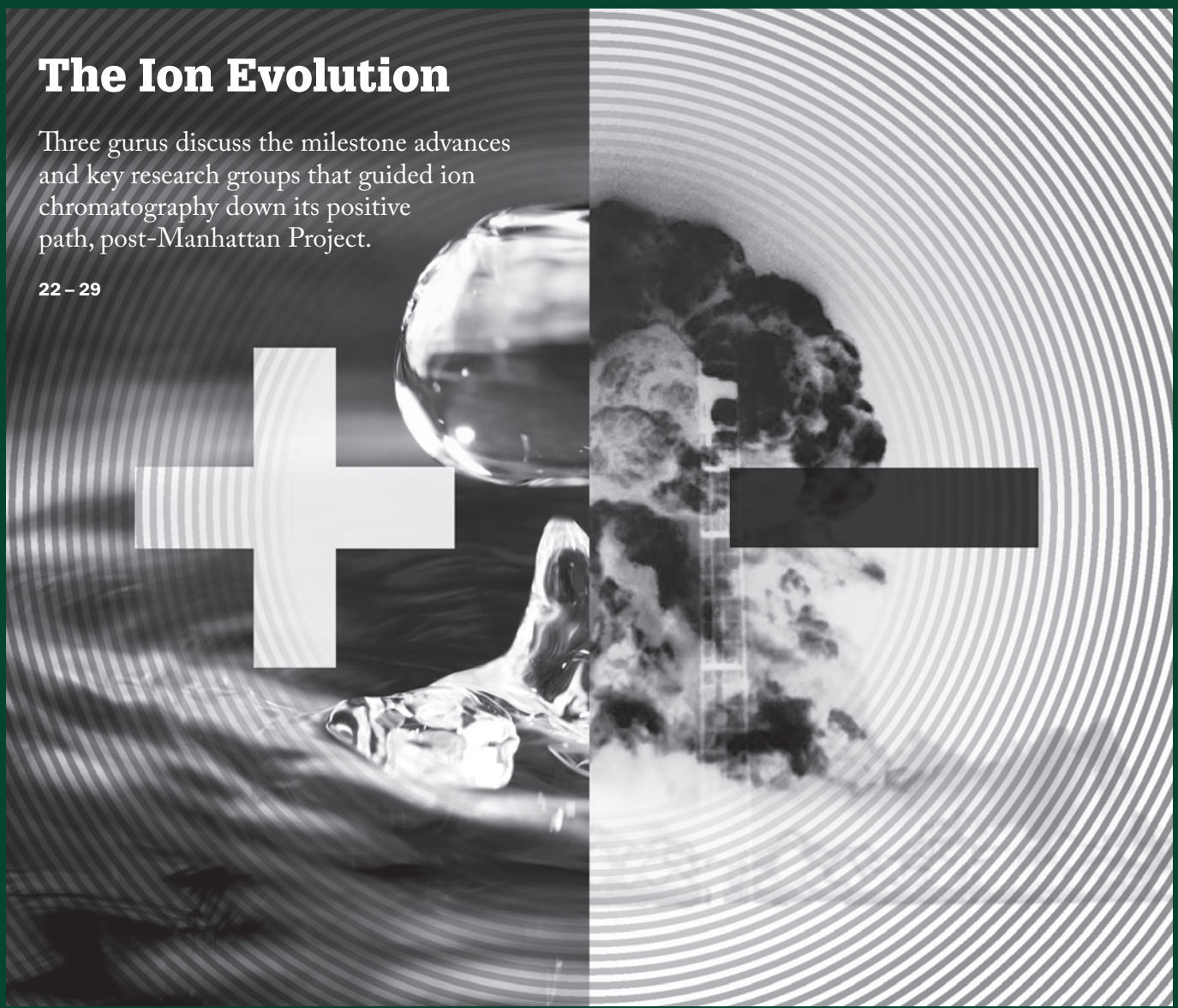
NOVEMBER 2014 # 22

the Analytical Scientist



The Ion Evolution

Three gurus discuss the milestone advances and key research groups that guided ion chromatography down its positive path, post-Manhattan Project.

22 – 29

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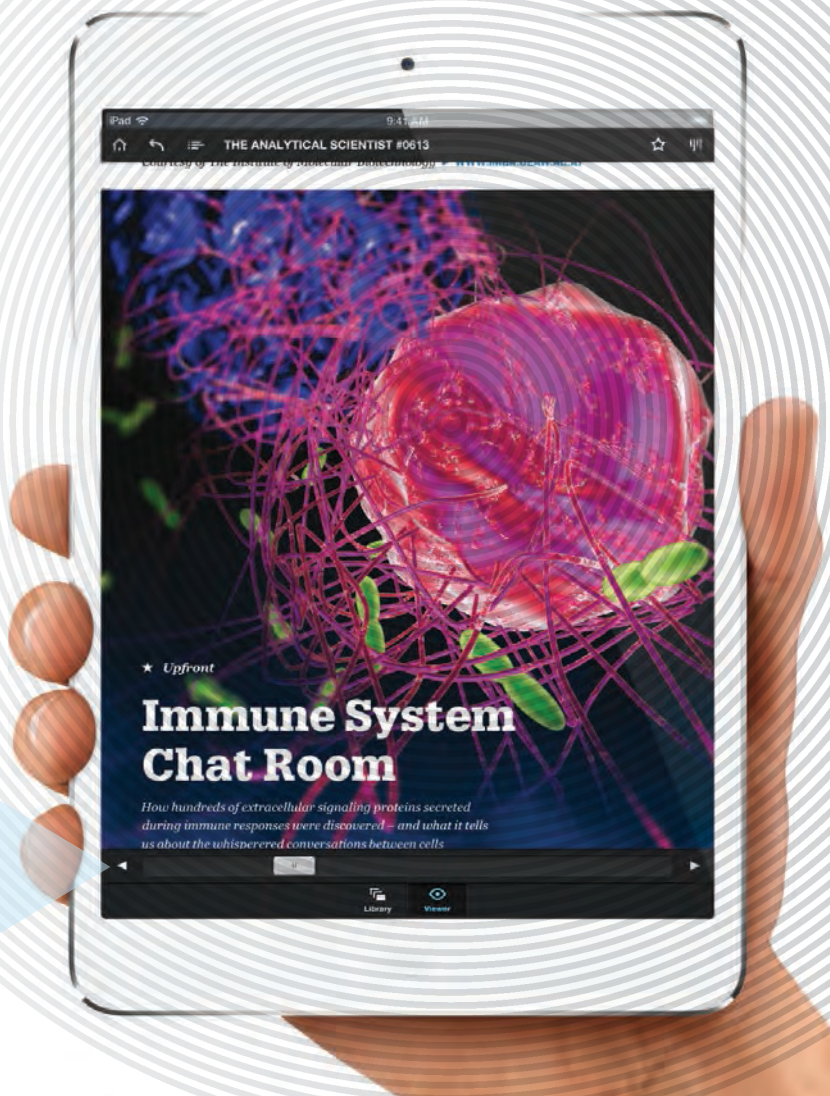

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I feel both honored and humbled to be sat in the editor's chair this month. It is certainly not unusual for us to feature the lives of great analytical scientists, but this issue is particularly rich in history and touching memories.

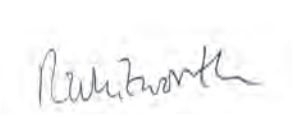
It was with sadness that I learned of Georges Guiochon's death. Georges finally succumbed to neuromuscular failure caused by Post Polio Syndrome on October 21, 2014. It is instantly clear that the field of separation science has lost one of its greatest pioneers. Georges was one of our early "Sitting Down With" interviewees, and though I only met him once very briefly, his name has come up in conversation countless times – and will no doubt continue to do so. To remember Georges, we could list his many accolades, count his top-cited papers, or refer to honorary degrees – but to do his memory true justice, we collected the thoughts of fellow scientists who had the pleasure of knowing him well. Of course, they were not simply colleagues; they were also friends. For me, the sentiments expressed on page 16 could not paint a better picture of a man who was so very fondly respected.

When we asked Georges about the favorite moments of his career in 2013, his answer was clear: personal interactions and helping people. "I brought people to work with me from Central Europe, Russia, Cuba, Iran, China – and that is what has given me the greatest satisfaction."

You will find Eva Smolková-Keulemansová in this month's final pages. Like Georges, Eva's childhood collided with the Second World War. Despite having her school years cut short by time spent in several infamous concentration camps, Eva returned to Prague to start an astonishing academic career in analytical chemistry. Her story – especially in light of her early years – is truly inspiring. Now well into her ninth decade, Eva offers several words of wisdom for "youngsters". Once again, the importance of camaraderie is strong; Eva's friends – past and present – include many esteemed scientists, and she notes that forging such relationships has been a great source of pleasure.

Though Eva's grounding philosophy states that "my eyes are in the front of my head; therefore, I need to look towards the future," it is clear to me that many of us could do well to take a look over the shoulder of the great and the good, to understand and learn from the path they once walked along.

Rich Whitworth
Editor



Bob Blackledge

Over thirty years in forensic science have taken Bob Blackledge all over the world to testify as an expert witness or to attend international conferences. "I feel extremely fortunate to have worked in an area where, every day, I looked forward to going to work and facing new challenges." Bob says that the term "Forensic Scientist" is very broad (and detractors might even say it's an oxymoron), so he describes himself as "an analytical chemist who specializes in forensic science." Although his interests are actually very broad, Bob's special love is trace evidence. He is the editor of "Forensic Analysis on the Cutting Edge: New Methods of Trace Evidence Analysis" (Wiley Interscience, 2007.)

A single photo inspired Bob to share his thoughts on the Coffee-ring Effect on page 18.

Steve Thomas

Graduating from Warwick with a Chemistry degree, Steve Thomas joined the NMR department of Merck's Neuroscience Research Centre at Terlings Park in 1990. "The wealth of experience in Medicinal Chemistry support made me analytically bilingual; speaking both NMR and Mass Spec." Closure of the site in 2006 led him to the Biotransformation and Drug Disposition group at GSK. "I have always loved puzzles and science," Steve explains, "and structural identification is a straight combination of the two". Having studied metabolic transformations his entire career, eventually, the lure of more challenging samples and close proximity to the development compounds that change people's quality of life proved too strong.

Steve urges us to share data and knowledge on page 20.

Victoria Samanidou

"When, as a child, I played in the square at the front of the chemistry building at Aristotle University of Thessaloniki, it never crossed my mind that I would spend the rest of my adult life there..." Victoria Samanidou started in atomic spectroscopy, but when working as a visiting researcher at the Institute of Ecological Chemistry in Germany using an HPLC instrument, a whole new world was revealed. Since then, she has dedicated all her research activities to the development of analytical methods using this "magic tool" for the determination of various substances in food matrices, as well as in matrices of biological, toxicological, pharmaceutical and environmental interest.

On page 19, she tells us how to inspire the next generation of analytical chemists.

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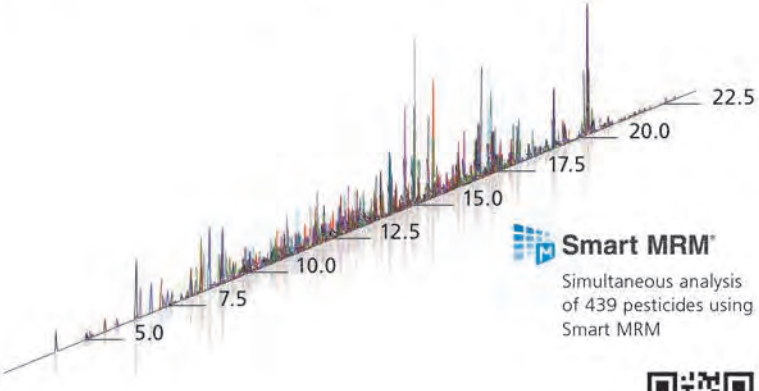

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Upfront

Reporting on research, personalities, policies and partnerships that are shaping analytical science.

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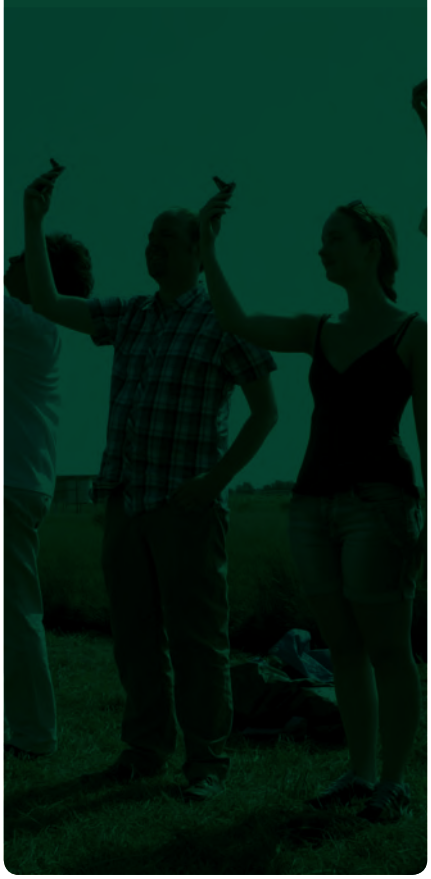

Smart Spectral Citizen Science

How can a network of citizens and smartphones measure atmospheric particles?

If you need a huge number of measurements, why not recruit the help of everyday citizens – and their smartphones? A research team led by Frans Snik of Leiden University did just that to gain data on atmospheric particles after developing iSPEX, a device that fits onto smartphone cameras (1). Snik hopes that the work will eventually help us understand the effect of atmospheric particles on health.

The smartphone camera acts as the detector, while the iSPEX add-on creates a spectrum of light that enters a slit with sinusoidal bands to directly measure polarization. Photos record the spectrum and linear polarization of light scattered by dust particles and the iSPEX app gathers information, records the location, and sends both to an online database (see promotional video: tas.txp.to/1114/iSPEXvid). Measurements were taken on three cloud-free days in the Netherlands and, when supplemented with ground-based and LIDAR measurements, the results provided a comprehensive overview of aerosol dynamics across the country on the measured days. In fact, Snik and his team say that iSPEX was able to discern smaller details than satellite data. We spoke to Snik to find out more.

How did you get the project off the ground?

It was pure serendipity. We were developing a highly accurate new method to measure atmospheric aerosols on a global scale with the SPEX (Spectropolarimeter for Planetary Exploration) satellite instrument. One afternoon, we were playing around with plastic optics to demonstrate this new method, and we used a smartphone to take pictures. We realized that the method was so robust that it even worked with a smartphone camera. From that afternoon onwards, things got increasingly out of hand. We had a couple of MSc students (in astronomy) produce the first iSPEX prototypes with 3D printing, and developed the data reduction. Around the same time, we started collaborating with environmental scientists to study how satellite-based monitoring could also produce valuable data for a ground-based network. But as it is simply unfeasible to install such expensive equipment to measure particles in the air at every street corner, we started thinking about crowdsourcing by making use of the technology that everybody carries around: smartphones.

It sounds challenging…

We had to figure out many things along the way, for example, how to set up the unique collaboration between scientists in many different fields (astronomy, space research, environmental science and meteorology), the collaboration with the several thousand citizen scientists, mass-producing scientific equipment, figuring out the data reduction… But by far the most nerve-wrecking aspect was waiting for a cloud-free day in the Netherlands!

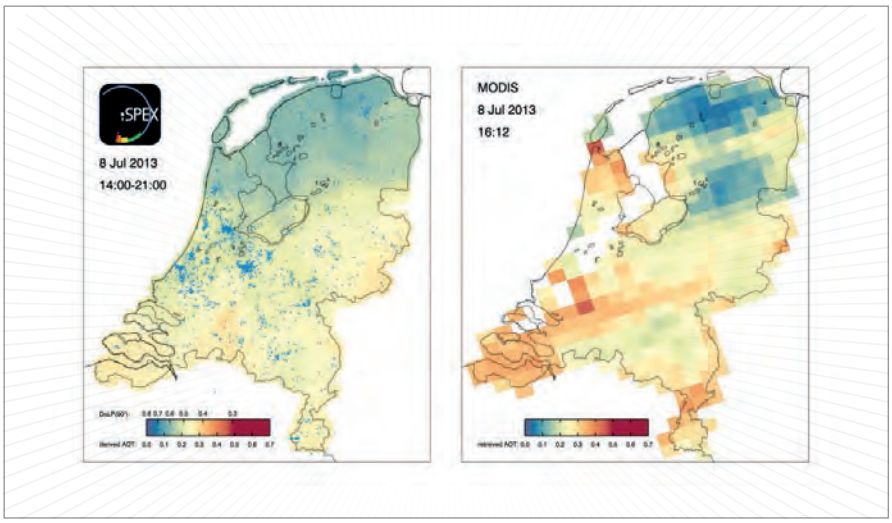

Figure 1. Left: iSPEX map from July 8, 2013, between 14:00 and 21:00. Each blue dot represents one of 6007 measurements that were submitted. At each location, the 50 nearest iSPEX measurements were averaged and converted to aerosol optical thickness (AOT), a measure for the total amount of atmospheric particles. Right: AOT data from the MODIS Aqua satellite for comparison, which flew over the Netherlands at 16:12 local time.

Were you surprised by the number of volunteers?

Yes and no. We invested a lot of effort to recruit participants and we also worked with several societal partners, such as the Dutch Longfonds Foundation for people with lung diseases. Nevertheless, when we announced the measurement day after a lot of preparation, the number of measurements really overwhelmed us. From 8:00am, a measurement was added to our live map every second! Many people also started doing their own little investigations; they were walking around town and performing measurements everywhere. They literally took our idea into their own hands.

We then started looking at all the submitted data. And the results were actually even more accurate than we had expected (see Figure 1).

What is the future of iSPEX?

We've proven that the iSPEX measurements are accurate, provided that a lot of people participate. To really add new information about air particles, we of course need more measurements on more days. Also, we are trying to retrieve additional information about the dust particles from iSPEX measurements. If we could gain information about the particle size and even the composition, it would be a total breakthrough, permitting detailed studies of health impacts. Furthermore, we are now going international; 2015 is the International Year of Light, and it is our goal to have people worldwide measure the light of the sky above their heads with their smartphones. With these data, we can really put local measurements in much more context, and study big patches of dust particles that are blown across the borders of many countries.

Our hope is that in the near future everybody can contribute to scientific measurements, using sensors attached to their smartphones.

Reference

Frans Snik et al., "Mapping Atmospheric Aerosols with a Citizen Science Network of Smartphone Spectropolarimeters", Geophysical Research Letters 41, doi:10.1002/2014GL061462 (2014).

Fighting Ebola

Technology gets an update to help combat the spread of the deadly disease

One problem seriously hindering the efforts to control Ebola is our ability to diagnose patients in the early stages of infection. Selim Ünlü from the College of Engineering at Boston University has spent several years developing a label-free, chip-scale photonic device with his team and believes that the technology could help. The device was recently shown to detect multiple viruses in blood samples, including viruses that had been genetically modified to mimic Ebola and the Marburg virus. The viruses are identified based on size variations caused by genome lengths and other factors.

"We call the device the single particle interferometric reflectance imaging sensor (SP-IRIS). Pathogens are detected by shining light from an LED light source on viral nanoparticles captured on the sensor surface by a coating of virus-specific antibodies," says Ünlü. "Interference of light reflected from the surface is modified by the presence of the particles, producing a distinct signal that reveals the size of each particle. The sensor surface is very large and can capture the tell-tale responses of up to a million nanoparticles. Thus, the viruses on the sensor surface are counted in a digital detection modality."

A precursor to the device was label-free microarray technology for bimolecular assays, which was developed in 2008. Ünlü and his team modified and simplified the original interferometric measurement by replacing the tunable laser with multi-color discrete wavelength sources, which made the technology cheaper and potentially more portable. At first, the team's efforts focused on the technology rather than applications, until John Connor, associate professor of microbiology at Boston University, became interested when his laboratory started testing various label-free technologies. "They tested the original IRIS instrument and then became involved in the development of the SP-IRIS. It was through his lab that we got involved in Ebola and other viral hemorrhagic fevers," explains Ünlü.

Label-free systems have been attempted by researchers before, but the SP-ISIS detects each viral particle on a large sensor surface and discriminates the particle from the noise by determining the size of each detected particle. The advantage, Ünlü says, is that the device detects viruses directly, where as many other technologies detect protein and nucleic acids specific to viruses.

The device is in the prototype stage, but requires little to no sample preparation and minimal processing. It takes about an hour to obtain the result. The team is currently working on making the instrument and the detection technology more robust so that it can be used in clinical laboratories. It's also being tested in multiple labs, including a Biosafety Level-4 (BSL-4) lab at the University of Texas Medical Branch. *SS*

Alcoholic and Nutritious?

Analysis of pottery shards from Teotihuacan reveals ancient alcohol experimentation

Organic residue analysis of artefacts from the Teotihuacan ruins in Mexico has revealed the first direct chemical evidence of alcohol production in Prehispanic Mesoamerica. The researchers focused on pulque, a milky alcoholic beverage made from the fermented sap of maguey plants, which has long been thought to be the liquor of choice for Teotihuacan people. A study led by Richard Evershed, a professor at the University of Bristol, sought to identify the elusive pulque

using a biomarker approach, which could potentially be used to detect other alcohols (1). Evershed is a master of biogeochemical analysis and previously gave us insights into the diets of the ancient Egyptians by looking at meat mummies (http://tas.txp.to/1114/mummy).

"Whenever we're trying to detect a new material, we first explore the basis of the production and the chemistry," says Evershed. "We found that pulque is based on a bacterial fermentation rather than the yeast fermentation of most alcohols. The bacteria involved (Zymomonas mobilis) produces high concentrations of lipids – bacteriohopanoids – in its membrane, offering a characteristic method of detection. Lipids are much better preserved than normal alcohol components, so it's a new way of looking at the material."

Teotihuacan, founded around 150 BC, was considered an important political, economic and cultural power up until 650 AD, but it was also located in a high-risk area for cultivating crops. The diet of the prehistoric civilization is thought to have been predominantly comprised of mainly maize and beans. Murals and other artefacts have suggested that pulque may have also been important. The maguey plant is quite hardy compared with maize, so it could have offered good insurance against failed crops. It's also high in nutrients and calories so Evershed describes it as an "interesting supplement" to the main diet.

Evershed's student, Marisol Correa-Ascencio, examined hundreds of different pottery shards and was at first interested in food residues; however, she also found several residues that had resinous compounds as the major components and discovered the presence of bacteriohopanoids. Coating pottery with resin is a well-known practice for waterproofing unglazed pottery and the bacterial compounds were only found in the resin-coated vessels. GC-MS with selected ion monitoring was used for targeted compound analysis. Of around 300 potshards, 14 revealed the characteristic bacteriohopanoid distributions.

"The bacterial compounds actually occur naturally as quite highly functionalized compounds, but it's been observed that they defunctionalize quite readily even at low temperatures," says Evershed. "We performed an ageing experiment with pulque and replica pottery to see if we could generate the same distribution of compounds. To be honest, I'm not a fan of artificial aging experiments […] there's the worry of introducing reaction pathways that might not have taken place. But the bottom line is that when Marisol extracted the pot she found exactly the same hopanoid hydrocarbons that she saw in the pulque vessels, which was fantastic."

Evershed adds that the bacteria examined are also involved in the fermentation of other alcoholic beverages, including beer, wine and cider, so the biomarker could be applied when examining other ancient alcohol residues. *SS*

Reference

1. *Marisol Correa-Ascencio et al., "Pulque Production from Fermented Agave Sap as a Dietary Supplement in Prehispanic Meso america", PNAS 111 (39), 14223-14228 (2014).*

Chicago Zoological Society's Sarasota Dolphin Research Program. NMFS Scientific Research Permit No. 15543

Blowhole Breathalyzer

Could bottlenose dolphin breath analysis help with conservation efforts?

We've covered human breath analysis before (1, 2) – and it's not as straightforward as it appears. Now, taking the challenge a step further, scientists based in California and Florida have developed an instrument that analyzes the metabolites in dolphin breath to determine health (3). The work is especially relevant after an unusually large number of dolphin deaths along the Atlantic coast of the US in 2013. Health assessments of dolphin populations could go a long way towards aiding conservation efforts by giving an early indication of serious problems, such as infectious diseases or contamination. According to the research team, breath analysis and comprehensive metabolite profiling have not been attempted in cetaceans before.

"The project started when I was introduced to Frances Gulland from the Marine Mammal Center and Stephanie Venn-Watson from the US Navy's Marine Mammal Program. We saw that breath monitoring concepts from humans could potentially be very useful in this population of animals," explains Cristina E. Davis, a professor at the University of California who led the study. "There is very little knowledge on the baseline breath metabolite composition of marine mammals, but several studies have shown that cetacean breath could hold diagnostic value."

Davis and her team designed an insulated tube that traps and chills the breath exhaled from a dolphin's blowhole. Breath samples were collected from both wild dolphins and dolphins from the National Marine Mammal Foundation in San Diego ahead of gas

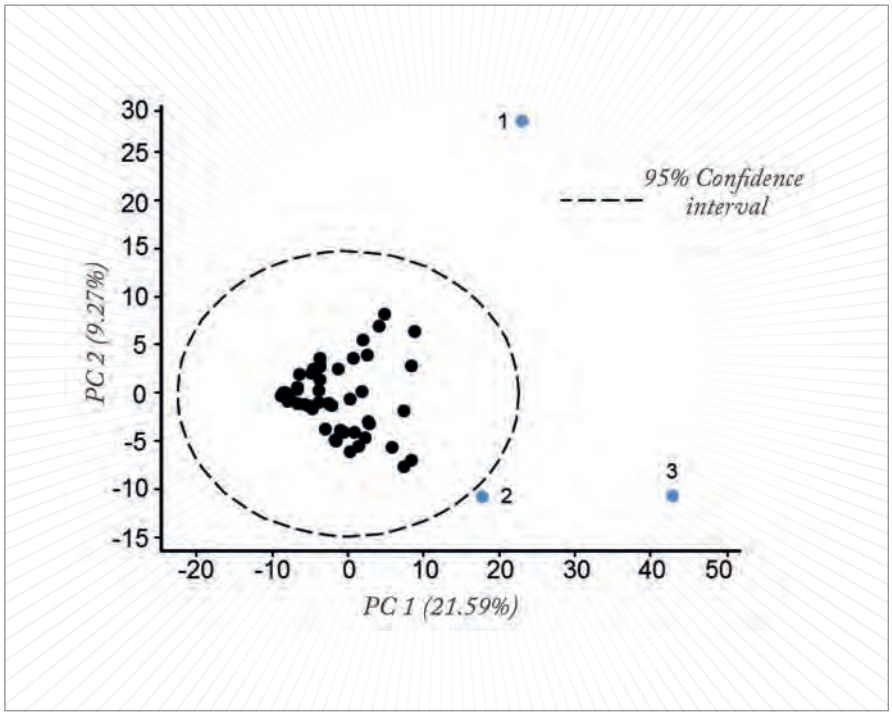

Figure 1. Principal component analysis (PCA) of LC-MS samples. Abnormal breathing behavioral or disease states were identified for each of the three dolphins outside of the cluster. All other dolphins were known to be healthy. (Reproduced from reference 3).

chromatography-mass spectrometry (GC-MS) and liquid chromatography (LC)-MS analysis.

"The device can maintain temperature control during the sampling process and was designed specifically for dolphin lung physiology. It allows us to get very reproducible samples even in ambient outdoor environments," says Davis. "When it came to profiling the exhaled metabolites, we chose an untargeted approach to broad spectrum metabolites, so that we could observe as many different biomarkers as possible," (see Figure 1).

Breath analysis is challenging because of the large variability and low abundance of exhaled metabolites, but dolphins and other cetaceans are particularly suited because they have separate digestive and pulmonary systems, helping to avoid contamination of the breath sample. Dolphins are also "explosive breathers" with rapid gas exchange. Davis admits that small variation may still occur and that follow up studies using chemical standards may be necessary to more precisely measure key metabolites.

"We are now working on a longitudinal trial to study how baseline metabolites are associated with animal health," adds Davis. "We are also working on expanding the veterinary applications of the approach." *SS*

References

1. *Lisa Spacek and Terence Risby, "Breath Analysis for Disease Diag nosis", http://tas.txp.to/1114/breath1*
2. *Terence Risby and Joachim Pleil, "CSI: Breathprint", http://tas.txp.to/1114/breath2*
3. *A. A. Aksenov et al., "Metabolite Content Profiling of Bottlenose Dolphin Exhaled Breath", Anal. Chem., 86921, 10616-10624 (2014). DOI: pubs.acs.org/doi/abs/10.1021/ac5024217*

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
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Remembering Georges Guiochon

"One of the giants of separation science," Georges Guiochon (1931–2014) will certainly be missed. Equally certainly, he will continue to inspire and educate future generations. Here, seven friends, colleagues and collaborators remember Georges with great fondness.

With the passing of Georges Guiochon, analytical chemistry – and especially the separation science community – has lost a major figure. In the field of fundamental chromatography, Georges was the leader by far, and his contributions have been seminal. He was active to the very end. In 2014, by October he had published a total of 27 papers, with others submitted. We were all impressed with his brilliance and enthusiasm for his research.

Georges' loss is very personal to me, as I have known him for over 50 years. We first met in the early 1960s, when he was interested in using data that I published with my PhD thesis on time normalization in GC. That moment started a lasting friendship. His mentor was Istvan Halasz, who also had a great influence on my career. I spent a sabbatical with Georges at the Ecole Polytechnique in 1972. Even in those early days he was recognized as a major figure in chromatography.

Georges moved to the US in 1984 – first to Georgetown and then, in 1987, to Tennessee. He used to joke, "how can a Frenchman possibly live in Knoxville?" It was around this time that he suggested that I start the HPCE symposium series. I followed his advice with the first meeting in 1989. In 1991, I wrote the preface for the special issue in the Journal of Chromatography, honoring Georges' 60th birthday (1). There you will find much about Georges career up to that point.

I have wonderful memories of the times spent with Georges and Lois in different places around the world. A special memory is the trip to Egypt in 1990 with Csaba Horvath and Fred Regnier. As Lois set it up through the US government, Georges was happy to be the tag-along spouse. Georges was a gourmet, especially a connessoir of wine, and it was always a joy to have dinner with Lois and Georges.

In closing, it's always hard to lose a colleague and friend. His contributions to the field of chromatography will remain for a long time with us. His impact on science has been enormous. He will be remembered as a great scientist, whom you knew was always correct. I am sure his memory will provide Lois with much comfort. May his memory be a blessing.
– Barry Karger

I was most impressed - as so often happens - at the very beginning, but unlike many other people Georges kept impressing again and again - long after I was a young student. Georges had a fantastic group at the Ecole Polytechnique, with Michel Martin, Henri Colin, Patrick Arpino, Anté Krstulović, and perhaps a few others who I fail to remember or whom I did not meet when Leo de Galan's group from Delft paid a one day visit to Georges' group in Palaiseau near Paris. That one day happened to be a Friday afternoon and a Monday morning. How can I forget? I don't think I met Pavel Jandera at that time, but the papers that I spelled out from Guiochon, Jandera and Colin did much to form me as a scientist.

My fondest memory is a dinner we shared a few years ago (2010) in Somerset, New Jersey. Georges and his wife Lois, my wife Dana and I, Jan Blomberg, Mark Schure, Ron Majors and one of his colleagues (if my memory serves me right). It was a small enough group and a quiet enough place to have a single conversation most of the time. Georges was at his most charming. We always shared many opinions on science. We did not have a common view on politics or a common taste in music, but there is one Joe Cocker classic that Georges would have appreciated: n'oubliez jamais. – Peter Schoenmakers

I was saddened to learn of Georges' passing last week. Georges was a true gentleman and a most exceptional scientist. He had keen insight into all aspects of the chromatographic process, from fluid dynamics to the kinetics and equilibria of retention. He has left us a legacy of so much fine work, and a great many students and colleagues whose lives he changed. The sum of his life's work is monumental. I have so many images of Georges in my mind; all of them are of a generous and gentle man, eager to share ideas, pose questions, and offer suggestions. Our field has lost a giant, and no one can fill his shoes. – Jim Jorgenson

My first contact with Georges was in the middle of the 1970s when he was visiting the laboratory of Maurits Verzele at the department of Organic Chemistry, Ghent University. I had to show him two chromatograms of urinary steroids on packed columns (I preferred the capillary GC chromatogram!). The first one was on a column packed with (porous) diatomaceous earth (Gas Chrom Q 100-120 mesh) and the second one was packed with (non-porous) glass beads of 100-120 mesh size. In both columns, the stationary phase was 1% polydimethylsiloxane. In a very elegant and brilliant way he explained the much higher efficiency of the latter column through mass transfer kinetics… and this to an organic chemist (!). Anyhow, this conversation motivated me to study the fundamental aspects of chromatography in detail, which determined my scientific career.

Forty years later, we still discussed mass transfer kinetics and fundamentals – at HPLC 2013 in Amsterdam on core shell and porous particles in LC, and at HPLC 2014 in New Orleans on the theory and practice of supercritical fluid chromatography (SFC), and the need to use the correct terminology to describe the use of carbon dioxide as mobile phase constituent. The years clearly did not affect his drive, motivation and enthusiasm. Fortunately, these latter discussions were in the relaxed environment of a high quality restaurant (Georges was Frenchman!), although Lois and my wife Martina most probably didn't always appreciate the discussions on the A- and C-term in the late hours.

Georges was also an amazing person outside of the chromatographic environment, and having a conversation with him on any topic was a unique, instructive and entertaining experience. I will miss him a lot. – Pat Sandra

Georges Guiochon was one of the few giants of separation science. He was a visionary; he could clearly see which area of separation science was going to succeed and he focused his research on that field. Then, with a rather thorough and disciplined research strategy, he studied every possible aspect of that yet unexploited territory. Besides exploring the essential details of separation mechanisms in analytical or preparative chromatography, he always looked and went beyond the frontiers to expand the limits of current technologies.

He had the most remarkable memory. He could recall all relevant papers published in chromatography and all of the important talks given at symposia. You did not need any source of information when you had Georges around.

Georges has had the most significant impact on the way I see and understand science and life in general. – Attila Felinger

The scientific community has suffered a great loss with the passing of Georges Guiochon. I first met Georges in 1964 when he was at the Ecole Polytechnique in Paris. At the time, both of us were interested in gas chromatography, but soon afterwards, liquid chromatography became of prime interest and the search for appropriate technology was on.

Because of his solid engineering background, Georges quickly assumed prominence in the theory of high-performance liquid chromatography and the quantitative relationships that he developed for many aspects of this separations method. He was relentless in attacking all phases of HPLC technology that needed quantitative understanding, and was a prolific writer, preparing more than a thousand publications in his career. Georges made a point to attend all the important symposia despite physical limitations, and was always generous in his interactions with fellow scientists.

I will strongly miss Georges and the interesting discussions and interactions that we had over many years. He was an icon in science and the world is much better because of his efforts. – J. J. Kirkland

With the passing away of Georges Guiochon, we sadly lost the long-term authority on the fundamentals of chromatography. Georges published – continuously over more than 40 years – the most essential contributions related to advancements of analytical and preparative liquid chromatography in theory and practice, thus becoming the highest cited chromatographer ever. Georges always fought for top quality scientific results and was strictly against soft compromises –

these criteria applied not only for himself but also for others.

For obvious reasons, Georges' lectures were extremely well attended and gave the chromatography symposia a special flavor, which we will very much miss in the future. Georges had strong opinions but at the same time he was warm hearted and very supportive to many students and post-docs who had the luck to work with him.

I had never worked with Georges on scientific projects but collaborated with him on diverse committees where it was always a pleasure to experience his quick and sharp analysis of certain situations. He continuously taught us new perspectives.

Behind a strong man is a strong women – his beloved wife Lois was exceptional in motivating Georges to live his life as actively as possible – an amazing couple we will never forget. – Wolfgang Lindner

Reference

1. *B. Karger, J. Chromatog., 556, XIII–XV (1991).*

The Coffee-ring Effect

A single photo got me thinking about sample preparation for mass spectrometry – that's the real beauty of art.

By Bob Blackledge, Forensic Chemist, El Cajon, CA, USA.

The photo, "Just a Drop" (page 26, Art of Analysis, August 2014 issue: tas.txp.to/1114/drop) showed four aqueous drops containing ink. Each drop had been deposited in the center of an area – a light blue circle – on a paper substrate. The blue areas had been treated to make them very hydrophobic. For this reason, the drops were spherical and showed no tendency to spread out on the surface.

I wondered about that circle. After the aqueous solvent had all evaporated, had the ink dye molecules or pigment particles been deposited in a "coffee ring" with most of the deposition along the ring rather than evenly distributed like a disk – or would they have a gradual increase in concentration towards the center?

The coffee-ring effect has interested me since the early nineties when I read the paper, "Liquid sample concentration technique using perfluorated polymer FILM for picogram analysis by FT-IR" (1). They defeated the coffee ring effect by depositing a drop containing their sample onto a flat, highly-polished stainless steel surface that had been treated so that it contained a very thin fluorocarbon layer. As the solvent in the drop evaporated, the contact angle of the drop with the surface caused any solute to be pulled along rather than being deposited along an outer ring. Eventually, you ended up with a small spot of concentrated solute rather than having it distributed around an outer ring. Since samples in forensic science may be quite limited, this appeared to be an excellent way of concentrating a limited amount of sample.

However, although both of these examples feature a treatment with a perfluorated polymer, one sample was deposited on a hard smooth surface and the other on a fibrous matrix. I wondered, although treated to make it hydrophobic, how could a fibrous matrix be able to defeat the coffee-ring effect?

A Google search led me to a video, "Hydrophobic Coating Technology Inspired by Butterfly Wings" from Richard Hammond's "Miracles of Nature" TV show (watch video: tas.txp.to/1114/butterfly). Hammonds explains that heavy raindrops do not harm the Morpho butterfly's wings because the microstructure of the material makes minimal contact with the water drops. He uses a balloon and frame model to illustrate that even if the balloon is in contact with the frame, there is actually very little contact between them.

In much the same way, the paper's fibrous cellulose matrix has minimal contact with the ink drop and the hydrophobic treatment prevents any wicking effect. As the solvent evaporates, the drop radius gets smaller. Presumably, as the drop becomes supersaturated with dye molecules, they are deposited onto the paper. In fact, there may even be a partial "inverse-coffee-ring effect" with a slight increase in deposited solute towards the center.

Although Xuan Mu envisions this effect as useful in paper microfluidics, it may also have application in mass spectrometry (MS). Something akin to the coffee-ring effect may happen in MS as a sample is deposited either on a desorption matrix or a substrate such as a silicon crystal. Sampling can be hit and miss, because you end up with hot spots (lots of sample) and dead spots (almost no sample).

It would be great to have my above reasoning tested. What would time-lapse environmental SEM show? What about MS imaging? Never stop reading the general scientific literature and keep an open mind; your next great inspiration may be sparked by something seemingly unrelated – like a beautiful photograph of ink drops.

Reference

1. *M. Ikeda and H. Uchihara, Applied Spectroscopy, 46 (9), 1431–1434 (1992).*

Inspiring the New Generation

Is the successful marriage of education and research in the challenging era of the financial crisis a reality or utopia?

By Victoria F. Samanidou, Associate Professor, Laboratory of Analytical Chemistry, Department of Chemistry, Aristotle University of Thessaloniki, Greece.

Academics, unlike other professors, have to deal with two issues: lecturing in the amphitheater and conducting research to cope with progress in the scientific arena. Teaching at a university is completely different from teaching in high school – professors have to communicate primary knowledge rather than reproduce material from books.

Fundamental chemistry is of course the foundation on which a syllabus is built, but new findings are the bricks for the continuation of building science. Even results of low impact or importance, or even negative results can be used for this building process. And who is going to continue this construction in the field of analytical chemistry? The answer is the new generation of analytical chemists.

Students in their early twenties are the ones who have the big task of first learning and then subsequently promoting research. But how can young scientists be challenged and inspired?

The new generation is fortunate as they live in a time when everything is very fast and easily accessible. They are all more familiar in using technology than their tutors are. Highly sophisticated instruments are available, which make life simple but sometimes boring. Many questions arise. What is the role of the analytical chemist? Is it solely the collection of data? What about the interpretation of results? Is it even still attractive?

Is the collection of raw data equally as fascinating as the formation of cobalt phosphate in an experimental tube? How impressed my students are when I show them precipitation of the colored sediments. How amazed they are to see those sediments disappear as the pH value changes. This is the magic of chemistry.

But is there still magic behind the numbers obtained during measurements? Yes – when considered in the right way; for example, when plotting numbers and seeing how well they fit to the line or curve, or using those numbers to support an initially stated hypothesis. Undoubtedly, students must be open to receiving the signals that instructors give them whilst using skill to create magic of their own. We all have to take into account that "the mind is not a vessel that needs filling, but wood that needs igniting" – a quote of Greek Philosopher Plutarch. In other words, we should create an impulse for them to think independently.

But, as academics, are we instructors or researchers? Which task must receive priority and how easy is it to find the right balance between the two? And, given the above, how can we spark excitement in young analytical chemists?

We certainly have to be up to date with innovative research, but we also have to attract the students' interest and pass the spirit to them. Students are not interested in publication numbers, H-index or G-index. Most of them are not even aware of these figures of merit. Students need inspiration. Discussing the results or the problems that arose during research in the class can be exciting for some students and less interesting for others. Sharing our passion can be determinative for those who can see the magic behind crude research findings.

Young scientists have to realize that doing research simply for research's sake is not the target. Research is a tool used to create solutions to analytical problems that can be applied in many important fields, such as the medical, pharmaceutical, environmental, or food industries.

But how can we reach this goal in a period of global financial crisis? How can we meet the challenge of making big progress with innovative and pioneering research despite shortages? Maybe from this point of view, magic is more necessary than ever. And let's not forget that the new generation of analytical chemists will confront the same issues.

Students should feel that their dreams can come true, even though they are pretty much aware that further studies may not be financially viable or that they may not find a job that matches their qualifications. We – the academics – must help the new generation envision a brilliant future – putting aside all shortcomings of the reality.

The point is that future analytical chemists, like all students, need some time dedicated to their concerns. Professors must be their mentors – not inaccessible legends. We must get close to them and feel the pulse of the present. In that way, we can use the wisdom of the past to build a better future. Remember what Confucius said: "If your plan is for one year – plant rice; if your plan is for ten years – plant trees; if your plan is for a hundred years – educate children."

Sharing Data – and Knowledge

How to use a knowledge repository that doesn't retire or leave you for a competitor.

By Steve Thomas, Investigator, GSK, Ware, UK.

Millions of dollars have been invested to harness data for intellectual property protection and regulatory purposes, but the industry is severely lacking systems that re-use the data generated in analytical laboratories on a daily basis. In fact, many organizations still rely on scientists' brains or interpretations scribbled on paper spectra when it comes to analytical data and knowledge, even though far more data is generated than can possibly fit in a person's head.

My colleagues and I are responsible for studying the metabolic fate of molecules in development for GSK's drug metabolism and pharmacokinetics department. We generate and consume a lot of data (analytical, structural and species specific) to build metabolite schemes that help us to understand the fate of molecules. Until a few years ago, a lot of our data were recorded on paper, so when I tried to discover if anything similar had been seen in another project or species, I had to ask colleagues or search through the paper files. I also had colleagues in the US, so sharing data in the days of paper records was extremely difficult, particularly as they used software to store analytical data, but not to map metabolic outcomes. I suspect you'll find a similarly fragmented approach to analytical data in other global companies.

Quite often the terms 'data', 'information', and 'knowledge' are used indiscriminately and interchangeably, but an understanding of these terms can help you identify where you have a gap.

- Data is raw and represents a set of discrete facts; it has no significance beyond its existence.
- Information is data that have been processed to derive meaning and purpose.
- Knowledge is the human understanding of the subject matter, acquired through study and experience, which helps us draw meaningful conclusions.

These terms form an ascending scale of value and context. The following metaphor makes the difference clear: Out shopping, you might spot an old colleague. The facial recognition represents data. The value is increased by information or metadata that begins to fill in the picture. You remember his dog's name and what his daughters were studying in school. Knowledge is how you recall that he is dreadfully dull! You quickly duck into a store to avoid him, thus using your acquired knowledge to guide your actions to a preferable outcome.

Several years ago at GSK, we set out to create a repository of knowledge that doesn't forget, doesn't go senile, doesn't retire, and doesn't leave the company for a competitor. Our goal was to capture spectra generated in sample investigations, as well as the context (associated metabolites, and project details) and insights associated with the data (interpretation and conclusions drawn), so that our investigators could share information easily and learn from past outcomes. The end result was very positive and allowed us to better manage our knowledge, which is why I am sharing it here.

Our solution was to implement software from ACD/Labs that could store, search and share analytical and metadata linked to structures in a biotransformation map. We were able to collect analytical data from different techniques (mass spectrometry and nuclear magnetic resonance are widely used in our research); connect it with metabolite structures and other information; and map the data onto biotransformation schemes that record the metabolism pathway, where the parent drug can turn into 100 metabolites. Importantly, it was a way of sharing data with colleagues worldwide so that we could all benefit from previous experiences when looking to develop compounds that could avoid a particular metabolic fate. The data could be searched from almost any facet; for example, by molecular mass, project, analyst, site, species, or structure. Sharing data is extremely important because access to colleagues' findings can give you confidence in your own conclusions, or reveal additional considerations when analyses have proven tricky.

We've configured the software to fully meet our needs and it's also provided other benefits beyond access to information. Reports that used to take weeks to compile, requiring cut and paste from various vendor software and data management silos, can now be created much more easily.

We haven't looked back, but I can see why others in our industry may be wary. Even when a new technology or piece of software offers benefits, the pharma industry is cautious of change, after all there is a mentality of: "if it ain't broke, don't fix it" – the change curve could bring about a dip in productivity. But many companies are perhaps unaware that if they don't facilitate the sharing of data and knowledge, they are already experiencing lower than optimal productivity.

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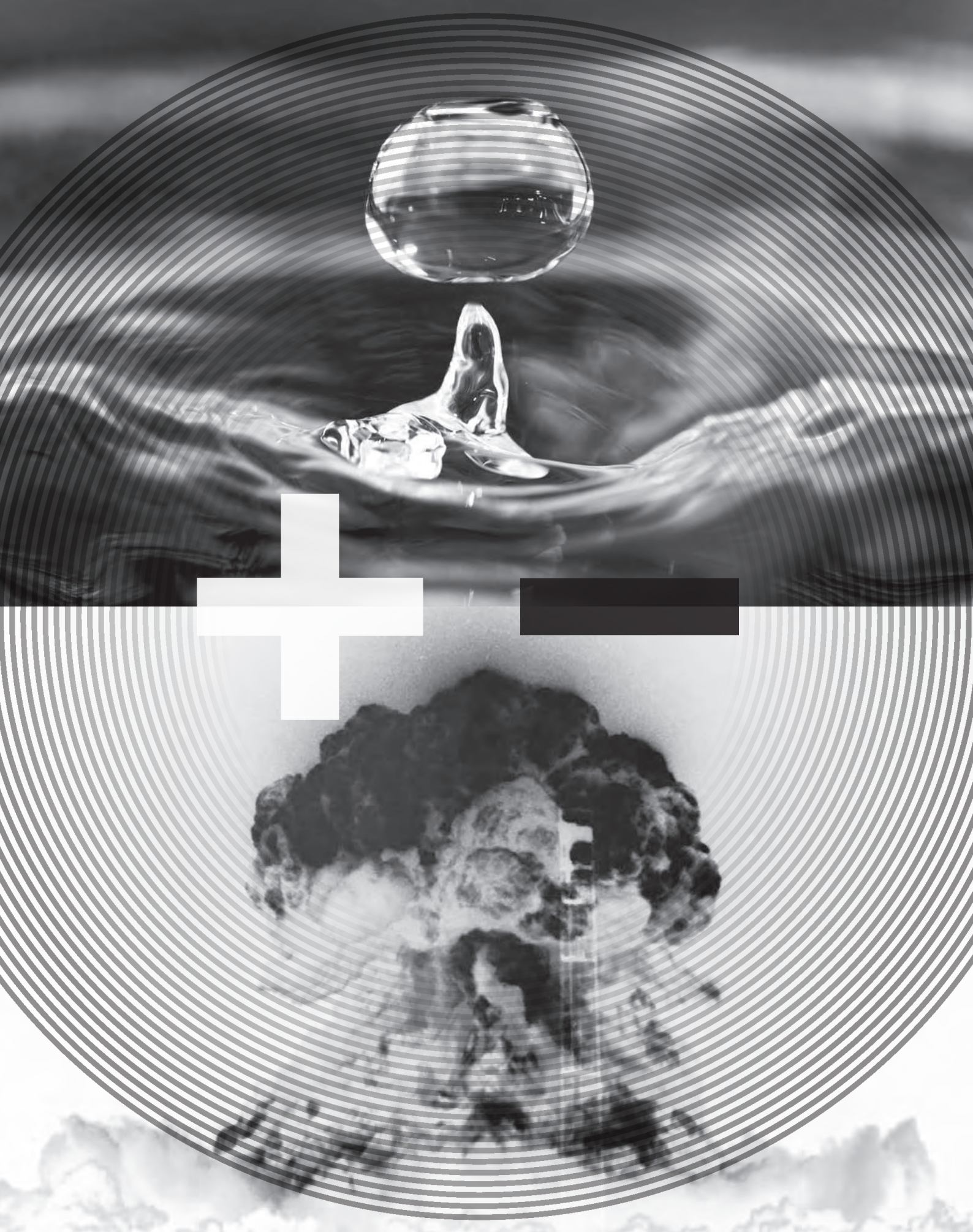

Three Gurus of Ion Chromatography

Ever since the innovation injection that was the "Manhattan Project", ion chromatography has defied probability by exhibiting almost continuous evolution into its current state-of-the-art form. Here, our three gurus – representing the two major commercial players and a significant academic contributor – reflect on the major milestones and future direction of this mature but still advancing technique.

Where does ion chromatography fit within the world of analytical chemistry?

Joachim Weiss: Over the past almost 40 years that encompass its birth and development, ion chromatography (IC) has become the most dominant method in ion analysis, replacing many time-consuming and laborious wet-chemical methods such as gravimetry, nephelometry, and turbidimetry. While in its earliest embodiments IC focused primarily on the analysis of inorganic anions and cations, today IC has an important role in the analysis of organic ions as well. Although part of liquid chromatography (LC), the term ion chromatography was created to make people aware of the fundamental differences in the separation and detection of ions as compared to traditional reversed-phase liquid chromatography (RPLC) of molecular organic species. Today, the term 'ion chromatography' can be defined as an umbrella term for all kinds of liquid chromatography techniques that are suitable to separate and detect ionic or ionizable species.

Paul Haddad: IC is unquestionably the premier analytical technique for the determination of low molecular weight organic and inorganic anions. It is also a very useful technique for the determination of low molecular weight inorganic and organic cations. I should note that for inorganic cations, modern spectroscopic methods, such as inductively coupled plasma atomic emission spectrometry (ICP-AES) and ICP-mass spectrometry (MS) are both faster and more sensitive.

Markus Läubli: Ion chromatography is a crucial method for the analysis of inorganic as well as organic ions. For anion determinations, I would agree with Paul Haddad and even describe it as the method of choice. With inline sample preparation techniques, it's possible to analyze ions in almost any matrix with high selectivity and sensitivity within a short time. While cations are often analyzed using spectroscopic methods, ion chromatography can be a more favorable alternative. For ammonium compounds, for example, IC is again the method of choice. Also, speciation analysis is getting more and more in the focus of modern ion chromatography.

The Gurus

Paul Haddad:

Paul Haddad is Distinguished Professor of Chemistry at the University of Tasmania, and from 2001-2013 was the Foundation Director of the Australian Centre for Research on Separation Science (ACROSS - see page 28). He has worked in IC for more than 30 years, with a special emphasis on the development of algorithms for computer-assisted prediction of retention times in IC.

Markus Läubli

Markus Läubli is Manager, Marketing Support IC at Metrohm's Competence Center IC, supporting IC sales and applications worldwide. He started on IC development for Metrohm almost 30 years ago after his thesis at Eidgenössische Technische Hochschule (ETH) in Zurich.

Joachim Weiss

Joachim Weiss is Technical Director of the Chromatography and Mass Spectrometry Division (CMD) of Thermo Fisher Scientific, where he is responsible for global sales support of the former Dionex liquid chromatography and sample preparation products.

Where are the strongholds of IC?

PH: I will mention four groups (excluding my own group!): First, IC research and development staff at Dionex (now Thermo Fisher Scientific), particularly Chris Pohl and Nebojsa Avdalovic. Over a period of almost 40 years, these staff have maintained a position at the absolute forefront of research and developments in IC, leading to numerous high-level innovations that collectively have maintained Dionex as the unquestioned leader in IC.

Second, the late James S. Fritz from Iowa State University, who made profound contributions to the non-suppressed form of IC, commercialized by Waters, Metrohm and others.

Third, Sandy Dasgupta from Texas Tech University and then University of Texas at Arlington, who has been responsible for a long list of highly innovative and highly original developments in suppressed IC.

Finally, Charles Lucy of the University of Alberta, Canada, who has been responsible for many insightful fundamental studies in IC.

ML: Besides the two main manufacturers of IC instrumentation, a few academic groups are working on further development in IC. Just to name the bright stars: Paul Haddad, Sandy Dasgupta, and Hamish Small.

Though it's true that Metrohm did not invent IC, I feel we brought it to a higher level. We made it the robust, versatile method that it is today, for routine as well as research applications. An important part of our contribution to IC has been in terms of user friendliness; take, for example, state-of-the-art Metrohm Inline Sample Preparation (MISP). Our focus on system quality started a competition from which everybody – manufacturer and user alike – has benefited and will continue to benefit.

JW: Actually, the list is long – I probably can't be definitive, but I can cover many of the bases… The heart of any ion chromatographic system is still the separator column. It is relatively unique for ion chromatography that the fundamentals in modern, pellicular ion-exchange stationary phase design were developed in industry by Hamish Small and colleagues at Dow Chemical in the US. Based on the groundbreaking work of Small's group, Christopher Pohl and his entire research team

at Dionex Corporation (now part of Thermo Fisher Scientific) are definitely the most significant contributors to IC stationary phase design up to the present day.

Other important contributions in the field of IC stationary phase design were made by Charles Lucy (as noted by Paul) who especially focuses on separator columns for Fast IC, Brett Paull and Emily Hilder (University of Tasmania, Hobart, Australia) as well as Kelly Flook (Dionex Corporation) in the field of ion-exchange monoliths, and lately also by Andreas Seubert (University of Marburg, Germany), to name just a few.

The most remarkable contributions in the field of IC detection were made by Dennis Johnson (Iowa State University, Ames, USA) who developed the concept of pulsed amperometric detection, which today is one of the most common detection techniques for carbohydrates and related components. William LaCourse (University of Maryland, Baltimore, USA), a former student of Johnson, further developed this technique by introducing the concept of multicyclic waveforms for the detection of amino acids, amines, and divalent sulfur compounds. The recently introduced concept of charge detection in IC is based on the groundbreaking work of Purnendu (Sandy) Dasgupta (University of Texas, Arlington, USA).

> *The overall success of ion chromatography in ion analysis can only be understood by comparing it with traditional wet chemistry used before the introduction of IC.*

General contributions in ion chromatography developments, such as the concept of Reagent-Free IC (RFIC), were made by Yan Liu and colleagues (Dionex Corporation), while Kannan Srinavasan's group (also Dionex Corporation) are very well known for their contributions in the development of electrolytic devices such as suppressors and continuously regenerated trap columns.

Last but certainly not least, Paul Haddad's group (also University of Tasmania, Hobart, Australia) have to be named for a huge number of contributions in the field of applied ion chromatography. Together with Peter Jackson, he also published an important textbook on ion chromatography.

What's the secret of success in IC?

PH: Not an easy question for me to answer – you should ask those involved! However, one thing in common is that all groups have worked collaboratively with others (in fact, with each other). In the case of Dionex/Thermo Fisher they have collaborated actively with academics (especially the academics on my list), whilst in turn the academics on my list have all collaborated with industry (mostly with Dionex). IC researchers are therefore all strongly interconnected and there is a strong sense of working together. You could almost say that IC researchers around the world form a single, large family exhibiting a high degree of cooperation rather than competition.

JW: I can't speak to the success of individual groups – certainly good collaboration is key as Paul notes. But I would like to say that the overall success of ion chromatography in ion analysis can only be understood by comparing it with traditional wet chemistry used before the introduction of IC. In contrast to atomic absorption spectroscopy (AAS), photometry, titration, and ion selective electrodes, IC offers simultaneous determination of many ionic sample components, even when concentration differences between sample components are very disparate.

The second reason for success of IC in comparison with wet chemistry is speed. Today, complete anion or cation profiles are achieved in significantly less than ten minutes. Determination of ions down to sub-µg/L levels utilizing large-volume injections indicate the enormous sensitivity that IC provides nowadays. Determinations down to single-digit ng/L levels after pre-concentrations became routine in the semiconductor and nuclear power industries. However, the only limiting factor is ubiquitous ions, such as chloride or sodium. The large variety of universal purpose and application-specific stationary phases as well as specific detection systems, such as suppressed

conductivity, amperometry, charge, UV/Vis, and hyphenated techniques (IC-ICP, IC-MS), account for the enormous selectivity that IC provides. Thus IC succeeded initially, because it provided a powerful solution to an unanswered question, especially in the field of anion analysis.

ML: The "secret of success in IC" for us should be defined much less in academic terms but rather in the practical dimensions of ion chromatography. "What can I do with it and will it work for me?" – these are the questions that customers are asking and, as a solutions provider, the ones that we are addressing. Instrument design, ease-of-use, and relentless application work for and with users are the factors that make or break success in IC. "Success in IC" in that sense has come over time. IC as a technology has been 'democratized'; today, it is a robust, highly sensitive, and affordable mainstream analytical technique that may be used by anyone. Smart engineering has brought the running costs of ion chromatography down, especially when compared to competing high-end spectroscopic techniques.

Instrument design, ease-of-use, and relentless application work for and with users are the factors that make or break success in IC.

Can you walk us through the last few decades of IC development?

JW: Upon introduction, ion chromatography was characterized by stationary phases of relatively low chromatographic efficiency due to the large particle sizes. Although the seven so-called standard anions were resolved to baseline, resolution was limited, and analysis times were around 30 minutes. Despite injecting large sample volumes of approximately 100 µL, lower detection limits than 100 µg/L were not possible by direct injection. Mono- and di-valent inorganic cations could not be analyzed simultaneously. Gradient elution techniques in combination with conductivity detection in both anion and cation exchange chromatography were unknown due to the lack of high-capacity suppression devices, and detection systems were limited to conductivity, DC amperometry, and UV/Vis detections.

The introduction of the concept of electrolytic eluent generation revolutionized IC not only in terms of ease-of-use, but allowed the application of gradient elution based on hydroxide or methanesulfonic acid eluents for anion or cation exchange chromatography, respectively. Modern stationary phases are based on much lower particle sizes, resulting in higher chromatographic efficiency and thus, in higher resolution and much higher sensitivity. The downside of using smaller particle size columns is the significantly higher backpressure. However, modern IC instruments are capable of tolerating up to 5000 psi (34.5 MPa) back pressure due to PEEK-based fluidics. Also, the range of specific and non-specific detection systems is much wider today. The higher degree of automation in combination with dual instrument configurations and software solutions provides a great deal of flexibility.

ML: Most importantly, ion chromatography has become more straightforward. Not only has the range of instruments from basic instrumentation up to sophisticated high-end systems grown, but also a lot of developments in automated sample handling and new features in the ease of use have arrived. Ion chromatography has moved from a sophisticated technique that required a lot of special skills to a straightforward routine method.

PH: IC has been around for almost 40 years and is now a very mature analytical technique. Changes have been profound and are too numerous to mention. These changes fall over the whole spectrum – stationary phases, instrumentation and hardware, suppressors, miniaturization, developments in theory, and so on.

What major milestones brought us to the current state-of-the-art?

PH: (i) The detailed understanding of IC theory and retention mechanisms made it possible to predict retention times under varying eluent conditions, and thereby optimize separations and extract the best possible performance from IC systems.
(ii) Exquisite understanding of the factors that influence separation selectivity in IC led to the design and synthesis of stationary phases of diverse and predictable selectivity, capable of separating highly complex mixtures.
(iii) Major developments in hardware, in particular electrolytic suppressors and electrolytic eluent generators, enabled IC to be performed with only water as eluent. IC must be the only chromatographic technique in existence in which the eluent and the final column effluent consist only of water. These features must certainly qualify IC as the "greenest" analytical chemistry technique in existence today.

ML: In contrast to HPLC, IC was not initially feasible with direct (conductivity) detection mode because of the high background conductivity of the eluents. Only the development of a post-column reaction – the so-called chemical suppression – made IC possible. The next important step was the development of high-performance conductivity detectors with highly shielded and thermostatted detector blocks. They opened IC to direct and suppressed conductivity detection, thus widening the application range from strong and medium strong acid anions to the full range of anions. For cation determination, columns that separated alkali and alkaline earth metal cations ushered in a new era. All the more because ammonium and amines – not detectable by AAS and ICP – could then be easily separated and determined.

A stronger focus has been placed on further detection modes such as UV/VIS, amperometry and the hyphenation with MS and ICP-MS.

With the introduction of inline dialysis, the era of automated inline sample preparation – that is to say, automatic sample preparation before injection – began. Techniques such as inline ultrafiltration, inline dilution, inline matrix elimination followed. Using intelligent software, logical decisions have become feasible, which allows us to check whether a result is within the calibration range. If not, the sample will be automatically rediluted with an appropriate dilution factor – all without user intervention.

JW: One of the first major milestones in the development of IC was the introduction of membrane-based suppressor devices in the mid-1980s. Membrane-based suppressor devices are continuously operated and have a much higher suppression capacity allowing for the use of high-capacity ion exchangers, which are important for analyzing closely eluting ions of disparate concentration levels. Around the same time frame, pulsed amperometry was introduced as a new detection method for sensitive detection of carbohydrates and related compounds. The concept of RFIC mentioned above is another important milestone introduced at the end of the 1990s. Until then, eluents and regenerants had to be prepared manually – a source of labor and uncertainty. RFIC involves the generation, purification, and suppression (for conductivity and charge detection as well as for hyphenation with MS) of eluents by means of electrolysis. Thus, high-purity, contaminant-free eluents are generated in situ with an electrical control of eluent concentration, which is a prerequisite for gradient

The Players

Metrohm AG manufactures precision instruments for chemical analysis in its own facilities in Herisau, Switzerland. Metrohm focuses on ion chromatography, titration and metering (for example, pH and conductivity), as well as voltammetry and spectroscopy. Metrohm sells and supports instrumentation and applications worldwide through exclusive subsidiaries.

Headquartered in Waltham, MA, USA, Thermo Fisher Scientific is a manufacturer in the genetic testing and precision laboratory equipment markets. Dionex Corporation – an ion chromatography products specialist – has been part of Thermo Fisher Scientific since May 2011.

elution techniques.

One of the most recent milestones in the development of IC was miniaturization down to the capillary level, which enables IC users to operate their systems 24 hours a day over the entire year without processing large amounts of liquids. Capillary IC saves the time for system equilibration and re-calibration, and offers a 100-times higher mass sensitivity. It is predominantly used in routine analysis, where chromatographic conditions are not changed over a long period of times.

The introduction of charge detection two years ago is a milestone in detection development as it provides larger response factors and an almost linear calibration for weakly dissociated ions that exhibit smaller response factors and a non-linear calibration behavior in suppressed conductivity detection. The introduction of high-pressure IC three years ago provides an instrument platform for high-throughput or high-resolution separations in IC utilizing 4 µm ion exchangers.

What are the big challenges that remain?

PH: (i) Miniaturization and field portability. (ii) Higher efficiency particulate stationary phases, especially using successful principles demonstrated by UPLC, such as sub-2 µm particles and core-shell particles. (iii) Increased peak capacity columns. (iv) Extremely rapid (sub-minute) separations.

ML: 'Ease of use' on instrumentation and software is still a large field for development, by which I mean that instruments and software need to check system suitability during routine runs. The user requires information about any change in performance immediately and before obtaining wrong results. On the other hand, we are looking forward to tracking any new developments in research and development.

I would also like to say that quality is not only how instruments are built and software is designed. Quality means ongoing work and a dialogue between manufacturer/supplier and user. Development of new and alternative phases for IC columns is still a challenge. Faster separations, a broader range of selectivity, and higher stability are still required.

JW: One of the biggest challenges in ion chromatography is separation of ions at very disparate concentration ratios; for example, saline samples in environmental (seawater), in petrochemical samples (formation water), and in industrial samples (brines), which can only partly be achieved by one-dimensional chromatography using selective stationary phases in combination with specific detection methods. The situation is even worse in the analysis of ionic contaminants in ultrapure chemicals that are used in the semiconductor industry. In the latter case, two-dimensional separations (2D-IC) and heart cutting techniques (IC×IC) are required, which are currently being developed. A lot of emphasis is also directed toward the integration of sample preparation processes into the total analytical solution in the form of in-line sample preparation techniques.

> *IC has defied the odds by evolving continuously into new formats and this evolution has maintained the vigor and freshness of IC as an analytical technique.*

Are those challenges being adequately addressed?

PH: I believe that the areas I mentioned are receiving some attention, but the level of this attention and the intensity of focus will be market-driven.

ML: Today, modern IC reacts to the user, as Paul says. In the past, it was often the other way around, not least because the previously mentioned detection/suppression tasks had to be solved. To address new challenges – faster separations, improved stability, and a broader range of selectivity – we need to be as close as possible to users and their problems. In

this way, we can allocate demands, to which our experienced R&D responds. In other words: tomorrow's products are fueled by the customer's needs and are realized with our knowledge and experience.

JW: Absolutely! Instrument manufacturers in cooperation with the respective industries have a high degree of interest to tackle these problems. While industry needs a solution for these problems, instrument manufacturers would like to expand the market for IC with analytical solutions. On the other hand, research groups like Peter Schoenmakers' (University of Amsterdam) are currently looking very intensively into two- and multi-dimensional separations to increase peak capacities in the minimum amount of time.

How do you see IC developing over the next 10–20 years?

ML: Ion chromatography will be applied to a broader range of applications, both in the lab and in process analysis. The analyst's dream should come true: place the sample on the autosampler, press the start button – and get the result. Sample handling will be much more automated with respect to sample preparation but also with respect to quality assurance. The software will focus more on plausibility and quality checks of results. Moreover, multiparameter analysis, the combined ion analysis, such as IC and titration (TitrIC), and IC and voltammetry (VoltIC) will become more important and will include more parameters.

PH: As mentioned above, IC has been available commercially for almost 40 years and is therefore a very mature technique. IC has defied the odds by evolving continuously into new formats and this evolution has maintained the vigor and freshness of IC as an analytical technique. One might expect the rate of developments of IC to moderate, but I suspect that this will not occur due primarily to the intensive efforts of Dionex research staff. It is also fair to say that the cohort of dominant IC researchers worldwide consists of researchers nearing the end of their career. A new cohort of active and talented IC researchers will therefore need to emerge to take the mantle from the outgoing researchers.

JW: Ion chromatography is a very well established and well-developed analytical method. Nevertheless, we have still seen some exciting new developments in recent years that are outlined above, such as Capillary IC, charge detection, and high-resolution separations based on smaller particle size columns. I think it's very difficult for anybody to predict the development of an analytical technique over such a long time of 10-20 years; however, for the near future I see a lot of potential for further development in a much greater degree of automation based on hardware and chromatography software developments as noted by Markus. Separation times will definitely decrease in standard ion analysis and high-resolution separations will become available on a routine base. As mentioned above, turnkey solutions for two-dimensional separations might become available very soon. A big gap could be filled with the discovery of materials that are as chemically stable as PEEK but with a much higher pressure tolerance – that would allow IC to follow more closely the transition from HPLC to UHPLC. Within the foreseeable future, it will be more and more difficult to differentiate between IC and RPLC. With the introduction of mixed-mode stationary phases, for instance, we see the same trend in separation science as in instrument hyphenation – that is to say, the combination of several retention mechanisms will enable us to separate ionic and/or ionizable analytes we presently cannot separate with either one of the retention processes.

I would say that column miniaturization down to microbore (2 mm and 1 mm) or even capillary (0.4 mm) will eventually replace standard bore (4 mm) columns due to the savings in eluent consumption and eluent disposal. This transition would also enable the operation of ion chromatographs over a long period of time. Hyphenation of IC with ICP or MS for element-specific or mass-selective information will definitely help to overcome the once very competitive thinking of scientists by combining the best of both worlds instead of pitting one against the other – it shouldn't be chromatography versus spectroscopy!

I don't see any IC technique falling by the wayside any time soon – analytical chemists love to have a huge toolbox to solve analytical problems!


Hobart
147°

Cutting ACROSS Separation Science

I've been running analytical chemistry research groups with military precision for decades. Over that time, I've had the good fortune to work with the brightest minds in the field on the most rewarding and exciting projects. My greatest hope was to leave a lasting legacy on retirement – and I'm proud to say that the Australian Centre for Research On Separation Science fulfills that dream. That's the end of the story, but clearly there is a beginning and a middle. Here I present my notes from a small island…

By Paul Haddad

At the age of 15, I was the youngest person ever to be accepted into the Royal Military College, Duntroon – a prestigious training institution for army officers in the Australian Capital Territory. And that's how my career began in 1966 – in the army – at the tender age of 17, studying mathematics, physics and chemistry equally…

It wasn't possible to graduate with a formal degree from the college back then. Duntroon was in the process of being affiliated with an Australian university, but, unfortunately, that was too late for me. I graduated as a first lieutenant, but somehow convinced the army to send me to the University of New South Wales (UNSW) in Sydney to complete my degree. And so, I found myself doing another two years of chemistry (1969–1971) towards the end of the Vietnam War. The Australian people were very opposed to the war and I was an army officer. It was a pretty hostile environment for people of my persuasion, but I succeeded in disguising myself by growing my hair and a beard (much like the editor of this publication).

During those two years, I discovered a real affinity for chemistry and decided that I'd prefer an academic career. I asked the army to allow me complete a PhD. They initially declined; however, troops were being withdrawn from Vietnam as the army transitioned into peacetime mode, so my application must have looked increasingly inviting. I completed my PhD in chemistry in 1975.

I spent the next 10 years or so trying to formally resign from the army, who were making my life very difficult. Eventually, we settled on terms of separation (no pun intended; I had to pay them back some money over a period of time). In addition to fighting with the army, I also found time to spend four years at the Australian National University in Canberra, after which I was appointed back in the department where I had completed my PhD at UNSW and commenced my academic career in earnest. I stayed there until 1992, progressing through the ranks to become a full professor.

I have to admit the only reason I started out in separation science in the first place is because, on my return to UNSW, my head of department told me that I must develop an interest

in chromatography – something that I knew virtually nothing about. In fact, my PhD was in an unrelated field – molecular fluorescence spectroscopy – and it's interesting to note that after my PhD, I never had anything to do with that field again. I guess that proves that a PhD is just a training program; at the end of it, you should be able to do anything you want as long as you've learned the correct way to undertake research – that's something I always stress to my students.

In any case, I started learning about chromatography and in the 13 years that followed, sure enough, I developed a keen interest in separation science. And because I had been trained as an inorganic chemist I became an inorganic chromatographer – hence my interest in ion chromatography (see "Three Gurus of Ion Chromatography", page 22).

To Tasmania!

I visited Tasmania for a conference with my wife and family in 1991. It was the middle of winter and we spent one week at the conference and another looking around Tasmania. At the end of the two weeks, I was hooked – there was something magical about it. I walked up to the University of Tasmania in Hobart and asked if they had any vacancies. They did – for a professor of chemistry – but applications had closed. Fortunately, they allowed a late submission.

In the interview, my narrow focus in chemistry was exposed – they asked questions about organic and physical chemistry that I couldn't answer. My entire career, I'd been a chemist in the analytical department of a big school of chemistry so clearly, I spent much of my time enjoying the company of other analytical chemists. It wasn't looking good. When I was asked at the end of the interview if I had any questions, I said, "Yes, you've asked me about every area of chemistry except analytical, and that's the area I know about – so can I tell you what's going on in analytical chemistry and why separation science is at the forefront of those developments?" Fifteen minutes later the committee told me I had got the job.

My colleagues back in Sydney thought I was absolutely crazy to leave the largest, most successful chemistry department in the country. But I felt it was exactly what I wanted to do. We moved in 1992 to a bit of a rude awakening. When I started at the University of Tasmania, I found myself the head of a chemistry department that naturally covered all aspects of chemistry (a steep learning curve) – but there were no other analytical chemists. Today, the department of chemistry is about 60 percent analytical – a profound shift! I felt that part of my appointment was to develop analytical chemistry – I think history shows that's what happened.

"I agreed under two conditions. The first was that I stayed in my office in chemistry, and the second was that the faculty paid for a post-doc position for every year that I was dean."

I'd only been head of chemistry for about 10 months when the vice chancellor asked me if I would consider being dean of the Faculty of Science. I was struggling with even being the head of department, which I'd never done before – I was learning about budgetary and curricular aspects and so on. I was shocked to be asked to lead a faculty that contained another 10 schools, covering natural sciences, physics, engineering, psychology, zoology… it was extremely diverse – and accounted for about a third of the university. I doubted my own ability and asked the vice chancellor why I had been chosen.

He said, "Frankly, you're the only one who hasn't been here long enough to accumulate some baggage – you've not upset anyone yet. We think you'd be a safe appointment." I do like a challenge so I agreed under two conditions. The first was that


Hobart


Ion chromatography

Waters Under the Bridge

While at UNSW, I had a couple of six-month sabbaticals. The first was in The Netherlands at the Delft University of Technology, where I first met Peter Schoenmakers – I made friends there that I still have today. The second was with Waters at its headquarters in Milford, MA, USA. It was 1987, and I had been collaborating with them for a while. Waters were trying to break into the ion chromatography (IC) space – Dionex had the lion's share of the market. Waters actually offered me a job, but I agreed to a six-month sabbatical to test the corporate water… I obviously decided to stay an academic, but I worked closely with them for about 15 years. It was a very productive relationship. We got on very well on a personal and scientific level and sold a lot of instruments, collectively penetrating the ion chromatography market – but not enough for the managers, who pulled out in the early 1990s. I was with them at the start – and the end.

Both sabbaticals had profound influences on my subsequent work. At Delft my interest was piqued by computer optimization of separations. Here we are, 30 years on, and I'm still working on that. And at Waters, we got a lot of support in terms of both instruments and ideas, which got us involved in some interesting areas. During those days I co-wrote a book on ion chromatography with one of my PhD graduates – Peter Jackson (1). The 900-page tome was published in 1990 and was pretty much the bible of the field for 10 years (cited about 700 times, I think), putting me on the map internationally as far as IC was concerned. And it almost killed me, though it is without a doubt the best thing I did in my career and I feel very proud.

I stayed in my office in chemistry, and the second was that the faculty paid for a post-doc position for every year that I was dean. The long and the short of it was that my research career was not impeded by being dean, evidenced by no slowing in publications over those nine years. I was blessed with good people around me. I had excellent post-docs, like Brett Paull. And I had extremely good students – Emily Hilder and Michael Breadmore (both in the Top 40 Under 40 Power List), for example.

2001: an ACROSS Odyssey

In 2001, I was offered a third term as dean. But I had decided that I needed to break away from the path towards the higher echelons of university management. I wanted to get back into research. So instead, I applied for – and won – a professorial fellowship from the Australian Research Council (ARC) that allowed me to do full-time research.

It was while being able to focus on research that I got the idea of forming the Australian Centre for Research On Separation Science – ACROSS. The goal was to bring together the major players in separation science around Australia, so that they could collaborate and support each other. Basically, I wanted to lift Australia's international standing in the field.

I approached Phil Marriot, who was at the Royal Melbourne Institute of Technology (RMIT) University, and Milton Hearn from Monash University. We each got some money together to set ACROSS in motion. There were two agendas – the first was to set up a good network between the universities, and the second was to build up to a critical mass in our individual founding nodes. In 2001, my ACROSS group had about six people – we now have 60 in Tasmania. I should note here that we later took on another node at the University of Western Sydney with Andrew Shalliker. Today, the nodes in ACROSS have a total of about 90 people, so while it is a national center, the critical mass is in Hobart.

I had been enjoying my full-time research position for three years before the Australian government came up with the federation fellowship scheme. In part it was created to prove that it was possible to be as successful taking the research path as it was moving through senior management at a university. The salary was very attractive

"The fact that the US came to Tasmania and essentially recognized us as leaders in the field is hugely satisfying and a great example of what ACROSS can achieve."

and the fellowships were offered to all fields, so it was very competitive; there were only 12 positions available annually, as I recall. It took a fourth (and, I had decided, final) attempt before being accepted in 2006 for the five-year position. My plans for ACROSS were clearly a huge factor in winning the fellowship. Once again, I found myself in the absolute luxury of working in a full-time research role, so, all in all, I had eight years completely focused on research, which cleared the way for me to concentrate on the development of ACROSS.

I had thought long and hard about the way I wanted to move forward with my career after stepping down from the deanship of the science faculty. I wanted ACROSS to be my legacy. It was a deliberate strategy and I felt confident that, if I could sell the idea to my university and ARC, I could deliver.

Forging a legacy down under

What characterizes the team at ACROSS is solid strength over a breadth of areas. At the beginning, we wanted to assign leaders to different programs within ACROSS so that we could build up expertise equally across the board. For example, Emily Hilder looked after materials science, I ran the retention modeling and optimization program, and Michael Breadmore looked after capillary electrophoresis (CE). We have about nine solid areas where we are very respected by the community. If you ask for the top people in polymer monoliths, for example, Emily will certainly be up there, and the same goes for Michael and Joselito Quirino when it comes to online sample enrichment in CE. We've got people at the top of their game.

It's difficult for me to pinpoint individual groundbreaking papers. Rather, I like to look at the collective body of work generated, which I think is outstanding. To prove the point, there is a system in Australia called Excellence in Research Australia (ERA) that ranks research outputs in certain disciplines – analytical chemistry is a recognized sub-discipline. All papers that are published in analytical journals over a six-year period are collected, their citations measured, and then they are compared to global benchmarks. This analysis, in addition to other factors, such as funding received, prestige of staff and fellowships, and so on, is used to produce a ranking of between 1 and 5, where 3 represents the global average. We've been assessed three times now and gained the top score of 5 on every occasion.

One of the key objectives at ACROSS was to get people working across the spectrum of separation science so that one individual or group didn't eclipse the rest, creating friction and unhealthy competition. We mapped out the territory and recruited people to fill the gaps without having too much overlap; for example, we've also got Mirek Macka in microfluidics, Rob Shellie in two-dimensional gas chromatography, and so on. We've ended up with groups that are more complementary than competitive. And having groups that get along and work well together is the root of our success.

We've got a very unusual structure at ACROSS. It's not the typical pyramid of power. When we started, I was the professor at the top with a cohort of people who started at the same level. They were all ambitious and talented; the natural tendency with those characteristics is to try to forge ahead and make your own mark. But working as a collective under the umbrella of ACROSS meant that they could all advance faster than if they were competing for the same resources. In 2001, we had one full professor – we now have six. All of the people that started in that original cohort are now at the top. In academia, it's typical for people to fight to build an individual empire, but at ACROSS we've built a collective empire.

ACROSS antiterrorism

I'd like to highlight one area where our collaborative approach has had an international (and national) impact: counterterrorism. It was about seven years ago, when we were first approached by the Australian government, but in fact, the story starts with a very large explosion in Bali. The perpetrators made it quite clear that they were targeting Australians – over 200 people were killed, of which 83 were Australians – and it was the first time our country had been exposed to terrorism in that way.

We're in the Army Now

I come from a military family. My father was a lieutenant colonel, and I have two brothers (one a year older, one a year younger) – and we all went into the army. At one stage, we were all officers at the same rank in the same ordnance corps, which looks after logistics and supplies. To make matters worse, my brothers were called Peter and Philip, so we were all Lieutenant P. Haddad – and we all sound the same on the phone. The army probably couldn't manage that, so maybe that's why I was allowed to go off to university! At any rate, I ended up with the lowest rank in my family. And in fact, I think I ended up doing what amounted to only six weeks of active service! Nevertheless, the officers' training course was four years, and I think it's had an effect on my career.

In particular, the army teaches you a lot in terms of leadership; there aren't many places where you receive such active instruction in that area. We looked at whether it was best to get people to follow you and do as you say, or if it was best to stand back and let good people do the work without getting in their way, sometimes clearing roadblocks ahead without them even realizing – we also learned when each 'mode' was necessary. As noted, I spent nine years as the Dean of the Faculty of Science – the largest faculty with the biggest budget and the most students, while trying to maintain a research career. I'm not claiming I'm the world's best leader, but I think the army did give me an advantage.

Any other lessons learnt? Attention to detail is also key in the army – dotting the i's and crossing the t's – and I think that's a useful skill in research. I also learnt how to write properly, as strange as that may sound. I remember when I handed my draft PhD thesis (handwritten I might add) to my supervisor, he gave it back to me an hour later without a single correction and told me it was fine. I've had about 70 PhD students and I've never given one back without a lot of corrections…

After the explosion, there was a very large hole in the ground, so it was clearly a big device – likely a ton of explosives, but no one had any idea what material it was made from. No doubt it was an improvised explosive – a fertilizer bomb, for example, which can be made from ammonium nitrate and fuel oil (ANFO). But without confirmation, it wasn't possible to start searching purchase records to aid in the investigation. It took the authorities quite some time to identify that the bomb was in fact potassium chlorate-based, with sugar and aluminum as the fuel.

We were tasked with developing field tests for post-blast identification of improvised explosives, which could be achieved with ion chromatography or CE. A group of about 10 of us came together to work on the project. We subsequently developed a number of techniques and instruments that have been patented and licensed for production. For example, we made a field-ready CE unit with four channels that can measure inorganic anions, inorganic cations, high explosives (TNT, RDX), and peroxide-based explosives all from the same sample vial.

Our efforts attracted a lot of attention. Some of the equipment and procedures we designed is being used routinely by the Australian Federal Police. Next, we were approached by the US Department of Homeland Security, which offered us more funding. Over the years, we've probably received about five million Australian dollars to develop not only post-blast but also pre-blast analyzers. Currently, ion mobility spectrometry-based devices are used in airports to screen for explosives. Such devices are very good for organic explosives but extremely insensitive for inorganics. We've now built a CE-based device that can identify inorganic explosives by measuring anions. It uses a very short capillary so analysis takes about 40 seconds – a pretty spectacular achievement. And we've made a confirmatory ion chromatography test for positive CE results that takes about three minutes. Once again, that technology is on the road to commercialization.

The fact that the US came to Tasmania and essentially recognized us as leaders in the field is hugely satisfying and a great example of what ACROSS can achieve by getting experts to work together.

Corporate collaborations

I long since understood that collaborations with industry and governments are essential for success. When Waters Corporation left the world of IC (see sidebar "Waters Under

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"I would have to attribute my success to two abilities: the first is being able to recognize an opportunity when I see one, and the second is being able to recognize (and attract) talent."

the Bridge"), it left me free to work with Dionex and later Thermo Fisher Scientific – a fantastic collaboration that has lasted over 20 years. Each year, we've agreed on a number of projects, and most of the support we received has come in the form of instruments and consumables, which has been vital in allowing us to grow as a center; we weren't constrained by the equipment we could afford. Dionex and Thermo have been extremely generous with their support and technical expertise, and we jointly hold a number of patents and products.

In 2006, I was approached by Pfizer. They had already set up PARC – the Pfizer Analytical Research Center – in Ghent in Belgium, where Pat Sandra was director. Pfizer was interested in setting up another PARC in the southern hemisphere, and Pat suggested that they talk to me. And so, I received substantial funding to set up PARC Hobart, which funded four PhD students, eight post-docs, and a technician for about six or seven years. The final PARC projects are now coming to a close and it's been another wonderful collaboration. Over the years, I've learnt a great deal about the pharmaceutical industry – for example, where the main problems were and what we needed to focus on to remain relevant.

Bringing everything together, we've currently got a government sponsored grant to work with industrial partners – Thermo Fisher, Pfizer and LC Resources – to work out a way to predict the retention of a compound on the basis of its chemical structure. It's a very ambitious project with a great aim. The software will predict the technique, the type of column and the mobile phase needed for a target compound. A pharmaceutical company like Pfizer spends a lot of time with trial and error searching; scoping out methods in this way could potentially save billions of dollars. The results have been extremely interesting, and I have a strong feeling that we'll be successful across three fields of chromatography – reversed phase, HILIC and ion exchange. So far, we've released just enough information to tantalize people – we've had correlations between predictions and actual retention times of 0.99...

But that's not to say we want to keep it a secret. In fact, we are actually looking into crowdsourcing the whole project after we've proven the concept. Clearly, the key is in having a big enough training database, so we will need lots of carefully measured retention times – and the best way to do that is to get people to contribute. If we have a consortium of pharmaceutical companies, we can build up the database – in return, they get to use the software. Pfizer is now moving very much into an open innovation environment. Gone are the days when a pharmaceutical company keeps everything to themselves; the only way that big pharma can continue is by working with each other. And this project is an excellent example of that.

It's a very exciting way to finish my career – it combines the things I like doing best: working with highly respected colleagues from two of my long-standing collaborators and my fascination with computer modeling and prediction, which started back when I visited The Netherlands for a sabbatical 30 years ago.

The end?

In 2013, I stepped down as director of ACROSS, and I've been phasing out my responsibilities and passing them onto the new director – Brett Paull. I will formally retire on December 31, 2014. Looking back over the years, I would have to attribute my success to two abilities: the first is being able to recognize an opportunity when I see one, and the second is being able to recognize (and attract) talent. If you can create a network to combine those two aspects, you get good people working on good opportunities – and the rest follows naturally. My role has been relatively minor. I put the structures in place – but the developments and successes come from the team who has worked at ACROSS. You cannot underestimate the power of Tasmania to attract and keep talent. Certainly, ACROSS offers an excellent scientific environment, but it's matched with a great physical environment. And so, the caliber of people is extremely high. I don't know many places where you have a collection of 10 really top class individuals with international reputations that are complementary to each other. That's a winning formula.

Reference

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Speaker

Andreas Thomas has been working in the Cologne Doping Control laboratory for more than 10 years. His main focus is the LC-MS analysis of peptides and other prohibited substances from different classes of compounds, using high resolution mass spectrometry or related techniques. After graduating in food chemistry at the University of Bonn and Münster, he worked for three years as an analytical chemist in the pharmaceutical industry before switching to the German Sport University Cologne, where he earned his PhD in 2008. Andreas has developed and published various methods for the detection of prohibited compounds in biological fluids for sports drug testing.

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Shifting the Landscape of Nanomaterial Measurement

Collaboration between the University of Montreal and PerkinElmer shows how the unique capability of single particle inductively coupled plasma-mass spectrometry (SP-ICP-MS) can be used to assess the environmental impact of engineered nanomaterials.

By Chady Stephan, Madjid Hadioui, Kevin J. Wilkinson, and Robert Thomas

The Problem

The recent surge in the use of engineered nanomaterials (ENMs) in consumer products has resulted in concerns about their release into the environment. To properly assess their impact on ecological and human health, it is necessary not only to predict exposure through modeling, but also to perform quantitative physical and chemical measurements. Historically, metrics like particle size have been carried out using techniques such as dynamic light scattering (DLS), nanoparticle tracking analysis (NTA) and transmission electron microscopy (TEM), while dissolved content has been typically measured by ultrafiltration followed by inductively coupled plasma mass spectrometry (ICP-MS). However, these traditional techniques have known limitations when it comes to quantifying very low levels of ENMs in the presence of natural colloidal species in complex waters. As a result, they are not ideally-suited for real-world environmental matrices, where nanoparticle concentrations are typically extremely low.

The Solution

Single particle inductively coupled plasma mass spectrometry (SP-ICP-MS) is an exciting new technique for detecting and characterizing metal nanoparticles (NP) at very low concentrations. SP-ICP-MS is fast and can provide significantly more information than other traditional techniques, including particle number concentration, particle size, and size distribution, in addition to the concentration of dissolved metals in solution. The added benefit of using SP-ICP-MS is that it can distinguish between particles of different elemental compositions.

SP-ICP-MS is based on the measurement of the signal intensity produced by a single particle. Nanoparticle suspensions are sufficiently diluted to make sure that only a single particle reaches the plasma at a time, where it is atomized and ionized, producing a signal of relatively high intensity that is measured as a pulse. Figure 1 shows particles (P) and dissolved analyte in the sample aerosol entering the plasma and being ionized. As the ions pass through the interface region into the ion optics where they are eventually separated in the mass spectrometer, particles are detected as individual pulses, whereas the dissolved analyte contributes to a continuous shift in the background signal. The frequency of pulses (events) provides the particle number concentration, whereas the intensity of each pulse is proportional to the mass of the nanoparticle. Because of the short transient nature of the pulse, very short integration times are necessary to maximize the detection of individual particles as pulses of ions after they are ionized by the plasma.

Figure 2 shows both metal nanoparticles and metal ions in solution being ionized by an ICP-MS system (NexION 350X, PerkinElmer Inc., Shelton, CT). The signal from the dissolved ions is represented by the continuous signal below the dashed line, while the ionized pulses of nanoparticles are represented by the individual spikes.

For this approach to work effectively, the speed of data acquisition and the response time of the detector must be fast enough to capture the time-resolved NP pulses, which are typically 300-400 µs (1). If the electronics are not fast enough, two or three pulses can easily pass through in one single integration leading to inaccurate particle counting and sizing. For this application, the ICP-MS should be capable of using dwell times shorter than the particle transient signal time, thus avoiding false signals generated from clusters of particles. In practice, for single element nanoparticle studies, this means using a dwell time of less than 100 µs and a settling time of zero, so the pulse can be fully characterized and precisely integrated using a peak area integration algorithm (2).

Once generated, the signal intensity as a function of time can then be processed using a theoretical approach, first developed by Duegeldre and co-workers (3). In this approach, a histogram of NP diameter versus number of events (NP frequency/number) is generated in order to characterize the NP distribution in the sample. Figure 3 shows a size distribution histogram of 60 nm gold particles, with frequency of ion pulses (events) plotted against nanoparticle diameter, generated using PerkinElmer's Syngistix Nano Application Software Module (4).

Beyond the Solution

SP-ICP-MS is a very exciting development for the identification and characterization of nanoparticles at environmentally-significant levels and is being used to improve our understanding of how nanomaterials are interacting with the outside world. However, the technique is continually being fine-tuned to improve its detection capability even further. One of the main difficulties in the data processing for SP-ICP-MS is the discrimination between the intensities of dissolved metal

and ENP. This is particularly difficult for high concentrations of dissolved metal and/or small ENP sizes. With no silver present in solution, the minimum detectable size is 11 nm, whereas a concentration of 2 µg/L of Ag+ would degrade the detection capability approximately five-fold.

It is therefore extremely important to be able to remove or at least reduce this limitation, given that the smallest ENPs are most likely to be of greatest environmental risk. It should also be emphasized that particle solubility increases with decreasing particle size, so the smallest particles will always be the most difficult to quantify.

While some groups have used statistical methods or iterative software algorithms to remove the contribution of the continuous signal, recent research activity has focused on coupling SP-ICP-MS with separation techniques such as field flow fractionation (FFF) (6), or hydrodynamic chromatography (HDC) (7). One of the more promising approaches is to use ion binding columns coupled to the SP-ICP-MS to remove the dissolved metal ions in solution (5). Figure 4 shows a mixture of 0.05 µg/L of 20 nm Ag nanoparticles, 0.09 µg/L of 60 nm Ag nanoparticles spiked with 1.2 µg/L of Ag+ in solution. Figure 4a shows the raw signal for the nanoparticle and dissolved silver analyte, while Figure 4b shows the derived particle size distribution. Under conditions of high dissolved metal, it is not possible to distinguish the smaller Ag nanoparticles from the background Ag. But by using ion exchange resins, it is possible to lower background noise sufficiently so that the 20 nm Ag nanoparticles are detected. For example, when a SiSH (SiliaMetS Thiol, SiliCycle) resin, which is highly specific for Ag+, was employed, the background signal decreased substantially, as seen in Figures 4c and 4d, resulting in the easy discrimination of the smaller nanoparticles.

Somewhat surprisingly, a similar result was achieved when the sample was passed through a non-ionic, hydrophobic resin (Amberlite XAD 1180N), which is typically used to remove natural organic matter (NOM) from aqueous samples. This is shown in Figures 4e and 4f. For both resins, dissolved silver decreased, significantly reducing the background signal, whereas the resolution and identification of the particulate silver fraction improved substantially, as demonstrated in Figures 4d and 4f. In both cases, the signal intensity attributed

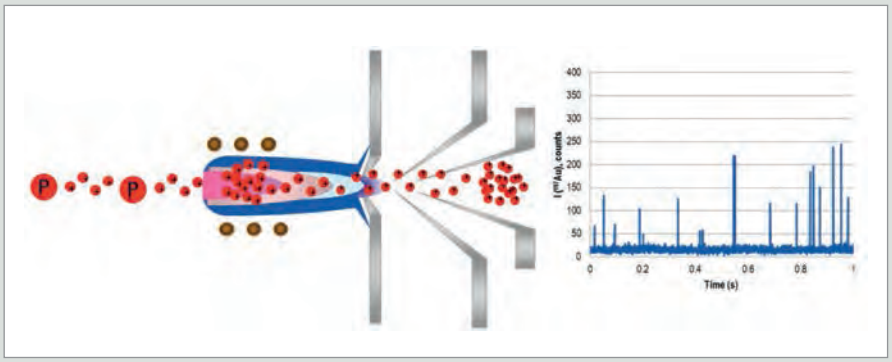

Figure 1: A suspension of nanoparticles (P) and dissolved analyte reaches the plasma where each particle is ionized, producing a signal that is measured as a single pulse.

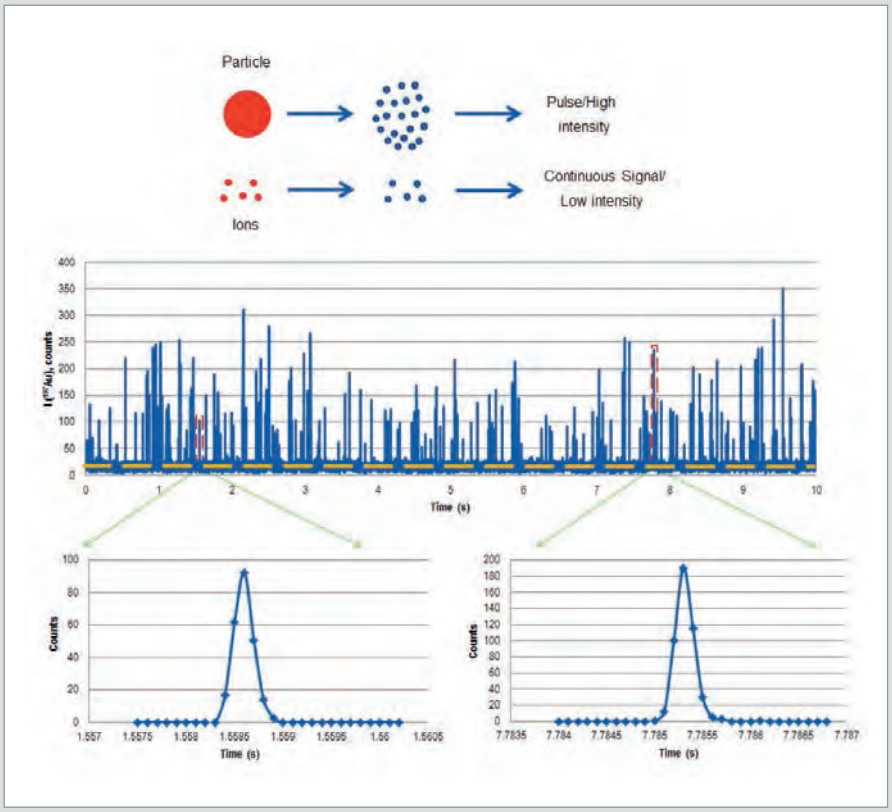

Figure 2: Metal nanoparticles (pulses) and metal ions in solution (continuous signal below the yellow-dashed line) being ionized in the plasma.

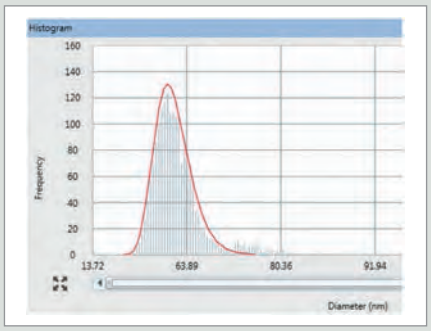

Figure 3: A size distribution histogram of 60 nm diameter gold particles showing frequency of ion pulses (events) plotted against nanoparticle diameter.

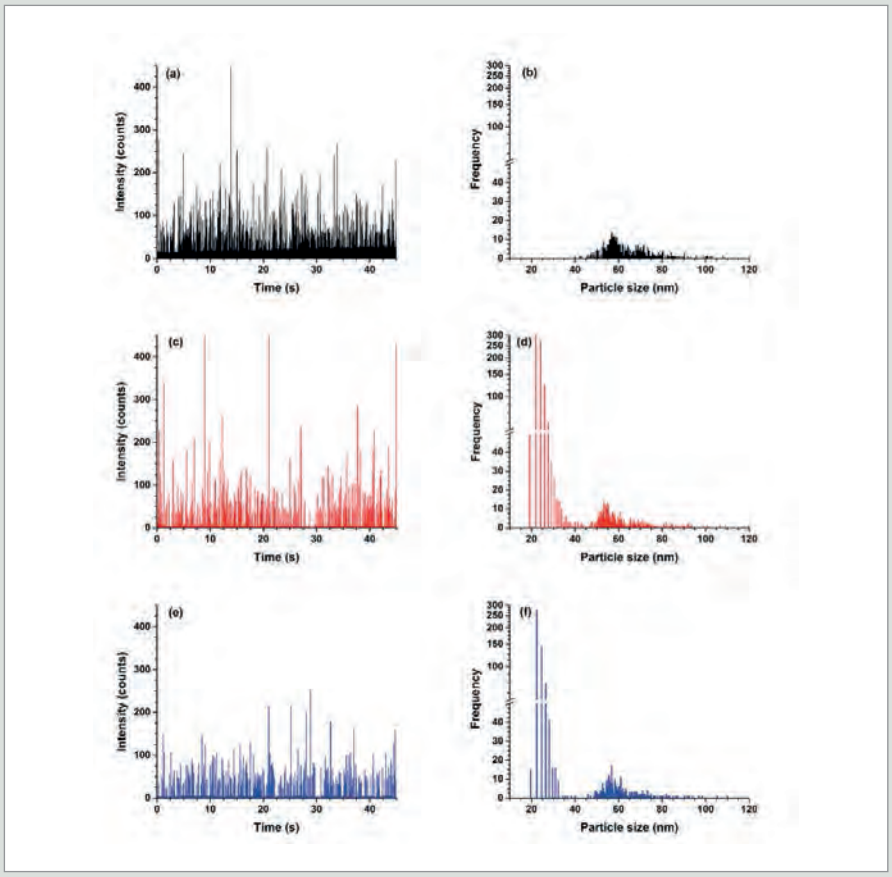

Figure 4: Signals from a sample mixture of 0.05 µg/L of 20 nm Ag nanoparticles, 0.09 µg/L of 60 nm Ag nanoparticles spiked with 1.2 µg/L of Ag+ in solution (4a), and the reduction of background signal from the dissolved silver after being passed through a SiSH resin (4c) and XAD resin (4e) demonstrating that greater discrimination of the nanoparticle pulses from the dissolved metal can be achieved. Derived size distributions are shown with no resin (4b), SiSH resin (4d), and XAD resin (4f) for the sample mixture described.

to the nanoparticles was constant, confirming that the nanoparticles did not appear to interact with either of the resins.

Much of the data presented in this study suggests that coupling ion exchange resins to SP-ICP-MS will further enhance the capability of SP-ICP-MS by detecting smaller-sized nanoparticles, particularly in the presence of other matrix elements. Under these conditions, where both the nanoparticles and ions were present, Hadioui and co-workers (5) have shown that this technique is capable of enhancing the sensitivity by a factor of 2-fold, resulting in a significant increase in the signal to noise ratio (5).

Summary

The excellent elemental sensitivity and specificity of ICP-MS makes it ideally-suited for the characterization of metal-based engineered nanomaterials at environmentally-relevant concentrations. In particular, using the technique in the single particle analysis mode allows more information to be obtained in a much shorter time, such as understanding how much of the nanoparticle has dissolved in the sample and how much is still in the suspended particulate form. In addition, recent developments of coupling SP-ICP-MS with ion exchange column technology is further improving its flexibility and detection capability and allowing this approach to be used for real-world environmental samples. However, it should be emphasized that measuring nanoparticles with ICP-MS in Single Particle mode is quite different to measuring dissolved species. For this reason, it is very important that the speed of data acquisition and the measurement protocol be optimized to detect and process these rapid transient events.

Chady Stephan is Manager, Global Applications-Nanotechnology at PerkinElmer, Inc., Shelton, CT, USA; Kevin J. Wilkinson is a Professor and Madjid Hadioui is a Research Associate, both at the Université de Montréal, Canada; and Robert Thomas is Principal Consultant at Scientific Solutions, Gaithersburg, MD, USA.

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The Talent Trap

Hiring gifted new employees is tough enough, but how do you keep them within your organization once you've got them?

By George Scott

Talent is one of the major challenges we face as managers or leaders. For example, in my industry – biotech – a recent study from Randstad suggested that over 50 percent of its talent is actively looking for employment elsewhere and that more than 65 percent would be likely to accept a new job offer (1). I suspect I could find equally compelling evidence in the analytical field, if I were to start digging. Dissatisfaction can arise from compensation issues to professional advancement to relationships with co-workers and supervisors. The cost of attrition can be counted not only in economic terms, but also a loss in productivity; recruiting and training activities may take weeks or even months, depending on the role. As managers in highly competitive industries, achieving stability in our organizations is important for future success. Two big questions need to be addressed. First, what makes an organization highly attractive for talented individuals? And second, what makes those individuals want to stay?

Numerous metrics regarding salary and compensation levels are published annually, and most companies ensure that they are at least competitive at the first level of talent engagement. In this article, I'll leave the obvious element of money aside to instead focus on the environmental attributes that contribute to a healthy, engaging and vibrant workplace that is attractive to both new and existing talent. After all, there's more to our working lives than money…

The right fit

Growth and attrition are the typical reasons that set us off down the path to find new talent, and there are at least three factors to consider in the hiring process. We need to understand what the candidate will bring to our existing talent pool, whether or not they can perform the role adequately, and how they will "fit in" with the existing team or even strengthen the group culture.

We often place a premium on high technical capability, believing it will transform our teams and give us a

"A manager whose first instinct is to blame and punish people when they make a mistake will stifle creativity"

competitive advantage. How many times, however, have we targeted a candidate based on expertise alone, only to find that our new superstar didn't contribute or fit into the organization in the way we'd hoped? "All too often," is a common answer. But many of us have also instinctively prevented disaster by holding back on a hire because we felt they did not fit. As unscientific and unquantifiable a feeling as this is, it is this powerful recognition of the non-technical attributes of potential hires that can make or break your team.

Technical capability is often a normalizing factor, in that the candidates applying for your position should all have the credentials to perform the tasks you need based on their CV alone. A PhD and 10 years of industrial experience may get you to the negotiation table, but it's a small part of the equation and needs to be balanced against their less tangible attributes. Technical expertise can be built or acquired; personal traits can only be coached. I believe that the emphasis we place on a candidate's technical capability and experience, and the balance between this and the ability to personally invest themselves in a new organization, is in fact the defining factor when it comes to successfully building – and retaining – your dream team.

The right environment

Hiring the right person is only the start. As I stated at the beginning, the next big question is: how do you keep them? When you have a great team, your priority should be to keep it intact. Compensation is not always the key factor. A pay rise wouldn't tempt most people to stay in a job with poor career development and poor colleague or supervisor relationships. In contrast, a highly engaged and motivated person, with room to grow in a collegiate and supportive environment of like-minded friends is unlikely to leave all that for a few extra dollars. Extreme non-equity in compensation will be a destabilizing factor, of course, but if this is normalized, the key elements of success lie in the working environment. A common mistake that many leaders and managers make is to assume that this environment is generated by their team while absconding themselves from responsibility. In fact, it starts and ends with you. To help, I offer five straightforward considerations to create the right environment:

1. Accept that you are not the smartest person in the room

And if you think you are, then don't feel the need to remind everyone. It is important to acknowledge that everyone needs to feel as if they are part of the solution when you are building your team. The best leaders recognize that there are facets of their organization in which they are not the expert, and their hiring strategy reflects the need to fill these gaps with motivated and empowered individuals. Put someone in the right place and then empower, challenge and trust them – I've seen people flourish in a way that is almost unimaginable.

2. Expect mistakes

Mistakes will be made, of course, so

"You will lose key talent, for family, health, geographic or other reasons, but what they take away with them will be as important as what they have left behind."

you should expect them, but view them as part of the continuum of learning and experience. A manager whose first instinct is to blame and punish people when they make a mistake will stifle creativity. An environment where mistakes are managed as learning events can alleviate the fear of failure and transform risk-averse conservatism to risk-based advancement. Having the trust of leadership can instill self-confidence and motivate an individual to feel truly invested in the team.

3. Know your team

I can almost guarantee that there is talent and capability embedded within your organization that you don't know about. Many of us have abilities and expertise that we don't harness in our daily roles, or that were a footnote in our role-targeted resumes. When transforming an organization, give opportunities to those who are currently invested in your company's success wherever possible, rather than recruiting externally. It is always a pleasant surprise to find out that someone in your organization is a six-sigma black belt just as you are planning to force-fit a process improvement initiative to an unsuspecting research scientist. In many companies, employees with diverse talent end up leaving because they feel unrecognized; often unintentionally overlooked through a lack of awareness.

4. Create lateral opportunities

Many of us see the logical progression of our career as a vertical ascendance. More often than we want to believe, lateral progression can be as fulfilling and even more rewarding. Expanding a role laterally can allow someone to step outside the boundaries of their current experience and build their skillset, develop a more integrated and inclusive viewpoint of their organization, and allow them to engage intellectually in a new environment with new people. In the scientific and technical fields, many do not want the extra managerial burden that comes through the acquisition of a higher title, but want to expand their experience through new and challenging opportunities. Those opportunities do not need to be vertical, and it is a common yet fatal mistake to "elevate" technical staff to a higher role that makes them lose their identity – there is no quicker way to lose a hardcore scientist than to make them a manager.

5. Remove obstacles quickly

One of the most damaging elements to the morale of a high-performing team is a toxic element left unchecked, for example, a poorly performing or obstructive individual. In many cases, the group itself can resolve the problem, but if the issue is unresolved and management fails to react, it can be a death sentence for team cohesion. When your team does not believe you have the capability or intention to remove obstacles, your credibility as their leader is lost, and they will find another leader that they trust and respect.

Parting on good terms

It is inevitable that attrition, like death and taxes, will exist at some level. You will lose key talent, for family, health, geographic or other reasons, but what they take away with them will be as important as what they have left behind. The experiences, opinions and perceptions of departing staff can have a huge impact on the reputation of both you and your organization, and will impact your ability to attract new talent. If someone has to leave an organization then we should plan that they leave with more than they started with, and that our team has had a positive influence on them both professionally and personally. The analytical community is well connected and it reflects well on you if the talent acquired from you is of a high caliber.

Developing a positive working culture of inclusion, empowerment and recognition undoubtedly comes at a high price; it's an all-consuming effort and it may take months or years for you to fully realize the benefits. Difficult decisions and candid discussions will be daily events, and your time investment will be considerable. But the rewards do outweigh the effort. It is worth remembering that friends do things for friends that others will not, and any organization that has a culture of celebrating each other's success will be a difficult place to leave. A team with this culture can only win, and nobody wants to leave a winning team.

George Scott is Vice President of Bioanalytical Services at inVentiv Health Clinical, Seattle, WA, USA.

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Time Saving, High Resolution GPC/SEC Using Micro Columns and Optimized RI Detection

As in HPLC before there is now a trend in GPC/SEC to separate low molar mass polymers and oligomers on columns filled with small particles.

Dr. Michael Krämer

Smaller particles are used to obtain higher resolution. If these particles are packed in GPC/SEC columns with smaller inner diameters (compared to traditional analytical columns) an efficient separation can be achieved that needs less mobile phase. Traditionally micro columns are run at a reduced flow-rate of 0.35 ml/min, but modern materials can be also operated in the flow-rate range of 0.5 - 0.7 mL/min to save not only mobile phase, but also time. Typical column dimensions for micro columns are e.g. 150 to 250 mm length and 4.6 mm inner diameter.

Unlike in UHPLC, in µGPC/SEC it is not the extremely high pressures that are a challenge but the cell volumes of the typical GPC/SEC detectors. If the cell volume is too large, the previously separated oligomers will be back-mixed in the detector cell and the advantage of small particle sizes and dedicated micro columns is lost. Cells with small volumes are already available for many UV/DAD, however for the most common GPC/SEC detector, the refractive index RI, only very few models are suitable.

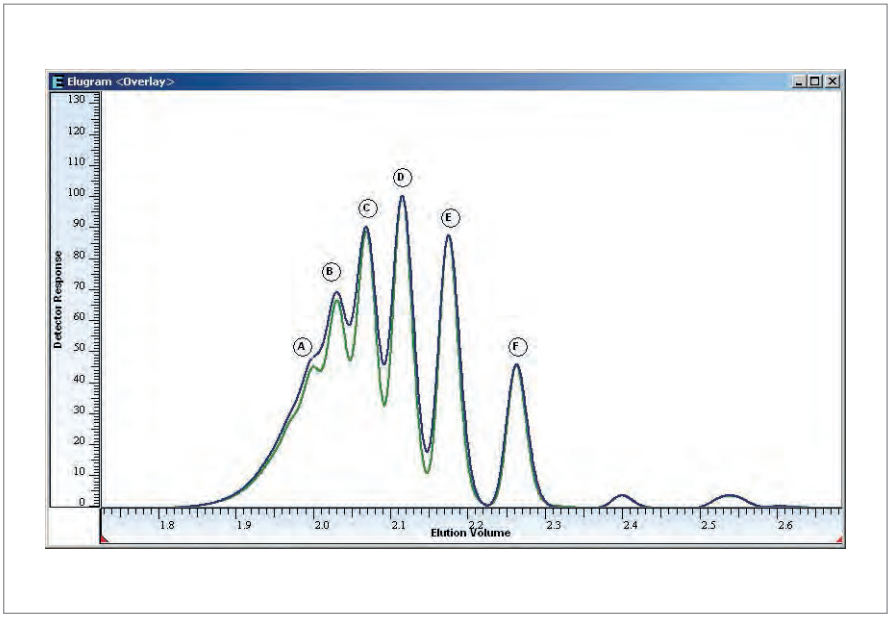

Figure 1. Oligomeric polystyrene separated in < 3 mL on PSS SDV micro column with 3 µm particle size using an analytical SECcurity RI (blue trace) and a SECcurity µRI (green trace)

With the PSS SECcurity µRI an RI with the smallest cell volume (1.7 µL), specifically designed for use with micro columns is now available. The detector can be seamlessly retrofitted at any time in PSS SECcurity GPC systems as well as in Agilent 1260/1290-Systems.

For this application note, PSS SDV columns, a polymeric crosslinked styrene-divinyl benzene material, have been used to investigate the resolution and the performance of the PSS µRI for oligomeric polystyrene. An advantage of polymeric stationary phases over silica based materials is that less interactions are observed. In addition, it is easier to construct a column set that is mismatch free to cover a wider molar mass range.

Figure 1 shows a comparison of the separation of an oligomeric polystyrene on two polymer based PSS SDV micro columns with small particles using a analytical SECcurity RI with 8 µL cell and the PSS µRI with a 1.7 µL cell. Although the analytical RI already has very good performance, it is possible to detect more oligomers with the

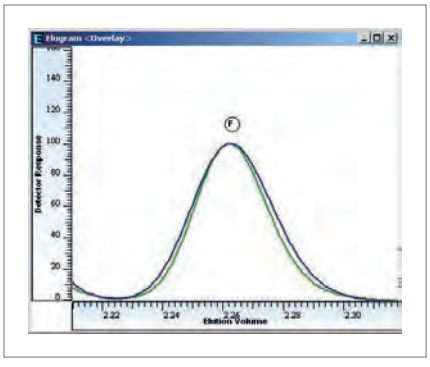

Figure 2. Comparison of dimeric styrene measured on micro columns with an analytical SECcurity RI (blue trace) and a µRI (green trace).

µRI detector. In addition to the higher resolution, the µRI detector also shows less signal broadening. Figure 2 shows a magnified comparison of the two traces for Peak F – it is clearly visible that the peak for the µRI is narrower.

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Sampling of Spirits

Spirit Sampling for counterfeit detection and brand authentication

Nick Barnett, Ph.D.

Incidents of spirits counterfeiting pose serious health risks to citizens and potential revenue loss to brand owners. The Ocean Optics Spirit Sampler can help to combat increased sales of illicit spirits. This application note describes measurements performed to demonstrate device reproducibility and outlines some of the instrument's quality control software features.

Experimental Conditions

The Ocean Optics Spirit Sampler scans clear, light and dark liquids, providing information about adulterated and gross counterfeit samples, as well as brand authenticity and quality. The system is based on an Ocean Optics UV-Visible spectrometer and light source, covering the wavelength range from 200-450 nm.

Spirit samples are introduced by injecting the liquid into the appropriate measurement channel using a plastic syringe. There are three channels corresponding to three different pathlength flow cells optimized for clear (e.g. vodka), light (e.g. whisky) and dark (e.g. rum) spirits. Rapid referencing and repeated automatic dark correction ensure optimum measurement stability and, therefore, maximum reproducibility in results.

Absorbance measurements from samples of rum, whisky and vodka were measured using 10 individual Spirit Sampler devices. Absorbance data was collected at 1 nm intervals between 220-450 nm. Each instrument was switched on and left to complete its initial warm-up. The flow cell was then flushed with distilled water in order to reference the instrument.

After taking a reference measurement, we injected the sample spirit into the appropriate flow cell and collected the absorbance spectrum. Absorbance measurements were collected for a total of 130 rum, 68 vodka and 184 whisky samples using the 10 Spirit Sampler units.

Results

All of the absorbance spectra for rum, whisky and vodka samples are shown in Figures 1, 2 and 3.

Analytical precision was investigated over the range 220-450 nm. The repeatability of measurements on one sample of brand ranged from +0.01% at 260 nm to +0.03% at 360 nm. Across the 10 instruments, the variability was still low at +0.03% at 260 nm and +0.09% at 360 nm.

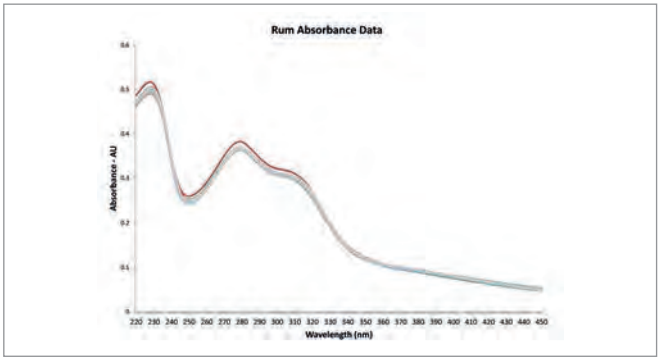


Figure 1: Absorbance measurements of rum showed great repeatability over more than 130 samples

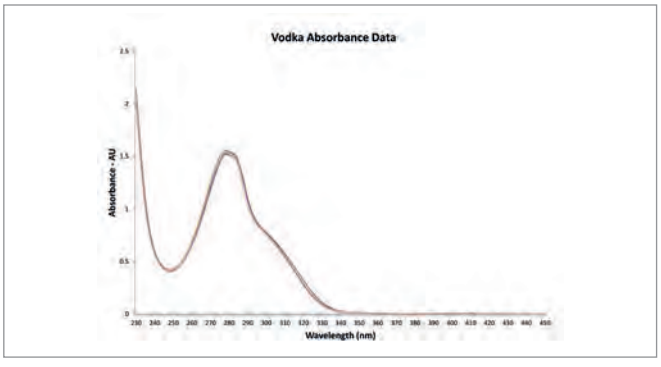


Figure 2: Strong UV absorbance response for vodka is shown over multiple measurements

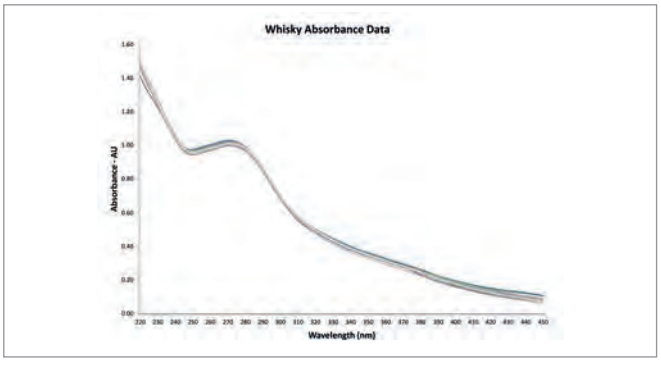
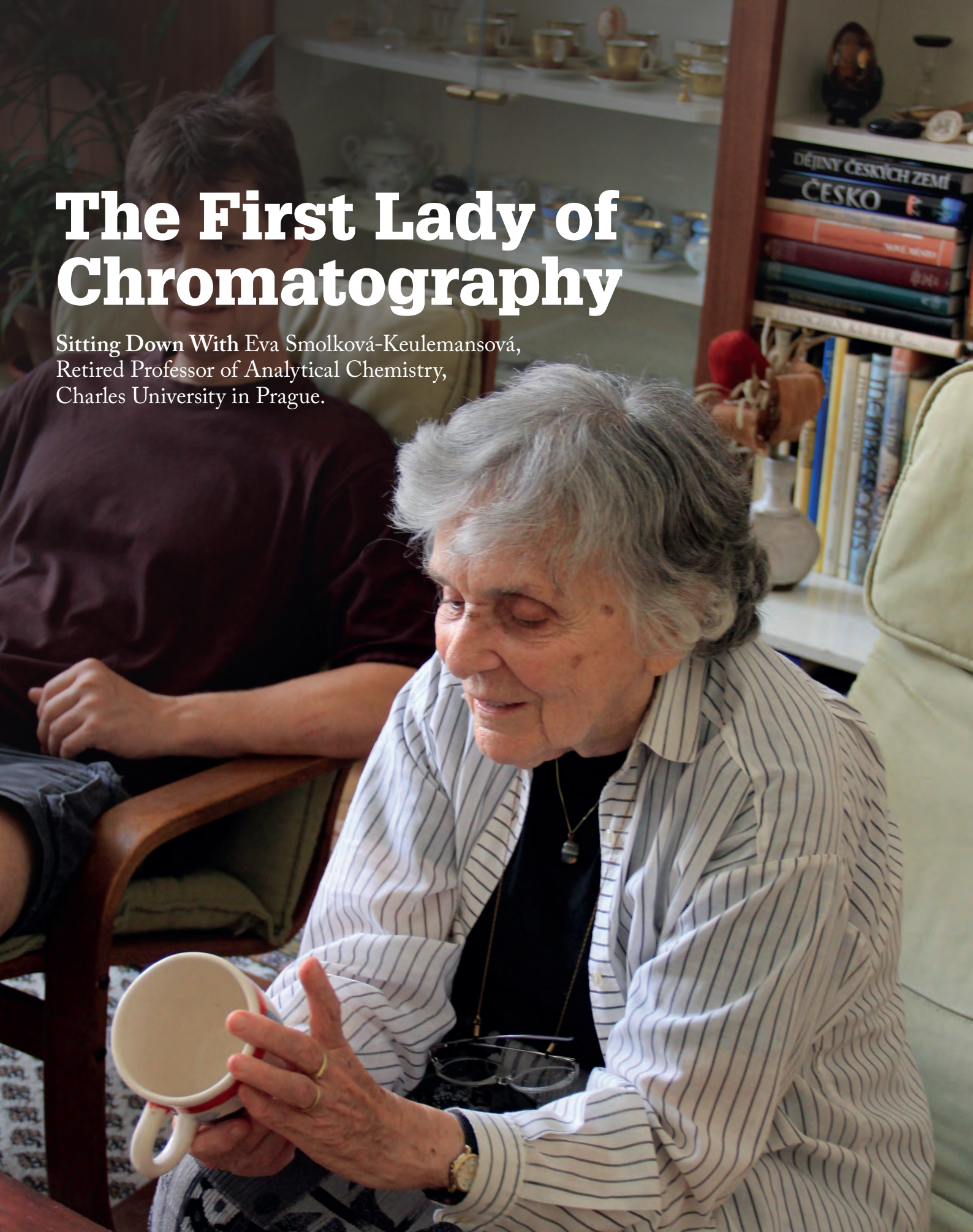


Figure 3: The Spirit Sampler demonstrates excellent repeatability for whisky sample measurements

Conclusions

The Ocean Optics Spirit Sampler provides reliable and reproducible data for authenticating spirits in the field or in the lab. Samples collected from different Spirit Sampler units can be combined to generate extremely accurate brand signatures with precise thresholds and tolerances.



The First Lady of Chromatography

Sitting Down With Eva Smolková-Keulemansová, Retired Professor of Analytical Chemistry, Charles University in Prague.

Take us back to the beginning...

I was born on April 27, 1927. I grew up in the suburbs of Prague with my beloved parents. I had a happy childhood and enjoyed school until the Second World War and the occupation of my country. I spent the wartime in Theresienstadt, Auschwitz, Hamburg and, finally, Bergen-Belsen. After the liberation, the Red Cross took me to Sweden for medical treatment – but my dream was to return to Prague to resume my studies. My dream came true in November 1945.

What happened on your return?

I considered medicine given my experiences, but actually started studying chemistry at Charles University in Prague. It was a great decision – and I've never regretted it. The first step of my scientific career was in the field of polarography, studying complex formations that could separate otherwise inseparable ions. During those early years, I was lucky to meet and befriend such personalities as Jaroslav Heyrovsky, who won the Nobel Prize in Chemistry for his discovery of polarography.

How did you start in gas chromatography?

In the early 1950s, a volumetric chromatographic device grabbed my attention at an analytical conference in Prague. We started to prepare our own device with volumetric detection. Next, we constructed a glass thermal conductivity detector, which was more universal and allowed us to analyze a larger variety of gas. At the time, it was a new idea, but I wasn't aware of that fact! Soon after, this detector became part of a commercially available instrument.

Where did that lead you?

In the early 1970s, I studied inclusion complex formations in selective analytical separations. Our first choice was cyclodextrins, but they weren't available, so we began with urea and thiourea for the separation of isomers. Thanks to József Szejtli, we were able to start research on cyclodextrins relatively soon after in GC, HPLC, and modern electromigration methods. Actually, I think we were the first to use cyclodextrins in isotachophoresis. Our research became popular, and we were asked to contribute several monographs on cyclodextrins – one that stands out in my mind was for a compendium on supramolecular chemistry edited by another Nobel Prize winner: Jean-Marie Lehn. Cyclodextrins are also responsible for my friendship with Dan Armstrong; it's a pleasure to follow his research.

Who were your other heroes?

In the 1950s (when I'd moved over to GC) there is no doubt that A. I. M. "Lou" Keulemans (Eindhoven University of Technology) had a huge influence on both my scientific and personal life. He put me in touch with a lot of other big names around the world. In 1968, he started an initiative called the Scientific Exchange Agreement, which enabled cooperation between laboratories in East and West Europe – a very special and significant development. In 1975, we married and he spent the last two years of his life with me in Prague.

A. A. Zhukhovitskii was also very important in my life. And over the years, I got introduced to many other great scientists. I knew J. J. van Deemter and Erika Cremer, and at Eindhoven University I spent a lot of time with A. J. P. Martin – Nobel Prize winner in chromatography. To be honest, I have a very long list of impressive people who I am proud to have called friends.

What motivated scientists in the 1950s?

It was a very special time. Many universities got closed during the war, but – like me – many young people were excited to resume studies. It's fair to say that there was a lack of instrumentation; however, there was a great deal of enthusiasm! We had to rely on home-made instruments and do research with simple equipment. I remember visiting Professor Willy Simon's lab at the University of Zurich and being amazed by the number of instruments. He told me, "Yes, instruments are important. But you can do chemistry with very simple equipment." Indeed, ideas are sometimes more powerful. I think scientific knowledge was higher back in my day – I think solving problems with minimal facilities demands a deeper understanding.

Today, we have fantastic instruments and results are produced very quickly – but the ideas are sometimes very old! I'm surprised when I hear about research that we did 50 years earlier... It's important to remember that modern science can benefit from a sound knowledge of historical research. Progress and advances that may have been forgotten could aid in a new discovery.

What else would you say to the younger generation?

I returned from my bad experience in the war with a key philosophy: my eyes are in the front of my head, therefore, I need to look towards the future. That's helped me throughout my whole life.

From my personal experience, I know that building relationships is not just a question of a mutual exchange of ideas; I gained a great deal of pleasure from the long-term friendships I forged with other scientists. And that includes students – who later saved me from the feeling of getting old!

I learned from Berzelius (the well-known 19th Century Swedish chemist) the important role that fantasy plays in science. It is often the way to new ideas.

Finally, I remember Zhukhovitskii's closing remarks at a Moscow symposium in 1959: "Gas chromatography is an excellent method to separate substances, and an excellent way to unite people."

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