pressure drop of the pre-column.

I began to imagine what might happen if the flow were reduced to only a few milliliters per minute. The pressure would not drop, but could not be controlled with the same precision, and the purge flow would be very low and inefficient.

So, why not use a secondary helium source, set to deliver only a few milliliters per minute at the tee through a tiny bore

capillary with a high input pressure? If the pressure drop of the pre-column is indeed low, the electronic pressure control of the inlet could be used to effectively control the pressure at the tee junction and the small backflow of helium would simply contribute to the helium already in the inlet. The actual flow to the tee would not be critical, since only the pressure is important to provide the correct column flow.

During this discussion with Paolo, it dawned on me that during backflush, a separate gas such as nitrogen, argon, or hydrogen could be used to purge the inlet and reduce helium consumption. As long as the backflow of helium was sufficient to occlude back-diffusion of the secondary gas, it would work. In fact, only a very short pre-column is needed to prevent back diffusion of the secondary gas. And this was the key to using this idea on an SSL without backflush. Rather than using a pre-column to cold trap and selectively backflush analytes at a specific oven temperature, a short heated pre-column could be used so that injected components would fully transfer to the analytical column, then "backflush" immediately following sample transfer. This modification would allow dramatic reductions in the consumption of helium if, say, nitrogen were used to pressurize the inlet.

Since the required "pre-column" is quite short, it is advantageous simply to incorporate it into the body of the injection port. This short back-diffusion barrier needs only be about a centimeter long to work with most capillary columns. Incorporation into the injection port allows the pre-column to be heated without a separate heating element. It also makes it possible to eliminate the separate tee piece in the oven.

In practice, we used a short segment of hypodermic needle stock with an internal diameter just larger than the outer diameter of the column. The needle stock is stainless steel, which has been treated using a proprietary process to render the surface inert. The inert coating and short residence time ensures no interaction with injected analytes. Furthermore, matrix residuals resident in the glass liner are precluded from out gassing onto the head of the GC column, reducing the frequency of column trimmings to restore chromatography.

The hardware solution consists of a subcomponent SSL injector and a specially modified insert. The helium delivered to the back-diffusion barrier is controlled using a solenoid valve set to deliver a residual purge of approximately 0.1 mL/min (nitrogen acts to transfer the injected sample), or approximately 4mL/min during analysis. Flow is not critical, as long as it is above the amount necessary to occlude nitrogen. The excess helium is simply diverted upward, where it contributes to the bulk nitrogen purge.

This helium-saving solution allows a cylinder of helium to last for 3.5 years with continuous use and up to 14 years, if put in standby mode overnight and on weekends. The standby condition consumes only 0.1 mL/min.

Beyond the Solution

Decoupling the purge gas requirements from those of the analytical gas as described above, opens up several possibilities. While nitrogen is the gas of choice for the purge gas, it might also be possible to use argon, methane, propane, or carbon dioxide. Liquefiable gasses, in particular, offer high capacity for a given cylinder size.

To prove a point, one day I attached a propane cylinder from my home barbeque grill (see photo). A regulator wasn't even required. (Note: Please don't try this. The olefins will eat your lunch. There are far too many impurities in fuel-grade propane!)

Alternatively, air might be used as the purge gas. The oxygen in air can cause column and sample degradation, but it is possible to plumb the pneumatics with a three-way valve such that helium is used for the short duration of the injection. This way, the column and sample are never exposed to oxygen.

A less-conventional application of the Helium Saver with a different gas cylinder… BBQ propane.

Another example of going 'beyond the solution' is direct coupling of a purge and trap concentrator. Instead of using helium to desorb the trap and act as carrier, one can simply use nitrogen for purging and desorbing, compounding helium savings. And for hard-core hydrogen users, the technology may reduce the potential for halogenated solvents or unsaturated analytes to react with hydrogen in the hot inlet. Nitrogen would be present during the injection, but then hydrogen would be used for the chromatography.

Since the hydrogen is restricted to only a few mL/min, there would be far fewer safety concerns. Generally, a column break near the injector results in high hydrogen flow into the oven, unless the flow is limited by a restrictor. This restrictor can hamper the maximum allowable split flow. In the case of the Helium Saver SSL, high split flows remain available because the split is accomplished using nitrogen delivered by separate pneumatics.

Development of the Helium Saver SSL is a shining example of how a problem in the back of a person's mind can sometimes be solved in unexpected ways, just through bouncing ideas off colleagues.

Ed McCauley is a Senior Scientist in Research & Development at Thermo Fisher Scientific, Austin, TX, USA.

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Understanding Graphene

Raman analysis of graphene on silicon dioxide wafers

By Yvette Mattley, Ph.D.

Carbon nanomaterials like graphene, fullerenes, carbon fibers and nanotubes have unique electrical, thermal, chemical and mechanical properties. These properties have inspired significant interest among researchers and innovators who are discovering new applications for graphene in fields such as aerospace, catalysis, photovoltaics, optoelectronics, microelectronics and sensors development.

Raman spectroscopy is ideal for rapid, non-destructive characterization of all sorts of samples, and is well suited for characterizing carbon nanomaterials. In graphene, Raman analysis of several key bands can be used to assess the number of graphene layers and the purity and quality of the sample.

The G band observed at ~1580 cm^{-1} is a good indicator of the number of graphene layers, with band position shifting to lower energy as layer thickness increases, dependent on doping and strain. The G' band at ~2700 cm^{-1} can also be used, albeit via a more complex relationship. The intensity of the D band at ~1350 cm^{-1} is related to the number of defects in the material (amorphous carbon), a marker of purity. It also therefore increases in intensity with the number of graphene layers.

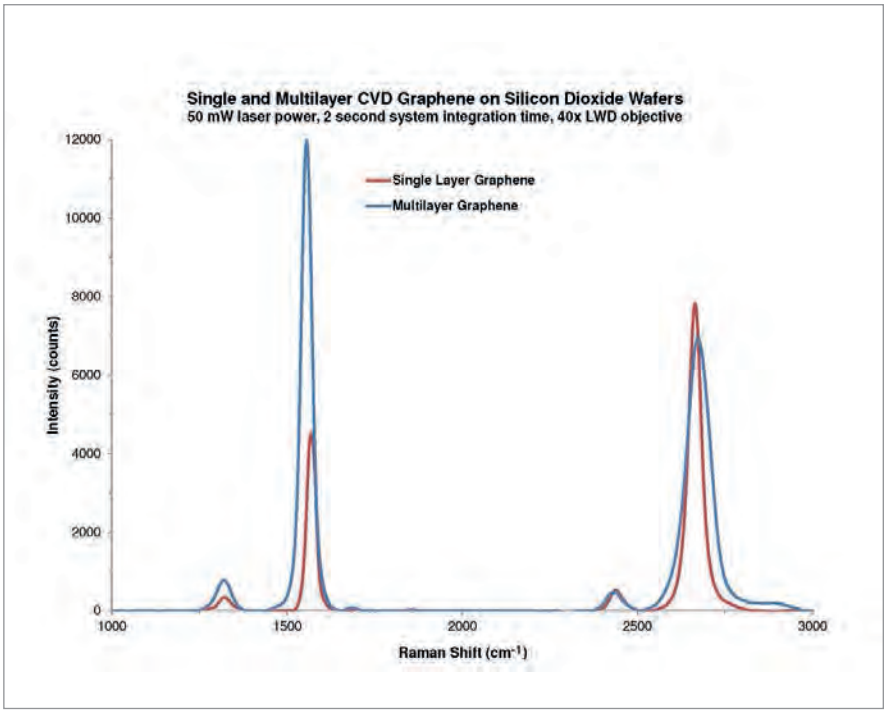

Figure 1: A shift of ~12 cm^{-1} was observed for the G band in comparing the single and multilayer samples, while the G' band width broadened from 39 cm^{-1} for the single layer sample to 60 cm^{-1} for the ~4-layer sample

Experimental Conditions

To assess the ability of the Ocean Optics IDRaman microscope with 532 nm Raman excitation to study graphene samples, we measured both single and multilayer samples across the system's full Raman shift range, 200-3200 cm^{-1}. We used the Clean Peak function in OceanView spectroscopy software to remove background artifacts, capturing high quality data from all three bands of interest. OceanView displays wavenumbers or the Raman shift relative to a specified excitation wavelength. For more sophisticated analysis, a chemometrics software package may be warranted.

Although the choice of a short-wavelength 532 nm laser meant autofluorescence generated in the sample could potentially degrade signal to noise and make Raman peaks difficult to resolve, this is not a significant effect for excitation of inorganic materials such as carbon nanotubes and fullerenes.

Results

A shift of ~12 cm^{-1} was observed for the G band in comparing the single and multilayer samples, while the G' band width broadened from 39 cm^{-1} for the single layer sample to 60 cm^{-1} for the ~4-layer sample (Figure 1). As compared to the 7 cm^{-1} resolution of the IDRaman micro system, this setup demonstrated performance that is fully capable of building an accurate model of layer thickness. Additionally, the appearance of the D band with an intensity of ~930 counts for the multilayer system demonstrates dynamic range that is more than adequate for assessment of defect levels.

Info@OceanOptics.com
http://oceanoptics.com/application-note-understanding-graphene/

Determination of Anions with Suppressed Conductivity Detection

Mareike Margraf, Dr. Silvia Marten, Wissenschaftliche Gerätebau; and Dr. Ing Herbert KNAUER GmbH, Germany

Determination of the common anions, such as bromide, chloride, fluoride, nitrate, nitrite, phosphate, and sulfate, is often needed in water analytics.

Conventional colorimetric, electrometric and titrimetric methods are available for the determination of individual anions, but ion chromatography provides a single instrumental technique that may be used for rapid, sequential measurement.

Here, we present the sensitive determination of anions in water samples using the isocratic AZURA Compact System with suppressed conductivity detection (Figure 1). Typically, anions can be rapidly and easily analyzed using conductivity detection with additional apparatus for the suppression of the eluent's conductivity.

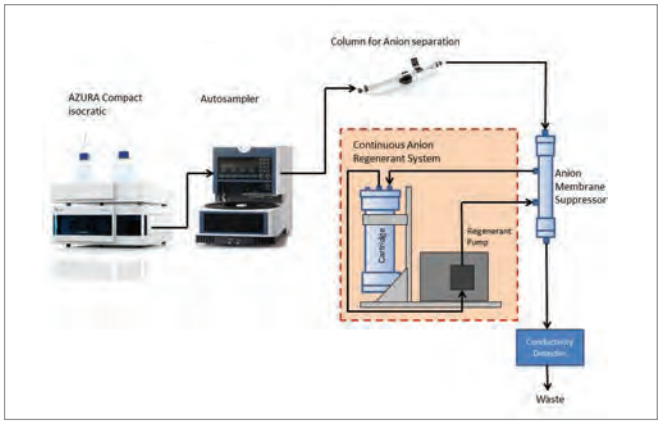

Figure 1: Flow chart of the IC system with anion Membrane Suppressor and Continuous Anion Regenerant System (4).

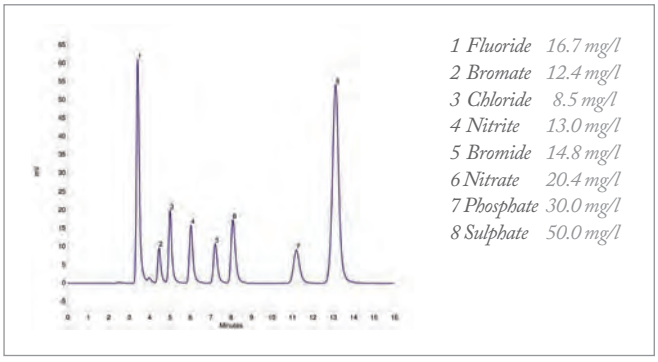

Figure 2: Chromatogram of the anion analysis.

Experimental

The stock standard solution was prepared by weighing in anion salt standards and dissolving them separately in deionized water. For the analysis of water, samples are often just filtered through a 0.45 µm syringe filter and injected to the IC system as also described in US EPA method 300.1 and the standard method 4110 (1,2). More complex sample pretreatment is required if very low concentrations of anions have to be determined or if matrix constituents are interfering with the IC separation (3).

Method parameters

Column	*Anion Column, 250 x 4 mm*
Eluent A	*4.5 mM Na2CO3, 1.4 mM NaHCO3*
Gradient	*Isocratic 100 % A*
Flow rate	*1.2 ml/min*
Injection volume	*50 µl*
Column temperature	*25 °C*

Detection Conductivity Detector CDD-10A VP (5 Hz, 0.02 sec)
SeQuant® SAMS™ robust suppressor for anion chromatography
SeQuant® CARS™ Continuous Regeneration System for SAMS™

Results

The isocratic AZURA Compact system in combination with suppressed conductivity detection was found to be well suited for the analysis of anions in mixed standard solutions even in the low ppm region. Under the chosen conditions, the applied anion column separates all anions within 15 min as shown in Figure 2.

The column was designed specifically for compliance monitoring of inorganic anions in accordance with US EPA Method 300.0 (A) and 300.1 and low molecular weight organic acids. Common inorganic anions can easily be separated in a variety of sample matrices including drinking water, wastewater, process streams, and scrubber solutions with an optimized operating temperature of 30 °C to ensure reproducible retention times.

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The American Irish Dream

Sitting Down With…
Apryll Stalcup, Director of the Irish Separation Science Cluster, Dublin City University, Ireland.

You didn't have the easiest route into science…

Right. I grew up in the USA in an era when women didn't really do science. I wasn't aware that it was even an option for me – I just knew that those were the classes I was most interested in. After high school, I followed a crooked path, attending four different undergraduate institutions (we were a roaming military family). Back then in the States, you could take classes without declaring a major, so I was taking graduate courses in quantum chemistry just because they sounded interesting – much to the surprise of my professors. As an undergrad, I supported myself with one, two or even three jobs per semester. It was hard to keep my grade point average up, but it worked out. I also had no idea that you could get paid to go to graduate school, so I worked in an environmental lab for a couple of years before starting.

Why did you become Director of the ISSC?

I had reached the point in my career where I was ready to take a leadership role. I had been approached three times about becoming the head of the chemistry department at the University of Cincinnati, but I wanted to take on a broader challenge; there are people here at the cluster working with monolithic media, microfluidics and in several other fields that I'd never had the opportunity to get involved with or support. To become a great leader you have to be a great advocate, so the role at ISSC really is my dream job. For me, it's all about separations; I don't care what the platform is, so our multidisciplinary cluster is a great fit.

What have been your major highlights en route to Ireland?

At the University of Hawaii, our group was the first to use sulfated cyclodextrin as a chiral additive in capillary electrophoresis (CE). We were also the first to use heparin as a chiral additive – and that's taken off in some really unexpected directions, particularly in light of the adulteration in 2008. More recently, we've revisited some of the earlier work we did on linear solvation energy relationships (LSER) and shown that we can distinguish between different cations and anions. Despite being exciting work, it was difficult to get funded…

Tell us about the ISSC.

The cluster was setup by Brett Paul in 2009 with funding from the Science Foundation Ireland, essentially because of the realization that separation science is a key enabling technology not only for the (bio)pharmaceutical industry, but also other sectors, such as food and the environment.

There are two core programs at ISSC: materials technology and advanced platforms. Materials science includes monoliths and the work of Jeremy Glennon (University College Cork), whose group created core-shell particles that formed the basis for materials now marketed by Supelco. There is also a strong biopharma component; Brendan O'Connor is working with glyco-selective ligands that are more robust and reproducible than Protein A. On the platforms side, Dermot Brabazon has been working on microfluidics and its application (along with Brendan's work) in sample clean up. Innovative instrumentation and detection systems are another focus.

What we have to remember is that it doesn't matter how sexy our materials or platforms are if they can't be scaled up and applied. Therefore, in most cases, we actively seek industry partners.

Commercialization is important then, but does that leave room for a more creative approach?

Actually, I have mixed emotions about focusing everything on commercial success. A key part of research is serendipity. For instance, Dara Fitzpatrick, one of our ISSC investigators at UCC, is a separation scientist, but is launching a company based on broadband acoustic resonance dissolution spectroscopy (BARDS). Having a total focus on one area is not always the right approach – flexibility is also important. It's funny, but the ionic liquid work that we're still doing came about because one of my graduate students misunderstood me and pulled the wrong chemical off the shelf!

On the other hand, researchers do need to be very aware of the interesting problems facing industry – and the need to engage with those problems to attract funding.

You will be co-chairing the 2016 International Symposium on Chromatography (ISC) in Cork, Ireland, with Jeremy Glennon – what did you take home from ISC2014 in Salzburg?

I saw a couple of things that really piqued by curiosity – but I'm keeping those to myself at the moment… More generally, one thing that struck me was the vibrancy of the European separation science community. I recently gave a talk in the US about my academic career and noted that I could often partition my academic colleagues into two camps: those who think separations are magic (or witchcraft!), and those who think it's trivial. I don't think the average scientist in the US understands just how exciting and pioneering separation science can be. That's why I'm really delighted to be a part of the community in Europe.

Salzburg is certainly a tough act to follow. But Ireland is a wonderful place to visit. With the support of the ISC organizing committee, resources available from Science Foundation Ireland and a strong program, I feel confident that it will be a great success.

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