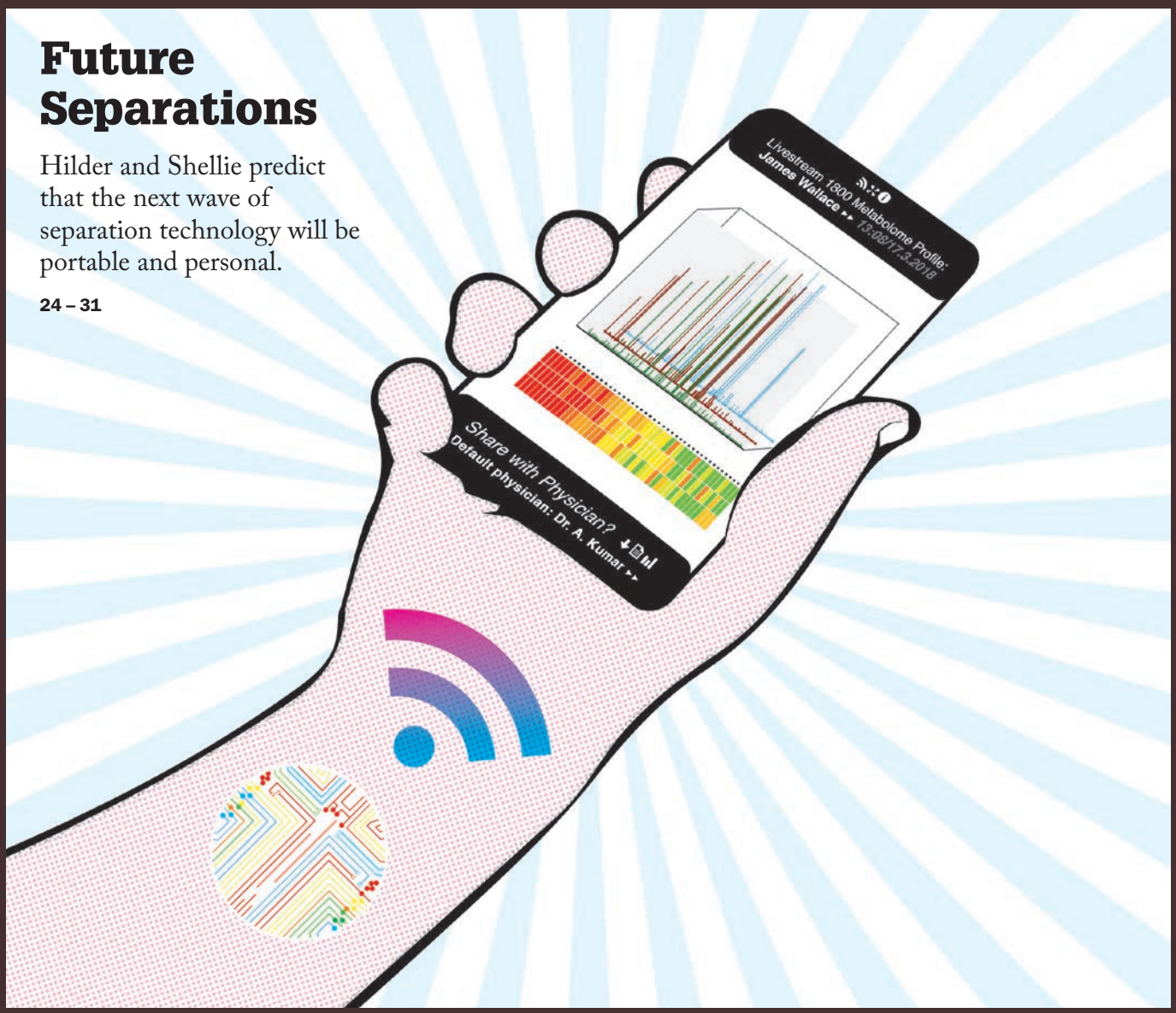
MARCH 2013 # 03

the Analytical Scientist

Future Separations

Hilder and Shellie predict that the next wave of separation technology will be portable and personal.

24 – 31



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Online this Month

Guest List

"If your name's not down, you're not coming in!"

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Legal Eagle

This month's editorial considers the variability of analytical measurements between laboratories and notes the increased uptake of analytical classes by lawyers interested in finding increasingly complex loopholes. Online, you can find a number of tips given to lawyers to cross-examine analytical evidence – how do you measure up?
theanalyticalscientist.com/issues/0313/104

Talking Point

The horsemeat scandal that recently swept the globe has raised some interesting questions about food authenticity and shocked a public that was assured improved transparency after the significantly more dangerous BSE outbreak. Once again, ruthless cost cutting appears to be to blame, but this time, the underlying problem is one of fraudulent labelling rather than safety. Are governments, the public, and the food industry overreacting or does the issue highlight a chink in either routine food analysis or legislation? Have your say: theanalyticalscientist.com/issues/0313/208

Analytical App

Download our iPad app and swipe your way through the contents of The Analytical Scientist and enjoy a rich and engaging multimedia experience. to boot. Learn more about the editorial from the horse's mouth and download the whole issue to read offline. link.theanalyticalscientist.com/app

Twitter

Thank you to all of our new followers. We're gathering pace on Twitter, but as the saying goes, there is strength in numbers. To find out what's popular and what's on the horizon, follow us at @tAnaSci.

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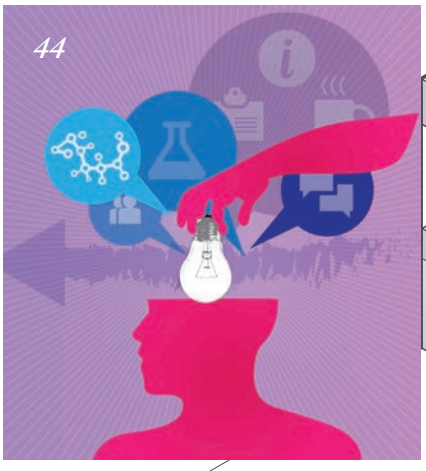

On The Cover

Concept: Personalized Analytical Instrumentation – visualizing the impact of miniturization, microfabrication and advanced technology on the future of separation science.

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the **Analytical Scientist**

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The Measure of Confidence

Agilent Technologies

How Well Do We Measure?

If any given sample was analyzed by five – or fifty – different labs today, how accurate and precise would the results be? And what would they tell us about the worth of analytical laboratories?

The same sample should yield the same results anywhere in the world. That's why standards organizations such as the European Committee for Standardization (CEN) and the International Organization for Standardization (ISO) exist. But, from what I hear, these methods are not being followed on a daily basis. "Very few of the standard operating procedures (SOP) manuals that I see are stained and dog-eared," an accreditation officer tells me. "In many analytical labs, the quality manual is consigned to the bottom drawer."

There are more unsavory issues. Let's say a standard method requires instrumentation that is unavailable at a commercial lab. Will the lab refuse the work or try to apply a slightly different method? What if a sample yields a higher level of contaminant than desired? Will the disappointed client find a lab that has reported low results in a proficiency test or will the first analyst be asked by his boss to repeat the analysis to gain a more pleasing result? If so, quality and integrity both go down the drain.

Last year, courses in gas chromatography (GC) were offered to lawyers in the USA. They sold out within hours. Lawyers know they can get clients charged with driving under the influence off scot-free, if they can shoot holes in the evidence; GC analysis of ethanol in blood offers one strong possibility, so they are studying the entire process, from sampling to end result. Was the procedure unclear or insufficiently documented? Was sample-handling imprecise or were the statistics not performed to the letter? Lawyers like GC. And they love sloppy lab practice. Woe betide us when they discover courses on LC-MS/MS…

The truth is, we don't really know how high standards are in analytical science. Collaborative studies measure variation in labs adhering to strict rules and identical methodology with staff on best behavior, but real-life variation could be far larger. Variability among laboratories is the dominating error component in the world of practical ultra trace analysis. Evaluation and validation of laboratory procedures are of paramount importance; international inter-laboratory proficiency tests using certified reference compounds and benchmarking exercises should be embedded in lab routine. Until that moment, perhaps the best way of assessing lab quality is to ask ourselves how confident we would be under the scrutiny of an analytical court case. If that keeps the field on its toes, perhaps we even have a reason to be grateful to lawyers!

Do you have a different opinion? Add your comment and read handy tips for lawyers (and analytical scientists) online: theanalyticalscientist.com/issues/0313/104

Frank Van Geel
Scientific Director

Ian Wilson

For his PhD, Ian Wilson used GC to analyze steroid hormones in insects. Much of his subsequent career has been in the pharmaceutical industry, working in discovery and development. In 2012, Ian moved to Imperial College, London. His research interests include separations science, particularly the development of hyphenated techniques and their application to problems in drug metabolism and metabonomics. Outside work, he collects old instruments, such as gas and liquid chromatographs. *See page 18.*

Emily Hilder and Robert Shellie

A Professor and ARC Future Fellow in the Australian Centre for Research on Separation Science (ACROSS) and at the University of Tasmania (UTAS), Emily Hilder's research focuses on the design and application of new polymeric materials in separation science. "I'm also interested in the development of miniaturised analytical systems, particularly for applications in clinical diagnostics and remote monitoring."

Robert Shellie is Associate Professor at UTAS and also a member of ACROSS. Having worked with hyphenated techniques in chromatography for more than a decade, Robert's research has largely focused on exploration of approaches for separation and characterization of complex multicomponent samples. More recently, he says, "I became actively engaged in the development and application of field-portable instrumentation, including miniaturized multidimensional gas chromatography." *See page 24.*

Janice Manzi Sabatine

From her days as a PhD student in biochemistry to her current role as an executive coach, Janice Manzi Sabatine has been intrigued by how important interpersonal skills are to success. "I saw my colleagues in non-technical fields receive management training and leadership development, and resented that those of us in the sciences and medicine, particularly in academia, did not receive those same benefits." To address that inequity, she became a certified executive coach, founded Avanti Strategies, and now provides this much-appreciated service to her technical colleagues *See page 40.*

Jean-Luc Jonniaux

"Screening for microorganisms collected from all over the world to optimize production in 100m^3 fermentors is fantastic training. You gain experience in genetics, physiology, process and analytics", says Jean-Luc Jonniaux of his experience in the food industry. From there, he moved to the pharmaceutical world and developed processes for virus, antibody and stem cell production for clinical trials. "The volumes processed are small but, like a bonsai tree, must be perfect," he explains. "It requires all your attention and deep knowledge of the art. Analytical tools are essential to master the drug." He put his combined experience to work in obtaining market authorization for a new biopharmaceutical. *See page 44.*

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Upfront

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

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Winter of Content

The remains of King Richard III are confirmed by DNA analysis, supported by micro-computed X-ray tomography and radiocarbon dating.

"Today, we bear witness to history. We peer 500 years into medieval times and literally reach into the grave." So began Richard Taylor, the University of Leicester's deputy registrar, at a press conference on February 4, preciptating a media storm over the discovery of the remains of King Richard III of England.

Image from 1955 film poster of Richard III, starring Laurence Olivier.

Richard III, made all the more famous by William Shakespeare's depiction, was King of England for two short years, from 1483 to 1485, at which point his death at the Battle of Bosworth snatched away his reign. The burial was without ceremony and the body was lost for over five centuries. Richard Buckley, the lead archaeologist on the 'Search for Richard III', said at the conference: "It is the academic conclusion of the University of Leicester that the individual exhumed at Grey Friars in August 2012 is indeed King Richard III, the last Plantagenet King of England".

The Analytical Scientist spoke with Turi King, lecturer in genetics and archaeology at the University of Leicester, who justified the use of mitochondrial DNA analysis: "We already had two distantly related female line relatives, and mitochondrial DNA also happens to be the DNA that is most likely retrieved from ancient remains." Mitochondrial DNA (mtDNA) is transmitted from mother to child and therefore has distinct advantages when tracing female lines of descent – 18 generation's worth in the case of Richard III.

"Ancient DNA is fragmented and so requires amplification of smaller fragments than would normally be used when typing modern DNA," said King who carried out the analysis on a tooth sample in dedicated ancient DNA facilities at the University of York, UK. She later traveled to the Université Paul Sabatier in Toulouse, France, where the work was verified – King was happily able to confirm a match. Coincidentally, King is based out of the

university's Department of Genetics, the birthplace of modern DNA fingerprinting (discovered by Sir Alec Jeffreys back in 1984).

The DNA analysis was just one part of an investigation that saw osteoarchaeology, geneaology, historical evidence, forensic pathology (including micro-computed X-ray tomography) and radiocarbon dating all coming together to build a full picture. Radiocarbon dating showed that the individual died some time in the second half of the 15th or early part of the 16th century, and had a high protein diet, including significant amounts of seafood – consistent with someone of high status. "Like I've said all along, the DNA work has to be taken alongside all the other evidence. Just as in any forensic case, a person wouldn't be convicted on DNA evidence alone; the same is true in this case. Fortunately, the mitochondrial DNA type is a relatively rare one, which further strengthens the case," says King.

There is still further confirmatory work to be done, though; King concludes, "I'm working on the Y chromosome analysis now and hope to then compare the DNA from the skeletal remains with those of putative male line relatives."

As a somewhat relevant but distinctly non-scientific aside, one camping shop in Leicester apparently quipped on a billboard after the announcement: "Now is the discount of our winter tents". I think even Shakespeare would smile at that. *RW*

For more information on the investigation, visit: www.le.ac.uk/richardiii

Nanomechanics

By combining the sensitivity of microcantilevers with the selectivity of IR spectroscopy, photothermal cantilever deflection spectroscopy (PCDS) opens up new opportunities for the detection, speciation, and quantification of explosive vapors in complex mixtures.

By Thomas Thundat

The Problem: Most vapor-sensing approaches based on immobilized chemical interfaces on sensor arrays fail to selectively identify target molecules when complex mixtures of molecules are present, such as in explosives.

The Background: Miniature sensors, such as microfabricated cantilevers, have very high sensitivity in the detection of adsorbed molecules, but do not have any intrinsic chemical selectivity. They require chemically selective interfaces to be immobilized on their surfaces to achieve selectivity in molecular detection.

But the nonspecific nature of chemical binding – especially those based on weak chemical interactions such as hydrogen bonds or van der Waals interactions – means that, despite their ability to detect molecules with extremely high sensitivity, miniature sensors, have had little market potential because of their failure to detect target molecules in mixtures.

The Solution: PCDS combines the sensitivity of microcantilevers and the selectivity of infrared (IR) spectroscopy. It delivers selective and sensitive detection of molecules even in the presence of interfering molecules with similar structures. In this technique, the physisorbed molecules on a thermally sensitive bi-material cantilever are excited with IR radiation in a sequential manner. IR energy absorption by the molecules results in a very small change in the temperature of the cantilever due to non-radiative decay, which in turn bends the cantilever.

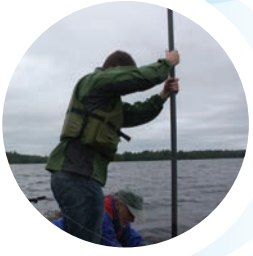
A bi-material cantilever can detect extremely small changes in temperature in a way not dissimilar to a thermostat. Plotting cantilever bending as a function of illumination wavelength mimics the IR spectrum of adsorbed molecules. PCDS can successfully detect target molecules even in the presence of interfering molecules with similar molecular structures since vibrational spectra can be treated as linear combinations of spectra from individual members in the mixture. The resonance frequency change of the cantilever due to molecular adsorption can be used for quantification.

Our paper in Nature Scientific Reports (1) highlights successful implementation of PCDS for the selective and quantitative detection of ternary mixtures of explosive molecules, such as trinitrotoluene (TNT), cyclotrimethylene trinitramine (RDX), and pentaerythritol tetranitrate (PETN), with picogram levels of mass resolution. The approach could have immediate applications in areas such as national security, forensics, and humanitarian demining.

Thomas Thundat is a professor in the Department of Chemical & Materials Engineering at the National Institute of Nano Technology, University of Alberta, Canada.

Reference:

1. *S. Kim et al., "Molecular recognition using receptor-free nanomechanical infrared spectroscopy based on a quantum cascade laser", Sci. Rep. 3, DOI: 10.1038/srep01111 (2013).*



Don't Wash Your Hands of This

Is triclosan, an antibacterial agent in soaps and body washes, damaging the aquatic environment?

Antibacterial soaps are marketed as preventing us from passing harmful germs to one another – they are generally accepted as a 'good thing'. Triclosan is a common antibacterial agent, but the US FDA "does not have evidence that triclosan added to antibacterial soaps and body washes provides extra health benefits over soap and water".

Futile scrubbing is no great issue. But what if common antibacterial agents, such as triclosan, are having a detrimental environmental impact on the water cycle?

To gather information that could help toxicologists, regulators and the public evaluate any risk, researchers at University of Minnesota examined the historical exposure of aquatic systems to triclosan, chlorinated triclosan derivatives, and the four dioxin congeners (polychlorinated dibenzo-p-dioxins) that arise from the photolysis of triclosan (and its derivatives). William Arnold, a civil engineering professor in the College of Science and Engineering and the study's lead author, told The Analytical Scientist: "We also wanted to look at the overall dioxin trends to determine if the four targeted dioxin congeners were, in fact, the result of triclosan releases."

The team studied sediment cores of a meter in length from eight lakes across Minnesota that had different levels of exposure to treated wastewater. The cores were freeze dried, spiked with C-13-labeled triclosan, and subjected to accelerated solvent extraction. The subsequent clean-up steps proved to be a major challenge, depending on the organic content of the sediment sample. "Extracts from low organic sediments could be cleaned up with silica gel. The 'dirtier' extracts had to be diluted into water, solid phase-extracted, eluted, and then run through silica gel," says Arnold.

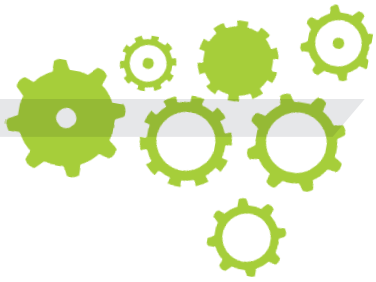
The team used an Agilent 1100 capillary LC with a Finnigan TSQ Quantum Discover MAX MS-Q3 tandem mass spectrometer, which allowed them to look for transitions that were specific to triclosan and its chlorinated derivatives. For the dioxins, Pace Analytical Services used high-solution gas chromatography–high-solution mass spectrometry, following a modified version of US EPA Method 1613B.

The analysis showed that all four dioxin congeners arise from photolysis of triclosan and its three chlorinated derivatives. Current levels of triclosan are site dependent but are in the 1–100 ng/g sediment range. Chlorinated triclosan derivatives are 100–2000 pg/g, and the 2,8-DCDD 40-6000 pg/g. In terms of accumulation, triclosan rates are currently 0.5–12 ng/cm^2 year. For 2,8-DCDD, accumulation rates range from 2 to 750 pg/cm^2 per year.

Though the study is centered on Minnesota, the findings should be relevant to other freshwater bodies receiving treated wastewater effluent. The major implication is that when triclosan is washed down the drain, it is not completely removed by treatment processes, with a fraction of it winding up in lakes or rivers potentially distant from the point of use. Potential impacts of triclosan and its derivatives on environment health are an area of exploration, but triclosan is known to affect the function of algal communities at environmentally observed levels. The effects of these dioxins are currently unknown. Arnold concludes, "We are looking at other sites, and we are also looking at other compounds that are structurally similar to triclosan, which could also be dioxin precursors."

Perhaps it's time we all started thinking a little more carefully about what gets washed down the sink, especially if the chemicals in question have no real benefit beyond marketing spin. *RW*

Full research paper: C. T. Anger et al., "Quantification of Triclosan, Chlorinated Triclosan Derivatives, and their Dioxin Photoproducts in Lacustrine Sediment Cores", Environ. Sci. Technol., 47 (4), 1833–1843 (2013).



In Sync

Notable partnerships in the world of analytics.

Designer Drug Wars

Who: Agilent Technologies and Florida International University (FIU)

What: A collaboration between Aglient and FIU's Department of Chemistry and Biochemistry and International Forensics Research Institute (IFRI) to identify and characterize designer drugs using advanced chromatography and mass-spectrometry systems.

Why: Designer drugs are synthesized derivatives of known illicit drug compounds that intentionally circumvent existing immunoassay drug-screening practices but produce similar 'recreational' effects. Routine methods are "unable to detect most of the hundreds of individual designer drugs that have been identified," according to Anthony DeCaprio, director of IFRI's Forensic & Analytical Toxicology Facility.

How: The team will develop advanced analytical methods using LC-QQQ-MS/MS, LC-QTOF-MS, GC/MS and GC/MS/MS for fast, accurate identification of designer substances in both ante- and post-mortem specimens, as well as expand the university's tandem mass-spectral library to approximately 300 designer drugs.

Forensics Focus

Who: US Department of Justice and National Institute of Standards and Technology (NIST)

What: The establishment of a National Commission on Forensic Science – part of a new initiative to strengthen and enhance the practice.

Why: "Forensic science is an essential tool in the administration of justice and needs to be continually evaluated as science progresses," said Deputy Attorney General James M. Cole.

How: The Commission will have 30 members, comprising forensic scientists, academic researchers, prosecutors, defense attorneys and judges. It will be charged with developing forensic policy guidance for the Attorney General and will also consider guidance on practices for federal, state and local forensic science laboratories developed by groups of forensic science practitioners and academic researchers administered by NIST.

NASAnalysis

Who: NASA and Ionicon

What: Researchers on NASA's $30 million DISCOVER-AQ mission are using Ionicon's new real-time trace gas analyzer to measure atmospheric air pollution.

Why: To understand more about air quality by using satellite-based Earth observations. The long-term aim is to predict air quality and reduce pollution. To do so, it is necessary to monitor how pollutants are vertically distributed in the atmosphere. "Satellite sensors have a hard time distinguishing air pollution that is close to the ground from pollution that is higher aloft," said instrument development project leader Armin Wisthaler who works at the Institute for Ion physics & Applied Physics, University of Innsbruck. "In this effort, NASA has put some of the most sophisticated air pollution monitors on two of its research aircraft. The cooperation with Ionicon has put us at the forefront of research".

How: To capture data, the aircraft will fly low-altitude spiral profiles from 15,000 feet down to 1,000 feet above ground level, and sample air along agricultural and traffic corridors at low altitudes. On board, along with other instrumentation and a selection of beverages and hot snacks, is a new compact proton transfer reaction time-of-flight mass spectrometer (PTR-TOF-MS).

Further mission information: http://discover-aq.larc.nasa.gov

Marking Territory

Who: Schauenburg and Markes International

What: The Schauenburg International Group has acquired the majority of the shares of the Markes International Group.

Why: "Markes will form a substantial part and a new technology segment within our Electronic Division with great potential and synergies. It will support and complement our efforts as technology and system supplier to the environmental monitoring market," stated Florian G. Schauenburg, CEO.

How: Markes International hopes to benefit from Schauenberg's 50 years' experience in sustainable technology investment but assures, "Business as usual". Its focus, facilities and all employees will remain in place, and the company's founders, Alun Cole and Elizabeth Woolfenden, will stay on as co-shareholders and leaders of the management team. *RW*

Mocking 'An Old Man in Military Costume'

Last month's "The Science of Art" followed four stories of analytical art appreciation. Here, we pursue another art mystery with one of the authors, Matthias Alfeld.

Why go to such painstaking extremes to create a mock up?

There is a hidden portrait underneath the surface of Rembrandt's 'An Old Man in Military Costume', which is of high art-historical interest. All previous imaging attempts failed to reveal details. We assumed that it could be visualized by MA-XRF, but several possible combinations of source (synchrotron or X-ray tube) and detectors were discussed and it was not clear which instrument would be best suited to the investigation of the original painting.

A comparison of different instruments was not possible with the original painting; it would be irresponsible to expose a unique precious work of art to the stress of transporting it to multiple experiments. Therefore, we decided to create a mock-up, analyze it with the different instruments discussed, and make an informed decision based on the results obtained.

How accurate is the fake in terms of chemical composition?

The creation of the mock-up was based on local, quantitative investigations of the original painting. So we assume the mock-up is accurate in terms of layering and qualitative (but not quantitative) paint composition. As well as allowing for a direct comparison of the imaging capabilities of each scanner, the mock-up also gave opportunities for logistical "dry runs." We were able to identify potential hazards for the painting during transportation, mounting, and the experiment itself, and thus plan to

minimize any stress the original painting might be exposed to in the "wet run".

Why is MA-XRF a good choice of technique in this instance?

Conventional imagining techniques for historical paintings, such as X-ray radiography, Infrared reflectography and Neutron Activation Auto-Radiography, had revealed the presence of a hidden portrait but failed to visualize the details. But preliminary investigations also revealed that Vermilion (HgS) has been used nearly exclusively in the flesh tones of the lower painting, suggesting that element-specific imaging might successfully reveal the hidden portrait.

Although MA-XRF is a recently established method, I believe it is best suited for the acquisition of surface and sub-surface elemental distribution images in historical paintings.

It seems as though there are some serious creative and scientific challenges involved…

To my best knowledge, the creation of a complete mock-up, including sub-surface layers, had never before been attempted and required detailed studies of radiography and cross sections of the original painting.

Furthermore, all instruments used in these experiments were of an experimental nature and assembled solely for these experiments – protocols for the acquisition and processing of data, as well as the presentation of the results, needed to be established.

How conclusive are the findings – will the analysis be equally effective on the original?

We have shown that synchrotron-based scanners and improved mobile X-ray tube based scanners are capable of visualizing the hidden portrait in the mock-up. I believe that an investigation of the original painting will provide additional information on the hidden portrait, but we cannot ignore the fact that heavy re-working of the paint layers above the hidden portrait may make the elemental distribution images more difficult to read.

Matthias Alfeld works in Professor Koen Janssens group at the University of Antwerp, Belgium.

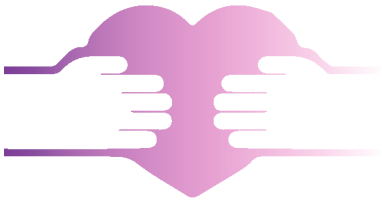
For more information, see: "Revealing hidden paint layers in oil paintings by means of scanning macro-XRF: a mock-up study based on Rembrandt's 'An old man in military costume'", Matthias Alfeld et al., Journal of Analytical Atomic Spectrometry 28, 40-51 (2013); DOI: 10.1039/C2JA30119A

Mass App-eal

A free iPad app allows you to run simulated mass spectrums of gas and vapor species without breaking a sweat… or even leaving your armchair.

The Mass Spectrometer Spectral Overlap Emulator from Hiden Analytical gives Apple fanboys in environmental, R&D and MS application setup labs the ability to select components from a quick mass peak look up table and view overlaps of multiple fragmentation spectra to identify the mass peaks with least interference. Certainly, a useful yet simple tool to get a first impression of MS optimization potential. It also looks great on your desk, if you need to look suddenly busy.

Up to 16 gas and vapor species (arsenic hyrdride, DP Oil DC705, Krypton…) can be assigned, with the ability to fine tune the relative concentrations of each. And it's so simple, a five-year-old could run it. In fact, a five-year-old produced the fine looking spectra above. Admittedly, it serves as a subtle marketing tool for Hiden Analytical, but given that it's free and with no obligation, what's not to like? *RW*



Science Dating

Need to conduct an experiment but lack technical capability? Got spare capacity in your lab or some sadly underused, budget-busting bit of kit? You guys should meet…

Science Exchange, the brainchild of Elizabeth Iorns (see page 50), Ryan Abbott and Dan Knox, originally popped up on the radar in August 2011 and has been garnering attention from the media as well as the research community ever since. Now, with a user base of over 5000 scientists at over 400 institutions, its mission to "improve the efficiency of scientific research by making it easy for researchers to access the global network of scientific resources and expertise" is taking good shape.

The service acts like a dating agency between researchers who need access to, say, mass spectrometry, RNA microarrays, or next generation DNA sequencing and providers. These service providers could be anywhere, from your neighborhood DNA sequencing lab to (potentially) the microgravity research facility on the International Space Station. And while the focus is currently on pre-clinical life science research, the marketplace is steadily expanding.

This service, unlike the majority of online dating agencies who try to hide fees, is transparent: it's free to subscribe, free to search and even free to list yourself as a provider. Researchers requesting services are charged a nominal one-time fee per transaction at the time of purchase, which is added automatically to the service provider's estimate – the price the researcher originally sees.

Researchers follow four simple steps:

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4. Monitor the status of ongoing projects via a project dashboard, with notifications of new status updates and the ability to ask questions or easily exchange data.

There are plenty of fish in this sea. *RW*

For more information and to sign up: www.scienceexchange.com

DNA Horse Play

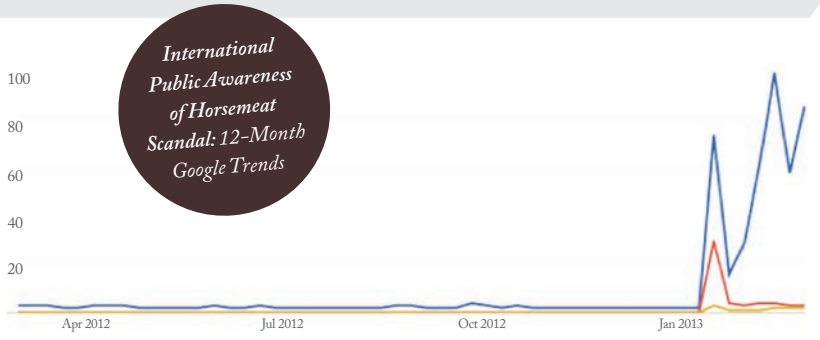
The ongoing horsemeat scandal has damaged customer confidence in authenticity and raised food analysis awareness.

"Public confidence has been badly shaken", according to European Health and Consumer Policy Commissioner Tonio Borg, and as evidenced by Google Trends, which tracks keyword search volume.

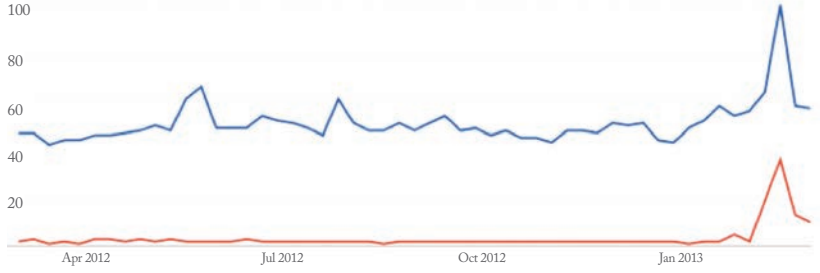
On Feb 13, Borg announced the Official Control Plan, which includes:

- Testing for presence of horse DNA in foods marketed or labeled as containing beef by Member States.
- Testing for presence of phenylbutazone residues in horse meat.

The findings from the first month of testing will be made public on April 15.



Horse meat (blue), horse burgers (red), horse DNA (gold). 100 on y-axis indicates peak search volume.



"DNA test" (blue), phenylbutazone (red). 100 on y-axis indicates peak search volume.

For more information on the scandal, visit: theanalyticalscientist.com/issues/0313/208

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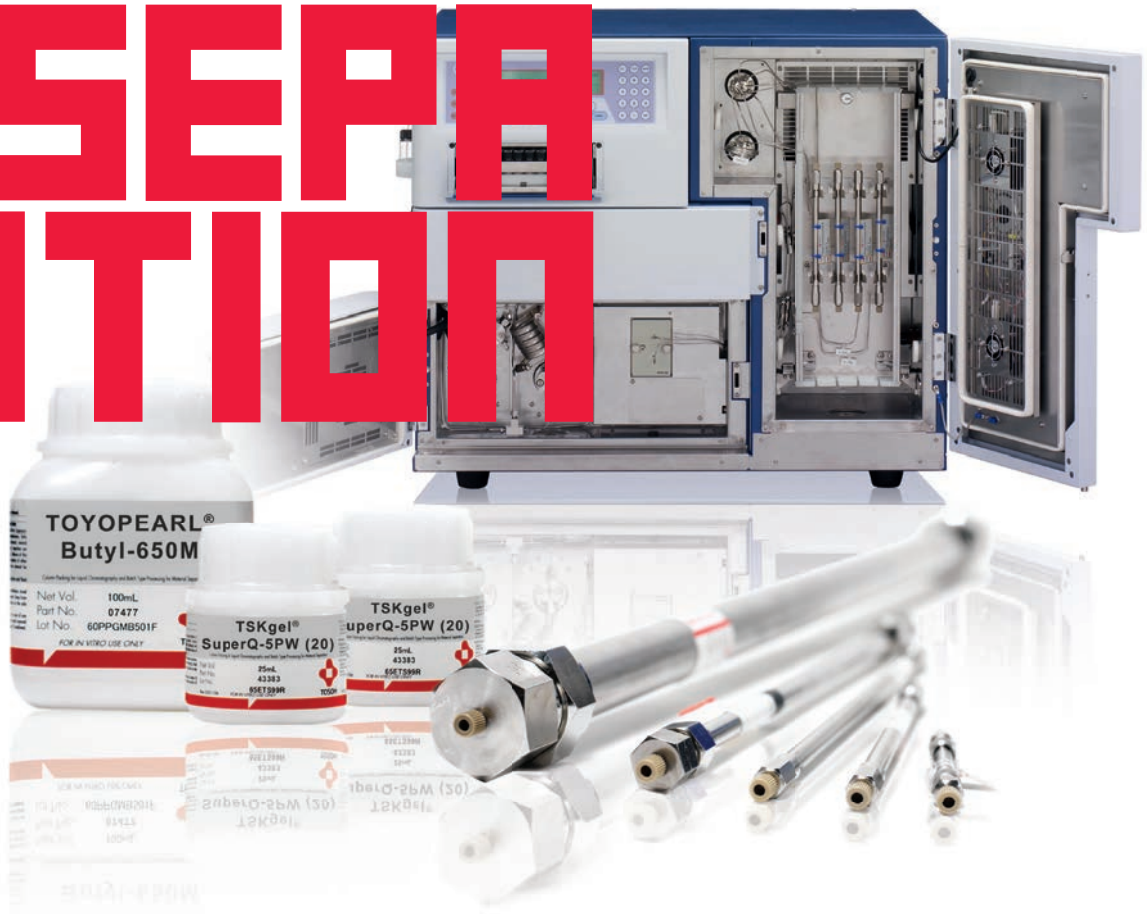

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A Catalyst for Scientific Progress

Rivalry with other researchers provides reassurance that the problem is sufficiently important for others to try for the same prize, and the impetus to raise your game.

By Ian Wilson, Department of Surgery & Cancer, Imperial College London.

One of the things that many of us forget about science is that it is an intensely human activity.

There is a notion that science progresses as a result of dispassionate intellects collecting, collating and sifting facts, developing a hypothesis to test and, via carefully thought-through experiments, ending up with a new worldview. It is, as any practicing scientist knows, (mostly) nonsense, although it is a useful myth because it makes us scientists appear a lot cleverer than we are…

There are, no doubt, many honorable exceptions, but anyone who has worked in a research lab for any length of time on a topic of general interest will know that second only to the joy of discovery is the very satisfying, but guilty, pleasure of beating a rival to publication.

I was reminded of this when I was given a present of "The Annotated and Illustrated Double Helix", by James D. Watson (1). Edited by Alexander Gann and Jan Witkowski, this edition was published in 2012 to mark the 50th anniversary of the award of the Nobel Prize to Watson, Crick and Wilkins. It includes a lot of extra material: letters from Watson to his sister, copies of Rosalind Franklin's laboratory notes and those for her 1951 colloquium, reproductions of relevant papers and many photographs, which all help to put the discovery of the double helix into context. The "Double Helix" is a great story, but as Watson himself wrote in his foreword, it's a memoir of how it felt at the time, not a history (2). Watson stated that he had "attempted to recreate my first impressions of the relevant events and personalities rather than present an assessment which takes into account the many facts I have learnt since the structure was found". It is his attempt to show, amongst other things, "how science is 'done'" and he is pretty accurate in the way that he covers both the process and the human face of science (I am aware that I am not the first person to say this!).

It is tempting to say that the events that Watson describes are a one-off, but actually I think it is very typical. A more recent, equally famous, example, where both sides have been able to put their views into print, is the race to sequence the human genome. In the publicly-funded corner we have "The Common Thread" (3) written by the Nobel laureate John Sulston and science writer Georgina Ferry, and in the Celera corner we have J. Craig Venter's "A Life Decoded" (4). Both books provide a fascinating insight into the competition that occurs in modern programs when the scientific prize is huge, and the funding is too, but it's not always obvious that they are describing the same race!

"Second only to the joy of discovery is the very satisfying, but guilty, pleasure of beating a rival to publication"

These books clearly reveal the intra- and inter-laboratory strains that inevitably accompany any race to solve an important problem. Reading them shows the intensity of the discovery process, the feeling of elation when you finally crack a particularly difficult problem and discover a piece of truth that (you hope) no one else has ever known. They also describe the fear of not being able to get something into print before that 'so and so' at the 'University of Somewhere Else'.

Such rivalry – let's call it 'creative competitiveness' – can be a spur to scientific advance, so long as it is not taken to excess. Rivalry provides both reassurance that the problem is sufficiently important for others to try for the same prize and the impetus to raise your game.

One thing to remember, however, is the other lesson from the Double Helix: Watson on his own would probably not have solved the problem, and neither would Crick. And if Wilkins and Franklin had got on as well as Watson and Crick, the story would have been very different. Rivalry as a driving force is very useful, but do remember to collaborate as well. The result is usually better.

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Why Isn't Chemometrics Center Stage?

There is a great need for more accurate planning of measurements and efficient extraction of relevant information from complex analytical data. The solution is at hand, let's start using it properly.

By Ewa Szymańska, Radboud University Nijmegen, Institute of Molecules and Materials, Analytical Chemistry, The Netherlands.

Today's chemical analytical technologies provide unprecedented quality and quantity of measurements. Datasets are invariably vast and multivariate, incorporating millions of individual measurements on great numbers of variables (analytes). Unfortunately, our ability to interpret this data has fallen behind our ability to generate it, especially in multidisciplinary projects at the interface of chemistry and biology, chemistry and technology, and chemistry and the real world.

Analytical scientists understand and use simple statistical tools in their daily work but these tools are rarely suited for large datasets. Chemometrics historically provided a valuable toolbox to mine information from complex data. However, despite the accumulation of forty years of experience with chemometrics, current levels of expertise - and popularity of the topic among analytical scientists - is shamefully low.

Why is this? After all, chemometrics tools enable and facilitate not only extraction of information from data obtained from hyphenated techniques but also integration of data obtained by many different analytical platforms. This is crucial to generating a full picture of the system being analyzed, for example, in advanced pharmaceutical and biomedical applications. Actually, chemometric tools can be put to multiple uses: in the design of experiments; in selection and optimization of analytical conditions; in quality control of series of measurements; and in data processing, including sample correction and compression, calibration, pattern recognition and classification. Given all this, chemometrics should occupy a position at center stage for analytical chemists.

It's not that the tools are not available. Most analytical chemistry labs are equipped with chemometric/statistical software. These user-friendly packages provide standard chemometric tools and a selection of result-visualization tools. However, slick packages also

present a danger: misuse and abuse. I know of examples that range from the disquieting to the truly appalling. Suffice to say, it is simply not acceptable to query data with all the software tools available with a click of the 'do all' button, and then choose the 'best' result (according to the user!). At best this is a pure waste of time, at worst it is malpractice.

The real problem is that chemometric software has become a 'black box'. Users are not familiar with the theoretical aspects of the tools, their assumptions or the requirements of the data sets. They don't understand what parameters to optimize or how to go about it. In short, they are selecting blindly. Applying chemometrics is not magic: 'garbage in, garbage out' holds, and poor data simply means poor results. Chemometrics is not 'a cleaning lady' at the Analytical Chemistry department; indeed, it should 'take a corner office,' because it has tools to reveal trends that are buried within the data.

"The real problem is that chemometric software has become a 'black box'. Users are not familiar with the theoretical aspects of the tools, their assumptions or the requirements of the data sets."

While chemometrics applies mathematical and statistical methods, the development of new methods is usually motivated by the pull of solving real chemical problems,

Analytical Fusion

Working in an environment that fosters cross-pollination between multi-disciplinary groups can be rewarding, both personally and professionally.

By David Collins, a PhD Researcher at the Irish Separation Science Cluster, Dublin City University, Ireland.

I originally graduated in 2000 with a B.Eng in Electromechanical Engineering, and spent the first few years of my career hopping between the aerospace and pharmaceutical industries while juggling an M.Eng. After a number of years in the pharma and medical device sectors and a period working for myself, an opportunity arose to return to academia on a PhD scholarship. The catch? The field was new territory for me – analytical chemistry. The bonus? It was with the Irish Separation Science Cluster (ISSC), an internationally renowned separations group based at Dublin City University.

Three years after joining the ISSC, I can look back at what has been a highly successful and fruitful experience, on many levels. The ISSC is a truly multi-disciplinary group, originally set up by Brett Paull, to work closely with industry in tackling analytical problems in the area of separation science. His vision was to create a separations group made up of chemists, engineers, physicists, and microbiologists to provide a platform through which new ideas could be shared and new challenges addressed by breaching traditional research boundaries. Indeed, the work being done with the ISSC is extremely diverse, from material science and research into new stationary phases, to instrumentation development and biotechnology applications. Clearly, a broad spectrum of projects such as this requires expertise from very different areas. And people with diverse perspectives are able to bring innovative solutions.

My own work has covered both high temperature liquid chromatography and the development of polymer monoliths, specifically in porous-layer, open-tubular formats. In the first case, I developed several prototype column ovens specifically for capillary and microbore scale – complementing a move by the industry towards faster, greener separations. Current column heaters are slow to heat and cool, so often cannot maintain pace with the separation when using gradients. By taking my knowledge of thermoelectric technology from a former life and applying it to chromatographic separation, we were able to achieve rapid heating and cooling rates (~400 °C/min as compared with 30 °C/min for the fastest column heaters) – perfect for fast separations. We now have a commercial prototype and are trying to make the technology more flexible. Watch this space.

My second area of interest is in the development of monolithic porous layer

rather than the push of mathematical and statistical sophistication. 'Fit-for-use' is a common approach in chemometrics, which tries to adapt statistics to chemistry instead of vice-versa. In general, the approach to analyzing data is a holistic one, taking a multivariate modeling approach. This can reveal unexpected patterns because the combined effect of all variables is taken into account, in contrast to traditional chemical and physical relationships, which usually consider just one or a few variables at the same time. The incorporation of wider effects can be especially advantageous for studying complex systems, such as in metabolomics and system biology. On the other hand, it can also be a disadvantage because complex analyses can be more difficult to interpret and translate to the scientific question at hand. This brings us back to the greatest challenge for chemometricians, which is to make the subject understandable and palatable to every analytical chemist, whether they have a mathematical/statistical background or not. Our goal must be to position chemometrics center stage.

"By taking my knowledge of thermoelectric technology from a former life and applying it to chromatographic separation, we were able to achieve rapid heating and cooling rates – perfect for fast separations."

open tubular (monoPLOT) columns, particularly for GC. This involves the in-situ fabrication of polymer layers inside fused silica capillaries. The process can be approached in a couple of ways, usually photo- or thermally-initiated polymerization; however, these methods can be quite complex with poor reproducibility. To make it easier (and crucially, more repeatable) we have developed some unique instrumentation to automate the process, and recently added in-process detection methods to further increase column-to-column reproducibility. Once again my engineering background is paying dividends. Successful patent applications were filed for both technologies and there has been considerable industry interest to date.

In 2012, I received the Royal Society of Chemistry Ronald Belcher Award for my work in high temperature capillary LC and capillary LC related instrumentation development. Hailing from a non-chemistry background, I feel highly privileged but also acknowledge that it would not have been possible without the guidance and advice of my more chemistry-savvy mentors (Brett Paull and Ekaterina Nesterenko) or my engineering background and experience. By becoming a researcher at the ISSC, I have been able to widen my own academic reach and find a creative niche: "analytical engineering". I am pleased to recognize, on a daily basis, the innovation and rewards that a multi-disciplinary approach can generate.

Fighting Organized Drug Crime

Why X-ray analysis should play a far bigger role in the detection of illicit substances.

By Peter Munk, Technical Product Manager at PANalytical B.V., Almelo, The Netherlands.

Counterfeit medicines. Falsified medicines. Drugs of abuse. Recreational drugs. Illegal narcotics. Steroids.

Not only are these substances extremely bad for health, taking thousands of lives every year, they are equally bad for the economy, tying up hundreds of billions of Euros/Dollars/Pounds/Pesos, etc. Yet, despite the frenzied response from governments worldwide, the reality is business as usual: enormous profits for the criminals who manufacture and distribute counterfeit medicines and controlled substances.

What can analytical scientists do to make the fight-back that little bit more effective? One way is to simplify identification of the substances involved. That means more effectively, quickly and cheaply sorting through an enormous range of structures that are compositionally often very complex and anywhere between purely natural to 100 percent synthetic. The diversity of chemistry and source challenges scientists to look across the boundaries of disciplines to solve the analytical problem. Usually, forensic and pharmaceutical laboratories combine several techniques, of which the most common are liquid/gas chromatography, mass spectrometry (including combinations) and nuclear magnetic resonance (NMR) spectroscopy. More recently, optical spectrometry techniques like near-infrared (NIR) spectroscopy, Fourier transform infrared spectroscopy (FTIR) and Raman spectroscopy have also gained a substantial role in the identification of illegal products.

Here, I want to make the case for the analytical power of X-ray diffraction (XRD) and X-ray fluorescence (XRF) spectrometry. The role for X-ray analysis has, to date, been remarkably limited; yet it can provide quick and unambiguous screening and identification of counterfeit drugs and illegal narcotics.

Take counterfeit drugs: modern XRD instruments are capable of highly accurate and robust identification of a wide variety of polymorphs in active pharmaceutical ingredients (APIs). They can discriminate among complex systems including hydrates, solvates, salts and co-crystals, which characterize solid-state pharmaceutical products (and controlled substances). Subtle variations in the final product, such as quantities of APIs and excipients, granulation, tablets, coatings and other dosage forms can all be detected non-destructively using current multi-purpose XRD with 1D, 2D and 3D (computed tomography) detection. Blister-packaged drugs can even be analyzed within their wrappings, checking for authenticity, thereby leaving the product "as is". Evidence of adulteration will be valid in court, while the original material is still available for other analytical techniques to support the results.

XRD is listed as a Category A technique in the latest (2011) recommendation document from the SWGDRUG (Scientific Working Group for the Analysis of Seized Drugs) meaning the "highest discriminating power" (1). The X-ray analysis company that I work for, PANalytical, will soon launch a unique XRD database consisting primarily of controlled substances, which was developed with data from the Drug Analysis Services of Health Canada. It contains a wide variety of illegal narcotics, steroids, clandestine drug products, related pharmaceuticals and precursors, that will support forensic, criminal investigation and customs laboratories in their challenging analysis tasks. Professional principal component analysis (PCA)-based clustering analysis even allows the origin of the material to be determined.

When it comes to fast and highly reproducible elemental analysis, XRF has a lot to offer to the pharmaceutical and forensic arenas. Apart from its high sensitivity for metallic residues in medicine, such as catalysts and impurities, new fingerprint methods are efficient in screening for counterfeits and identifying controlled substances. Sample preparation, if required at all, is very simple. Several studies have illustrated the capability of discriminating the place of origin of foods (2, 3) and plants (4), based on very subtle differences in trace element footprints, which is equally applicable to seized counterfeits and drugs.

The time has come for forensic labs to open their doors to XRD and XRF. It's a step in the right direction towards fighting drug crime.

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Winning the Sports Drug Arms Race

With a constantly growing pool of alternatives available to cheating athletes, more comprehensive doping control analytical assays are needed.

By Mario Thevis, Center for Preventive Doping Research / Institute of Biochemistry, German Sport University Cologne, Germany.

Recent confessions by convicted athletes have exposed the impact of drugs on sport. They have also demonstrated the influence that sport drug testing analytical strategies have on doping behavior. Although there is no guarantee that the confessions are comprehensive and exhaustive, the trend is clear: Whenever a doping method/drug becomes detectable, ways to undermine the improved control system are sought. It's doping's own form of an arms race.

Doping control analysis is a constant challenge. An important and integral part of international anti-doping efforts, the analyses must cover an ever-increasing collection of opportunities, both theoretical and practical, that are open to cheating athletes. These include the misuse of approved drugs, such as anabolic agents and erythropoiesis-stimulating substances; the administration of emerging drugs – with and without clinical approval; designer substances; and methods of doping such as blood transfusion and gene doping. Tests are required that, for example, unequivocally identify long-term metabolites of xenobiotics; that distinguish minute differences between naturally (and endogenously) occurring compounds such as testosterone, growth hormone, or erythropoietin, and their synthetic analogs; and that discriminate trace amounts of drug-derived banned substances from the naturally produced counterpart from urine or blood. All this is required on a routine basis - a demanding, while at the same time logical and indispensable, task.

"The analyses must cover an ever-increasing collection of opportunities, both theoretical and practical, that are open to cheating athletes."

How has the arms race developed? When testing was first introduced, out-of-competition controls were rare. So, doping athletes simply allowed sufficient time for prohibited substances to be eliminated from their bodies to ensure negative tests during and post-competition. With the introduction of unannounced out-of-competition controls, new/alternative drugs that were not detectable became favored. A prominent example is erythropoietin (EPO), which was not distinguishable from the natural hormone for almost a decade after the therapeutic agent was launched. Many athletes (they now admit) abused the drug to a considerable extent, illicitly increasing their endurance performance without risk of being discovered in doping control tests.

When analytical methods became available to identify recombinant EPO in human urine and blood, homologous blood transfusion (HBT) became a method of choice, followed by autologous blood transfusions (ABT) when HBT could be successfully tested for. Now, the implementation of the Athlete Biological Passport (1), which detects non-natural alterations of an individual's blood parameter profile, severely limits the options on blood doping.

There have been comparable developments with anabolic-androgenic steroids. Analysis of xenobiotic steroidal substances has been significantly improved, with the development of new instruments and identification of long-term metabolites. This has led to increased misuse of testosterone and, arguably, of unknown (and hence undetectable) 'tailor-made' steroids. Fortunately, steroid profiling and isotope-ratio mass spectrometry have now been established as a reliable means to reveal these doping practices.

Despite these substantial improvements to doping controls over the last decade, new loopholes are constantly being exploited by cheating athletes. One anti-doping tool of great utility, therefore, is long-term sample storage with the option of re-analysis. There are a number of examples of applying optimized test methods to long-term stored specimens to uncover doping. In combination with legislation on the consequences of adverse analytical findings, this strategy should effectively deter and discourage drug use, hopefully drawing the doping arms race to a successful conclusion.

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Livestream 1800 Metabolome Profile:
James Wallace
13:08/17.3.2018
Share with Physician?
Default physician: Dr. A. Kumar

Future Separations

Smaller, faster and smarter, the next wave of separation technology will be portable and personal.

By Emily Hilder and Robert Shellie

Separation science is an enabling science, the fundamental physical processes of which were identified over a century ago (see "Past Separations", page 28). Progress in the field is driven by the demands of biological, pharmaceutical, environmental and forensic scientists, and is realized by developments in technology. Materials science, electronics and engineering are each contributing to the technology truly becoming smaller, faster and smarter.

We believe that this progress will continue. In fact, as members of the first generation to grow up with computers, we see the development of computers from large, immovable objects to mobile devices as a model for the development of our field. Over the last 50 years, we have seen a move from classical chromatography to high pressure, high performance systems. API Project no. 6 (see "Classical Chromatography Limits", page 28) illustrates the limits of classical chromatography showing the lengths that were required to achieve what is now modest performance using modern systems. In the same way, we expect similar levels of development over the next 50 years, pushing new boundaries. Below we present a snapshot of separation science as it is today, as a prelude to our predictions for future development.

Current separations

As we inch ever closer to theoretical limits, we must ask: "Will chromatography shortly come to a standstill?" To answer this question, let's first give some brief consideration to the current state of the art.

Evergreen HPLC

High pressure liquid chromatography (HPLC) is one of the most frequently used and relied-upon analytical technologies in the chemical and pharmaceutical industries today, with reversed-phase HPLC at the top of the heap. To achieve this, a great deal more has been invested into enhancing the efficiency of instrument design and in the synthesis of sorbent materials with smaller particle sizes than in new selective materials. Ultra high-pressure pumps and the development of small particles that can withstand such pressure give some UHPLC practitioners the ability to achieve separations with GC-like efficiency (see Figure 1). Meanwhile, numerous

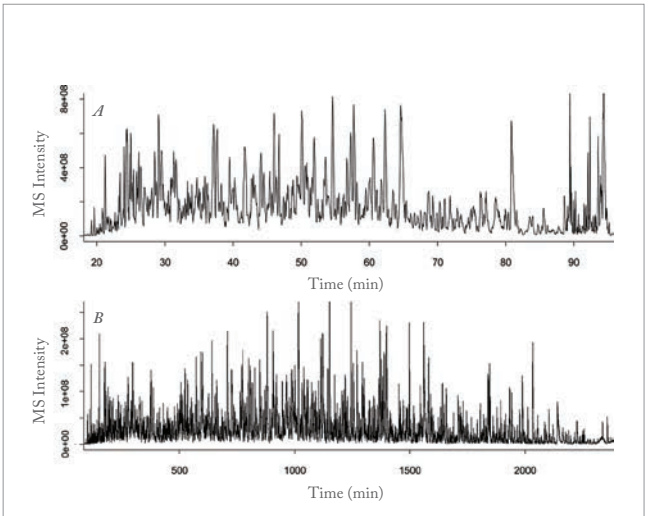


Fig1: Base peak chromatograms for the analysis of E. coli. cell lysate using a 15cm silica particle packed column (A) and a 350cm long monolithic silica C18 column (B). Reproduced with permission (see ref. 1).

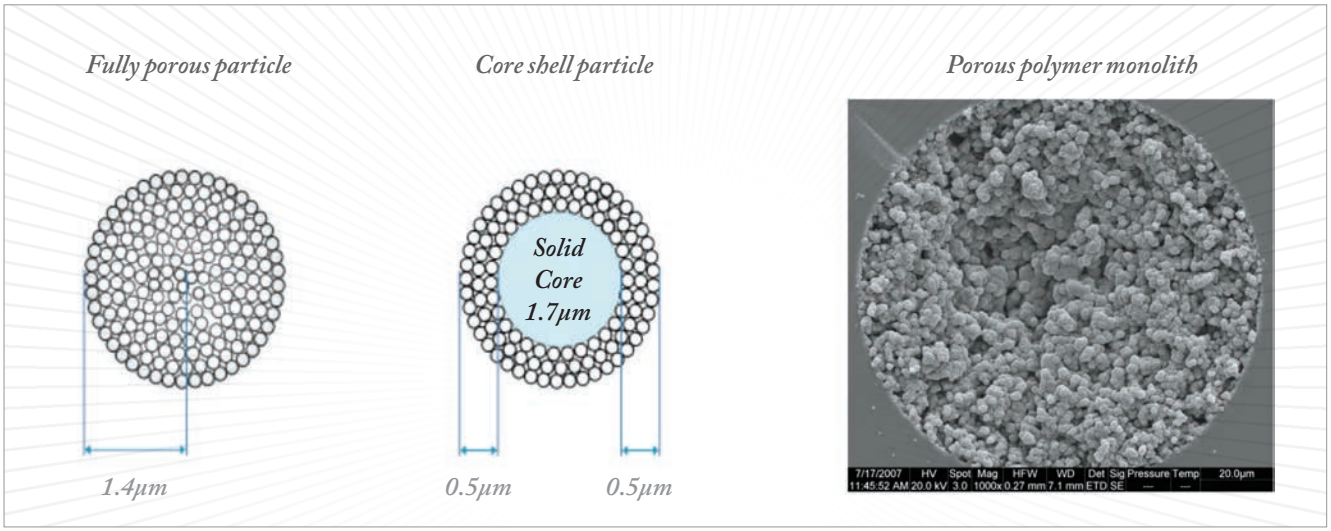


Fig 2: Comparison of different types of stationary phases commonly used in LC

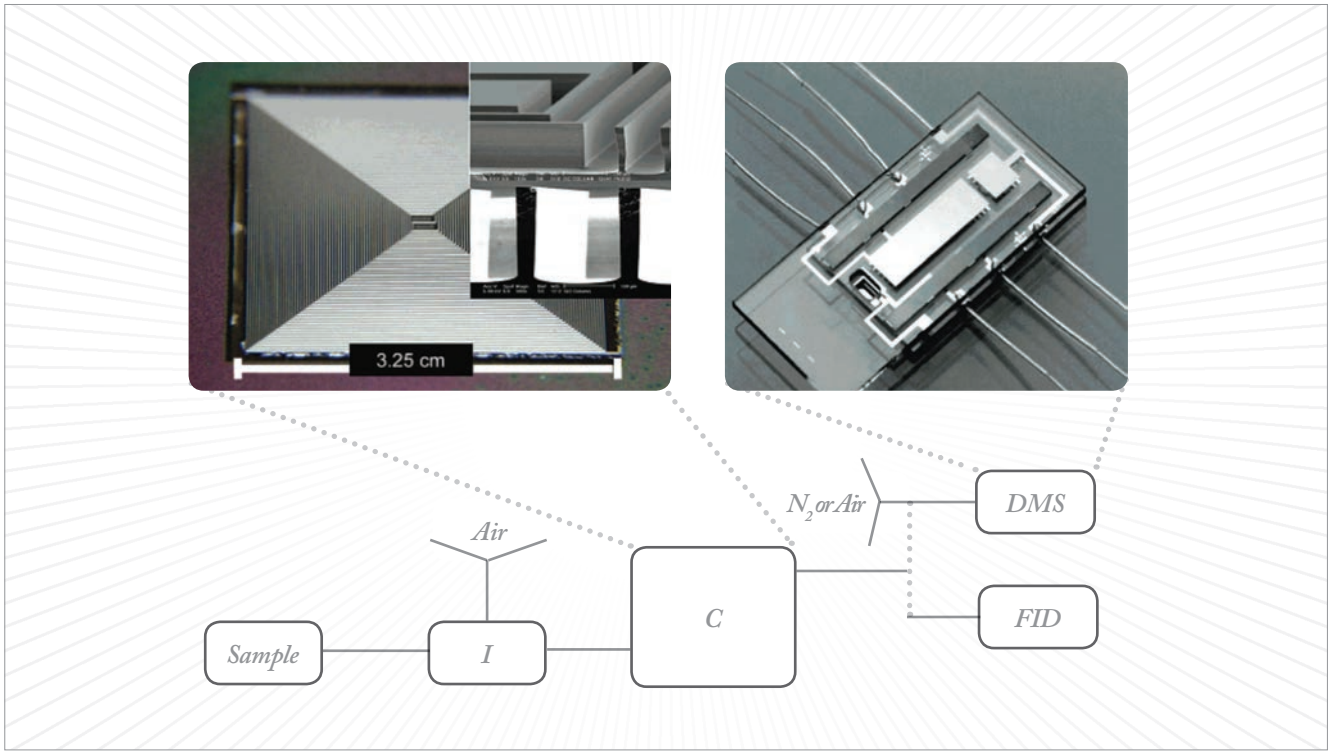


Fig 3: Example of micro-machined instrumentation using a gas chromatography platform. I: split inlet, FID: flame ionization detector, C: microfabricated column (photo inset), DMS: microfabricated DMS cell (photo inset). Reproduced with permission (see ref. 2).

significant developments in materials science have given us both core shell particle technology and monolithic columns (see Figure 2), both of which have led to improved separation efficiency using relatively low pressures.

Notwithstanding the heavy focus on efficiency, one of the outstanding features of HPLC is still the incredible diversity of stationary phase chemistries that can be synthesized, which provide real control of separation selectivity. Reversed phase HPLC has been, and will remain, the workhorse of LC, but new developments in stationary phase chemistry, for example, hydrophilic interaction chromatography (HILIC), have extended its reach into exciting new areas such as metabolomics.

Steadfast GC

Analytical gas chromatography (GC) is an indispensible tool in the petroleum and chemical industries. Widely viewed as a mature technology, it relies heavily on the use of wall-coated open-tubular fused silica and, to a lesser extent, metal capillary columns using convection ovens. Most users rely on high plate number rather than subtle selectivity manipulation to achieve separation of complex mixtures, and development of new stationary phase materials is relatively stagnant. Innovative materials development is often reserved for specific applications; for example, columns for separation of polycyclic aromatic hydrocarbons appeared recently. The recent introduction of a new class of ionic liquid stationary phase is also an area to watch as niche separations are developed using these materials. Analysis and characterization of biodiesel blends is a case in point, where ionic liquid stationary phases provide unique selectivity advantages.

A decade ago, the focus of column R&D was on bleed; the war against bleed is largely over (1 pA over the range from 40 to 300 C). Currently, a focus on reducing column activity is leading to emergence of highly inert capillary columns enabling specific analyses at trace levels that were hitherto impossible (3). Further development will likely broaden applicability to a wider range of polar solutes like amines and diols. Despite the speed and efficiency advantages of using narrow-bore capillary columns, there are relatively few commercial offerings of sub 0.1 mm internal diameter capillaries today and further developments should also be expected in this area.

"These instruments offer smaller footprints, lower energy demands, ultra-fast analysis, and high throughput. Most importantly, they open the door to portable instrumentation, alleviating critical sampling and sample-integrity challenges."

Miniaturization

Recently, progress in micro-machined instrumentation has been buoyed by the maturation of microfabrication technologies (see Figure 3) These instruments offer smaller footprints, lower energy demands, ultra-fast analysis, and high throughput. Most importantly, they open the door to portable instrumentation, alleviating critical sampling and sample-integrity challenges. Micro-machined sample inlets and detectors will couple with separation columns of smaller internal diameter. Most of these miniaturized systems rely on resistive column heating; a new application of an old technology. Moving away from the convection oven has been a major step towards miniaturization.

There have also been exciting developments in detection technologies for miniature GC. Two notable examples are the chemiresistor detector developed at the University of Michigan (4) and the use of miniature photoionization (normally used as standalone sensors) in GC detection by researchers at the University of York (5). Miniaturized mass spectrometry is also experiencing a burst of research and development interest.

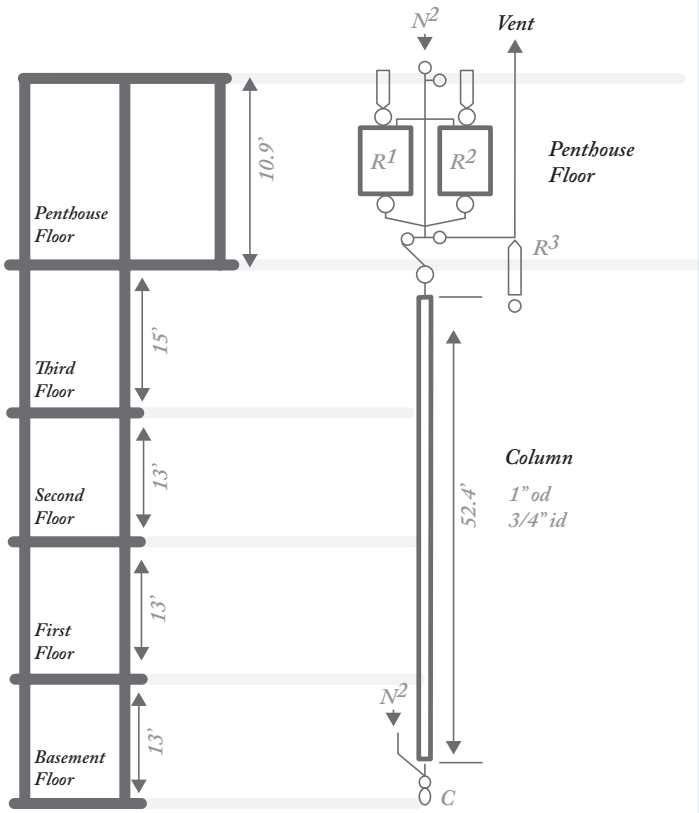
Multidimensional progress

Access to a wider range of HPLC stationary phases has given us multidimensional chromatography, which uses the

Classical Chromatography Limits

In 1927, the American Petroleum Institute established Research Project No. 6 at the National Bureau of Standards to identify chemical constituents of petroleum fractions. In 1960, F. D. Rossini, project director at that time, estimated that the project had consumed around 500 man-years of work as the system moved from systematic distillation through simple adsorptive filtration to true chromatographic separation. The project was terminated in 1967.

16 m adsorption column (25.4 mm stainless steel tube containing 3.7 kg silica gel, insulated with electric resistance heating wire) housed in 18.9 m shaft. Reservoir R1 holds sample, R2 holds desorbing liquid (alcohol). Typical sample volume: 500 ml. Receiver C collects fractions. Entire system can be placed under inert gas (nitrogen) pressure from top or bottom. Desorbing liquid regenerated by heating column to 200 °C and collecting evaporated alcohol in receiver R3.



Past Separations

The documented history of separation science extends back over 200 years. Here, we sumarize the key developments in the field.

1807

Ferdinand Frederic Reuss observes that application of an electric field caused clay particles to migrate in water, the basis for modern electrophoresis.
Several German chemists explore the phenomenon first observed by early dye chemists: dye solution migrates up an inserted material by capillary action, with the dye components separating into distinct bands.

unique selectivity of each dimension to maximize separation power. Without such developments, 'omics' – in particular, proteomics and metabolomics and related medical research – would simply not be where it is today.

There is also growing interest in multidimensional GC (MDGC) separations. The recent introduction of planar microfluidic column connections, which meet important requirements for MDGC interfacing such as low dead volume, a high degree of inertness for reactive solutes, and efficient sample transfer, has made the approach more amenable. However, data handling, although vastly improved recently, continues to be a bottleneck.

"It is also clear that partition chromatography is far from obsolete as an analytical method, as technological improvements have overcome many of the initial drawbacks."

Looking ahead

The separation science pioneers of the past were largely botanists and biochemists. As noted by Richard Synge in his 1952 Nobel Lecture on partition chromatography:

"And here of course the method is giving to the biochemist something akin to what the microscope gave to early biologists […] When the effects with small molecules are better understood we shall be much better able to understand from the structure of the larger molecules what is their function in terms of such intermolecular forces. Then chemistry really will begin to merge with biology as closely as it has already merged with physics. Partition chromatography may well be remembered for this contribution long after it is quite obsolete as an analytical method."

We see the importance of separation science as a tool in the life sciences continuing, even strengthening. The demand to understand biology in terms of chemistry has only increased. It is also clear that partition chromatography is far from obsolete as an analytical method, as technological improvements have overcome many of the initial drawbacks.

As always, future developments will be strongly driven by the demands of end-users. And, while the underlying demands have not changed greatly, advances in technology have led to much greater expectations. To meet these, separation science should focus on:

- The ability to separate more species in reasonable time frames
- The ability to handle increasingly large amounts of data
- Achieving highly selective and

1861

Frederich Goppelsroder first to describe separation of dyes by capillary action as 'capillary analysis'.

1901

Russian botanist Mikael Tswett coins the term 'chromatography' from the Greek words meaning colour writing to describe the physiochemical basis for the separation when he used calcium carbonate (chalk) to separate chlorophylls and carotenoids in plant extracts. Tswett's work is mostly unknown within the field as he publishes his findings only in Russian or German botanical journals.

1930

Richard Kuhn and Edgar Lederer 're-discover' Tswett's technique for the separation of carotenoids and amino acids

sensitive analysis of specific targets
- Smaller, cheaper, portable and, perhaps, personalized analytical systems.

Dimension X

The ability to separate more and more species as we continue to probe complex biological systems will remain a critical challenge. We believe that this will be best addressed through multidimensional separation approaches.

Mature techniques, such as GCxGC and LCxLC, will continue to be developed and refined. Other combinations, with the potential to re-define how we view multidimensional separations, will also appear on the horizon. Two-dimensional combinations, such as LCxGC, LCxCE and CExCE, have already been explored and the proof-of-concept for three-dimensional approaches (GCxGCxGC, LCxLCxLC) has been demonstrated (6, 7). It is reasonable to think that we will soon move even beyond these, with the number of coupled dimensions limited only by our own imaginations. At the same time, this unlimited dimensional progress highlights the great challenge to such systems: handling the sea of data produced.

"The widespread availability and speed of technological development in mass spectrometry has expanded the range of detection options for separation science beyond what many would have believed possible."

At a recent meeting of the Australian and New Zealand Society for Mass Spectrometry, we separation scientists were challenged by mass spectrometrists to justify why sophisticated separation technology is still necessary for many applications. The program at the upcoming HPLC meeting in Amsterdam in June 2013 promises to challenge chromatographers with similar questions. We argue that separation science will be more critical than ever. The widespread availability and speed of technological development in mass spectrometry has expanded the range of detection options for separation science beyond what many would have believed possible. At the same time, it is well known that one of the critical limitations of mass spectrometry is its susceptibility to matrix effects; sample preparation or pre-treatment is a key factor in reducing this. The development of highly specific and selective separation phases offers the opportunity to realize the true power of MS technology for the targeted analysis of complex samples, leading to the next generation of multidimensional analysis.

1941–1952

Initially, Archer Martin and Richard Synge use chromatography to study the amino acid composition of wool. Their method, defined as partition chromatography, is physically the same as Tsvett's yet conceptually different in that one liquid is bound to a finely grained solid (usually silica gel) packed in a glass tube, and a second, immiscible liquid is percolated through it. Separation is based on differential partitioning of molecules between the stationary liquid and the mobile liquid phase. Lack of reproducibility in the properties of silica gel and packing glass tubes also leads them to consider new formats, the most successful using paper as the stationary medium. Interestingly, in their initial publications they also describe what they believe, based on their theory, will be the future potential developments and limitations of this technique. Many of these could be not tested with the technology available at the time, but have since become a focus of some of the most exciting new developments in separation science.

1952

Archer Martin and Richard Synge are awarded the Nobel Prize in Chemistry "for their invention of partition chromatography" and introduce a model that suggests other systems, such as using gas as the moving phase, a mathematical theory and its application to the separation of amino acids and peptides with wider impact in biochemical studies. Archer Martin and Anthony James extend Martin and Synge's concepts to the use of a gaseous mobile phase.

Personalized analytical systems

In our view, the most exciting and significant developments in separation science will stem from miniaturization. Truly portable, and eventually personal, analytical technology is where we are headed.

Developments in 'lab-on-a-chip' and similar miniaturization technologies have not yet provided the outcomes that have been promised. The underlying technology, for example, in fabrication and electronics, has been one contributing factor. As fabrication technology develops and improves, we are confident that technologically more 'difficult' approaches, such as shear-driven chromatography and microfabricated columns, will emerge as practical alternatives in separation science.

Opportunities to develop from existing portable technology are among the most exciting to us, although they are admittedly far from commercial reality. The number of mobile phone subscriptions worldwide has increased from one to six billion within the last 12 years; this means that 75 percent of the world's population now has access to a mobile phone, and many of these devices have progressed well beyond simple voice communication to become sophisticated, personalized data handling tools. Using mobile phones for chemical sensing is already possible and with the current rapid pace of developments in mobile technology, it is likely that the impact of smartphones will be felt in separation science sooner rather than later.

In the immediate future, some of the factors driving progress in separation science are far more pragmatic. There is a continuing conflict between pushing a technique to its theoretical limits for maximum performance and the commercial desire for a quick return on investment (8). Certainly, with the numerous demands of the life sciences, as absolute limits are reached, new approaches will need to be explored.

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1957

Michael Golay, a consultant for the Perkin-Elmer Corporation, develops modern gas chromatography when he identifies from theoretical work that a very long capillary column with its wall coated in a thin film of liquid can yield superior separations – the first description of capillary gas chromatography.

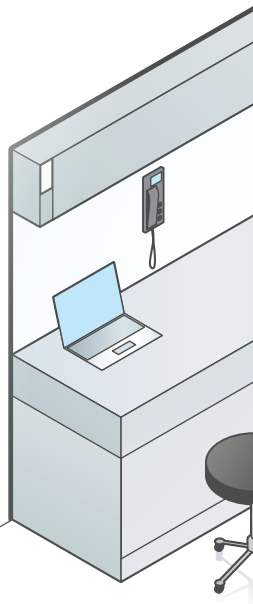
1960s

J. Calvin Giddings and Csaba Horváth provide the theoretical and practical basis for modern high performance liquid chromatography (HPLC), requiring the development of pumps that could deliver liquid at a defined flow rate at high pressures and detectors that could sense small sample sizes.

1970s

Lloyd Snyder, John Dolan and Russel Gant develop a theoretical framework for gradient elution as a solution to the 'general elution problem' (i.e., that, for most samples, no one set of conditions is suitable for elution of all components in a reasonable time and with reasonable peak shape). Snyder also provides a description of various solvent properties important in liquid chromatography to control separation selectivity: the acidic, basic and dipolar nature of the solvents, and plots them as a triangle, often described as the Snyder solvent selectivity triangle.

WHY WE DO WHAT WE DO



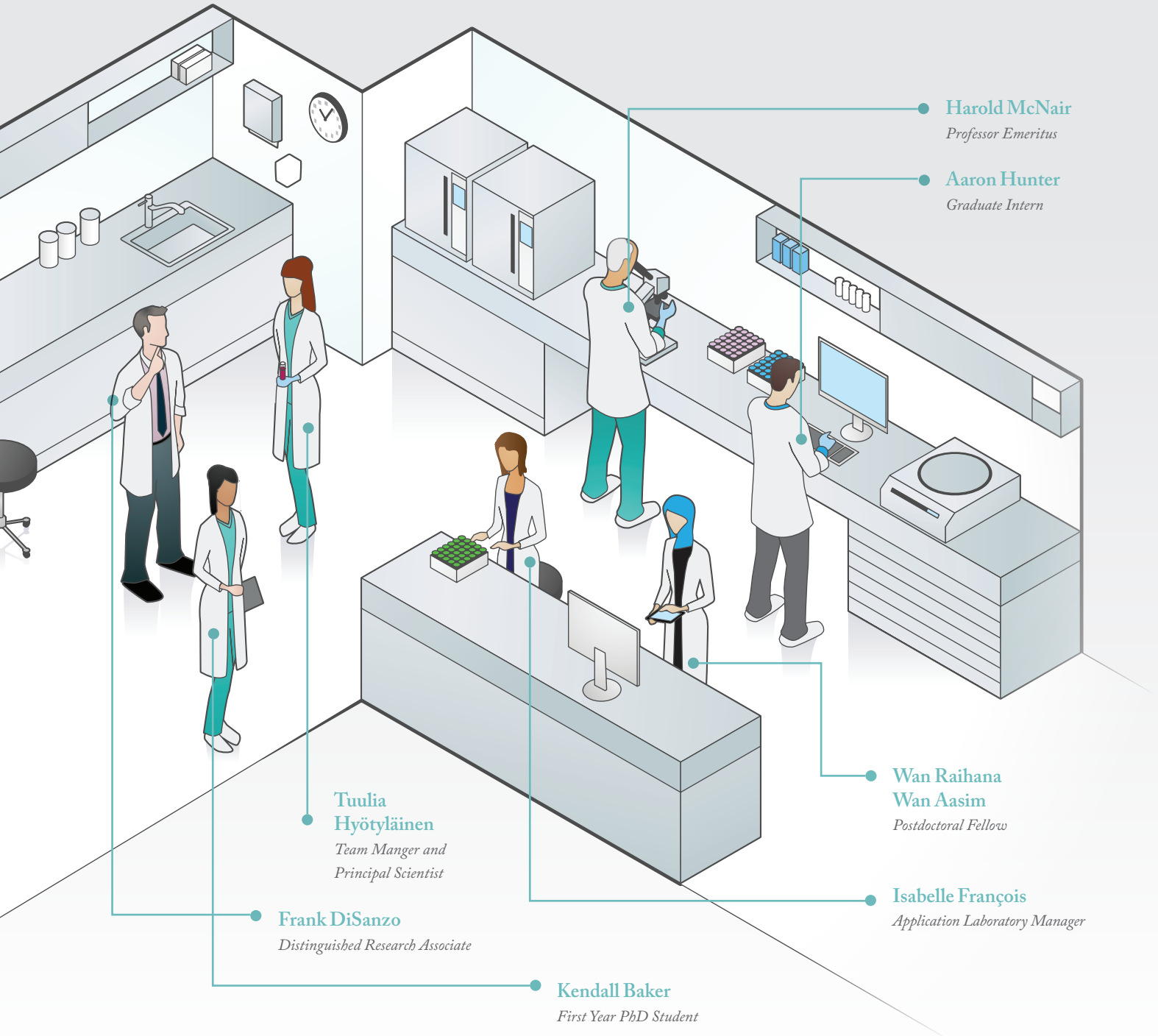
Analytical scientists love what they do. They relish a practical challenge, see the field to be the very foundation of good science and consider its place to be at the heart of modern society.

By Richard Gallagher

If you are reading this, the chances are that you are professionally involved in analytical chemistry or a related field. What does that mean to you? Do you have a sense of belonging to an enterprise or movement that shares a set of ideals? If so, what are its defining characteristics, who are its members and what is their motivation? Do you feel happy, lucky even, to be involved? How did you get drawn in in the first place?

One way of approaching these questions would be through a poll that surveys the opinions of a large sample, to establish the zeitgeist of the field. We'll do that later in the year, when this publication is more established.

A complementary approach is to drill down into the motivation of individual analytical scientists, and that's what I've done here. Seven people feature in this article, all of whom describe themselves as analytical scientists. To the extent that it's possible, I've tried to select people who are 'representative'. Two are recent graduates, one of whom has just started a PhD; another is a postdoc; two others are in their working prime, holding senior positions, one in academia and one in industry; and the final two are late in their careers, and can



reflect back on three and five decades of experience. Four of them are women. Two are currently based in the United States, four in Europe and one in Asia. Find out more about them in their profiles.

The seven were asked the same set of questions. The intention was to explore how they became involved in analytical science, what they enjoy and dislike about their work, how they feel that their chosen area is viewed by scientists in other fields and by the wider public, and what their sense is of the contribution of analytical science to society. Some trends emerge, as illustrated in the selection of answers here and in 'Recognize Yourself?' on page 35.

There are as many experiences and views on these topics as there are people in the field. Why not share your views on what being an analytical scientist means to you? You can join the discussion at theanalyticalscientist.com/issues/0313/402 or follow the twitter conversation @tAnaSci #whywedowhatwedo.

If you'd like to submit a short video response to be posted on our website, get in touch with me at richard.gallagher@texerepublishing.com.

What do analytical scientists contribute to research and to wider society?

There is strong consensus that analytical science plays a central role in research. For Harold McNair, it's all-encompassing. "If you cannot quantitate and measure, you are not really doing science," he says. "There are three hundred gas chromatographs on this campus alone (Virginia Tech), everyone has one. No lab in the world could live without GC, LC, mass spec, NMR, AA, UV, visible and IR." Aaron Hunter views things in the same way: "Analysis and the inferences we draw from it are the beating heart of science. From the most minute composition of our bodies and environment to the respective histories of our species and the entire planet, the ambition and sheer scope of analytical methods is inspiring."

Tuulia Hyötyläinen, commenting on her specific field, which is metabolomics, notes that "Analytical methods are essential for the early detection and diagnosis of disease, and in the development of better therapies and drugs for various diseases".

Turning to the societal impact of the field, Frank DiSanzo observes that "Analytical scientists have made significant contributions in areas of the environment, food safety, energy and fuels research, and deployment. I think analytical chemistry surrounds most aspects of current society and the sciences." One personal example, from agriculture, is provided by Kendall Baker. "I am studying the degradation of phytate," she explains, "which is an 'anti-nutrient' in the feedstuffs of non-ruminant animals, and a source of phosphate pollution in the environment, during the germination of barley. It has huge relevance to the agriculture industry."

"Analytical scientists have made significant contributions in areas of the environment, food safety, energy and fuels research, and deployment. I think analytical chemistry surrounds most aspects of current society and the sciences."

Where would we be without it? "I believe that there would be no 'society' without analytical science," says Wan Raihana. "It is what keeps us safe, healthy and secure as well as helping us to understand our past, present and future."

Who or what sparked your interest in analytical science?

More often than not, a specific person played a decisive part in getting this group involved in analytical science. Another recurring way of getting hooked is the sense of play that working with complex instruments can provide; more than once, they were referred to as 'toys'.

For some, the die was cast early in life. "My father was a physics teacher, so I have been getting detailed explanations of natural phenomena since I was a kid," Hyötyläinen explains. "I really liked, and actually still like, the experimental part. In fact, I now do basic chemical experiments with my own kids."

"I grew up in a small town in Arizona," says McNair, "where a schoolteacher, John Marshall, taught us one year of physics and one of chemistry. That would be 1950-51. He had a master's degree from MIT and was very tough. There were two of us in the class, myself and Joe, who were given 'one more extra problem'. For me, that was the start of it."

Others got the bug slightly later. "I had been interested in chemistry in high school, but did my degree in industrial engineering," explains Isabelle François. Two interactions caused her to change course. "During my master's thesis, I was challenged to improve my analytical thinking by Joeri Vercammen, who also encouraged me to start a PhD. I did so under Pat Sandra, and the independence he offered let me grow as an analytical scientist: his stimulation, dynamism and knowledge were highly motivating. Sandra provided the foundation for who I am today as an analytical scientist, for which I am very grateful."

Raihana also cites a PhD research supervisor, Tan Soo Choon. "He was encouraging when it came to me experimenting with analytical instruments. I learn best with a 'hands on' approach, so his willingness to give me free rein to 'play' with the different instruments helped me tremendously. They may look scary and they are definitely expensive, but once you get the hang of them, running the instruments becomes second nature."

Instrumentation also caught the imagination of DiSanzo. "Following my undergraduate degree, I worked for a short time in an analytical chemical laboratory," he recalls. "I was intrigued by analytical instrumental analyses, such as separation science and thermal analyses. Following that, I pursued my PhD at a university where analytical chemistry was very strong. I was impressed by the fact that graduating students had a big impact in industry and the professors were working at the frontier of the field of research."

What have been your best and worst moments as an analytical scientist?

The most mentioned topic is successful problem-solving. Labwork generally is the key: it's a shining highlight when it goes well and a source of gloom when it goes badly.

"I have enjoyed opportunities to develop and deploy many of the state-of-the-art technologies for research and complex problem-solving," says DiSanzo. "This has involved international travel to assist on key company projects as well as close interaction with highly motivated fellow scientists in the field to stimulate new ideas."

Raihana concurs. "The best thing about being an analytical scientist is the constant challenge," she says. "Whether it is coming up with an approach to a particularly difficult chromatographic separation, or troubleshooting why the GC-MS just won't work, life is a series of problems that require solving." Her best moment? "The day that I completed the lab work for my PhD project. I had reached the end of a long journey that, while full of challenges, was ultimately satisfying. I felt a great sense of achievement."

For the two younger scientists, that's still to come. "In my short career, I've most enjoyed when a protocol I have devised, planned, implemented and perfected gives significant results," says Baker. Hunter agrees. To him, "The satisfaction of clear and unambiguous results is a definite contender for the best thing, especially when they arrive along with a resolution of a frustrating problem! However, my experience so far has mainly been troubleshooting and calibrating a home-spun effort to construct a phenol-detecting HPLC setup – I'm sure any opportunity to innovate or discover something new would pip my example to the top spot."

Anyone recalling the first time that they waded into laboratory research can all empathize with Baker and Hunter's low points. "The thing I have found most frustrating is when results of replicate experiments are not consistent," says Baker. "It can be very stressful; you sometimes feel like every step forward is counteracted by a huge step backwards." For Hunter, "When you think everything's right, know it's been done properly elsewhere, but still aren't getting that clear data: those are the worst moments."

For her worst moment, Raihana chooses an event that sends a shiver of horror down the spine. "I had spent the entire day in the lab doing sample prep on more than a hundred samples," she recalls. "My hand was cramping from all the pipetting and my back hurt. The final step was to stick the samples in the GC-MS autosampler and head off for a good night's sleep... I dropped the entire tray, scattering tiny autosampler vials every which way. That meant spending several more hours crawling on the lab floor and then arranging them in order."

Despite his "Fifty-six years of making injections into a gas chromatograph", McNair can pinpoint precisely his high and low points. The most exhilarating: "In 2009 at Pittcon, I was on the front page of LCGC magazine, nominated as A Pioneer in Chromatography. That was satisfying. But on the same page, my last PhD student, Kevin Shug, was named the International Emerging Leader in Chromatography. That just tickles me to death; I got more satisfaction from him winning than from all of my awards". His worst moment came in teaching freshman chemistry. "One time, in a 1½ hour class of 450 students, the two back rows left after 15 minutes. I'd just said what would be in the exam, and one of them shouted out 'who gives a shit', and walked out. That lack of interest and dedication among first-year students is my low".

Recognize Yourself?

We had an independent expert consider the responses and come up with a profile of a typical analytical scientist. Here's what that person looks like:

- Wants to be a 'force for good', but keen to avoid the spotlight
- Is fascinated by what is real, but hidden from plain view
- Loves puzzles, has an analytical mind
- Has a tendency to look inwards, not outwards
- Prefers science hands-on, rather than theoretical
- Likes to 'play' with sophisticated technology
- Has a strong urge to mentor.

Do you fit this prototype? What other characteristics help make up analytical scientists? Join the discussion at theanalyticalscientist.com/issues/0313/402

Harold McNair

Age: 69
Position: Professor Emeritus, Department of Chemistry, Virginia Tech, Blacksburg, VA, USA.
Background: BS in chemistry/physics, University of Arizona, MS and PhD in Analytical Chemistry, Purdue University, West Lafayette, Indiana. Following a Fulbright Scholarship spent in The Netherlands, I held positions at Esso Research and Engineering, F & M Europe and Varian Aerograph before embarking on a forty-five-year career at Virginia Tech.
What makes me tick: Science is not easy, you have to work hard. If people are going off course, I tell them.
Outside of science, I… still play tennis, it keeps me alive. And I really enjoy teaching short courses in interesting places.
My alternative career would be… nothing else. After going to graduate school in chemistry, I was committed to a lifetime as a scientist.

Wan Raihana Wan Aasim

Age: 32
Position: Postdoctoral Fellow at the BRAINetwork Centre for Neurocognitive Science, Universiti Sains Malaysia, in Penang, Malaysia.
Background: BSc chemistry and PhD at Universiti Sains Malaysia (currently awaiting PhD viva voce to be scheduled). I also worked part time in a lab that screens race-horses for doping.
What makes me tick: I hate to give up on something.
Outside of science, I… am really interested in computers. I've taught myself programing, website design, graphic design and video editing. Currently, I am involved in a community project to empower the indigenous people of Malaysia by providing them with computer and information technology skills.
My alternative career would be… honestly, I can't imagine doing anything else.

Isabelle François

Age: 32
Position: Benelux Application Laboratory Manager at Waters Corporation, located near Brussels, Belgium.
Background: Master in Industrial Engineering from Hogeschool Gent. I was responsible for the chromatography team at ExxonMobil Chemical Europe, Inc. before joining Waters two years ago.
What makes me tick: A combination of flexibility, innovation, and using my skills and knowledge within a team. I am motivated by exploring new analytical technologies to find the right solutions for customers and to create business impact.
Outside of science, I… enjoy getting to know other cultures by traveling, and spending time outdoors.
My alternative career would be… something very different, maybe language- or art-related.

Being fresh from his undergraduate years, Hunter has a suggestion for building that interest. "We did not get to grips with techniques in sufficient depth, and learning though practice is invaluable," he says. "So, more teaching of the 'how' instead of just the 'why', 'what' and 'who', please!"

Hyötyläinen also cites the mentoring role as a most satisfying one, alongside her current research. "We have exciting results on a biomarker for early prognosis of Alzheimers disease. Without the sophisticated analytical technique used for this study, we would not have been able to find this specific metabolite, which is a strong biomarker for the disease." One gripe from Hyötyläinen is that "The role of the analytical chemist is not appreciated enough. People have been watching television programs like CSI, and they seem to think that results come very easily. It can be a bit challenging when this attitude comes from collaborators. Those who don't have much of a clue about chemistry assume we have an unlimited range of methods ready and tested for all possible types of samples. We don't. Testing takes time and money."

"The role of the analytical chemist is not appreciated enough. People have been watching television programs like CSI, and they seem to think that results come very easily"

François offers two highlights, one from her PhD and one from her current role in industry. "I really enjoyed doing in-depth research and had moments of great excitement with certain novel 2D configurations which illustrated proof-of-principles," she says of the first category. "In my current role in industry, my skills and knowledge are used in a more practical way, in a manner that matters more in 'real' analytical life." Being an analytical scientist, François says, "Provides a high degree of job satisfaction. But, from the work/life balance perspective, it is sometimes hard to stop or say no. The analytical thinking process in your mind doesn't stop at five pm. Sometimes the urge to identify a better solution, to find lower detection limits, or to meet business targets can push you that little bit too much. Of course, this depends on your personality."

Do analytical scientists get the credit that they deserve?

There appears to be some mild frustration among analytical scientists about how they and their field are perceived.

"Many people really do not understand that analytical chemistry is not just putting samples into some instrument and getting results," says Hyötyläinen, finishing her thought above about fellow researchers. François believes that the wider public also misses the significance. "People often underestimate the work that is done in the lab. We may not be directly saving lives, but analytical science helps significantly in, for example, identifying medications, their effective doses, and their side effects; in recognizing the toxic substances that migrate from plastic wrapping; in water and air quality assessment, and in many other things."

If it is true that analytical science gets a bum rap, Hunter may have the explanation. "The details get lost in the 'big picture' thinking that's encroaching on the scientific community. This is clearly bad news for analytical science, which is all about the details."

Others have a more sanguine view. "People who are familiar with research do understand the significance of what analytical scientists do, and give credit accordingly," says Raihana. And McNair agrees. "For the most part," he believes, "all scientists get the respect they deserve – based on their students and their publications." He does, however, think that the term 'analytical chemistry' is a misnomer that may be holding back the field. "That term goes back to when they did titrations and gravimetrics. Talk about 'instrumental methods' and then you see the real value."

Whatever it is called, Baker has observed a certain respect being accorded. "Researchers from across the university often come to our lab asking for advice from my supervisor … and sometimes about the use of our equipment."

What type of reaction does your work evoke from family, friends and neighbors?

The consensus is that the role of the analytical science is neither widely understood nor a great source of interest to most people – and perhaps it should be. It is, however, a respected profession.

"Friends that are into science understand my job content, and talk about research is never far away in when we get together," says François. "However, the majority of my friends and family do not fully understand what I am doing. That's okay. I enjoy having the different influences of their culture, personalities and other insights. And if I describe my job using a practical example, such as the ability to detect and quantify the amount of doping products that are retrieved in

Frank DiSanzo

Age: 59
Position: Distinguished Research Associate at the ExxonMobil Research and Engineering Company (EMRE) in Greater Philadelphia, PA, USA. I provide broad analytical chemistry expertise and advise research programs in support of the company's global operations.
Background: Undergraduate degree in chemistry at the University of Connecticut; Masters and PhD in analytical chemistry from the University of Massachusetts-Amherst. For nearly 30 years, I have worked for EMRE.
Outside of science, I… enjoy international travel, learning about ancient civilizations, new cultures, and archaeology.
My alternative career would be… in another science field. As a child I enjoyed geography and later developed a strong interest in civil engineering, which eventually lead me to my current area of expertise.

cyclist's blood or urine, that is usually sufficient to get across what I do."

DiSanzo reports a similar experience. "Usually family members are closest in understanding what role we play," he says." My children have had significant exposure to science and interestingly we can have discussions using true scientific terminology. To non-scientific friends and neighbors, we are seen as someone with a very interesting career in a challenging field."

"At a cocktail party," McNair says, "about thirty percent of people know what's going on; the other 70% say they flunked chemistry, end of story."

"General approval, mixed with incomprehension," is the reaction that Hunter gets too. "That's why," he says, "effective communication is especially important when it comes to analytical science; the details of how a given result was actually obtained often gets lost in the hyperbole about theories and implications, when in fact it's the techniques themselves that can lead to a real paradigm shift."

What would you say about analytical science to inspire people?

Accepting that the field could benefit from a higher profile, what stirring messages would the group propose to attract people to the field?

Frank DiSanzo gets to the heart of the subject: "Analytical chemistry is a discipline focused on solving issues that puzzle much of our society. Our focus is not just simply analysis, but a combination of research and problem solving. To quote Jules-Henri Poincare: 'somewhere, something incredible is waiting to be known'."

Aaron Hunter combines the inspirational with the practical: "Just think of the possibilities! Analytical science produces some of the most real and cutting-edge research there is, and the sheer transferability of the techniques you'll learn will provide a diverse set of options for developing your career."

Tuulia Hyötyläinen suggests that how analytical science is done should be a selling point: "It combines a very practical way of working with a scientific viewpoint; the range of applications are wide, so you have a lot of choice in what to do. It also is multidisciplinary, so you always have an opportunity to learn something new."

Wan Raihana also promotes variety as a strong point: "It constantly offers new opportunities. There are not many areas of science that don't depend on analytical scientists to get the answers that they need."

Isabelle François lays out the benefits: "Next to the fact that it provides a high degree of job satisfaction, there is currently a need for good scientists across industry and in universities. Career prospects are good."

The last word goes to Harold McNair and is a demonstration of inspiration, rather than a description. "I've taken on in my lab three people who were working in fast food restaurants, one of them recently. I just got chatting to them: all had college degrees and I felt that in waiting tables they were wasting their lives. I followed up with long discussions, and the first two that I gave part-time jobs are both now on PhD programs at other universities. The current one, if he picks up some GC and GC-MS experience, can get a job in about six months, or go on to study elsewhere." Now, that's inspiring!

Aaron Hunter

Age: 22
Position: Graduate intern, University of East Anglia (UEA), Norwich, UK. Working to identify optimal growth conditions for phenol-degrading microorganisms with the aim of remediating wastewater.
Background: BSc degree in Biological Sciences. About to begin an internship with Nesta, the UK's 'foundation for innovation'.
What makes me tick: I want to succeed, and to contribute something new, wherever I end up!
Outside of science, I… do a lot of reading – seeing through the eyes of others can be some of the most illuminating analysis we can perform.
My alternative career would be… working in policy, communications or teaching. I'm something of an evangelist for scientific, analytical thinking.

Tuulia Hyötyläinen

Age: 42
Position: Team Manager and Principal Scientist at VTT Technical Research Centre of Finland, near Helsinki, Finland.
Background: MSc in chemistry and PhD in analytical chemistry at University of Helsinki. After that I continued at the University of Helsinki in various research positions. I moved to VTT in 2009, where I am currently leading a metabolomics laboratory.
What makes me tick: In research, motivating and challenging work, and nice coworkers. There are always problems to solve in experimental work, and I like that.
Outside of science, I… enjoy spending time with my family, particularly with my very active kids, cooking and reading. I also enjoy painting.
My alternative career would be… in some more artistic field, perhaps a designer or an architect.

Kendall Baker

Age: 23
Position: First year PhD student at University of East Anglia (UEA), Norwich, UK.
Background: BSc in biochemistry from UEA. I then spent one year as a medical laboratory assistant before returning to study for a PhD.
What makes me tick: I thrive in an environment where I have goals and can gain a sense of personal achievement.
Outside of science, I… like to get away for weekends. I play volleyball with a local club.
My alternative career would be… in a different analytical role, working with numbers, perhaps in the financial or insurance industries.

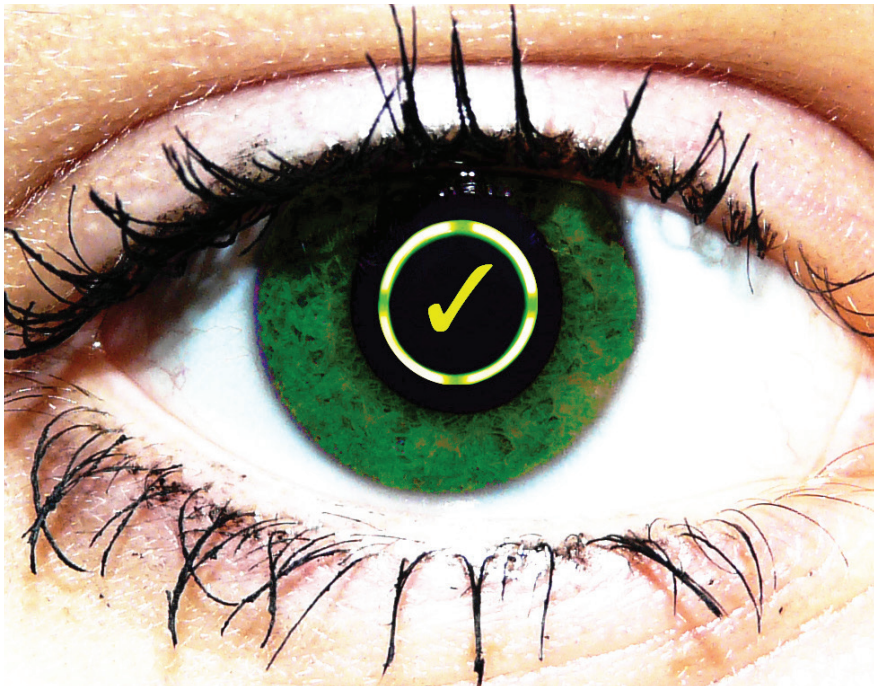
Business

Economic drivers
Emerging trends
Business strategies

Eye on the Prize

The analytics required to prepare a Chemistry, Manufacturing, and Controls (CMC) file for a new bio-pharmaceutical takes expertise, organization, time and money. Here's one company's story of success.

By Jean-Luc Jonniaux



The story of Jetrea started back in 2000. At ThromboGenics, the proteolytic region of human plasmin was being investigated for its ability to dissolve blood clots during thrombosis (hence the company name), in collaboration with Desiré Collen at the Katholieke Universiteit Leuven, Belgium. A phase I clinical trial was initiated in 2002 using ocriplasmin, a genetically engineered version of the proteolytic region of human plasmin.

Then, physicians discovered that ocriplasmin can cleave the proteins that cause vitreous macular adhesion (VMA; see 'Seeing is Believing' on page 48). A phase II clinical trial was designed in 2004 to define the therapeutic window for the VMA treatment. When the decision was made to undertake a phase III clinical trial, ThromboGenics needed to scale up the manufacture of ocriplasmin drug substance (DS) and to invest in validation, in order to comply with good manufacturing practice (GMP). To achieve this, in 2007, manufacture was transferred to a new contract manufacturing organization (CMO) with larger capacity. The process was designed and characterised between 2007 and 2009, a period that included three production runs to validate commercial-scale production. This material was used for the phase III trial, which was completed by the end of 2009. The trial results were encouraging, and the decision was made to submit a biological license application (BLA) to the US Food and Drug Administration (FDA) and a marketing authorisation application (MAA) to the European Medicines Agency (EMA). The analytical data in these submission dossiers has a very strong bearing on the positive outcome of the process.

Analytics for Approval

Ocriplasmin is a small protein (27kDa) with several disulphide bridges. The challenge it presents is that it has autoproteolytic activity. Controlling the levels of product-related variants throughout the manufacturing process is essential.

Table 1 summarizes the properties that we measured, using validated methods, throughout the ocriplasmin production process. The critical raw material in recombinant protein production is

the genetically manipulated cells themselves. Analytical requirements for these are well documented in the quality guidelines Q5 and Q6 from the International Conference on Harmonization of Technical Requirements for Registration of Pharmaceuticals for Human Use (ICH; see www.ich.org/products/guidelines/). ICH is a useful organization that brings together the regulatory authorities and pharmaceutical industry of Europe, Japan and the US to discuss scientific and technical aspects of drug registration.

Briefly, we performed routine monitoring by in-process sample testing (Table 2). The production process consists of more than 10 steps and the submission dossier demonstrated the consistency of each step using validated purity and content methods. The absence of contaminants like endotoxin or host cell protein was also assessed by several validated methods. The quality attributes of the drug substance (DS) and drug product (DP) were verified with validated methods comparable to those for monoclonal antibodies, as laid out in the European Pharmacopoeia 5.2.

We also defined the stability of drug substance (DS) and drug product (DP) in the scaled-up product batches, demonstrated the photostabililty of these batches and compared them to the batches that had been generated for the clinical trials, which had been produced on a much smaller scale than those for commercial process. Additional characterization of ocriplasmin included solving the 1D, 2D and 3D structures, measuring the physico-chemical properties and assessing potency to hydrolyse physiological substrates.

All existing and potential product-related variants had to be prepared and

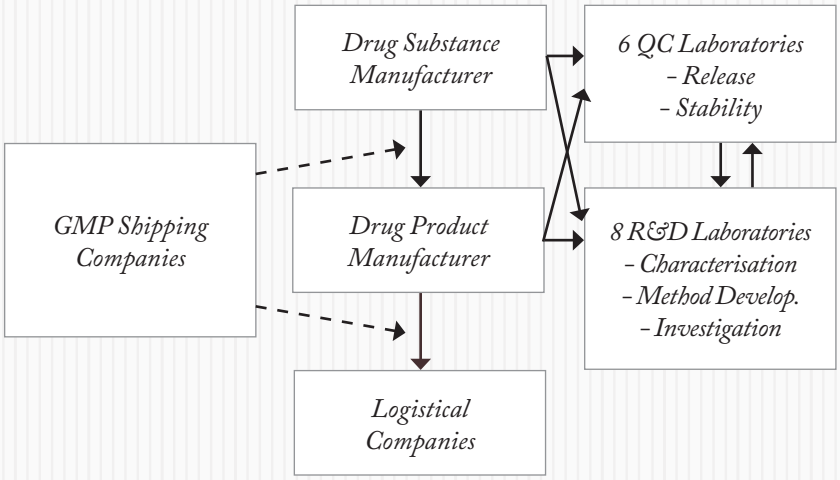
Table 1: Studies Required for the BLA/MAA Quality Dossier

Studies	*In Process*	*DS/DP*	*Ref. Standard*
Process Design Space	(X)	(X)	
Characterisation	(X)	X	X
Release	X	X	X
Stability	X	X	
Photostability		X	
Comparability clinical batch – commercial batch	X	X	
Extractable / leachable		X	
Specifications	X	X	X

Table 2: Properties Controlled During Manufacture of Jetrea

Process	*Purity*	*Potency*	*Content*	*General properties*	*Identity*	*Contaminants + Impurities*
Raw Materials					X	X
Cell	X	X	X		X	X
DS Process step	X		X			X
DS	X	X	X	X	X	X
DP Process step	X		X			
DP	X	X	X	X	X	X

Fig 1. Interactions Between Internal Staff and External Companies to Meet Analytical Requirements.



Seeing is Believing

Jetrea is a Non-Surgical Treatment of Vitreo-Macular Adhesion.

As people age, anatomical changes to the eye are common. One common ocular change is posterior vitreous detachment (PVD), in which the jelly-like part of the back of the eye, known as the vitreous, separates from the retina, which is a vital part of the eye that converts light into images by sending signals to the brain. Uniform detachments don't disturb the vision but if the vitreous remains attached to a small area of the retina it can create a hole. When this happens in the retinal region specialized for sharp color vision, the macula, the vision of aged people is distorted with a decrease of visual acuity, leading possibly to blindness. This pathology is known as symptomatic vitreous macular adhesion (VMA).

tested. These were either isolated from the process, or generated by accelerated degradation or de novo production using mutated genes. The variants were identified by mass spectroscopy (LC-MS) and their potency measured, in order to classify them as product-related substances or product-related impurities. In addition, the potential for ocriplasmin aggregation was investigated using several orthogonal methods. This is because recombinant protein aggregation is a known concern because of the immunological reactions they can induce, even though Jetrea is injected into the eye vitreous, where less immunological problems are anticipated in healthy individuals.

Such characterization data and the validated methods used to generate them are key components of the submission dossiers. They demonstrate an extended knowledge of process and properties pertaining to the molecule of interest.

ThromboGenics submitted the Jetrea dossiers in September 2011 to EMA and in April 2012 to FDA. Previous meetings with both agencies meant that we had aligned submission with the expectations of the agencies. However, as always when a drug is about to be commercialized, questions arose. The first set of EMA questions (called Day 120 Questions) were communicated in February 2012, and from the FDA in May 2012, and further sets of questions were received over the following four months. Concurrently, the FDA conducted pre-approval inspections at the CMOs contracted by ThromboGenics.

Finally, in October 2012, the FDA approved Jetrea for the US market. Some post-marketing commitments were made at this time. The very next day, EMA sent their second round of questions (Day 180 Questions), to which the company responded. The European Committee for Medicinal Products for Human Use (CHMP), which is responsible for preparing the Agency's opinions, then communicated a positive opinion on Jetrea. That was in January 2013; a decision from the European Commission is expected in March or April, 2013. In all, more than 100 CMC-related questions were handled during the assessment processes, most of them requiring additional analytical data.

Organizing for Success

We ascribe the success of the Jetrea submission to the positive clinical trial results, of course, but also to our strategy for fulfilling the expectations of FDA and EMA. To ensure maximal flexibility in responding to questions from the authorities and to provide validated methods in a timely manner, several QC and R&D laboratories were contracted to provide manufacturing and analytical services (see Figure 1). At any point in the process, work planned for one CMO could be transferred to another one to avoid delay.

This complex structure gave us the capability of delivering dossiers and responses in a timely fashion. Our rapid, coordinated approach was led by a small team that was solely dedicated to this purpose. The team members combined knowledge of, and experience in, regulatory, quality assurance, QC laboratory, GMP production, logistics and fundamental sciences. The group conducted short, efficient meetings with other parties that got right to the point. It had financial and operational freedom inside the company, and was allowed to take decisions and actions in less than a couple of hours. The ThromboGenics team was located in physical proximity to each other and had one common goal.

This type of business model is pivotal for a company the size of ThromboGenics. It is the only way to compile comprehensive BLA and MAA dossiers with the necessary complex, high quality information that is required to respond to the 100-plus CMC-related questions with speed and accuracy and, ultimately, to gain approval for a new therapy in the United States and Europe in parallel processes.

Jean-Luc Jonniaux is CMC senior manager at ThromboGenics nv, Leuven, Belgium.

Profession

Leadership
Talent Development
Career Planning

Coaching as a Leadership Tool

Increase your influence, and the efficiency and morale of your staff, by listening actively and asking probing, powerful questions.

By Janice Manzi Sabatine

Being highly skilled and trained in a technical discipline are key attributes of successful analytical scientists. However, they do not automatically make you a strong and effective leader. To achieve that, you must help to create an environment that empowers employees to think independently and creatively. Traditional, proscriptive management styles do not achieve this. One approach that does, and that will help make you an influential leader, is coaching.

What is coaching?

Coaching explores the strengths and goals of your staff, stimulates creative thinking in them, and addresses their behaviors and attitudes. As a coach, you act as a conduit to elicit greatness and empower the staff that you manage.

Coaching as a profession has gained attention in the business world for its impact on workplace satisfaction, goal attainment, and increased productivity. Professional, accredited coaches undergo unique academic and practicum training, engage in continuing education, and follow an established code of conduct. However, you do not need to become an accredited professional coach to use some basic coaching skills to improve your effectiveness as a leader.

The first thing to remember is that coaching is distinct from mentoring. As a mentor, you might engage in some of the following activities:

- Offer advice about career activities and advancement
- Share stories about your own experiences or knowledge

- Provide networking opportunities
- Make introductions to key leaders
- Suggest professional development programs

All of these activities can be vitally important to a more junior employee's success and advancement. There are times, however, when a coaching approach is needed. When acting in a coaching capacity, you must:

- Refrain from offering advice
- Assume your employee can find the answer
- Keep your focus on the employee; don't talk about yourself
- Remain curious and suspend judgment
- Focus on possibilities instead of problems
- Stay focused on the person, rather than on the issue
- Stay positive and take time to celebrate successes

When to coach?

Before delving into specific coaching behaviors in more detail, it's important to point out that not all situations at work are coaching moments; for example, when an employee does not have the skills needed for the position he or she holds, or when the behaviors are questionable from a legal or ethical perspective.

Ideal coaching moments may present themselves when an employee approaches you with a question or problem. Here, you should avoid automatically answering the question or solving the problem, which potentially adds something to your own to-do list. Employing key coaching skills can often lighten your workload as well as increase the motivation of your employees, and it results in creative and innovative solutions you may not have come up with yourself.

Once you've established that your employees have the skills to do their jobs, it is important to approach them with the fundamental mindset that they are capable and wise. Your job will be to probe their thinking in a way that stimulates their creativity and sparks their motivation.

The brain is a social organ

Understanding a bit about how the brain works will help you to understand why certain coaching approaches are so effective. The brain is a social organ: its physiological and neurological functions are directly influenced by social interaction. Brain scans indicate that the areas of the brain associated with physical pain are the same as those associated with social rejection, resulting from arousal of the sympathetic nervous system. The result is decreased cognitive ability and perceptual openness, decreased immune functioning, and feelings of nervousness and anxiety. In contrast, when a reward response is prompted, the parasympathetic system is aroused, a state in which cognition is optimal and new neural tissue can be created, which allows for new learning and an openess to new ideas. As leaders, how you approach and interact with your employees can have dramatic consequences. When your actions prompt a reward response rather than a threat response, your employees become more effective, open, and creative. The coaching behaviors described here align with those behaviors known to elicit a reward response.

Key coaching behaviors

So, what are these coaching behaviors? While trained professional coaches must demonstrate proficiency in multiple competencies, you can focus on these key elements: listening actively and asking probing, powerful questions.

To be an influential leader, you need to know what your employees are thinking, which means you have to be a good listener. Communicating your trust in them and sparking their motivation by probing their thinking with powerful questions will create an environment in which they are eager to perform. This seems simple enough, yet it can require a bit of mindful practice. More importantly, it may require a change in your attitudes.

Listening actively

Good listening is related to interpersonal influence. So, if you want to be influential, you need to be a good listener. Listening actively is fundamentally a state of mind. To be effective you must have a sincere interest in the other person. It requires that you try to get inside the head of the speaker, to try to see things from his or her point of view. More importantly, you must convey to the speaker that you are seeing things through this point of view.

Listening actively means you are listening for the complete message. Their message includes both content and feelings or attitudes. It is crucial to understand not only what they are saying but also how they feel about it. In fact, sometimes the content is much less important than the attitude that accompanies it. To hear the complete message, you must also pay attention to tone and inflection of voice as well as non-verbal cues such as facial expressions, posture, hand and eye movements, and pace of breathing.

To listen for this complete message means you must give the other person your undivided attention. Put everything down, turn toward the

Quick Coaching Tips

Features of Active Listening

- Listen for the complete message
- Reserve judgment
- Reflect and clarify
- Be patient

Examples of Powerful Questions

- How important is this for you?
- What is your thinking on this matter?
- What do you want the outcome to be?
- How will you make that happen?
- What is possible?
- What is the real issue?
- What else are you thinking?
- What matters most to you?
- What do you want me to know?
- What input do you want from me?

speaker, and make eye contact. Giving someone your full attention conveys the powerful message that you are interested in what they have to say and that you respect their thoughts. In fact, demonstrating this message through your behavior is much more powerful than conveying it in words.

Listening actively also requires that you reserve judgment, either favorable or critical, so that the speaker can communicate freely. You can't get the total message if the speaker shuts down because he or she feels judged. Being non-judgmental and open arouses the reward response rather than the threat response. Make sure you understand by reflecting back or by asking clarifying questions. These behaviors ensure that you not only get the message right but also demonstrate your sincere interest in what the speaker is saying. Further, it is crucial that you are patient. Provide an unhurried environment for the speaker to think clearly withthe opportunity to elaborate.

Asking probing, powerful questions

You can empower your employees to reach their goals and be high performers, not because you have all the right answers, but because you have the right questions. Good questions help your employees access their personal strengths and creativity. Asking questions rather than supplying answers or dictating actions implies a level of confidence and trust that precludes the threat response in your employees. Instead, the parts of the brain associated with openness and creativity are stimulated.

What do probing, powerful questions look like? Where do they come from? Let's answer the last question first. Good questions come from good listening. When you start from the basic attitude that your employees are capable and wise and assume an attitude of openness and curiosity when you listen attentively to them, the questions will present themselves. It may take some practice, but it will eventually become second nature.

As for structure, powerful questions are those that are not strictly for gathering facts and information. Rather, they are geared at helping others to access personal strengths, reflect on beliefs and attitudes, and access their own knowledge, wisdom, and creativity. Although there is no strict formula for powerful questions, they generally share some of the following characteristics:

- Open-ended
- Short and simple
- Focus on possibilities instead of problems
- Incorporate the other person's words
- Do not imply a correct answer
- Do not include a conclusion or suggestion

See 'Quick Coaching Tips' for some examples of simple yet powerful questions. Although you will naturally develop your own questions based on the specifics of your situation, the people you lead, and your preferred style, these examples may be helpful as you practice a coaching approach.

The benefits of a coaching approach

My clients who have adopted a coaching approach tell me about the positive impact it has on their effectiveness as leaders. They have found renewed passion for their jobs and have become more influential leaders in their companies and organizations. Many report that they feel less stressed at work. One client was amazed at her ability to leave meetings without other people's responsibilities added to her task list. Another client who had recently been promoted from a strictly technical position to one of leading a group was considering giving up the position. As we worked together and he learned that he did not have to have all the answers and began to listen carefully, pose the right questions, and handle conflict effectively, he truly embraced the new leadership role and was given rave reviews by the company executives.

Increase your influence as a leader. Create new habits. Learn to coach!

Janice Manzi Sabatine is President, Avanti Strategies LLC, which provides executive coaching for physicians and scientists.

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Saccharide and Polysaccharide Analysis

Polysaccharides are very important in nature, occurring in food (starches in rice, wheat etc.) and plants (cellulose). Some polysaccharides are also produced commercially e.g. Dextrans, which are manufactured through the fermentation of sugar solutions. These are higher molar mass polysaccharides.

Dextrans are used in clinical and technical applications, where molecular weight is critical in determining the properties of the final product. Accurate determination of the molecular weight distribution is vital.

On the other hand, low molar mass saccharides are also very common and can be found in food, such as fruits, honey and sweets. Examples for low molar mass sugars are mono- (glucose, fructose), di- (lactose, isomaltose, trehalose) and trisaccharides (maltotriose, isomaltotriose). The separation and identification of low molar mass polysaccharides is a challenge as the compounds have the same chemical formula and only small differences in structure, e.g disaccharides maltose, isomaltose, gentiobiose cellobiose and trehalose $C_{12}H_{22}O_{11}$.

Experimental Conditions

Eluent:	$NaNO_3$ 0.1M
Columns:	PSS SUPREMA 5 µm 3 x 100Å (8 x 300 mm) + precolumn
Data acquisition:	PSS WinGPC UniChrom
Detectors:	SECcurity GPC 1260 RI
Flow-rate:	0.25 ml/min
Concentration:	4 g/l
Injection volume:	5 µl
Sample Fig 1:	Dextran T1, Glucose
Sample Fig 2:	Disaccharides

Results & Discussion

A high resolution and therefore a good separation on the column is necessary for precise analysis. This is particularly important when new analytical LC coupling methods like GPC/SEC-ESI-MS are used, as the MS detector requires the columns to have a much higher resolution power within an overall smaller column volume.

The new SUPREMA column, with a reduced particle size of 5µm, offers a significant improvement in performance compared to traditional 10µm materials and provides outstanding additional resolution, especially in the low molecular weight area, which is a major consideration when analyzing oligomeric polysaccharides.

The analysis of dextran T1 shows the separation power when a combination of three SUPREMA 5µm 100Å columns is used. The oligomers in the low molecular weight are able to be resolved up to P10. A glucose separation is overlaid, as a reference.

The analysis of different disaccharides shows the ability to separate compounds with the same chemical formula and with only small differences in structure and hence size in solution.

PSS SUPREMA 5 µm columns can be used for numerous neutral and anionic aqueous applications in the molecular weight area between 100 Da to around 5 million Da. The columns are available in analytical (ID: 8mm) and micro (ID: 4.6mm) dimensions with different porosities. Linear or mixed columns are also available.

For more information, please see the full application note at: theanalyticalscientist.com/issues/0213/702

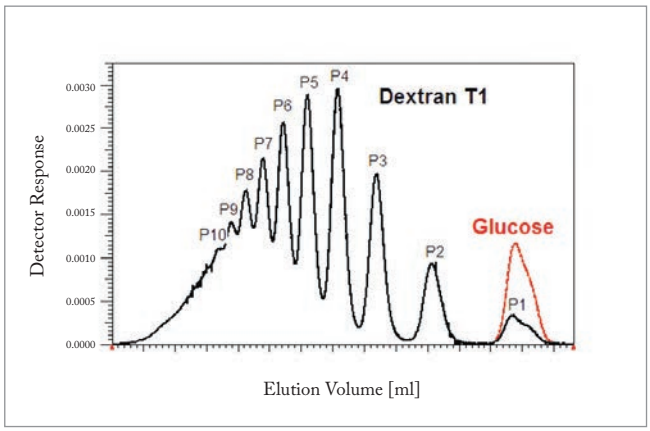

Fig 1: Overlay of elugrams of a glucose (red curve) with a low molar mass Dextran T1 (black curve)

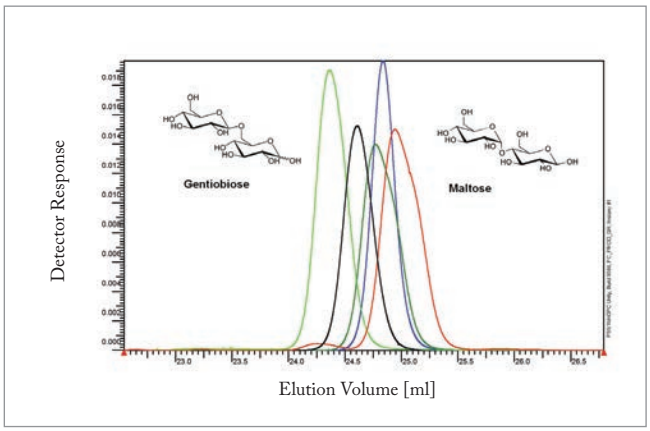

Fig 2: Overlay of elugrams of isomaltose (black), maltose (red), gentiobiose (green), cellobiose (dark green) and trehalose (blue).

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Bringing The Market to Science

Sitting Down With **Elizabeth Iorns**, CEO of Science Exchange.

Describe your company in the length of a tweet.

Science Exchange provides efficient access to the world's scientific expertise via an online marketplace of 1000+ expert providers

How does it work?

We are creating a big online network of scientific experts (providers) who can conduct experiments on a fee-per-service basis. The network comprises about 60 percent academic labs, 30 percent commercial and 10 percent government, and each one is verified by the Science Exchange team.

The website was designed very much with e-commerce in mind; we have advisors who have been part of successful online marketplaces like eBay, Open Table and Airbnb. It's easy to use, and that has resonated with the community.

What kinds of projects are being arranged on Science Exchange?

The most popular categories are next-generation sequencing, microarray analysis and mass spectrometry; other areas of interest to analytical science include HPLC and isotope analysis. The scope is wide, but also includes specialized techniques where expertise is rare. We want to be thoroughly inclusive: the more people that get involved, the better.

Where did the idea come from?

It came out of my own experiences in breast cancer biology research. All of my projects involved collaborations, either with core facilities at the university or external commercial service providers. But it was inefficient. I didn't know who the best partners were, and I didn't have information on pricing or reputation. What I needed was an easy-to-use website that gave a range of providers for a particular experiment, and feedback from previous users. So, I decided to create it.

"It turns out that the tools that we thought defined quality – publication in a high-impact journal, mumerous citations, even multiple papers showing a similar result – do not identify reliable data."

At that point you changed your career direction? Big change!

Yes. I got accepted into Y Combinator, a startup accelerator program in California. This helped with the transition, taking me through all the steps to start a business. I have also continued to do scientific experiments using the Science Exchange network and have published papers on the work, so I have not cut all ties to the lab.

We were incorporated in May 2011. We now have six employees and are just about to expand. More than 400 of the top 600 US universities have listed providers on our site, so we're strong in the US academic research space.

I've really enjoyed growing the business. It's in many ways similar to running a lab. Both have a management component, an experimental component and a money-raising component.

How have research funders reacted?

Funding agencies and foundations, like researchers themselves, have been very supportive. They want to ensure that their funding is used as efficiently as possible and see how Science Exchange can help. Let me give a couple of examples: one is the engagement of expensive equipment that would otherwise be idle. Another is where techniques that take a long time to perfect are being learned for one-off experiments; it is much more efficient to outsource to experts.

An additional string to the Science Exchange bow is in verifying scientific reproducibility. How did that come about?

I noticed that there had been several interesting use cases at Science Exchange in which venture capital companies were using experts to validate experiments independently, prior to making their investment. From there, I began to figure out an initiative to allow academic researchers to validate their research. The result is The Reproducibility Initiative, a partnership with a publisher (Public Library of Science), a data storage company (Figshare) and a literature analytics company (Mendeley) that provides a stamp of reproducibility for research studies, methods and reagents.

The literature is a vast body of knowledge but at the moment we are unsure what parts of it are really true. It turns out that the tools that we thought defined quality – publication in a high-impact journal, numerous citations, even multiple papers showing a similar result – do not identify reliable data. The Reproducibility Initiative is a positive incentive system for validating research.

Where do you think research is headed, what are the big trends?

In the past five years, the two key breakthroughs have been open access and data deposition; in the next, the focus will be on quality and efficiency. Science Exchange is well positioned to contribute to both these things. In five years' time, researchers will be producing high-quality research that can be independently reproduced, and they will be recognized and rewarded for it.

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