JULY 2018 # 66

the Analytical Scientist™

Curiosity Uncovered

Why the desire to know more lies at the heart of all science.

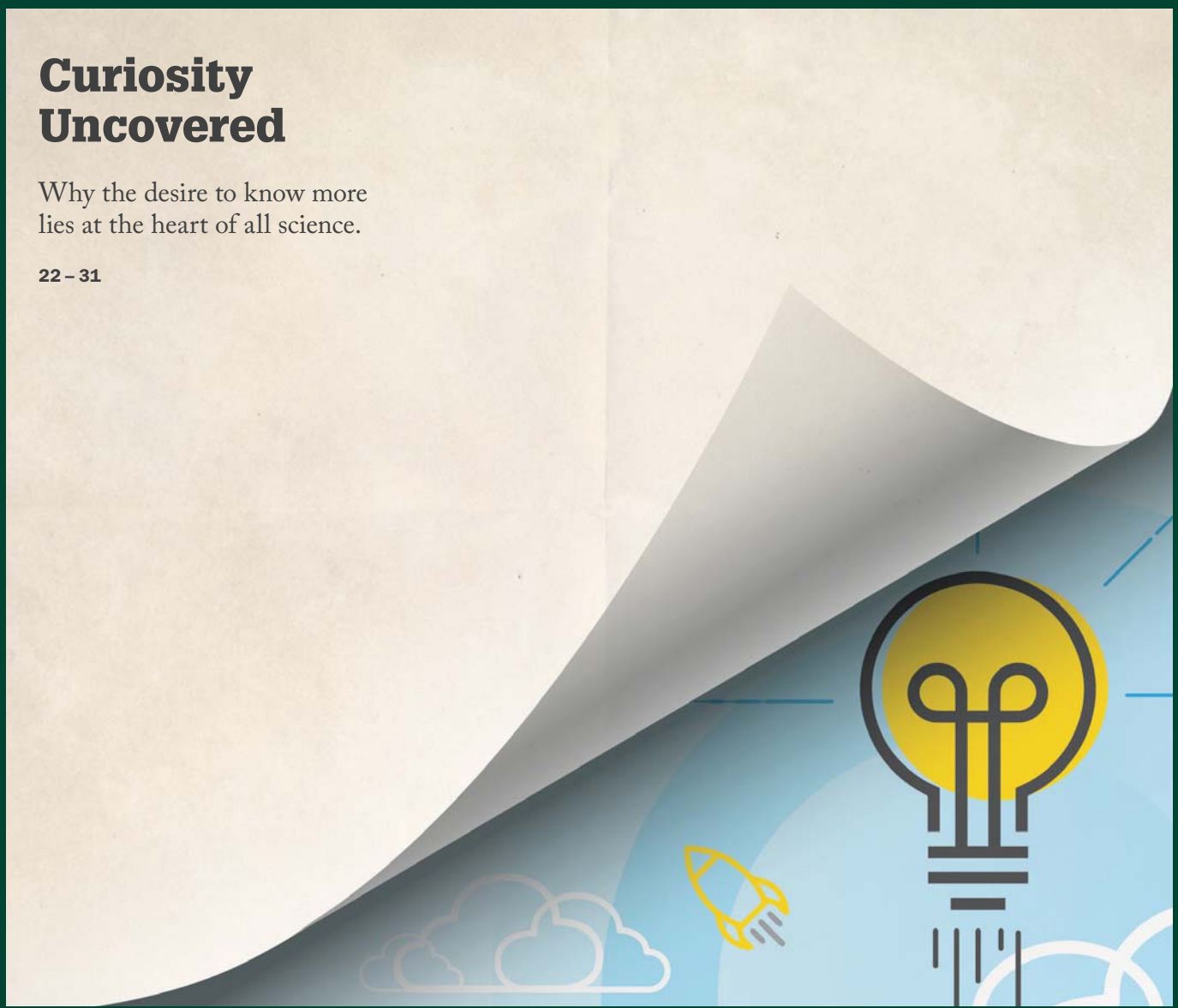

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Image of the Month

GC on the Fly

Is it a bird? Is it a plane? No, it's a flying gas microchromatograph. Scientists at Samar University, Russia, have developed a device capable of performing analysis within three minutes, while in the air. With its ability to measure concentrations of substances at a two km radius from the source and at an altitude of up to 1,000 meters, it could prove a valuable alternative to lab-based equipment in the environmental analysis field – especially in sampling situations that could be dangerous for humans.

Watch the video online: https://bit.ly/2Ieg8bb

Would you like your photo featured in Image of the Month? Send it to charlotte.barker@texerepublishing.com

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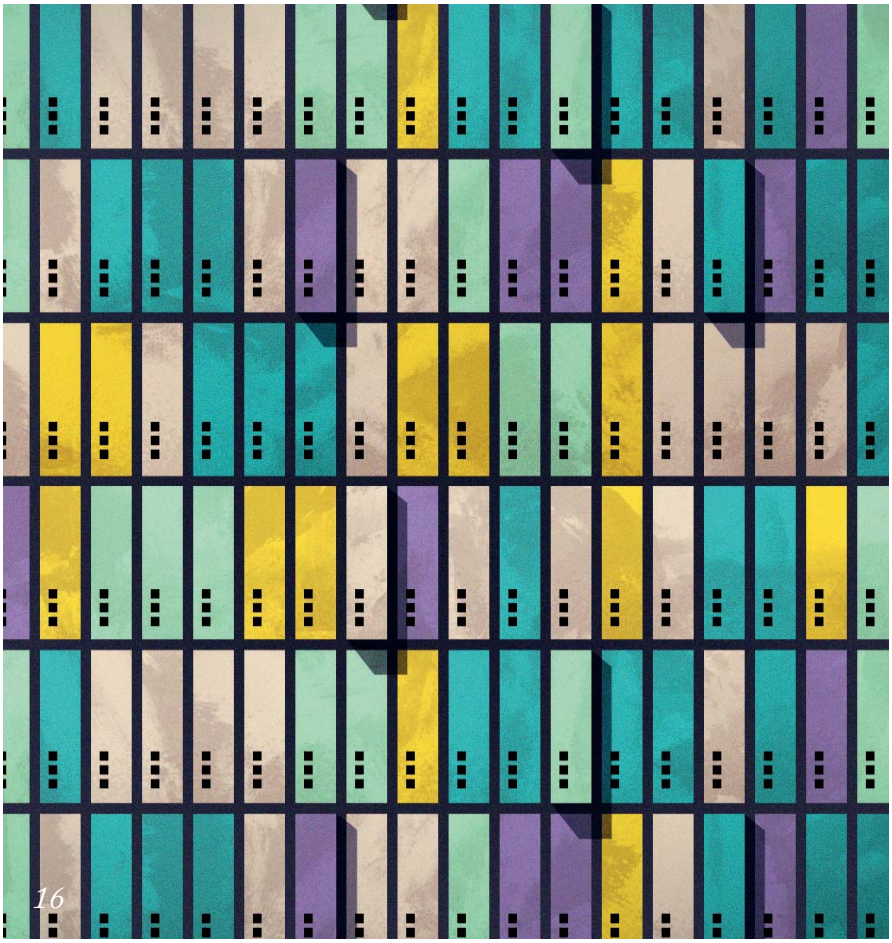

On The Cover

Don't keep your curiosity under wraps – turn to page 22 to see the full image!

Upfront

the Analytical Scientist

ISSUE 66 - JULY 2018

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Celebrating Curiosity

Curiosity is a fundamental trait of all scientists – but it must be nurtured.

Do ants sleep? How do squirrels find their acorns again? Why can't I eat two ice creams?

All excellent questions – none of which I could answer to the satisfaction of my friend's preschooler. In four-year-olds, at least, curiosity knows no bounds.

Like small children, good scientists never get tired of asking "Why?" And analytical science feeds further curiosity – not only by providing answers to burning questions, but also by opening up fresh lines of enquiry. This month's cover feature explores the topic of curiosity through interviews with four analytical scientists, who share its importance in their work, and in the wider world (page 22). The common theme: we are all born with innate curiosity about the world, but society often does a poor job of nurturing those feelings into adulthood.

There are challenges to maintaining our natural curiosity in the technology-rich 21st century. But is constant, easy access to information and entertainment sapping our curiosity? If we always have an answer to hand (albeit a potentially superficial one), are we tricked into believing we already have knowledge, when we have only trivia? Will children of the Internet age lose the ability or desire to think for themselves?

Personally, I don't think there is much danger of curiosity being extinguished from the human race. After all, our questioning nature is a big part of what makes us the world's most prolific (and dangerous) animals. From prehistoric humans making fire to Jonas Salk being the guinea pig for his polio vaccine to the discovery of CRISPR gene editing, we are a species of questioners and innovators, meddlers and thinkers.

Accepting the Biemann Award at ASMS in June, Benjamin Garcia noted, "It takes a society to raise a scientist." And the scientists in our feature believe we can do more to encourage creativity and curiosity in students, from preschool through to university. "When we teach younger students about science, we are too apt to give them a recipe," says Rick Yost.

How do you keep curiosity alive – in yourself and in your students? I'd love to hear your thoughts – charlotte.barker@texerepublishing.com.

P.s. Judging by the deluge of nominations we are receiving for this year's Top 40 Under 40, there is no shortage of innovative young researchers in analytical science. But feel free to make the judging process even harder at: tas.txp.to/powerlist2018.

Charlotte Barker
Editor

Upfront

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

We welcome information on interesting collaborations or research that has really caught your eye, in a good or bad way. Email: charlotte.barker @texerepublishing.com

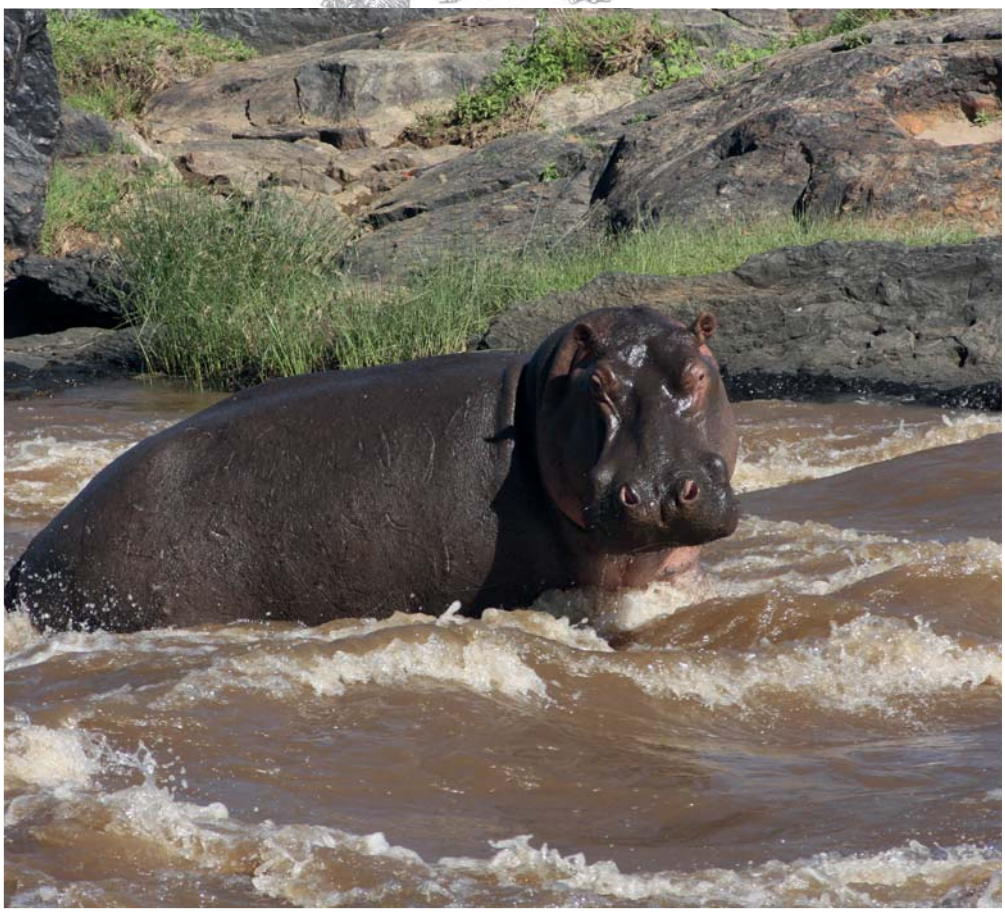
Hippo No-No!

"River horse" waste spells death by defecation for Kenyan fish

Chris Dutton and Amanda Subalusky first started working on the Mara River in 2008, participating in an environmental flow assessment to determine the amount of water that needs to stay in the river to keep the ecosystem functioning. As part of the project, they spent a year doing intensive water quality and macroinvertebrate sampling throughout the basin.

They first realized that something weird was going on in the river when they installed a water quality sonde that measured dissolved oxygen to look at the fluxes of sediment. "We noticed that there were some extreme drops in dissolved oxygen during some flood events and that, sometimes, there would be a massive fish-kill event. We began to wonder if the resident population of hippos had something to do with it," says Dutton.

They began with a few small experiments to see what effect hippo waste has on oxygenated water, before scaling up to a large experimental stream array and ultimately to an entire ecosystem manipulation. "We ended up experimentally flushing a hippo pool to understand the mechanisms responsible… And with



the experimental evidence, coupled with the modeling and in-situ data, it became pretty clear that the flushing of hippo pools was responsible for the crashes in dissolved oxygen," he says.

A variety of methods were used to tease apart the detailed biogeochemistry of these events, says Dutton: "We measured BOD (biochemical oxygen demand) using short-term incubations in bottles and physicochemical parameters (dissolved oxygen, temperature, pH, ORP, conductivity, turbidity) in-situ using a large water quality sonde. We captured samples using automated loggers and analyzed them on a custom-built flow injection analyzer. We measured the stratification of hippo pools using a custom-built remote-controlled boat and a custom-built conductivity logger." They also used a spectrophotometer to measure the amount of hydrogen sulfide and ferrous iron in samples, and captured samples of methane, carbon dioxide and nitrous oxide using evacuated exetainers before analyzing those samples in the lab using GC. "It is really hard to do good science in the bush!" says Dutton.

They confirmed that in a natural river with a large population of resident hippos, the amount of fecal matter generated is enough to cause frequent crashes in dissolved oxygen during floods.

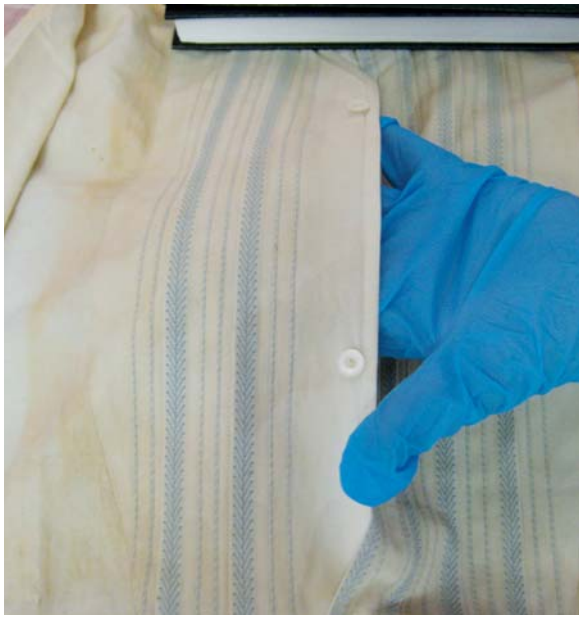
Hippos 1, Fish 0.

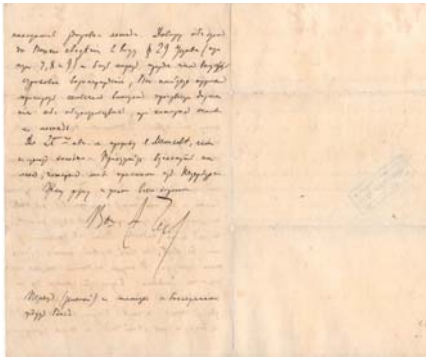
However, this doesn't mean we're up the proverbial creek without a paddle, says Dutton. "Increasing habitat heterogeneity due to these hypoxic episodes could increase biodiversity in the river system – as it results in spatial variation in physicochemical conditions that affect aquatic animal communities."

Next, to understand the detailed biogeochemical and microbial dynamics, the team will be taking a deep dive ("only figuratively!") into the hippo pools themselves to understand how they transition from aerobic to anaerobic states. But for now, it's a good job there's plenty more fish in the sea…

Reference

1. *CL Dutton et al., "Organic matter loading by hippopotami causes subsidy overload resulting in downstream hypoxia and fish kills", Nat Commun, 9, 1951–1960 (2018).*





TB on Your Collar

Metaproteomics study throws doubt on Chekhov's cause of death – and paves the way for future cultural heritage analysis

They say dead men tell no tales – but mass spectrometric analysis of their clothing might. Russian playwright Anton Chekhov is thought to have died of tuberculosis, and the shirt he died in – with a brown stain believed to be his blood – is now preserved in the State Literary and Memorial Museum-Reserve A.P. Chekhov, Melikhovo, Russia.

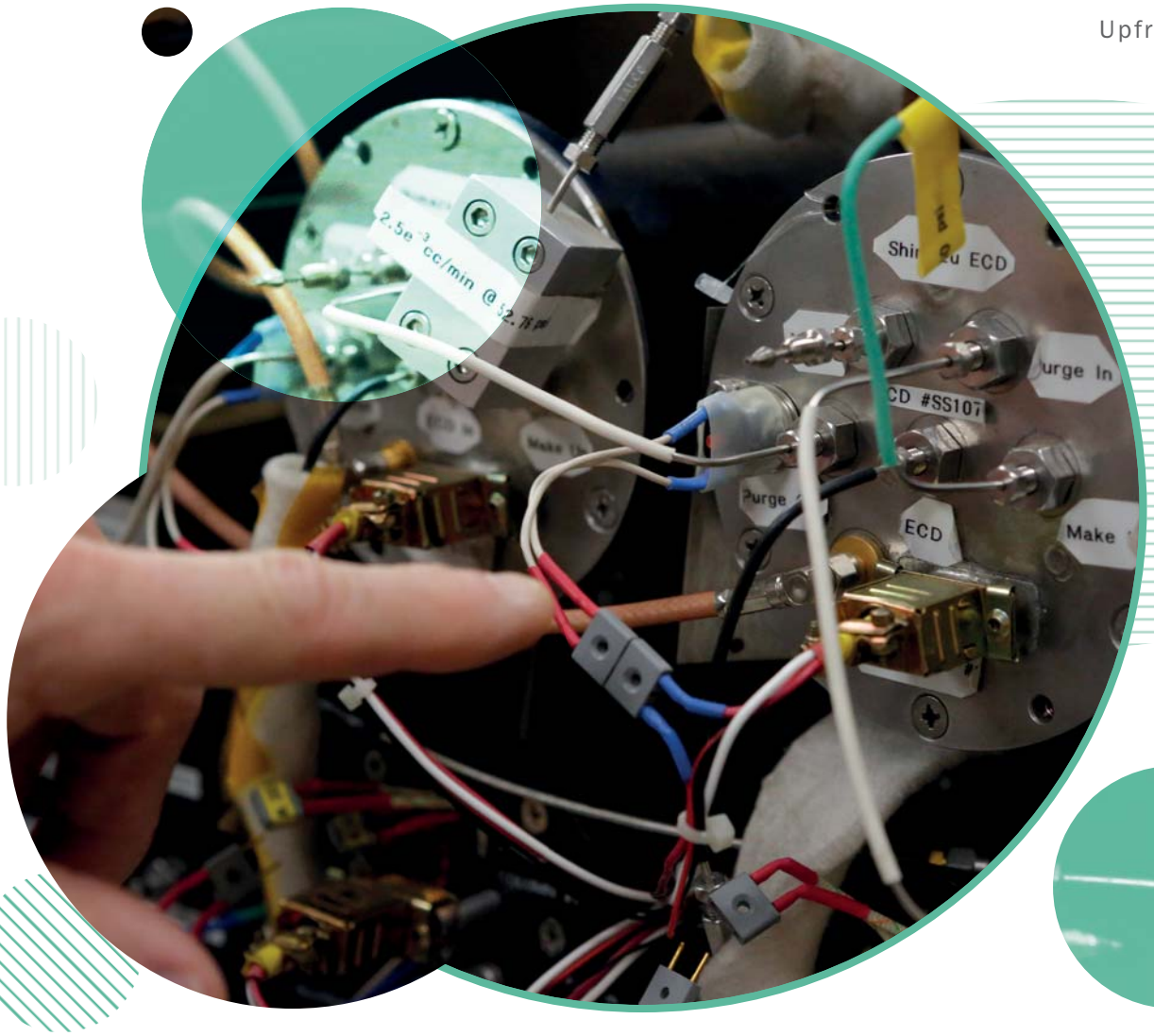
A multinational team from Italy, UK, Russia and Israel have now used an ethyl-vinyl acetate (EVA) disk to sample proteins from the suspected bloodstain, hoping it would provide evidence of his fatal illness. The disk is specially designed to extract sufficient material for analysis without damaging sensitive artifacts. After laying the disk on the stain for between 60 and 90 minutes, they analyzed extracted proteins by mass spectrometry before matching them against the SwissProt Bacteria database and the UniProt M. tuberculosis database.

Of the 108 human serum proteins detected, eight were found to be related to M. tuberculosis, supporting the previous theory. However, they also detected ITIH4, a protein associated with blood clot-induced strokes – meaning a hemorrhage could have been the cause of death.

The researchers now plan to further analyze the protein mixture for a more detailed reconstruction of Chekhov's health and demise. In time, they believe the EVA film – also used in their previous research on Mikhail Bulgakov (tas.txp.to/0417/bulgakov) – could become the technique of choice for analyzing cultural heritage artifacts.

Reference

1. *A D'Amato, "Anton Chekhov and Robert Koch cheek to cheek: A proteomic study", Proteomics, 18 [epub] (2018).*



CFC What Happens

Levels of ozone-depleting CFCs are on the rise – despite a global ban

In 1987, the Montreal Protocol called for an end to the use of chlorofluorocarbons (CFCs), including trichlorofluoromethane (CFC-11) – the second most ozone-depleting gas. With decreasing emissions, concentrations of CFC-11 were expected to fall rapidly from 2010 onwards.

However, scientists have discovered evidence of increased emissions after 2013; in fact, they were 25 percent higher between 2014 and 2016 than between 2002 and 2012. Stephen Montzka, a Research Chemist at the US National Oceanic and Atmospheric Administration and co-author of the paper (1), says: "It was and is the most unexpected observation I've made during my 27 years of making global-scale measurements. How can emissions of CFC-11 have increased, a decade after its production had been phased out for more than 10 years?"

The researchers used gas chromatography with electron capture detection and mass spectrometry with a 60m DB-5 1um column and cryogenic column cooling. "The inlet was a custom-built cryo-trapping device to allow for quantitative and artifact-free sampling of air, and standards were prepared at ambient mole fractions using gravimetric techniques," Montzka explains.

More specific measurements are needed to discover where the increased emissions are coming from, though analysis of polluted air over Hawaii – and factoring in wind speeds and direction – suggest "fairly definitively" that it is from East Asia.

Crucially, if emissions persist, Montzka believes that they could contribute directly to global warming – and prevent recovery of the ozone layer.

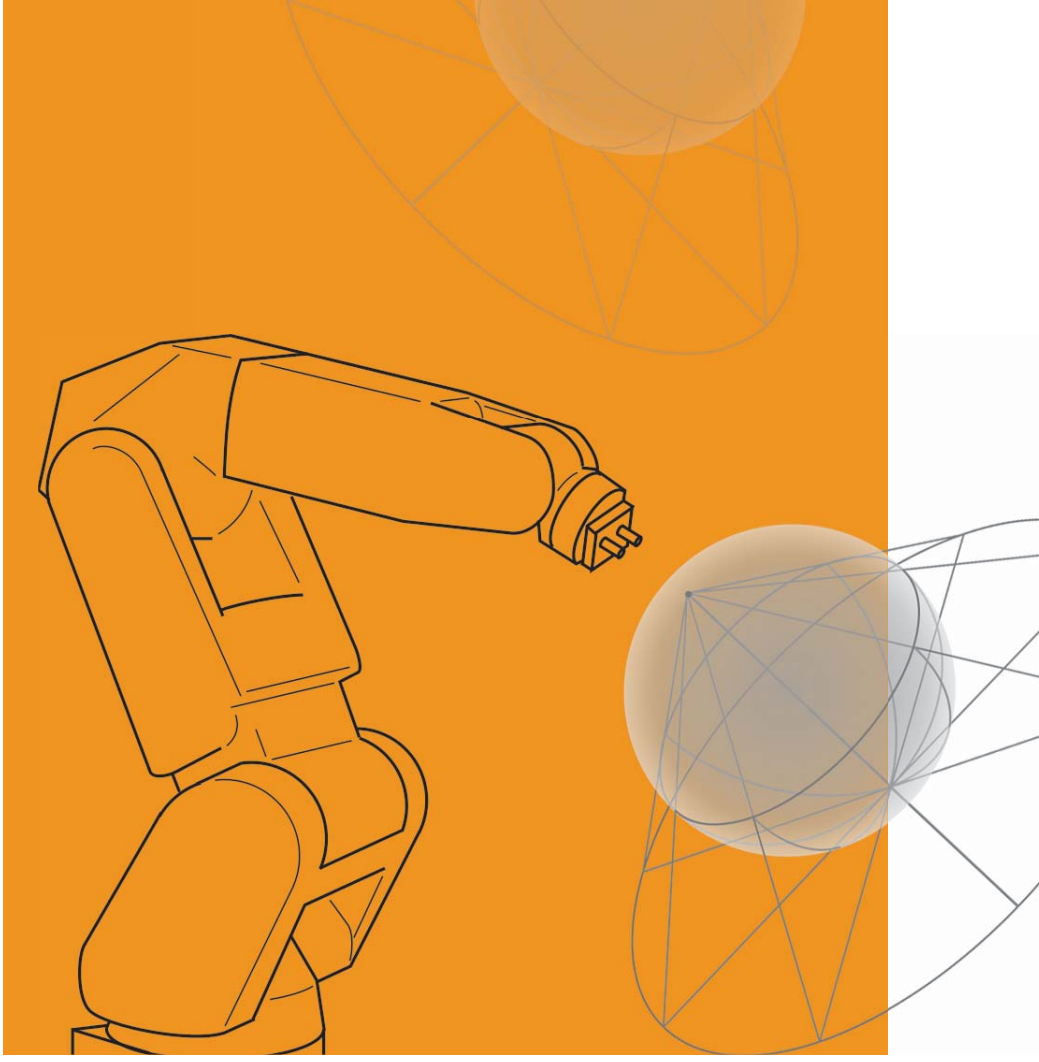
Reference

1. *SA Montzka et al., "An unexpected and persistent increase in global emissions of ozone-depleting CFC-11", Nature, 557, 413–429 (2018).*

The Future's RoSA

A robotic arm takes mass spec analysis of 3D objects to the next level

Chemical sampling of 3D objects has become increasingly important, particularly in forensics and drug screening. After a proof-of-concept study using a 3D camera and robotic arm to take samples for plasma ionization (1), Facundo Fernandez and his team at Georgia Tech wanted to improve on the analysis, by combining robotic surface analysis – or RoSA – with mass spectrometry. Fernandez tells us more.



Why combine robotics and mass spec in this way?

As mass spectrometers have grown more user-friendly and powerful, the bottleneck in the analytical pipeline has become the sampling process. I feel it's time to marry advances in automation and machine learning with mass spectrometry, opening new possibilities in analytics of complex systems. Electrospray ionization (ESI) and matrix-assisted laser desorption ionization (MALDI) can be seen as the first mass spectrometry revolution, and ambient methods, such as desorption electrospray ionization (DESI) and direct analysis in real time (DART), can be seen as the second – I foresee that the third revolution will involve the "rise of the robots!"

How does RoSA-MS work?

A 3D laser scanner mounted on a robotic arm scans the object, producing a 3D representation. The user then selects points to be sampled on the surface of this representation using custom-built software. The robotic arm moves sequentially through each one of these points, "touching" the surface with a sampling probe (a spring-mounted thin needle), then placing this needle into an open sampling port that washes away the material collected. The material is dissolved by the carrier solution, and directed to an ESI ion source, where it is ionized and then mass analyzed, giving the user a mass spectrum for each point. Because ESI is such a broadband ion source, many compounds can be studied in this way – and less polar compounds can be investigated by using a different ion source, such as a photoionization or chemical ionization.

What's the potential impact?

The sky's the limit! In the pharma industry, for example, it could detect substandard products in an assembly line by rapidly using the computer vision capabilities of the system to scan 3D objects (such as a tablet), and then probing its composition quickly without having to crush, dissolve, and analyze by HPLC. It could also be used to map tissue samples in 3D, or investigate the composition of small volumes of precious biofluids on non-planar surfaces.

Any plans for further advancement?

We are planning on arming the robots with lasers! We would like to develop a next-gen system that uses a laser ablation probe for sampling the surface, which should increase our spatial resolution and generate more detailed images. We would also like to investigate its clinical applications in the fields of high throughput diagnostics and metabolomics.

References

1. *RV Bennett et al,"Robotic plasma probe ambient ionization mass spectrometry imaging of non-planar surfaces", Analyst, 139, 2658-2662 (2014).*
2. *Anyin Li et al., "Robotic surface analysis mass spectrometry (RoSA-MS) of three-dimensional objects", Anal Chem, 20, 3981–3986 (2018).*

Poles Apart

Researchers develop a generic and potentially inexpensive method of separating chiral molecules using magnets

Chiral molecules have the potential to "flip" and exist as different enantiomers – non-superimposable mirror images of the original molecule with an identical chemical structure. Though these molecules look identical, their different "handedness" can have dramatic biological effects – as was made painfully clear by the thalidomide scandal. In the 1950s and 1960s, the drug was marketed to pregnant women to treat morning sickness, which its "right-handed" enantiomer did well. But the "left-handed" enantiomer caused thousands of babies worldwide to be born with malformed limbs.

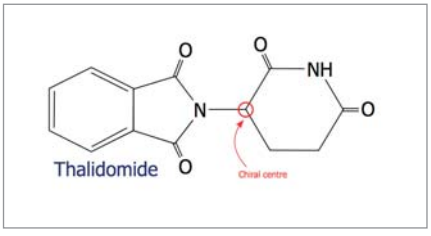
Today, the separation of chiral molecules is an expensive process, but an international team of researchers has developed a generic and cheaper method of separating chiral molecules, using magnets (1).

"We found that the interaction of chiral molecules with magnetic substrate is enantio-specific," says Ron Naaman, Professor in the Department of Chemical and Biological Physics, Weizmann Institute of Science, Israel, and study co-author. "One enantiomer interacts more strongly when the magnet is magnetized in one direction, while the other enantiomer interacts more strongly with the substrate when it is magnetized in the opposite direction."

Current methods to separate chiral molecules are specific to each molecule. "Where high performance liquid chromatography (HPLC) is used, columns must be refreshed once a certain amount of material is passed through them," says Naaman. "This is time consuming and expensive. In some cases, there are no good methods for separation."

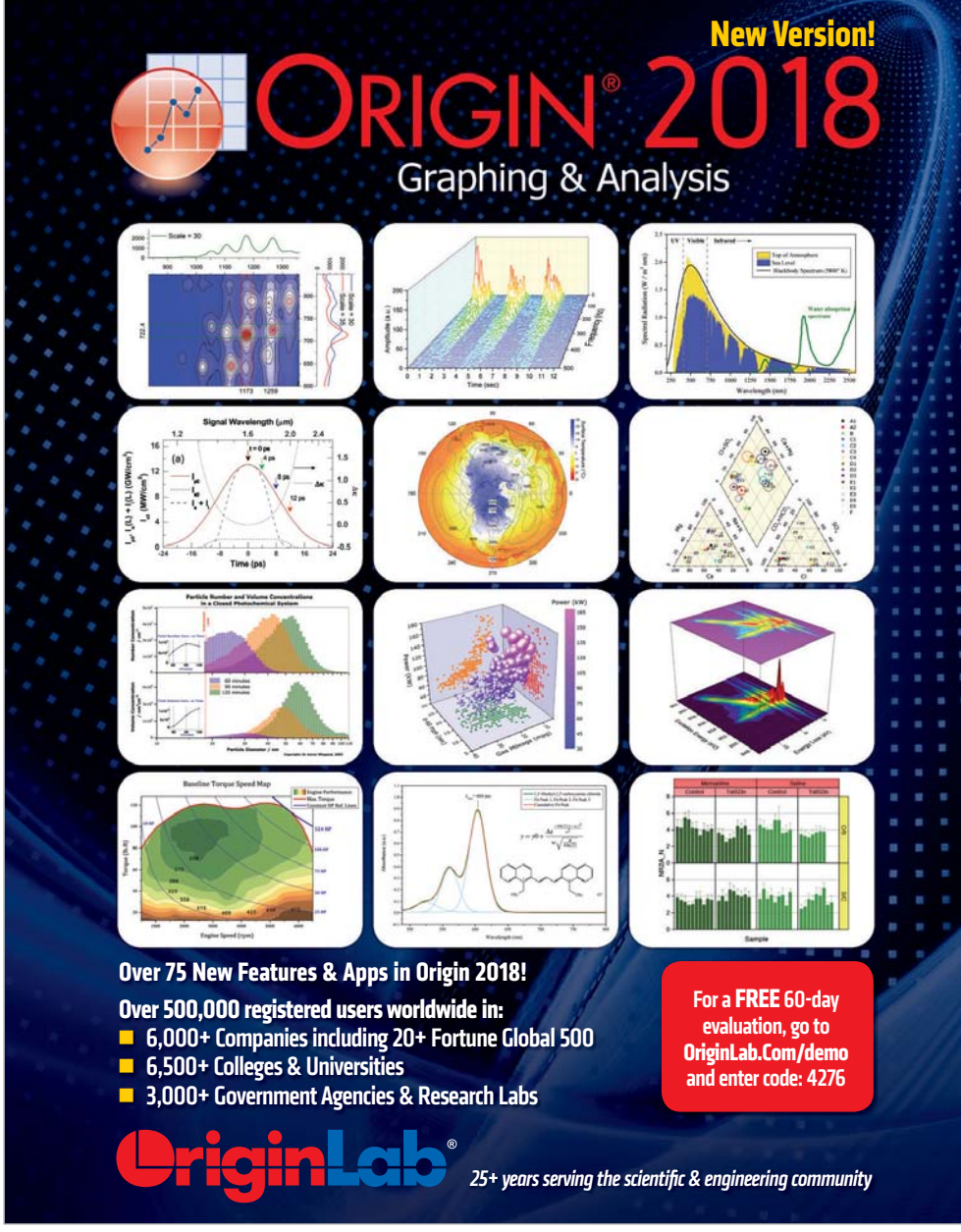
The new method is based on "chiral induced spin selectively," which Naaman, and his Hebrew University colleague, Yossi Paltiel, has been working on for the past decade. Electron spin has two directions, often called "up" and "down," and two electrons can only form a bond if they have opposite spins. If a substrate contains electrons, orientated with a uniform spin – as in magnetic material – then the strength of the interaction between the chiral molecule and the substrate will depend on the spin. Because electron spin orientations differs in chiral pairs, a perpendiculary magnetized substrate can be used to separate chiral pairs.

This method could allow the separation of chiral molecules from a mixture of molecules, either chiral or achiral – potentially eliminating the need for expensive and time-consuming HPLC.



Reference

1. *K Benerjee-Ghosh at al., "Separation of enantiomers by their enantiospecific interaction with achiral magnetic substrates", Science (2018). PMID: 29748324.*



Green Chemistry and Government Science

Business in brief: What's going on in analytical science?

Collaborations and acquisitions

- Waters Corporation and Hamilton Robotics have joined forces to launch STARWorks, an assay-ready automated sample preparation system.
- Waters has also worked with the National Mass Spectrometry Facility at Swansea University to develop the ACQUITY QDa Practical MS Education Package, which aims to educate undergrads in the fundamentals of mass spec.
- Nightingale Health and UK Biobank have launched a major initiative to analyse 500,000 blood samples. UK Biobank's PI said that gathering this metabolic biomarker data will provide opportunities to "benefit patient care and public health."

Company and people updates

- The UK government has appointed a new Government Chemist: Julian Braybrook, currently Director of Measurement Science at LGC and with a PhD in magnetic resonance spectroscopy. Braybook said he was "truly honored" to take on the role.
- DuPont Industrial Biosciences has unveiled its renovated headquarters in Delaware, USA, as part of a $200 million upgrade.
- A "Sustainable Chemistry" symposium was recently hosted by The ACS Nigeria International Chemical Sciences Chapter. Topics included sustainable industrial processes, chemical monitoring and new agrochemicals.

Products and launches

- 1st Detect has announced that its TRACER 1000, a new MS-based explosives trace detector, will enter the evaluation process for ECAC, the European regulator for aviation security.
- See page 17 for a roundup of new launches at ASMS.

For links to original press releases, visit the online version of this article at: tas.txp.to/0718/BUSINESS.

ASMS Advances

What new tech did vendors unveil at the year's biggest mass spectrometry conference?

ASMS always sees a raft of new hardware and software launches in the mass spectrometry space, and this year was no exception. Read on for a (highly selective) re-cap of some of the top tech innovations that made their debut in sunny San Diego.

- Bruker released the "game-changing" scimaX™ Magnetic Resonance Mass Spectrometer, plus the timsTOF™ Pro.
- Thermo Fisher Scientific had a busy show, with a number of new launches, including:
  - Q Exactive UHMR Quadrupole Mass Spectrometer, combining high-resolution, high-sensitivity MS2 and pseudo-MS3 capabilities;
  - Orbitrap ID-X Tribrid Mass Spectrometer, combining Orbitrap and linear ion trap mass analyzer technologies;
  - TriPlus AutoSampler and Liquid Handling System with Robotic Tool Change.
- SCIEX launched its SCIEX OS 1.4 software, for quantitative and qualitative analysis on the majority of SCIEX instruments.
- Shimadzu announced the release of the Cell Culture Media Analysis Platform, C2MAPTM-2000, a fully automated and integrated sample preparation workstation for the analysis of cell culture media. The company also premiered their new quadrupole time-of-flight (Q-TOF) LCMS-9030 system and the Nexera Mikros Microflow Liquid Chromatography Mass Spectrometry System.
- Waters took the opportunity to preview its new Xevo TQ-GC mass spectrometer, GC-MS/MS system for food safety and quality laboratories, which will officially be launched later in the year. Also showcased at ASMS was the new direct-from-sample analytical system from Waters in collaboration with IonSense, Inc – the DART QDa System with LiveID.
- CEM Corporation introduced the new EDGE™ extraction system for rapid sample preparation of GC/LC samples.
- Peak Scientific used their first ASMS hospitality suite to promote the launch of the new GENIUS XE gas generator.
- 908 Devices announced expanded compatibility for their ZipChip™ application, which can now be used with mass spectrometers including Thermo's Lumos and SCIEX's QTRAP instruments.
- Advion, Inc introduced a new molecular identification software suite, TAMI, for their expression Compact Mass Spectrometer.

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In My View

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

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

Contact the editors at charlotte.barker @texerepublishing.com

Trial But No Error

Despite technological advances, the forensics field remains conservative – for good reason.

By Kenyon Evans-Nguyen, Associate Professor, Chemistry, The University of Tampa, Florida, USA.

Every year at ASMS we host a forensics workshop. Many of the conference attendees are from academia and industry, so we try to make sure we have practitioners on the panel for balance; this year, we had representatives from the DEA (Drug Enforcement Administration), the FDA (Food & Drug Administration) forensics labs, and the Dallas County Crime Investigation Lab – Glen Jackson (page 50) provided an academic perspective on the panel. This year, much of the discussion centered around the need for the field to move forward and make use of the exciting new tools that are now available to us in forensic identification.

In forensic drug analysis, GC-MS is the gold standard – and has been for a long time. When I first got to run GC-MS, I thought it was so fascinating that you can take a complicated mixture and find out everything that's in there with very little effort. But one technique can't do everything. For example, some designer drugs are very closely related compounds, such as ring-substituted isomers. These can be difficult to distinguish with mass spectrometry. What we don't yet have is a measure of how confidently we have identified a drug based on a spectrum. GC-MS has been around long enough that experience dictates identification with those spectra is definitive, but there aren't established statistical measurements to put a number to that.

This is also an issue in adoption of the exciting techniques coming up in mass spectrometry (MS), particularly in ambient mass spectrometry (AMS) – for example, DART-MS (direct analysis in real time). There's an inertia in forensics when it comes to adoption of new technologies – and perhaps for good reasons.

First, cost has to be considered. Forensic science is largely taxpayer-funded, and nobody wants to pay taxes. In the workshop, some delegates suggested using accurate mass instruments with fragmentation – but others pointed out that no one in forensics can afford the technology required (though in the long run it saves money because it cuts back on time and labor). In the US, the FBI has the high-resolution instruments capable of fragmentation, such as the Thermo Q Exactive, as do federal FDA labs – but state labs don't have that kind of equipment.

The second concern when adopting new technology is validation. With these new techniques, how confident are we in the identification? Are they definitive techniques, or better suited to screening? DART has taken off in drug identification,

"First, cost has to be considered. Research is taxpayer funded, and nobody wants to pay taxes."

"VUV certainly isn't replacing mass spec, at least not any time soon. As my mother says, 'Not everything is a competition.'"

but at the moment it can only be used as a screening technique, with GC-MS used for confirmation. Validation plays a big part – when you go to court you have to have some sense of how certain you are.

And that brings us to the third 'barrier' that emerged in the workshop. Forensics is unique in that it's so applied, but the transition from academia/research into practice is challenging because our adversarial court system makes the stakes so high. Because of this there can be a big divide between those doing research, and those on the 'frontline' of forensics.

To the audience at ASMS, made up chiefly of academic and industrial research scientists, practitioners look very conservative (the word 'overkill' comes up a lot!). These researchers get understandably frustrated when their new techniques and products are adopted by other sectors, while forensics labs stick with older technology.

In contrast, practitioners may think researchers don't appreciate the high threshold of evidence required in the courts. They admit that theirs is a conservative community, but there's a good reason – the scientist who's doing the analysis may at some point have to sit up on the stand and face cross-examination by hostile lawyers. They have to have 'overkill' data to defend themselves; in most other situations, you're not going to have people challenging your science for the sake of it.

When we consider these factors, it's no wonder that the field is conservative – it has to be. The ASMS workshop was a good starting point for discussing (and perhaps one day overcoming) these hurdles; it stimulated helpful conversation, and I hope the audience gained some understanding of why criteria for identification are so much more rigorous than in other disciplines or fields. But when it comes to moving things forward, does the change needs to happen in the labs or the court? The jury is out.

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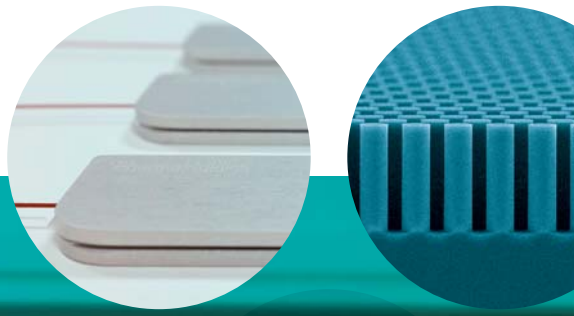

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We Don't Need No Separation, We Don't Need No Peaks at All

How much emphasis should we put on chromatographic separation in today's hyphenated techniques?

By Alex Hodgson, Applications Chemist, VUV Analytics, Inc., Austin, Texas, USA.

I concede that the claim I make in the title of this article might be hyperbolic (and the pun would definitely make Roger Waters recoil). However, I believe that the time has come to re-evaluate the role of chromatographic separations in some applications. It's not 1960, when mass spec was still in its infancy and people were relying on non-selective detectors, like TCD and FID, as their sole sources of data. The data are no longer based on a simple two-dimensional relationship: time versus response. That "response" now has a life of its own: in mass spec it's the collective intensity of individual and identifiable mass fragments entering the detector; for VUV it's the sum of the absorbances at each wavelength for molecules passing through the flow cell. In both mass spec and VUV, the spectrum collected at each time point gives information that can be used to identify a compound – a spectral fingerprint. This third dimension of information allows for substantial selectivity. If we have all this extra data, why is there still so much emphasis on improving chromatographic separation?

"If we have all this extra data, why is there still so much emphasis on improving chromatographic separation?"

Mass spectrometry's selectivity is well documented and a significant factor in its dominance of the analytical market. However, this selectivity must happen on the front end of the run, selecting specific mass fragments in SIM (selected ion monitoring) or ion transitions in SRM/MRM (selected/multiple reaction monitoring), so you better be sure you know what you're looking for. If you don't, you're fishing for analytes in the deep seas of a full scan (and some mass spec deconvolution software is the equivalent of a piece of string attached to a stick...). When those coeluting compounds have overlapping major ion fragments, achieving an accurate deconvolution can be tricky. And if they are isomers? Forget it!

Chromatographic compression is somewhat limited in mass spec because the vacuum state required for the detector restricts higher column flows. Conversely, the VUV detector is at ambient pressure, allowing for significantly higher flow rates. And because all non-enantiomeric compounds have unique VUV absorbance spectra (except carrier gases, conveniently), we can deliberately compress our chromatography. Peaks can then be linearly deconvolved (following Beer's Law) post-run with a high degree of accuracy using these unique absorbance spectra.

"VUV certainly isn't replacing mass spec, at least not any time soon. As my mother says, "Not everything is a competition."

VUV has even removed the need for GC altogether for some applications. A permanent gas mixture from a process line can be streamed through the flow cell, and any fluctuations in relative concentrations can be evaluated in real time. Less emphasis is placed on having symmetric, baseline-separated peaks… or any peaks at all!

VUV certainly isn't replacing mass spec, at least not any time soon. As my mother says, "Not everything is a competition." In fact, the two technologies are complementary, working in tandem and using different strengths to provide much more powerful data than either one alone could.

My ultimate point? The qualities that VUV spectroscopy brings to analytical science may change our perspective on the importance of "good chromatography" in general.

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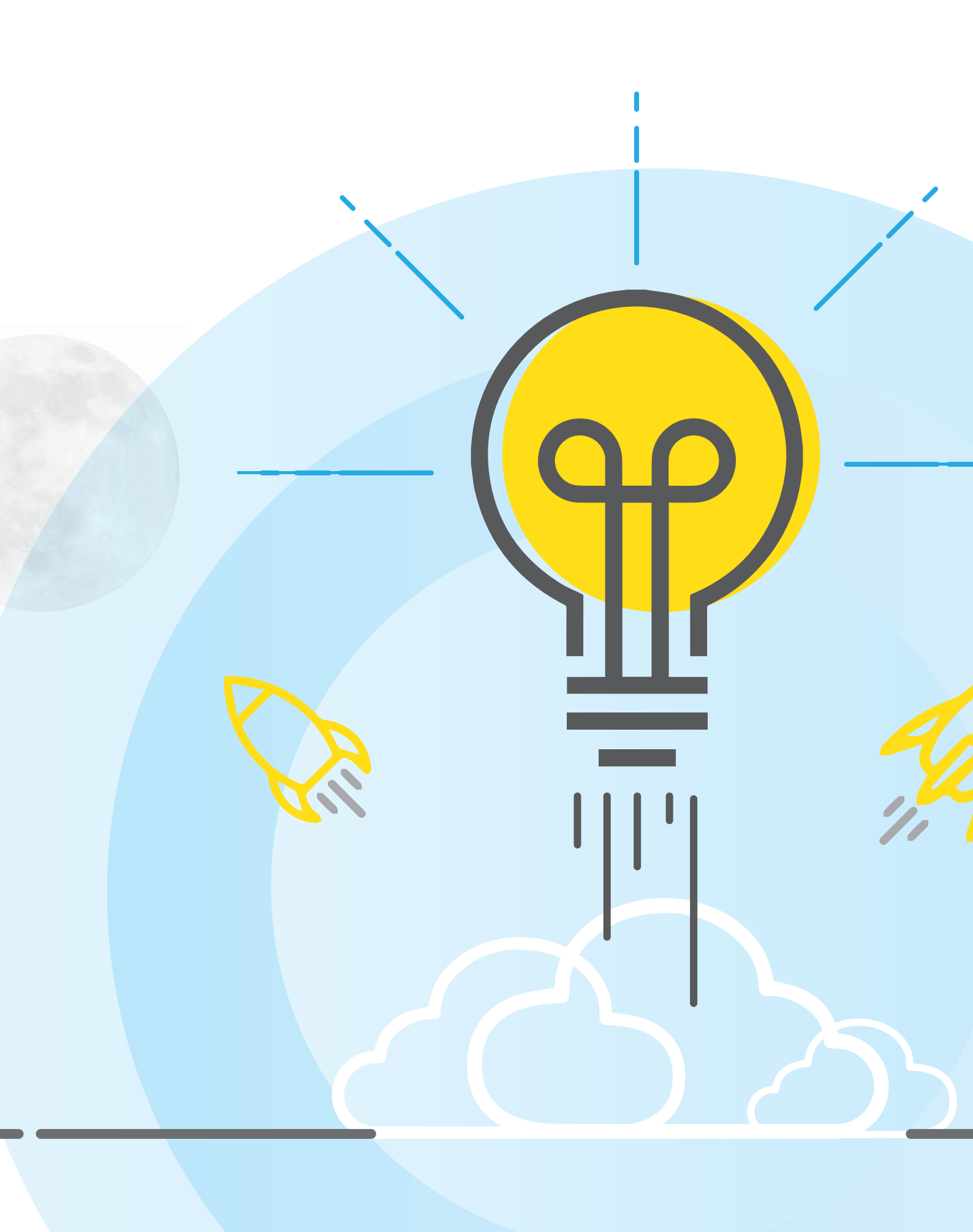


OURS IS TO QUESTION WHY...

...and what, and how. If curiosity is a fundamental part of good science, how do we nurture it – in ourselves and in others?

By Michelle Reid (graduate student) and Michelle Nolan (postdoctoral researcher) at the University of Florida, Gainesville, Florida, USA.

As researchers, we are accustomed to hearing about big personalities, exciting results, and revolutionary discoveries. However, the process by which great scientists unearth their claims to fame seems shrouded in mystery – and it's outright intimidating to aspiring scientists. One concept, though, appears to link all major breakthroughs: an initial spark of curiosity that, through dedication and perseverance, grows into a fully developed research project. We wanted to discover more about this leap from initial fascination to scientific innovation. Rather than discussing only the methodology and results of their research, we wanted details of the creative journey other scientists have taken to develop a project from conception to fruition. In particular, we wanted to learn about early preconceptions, unexpected developments and, most of all, how excitement about unknown chemistry came to shape the story's development.

We asked top analytical scientists, who between them have worked across government, academia and industry, to explore the nature – and importance – of curiosity.

EXPECT THE UNEXPECTED

An interview with Rick Yost

Rick Yost is Professor and Head of Analytical Chemistry at the University of Florida, USA. He is recognized internationally as a leader in the field of analytical chemistry, particularly tandem mass spectrometry (MS/MS). Probably best known for inventing the triple quadrupole mass spectrometer, he still loves teaching undergraduates and graduates in the classroom each semester.

Children are curious by nature. There are always those kids who take apart the toaster to see how it works, and I think we should promote that, making sure we don't quash that desire in school. It's unfortunate that kids rarely get chemistry sets for Christmas anymore (or do experiments in the basement with dangerous chemicals, like I did!). I was always a curious young person; playing with electronic components in the backyard probably generated my interest in instrumentation. I was fortunate that for all four years of high school I was an assistant in a biology lab, and was able to do routine tasks, such as preparing reagents and fixing microscopes, but was also encouraged to try different experiments. For example, I built an eight-foot-long terrarium as a closed biosphere, with salamanders and insects living inside.

Curiosity is what leads us to new scientific discoveries, and what creates passion in young scientists. I think we need to pique their curiosity and then let that drive where they are going. But I have concerns about science education today – when we teach younger students about science, we are too apt to give them a recipe; if they do the recipe right, they get the right answer. I don't think that breeds curiosity. Similarly, much of the way we conduct research today is built around forming and then proving a hypothesis, instead of letting curiosity take the driving seat.

With undergrads, the classroom is where, ideally, we teach critical thinking. Oftentimes we don't teach it very well, sharing facts without context and without explaining why students should care – beyond getting a passing grade on the exam.

The primary reason for educating PhD students should be to produce outstanding scientists – and on the way we should be producing outstanding science. Only allowing them to be a set of hands in a laboratory, washing bottles or taking data, does a poor job of preparing students to be successful scientists in the future. At the graduate level, students should have developed a curiosity and ability to devise and direct projects, and should pursue their own ideas and questions – at least for part of the time! If we're going to produce great scientists, it's important for them to discover for themselves how the scientific method works. The best faculty are not only productive in their own work, but also produce outstanding young scientists who are filled with curiosity and passion for their work.

Real scientific advances rarely come about by slogging through experiments in a linear fashion. We have to provide opportunities for an unexpected result – that's where real discoveries come from. You need the freedom to follow the advances in the field as improvements are made, moving forward as the instrumentation does.

In grad school, my PhD supervisor, Chris Enke, was willing to let me pursue something very different from his own research. In an instrumental analysis lecture when I was an undergrad, Bonner Denton passed a quadrupole mass filter around. He talked about it as a new kind of mass spectrometer, but also mentioned other things you could do with it – transmitting ions, for example – and to me that was a fascinating alternative to current mass spectrometers. After a fortuitous conversation late one night, Chris and I came up with the crazy idea of building a triple quad – and he told me I should write a grant proposal. It clearly defied the accepted knowledge of mass spectrometrists at the time – that's the advantage of coming in with a fresh perspective, not steeped in folklore. It's one of the reasons companies hire young scientists – it's not just because of the facts they know, but because they tend to be naturally curious, passionate and innovative (not that older scientists can't be all of those things!).

The popular saying, "It takes a village," applies to educating young scientists – and to conducting research. These days, science tends to require interdisciplinary teams, so part of graduate education should be developing the skills to work in that way. To me, the ideal research professor gives students enough guidance to get them going, then gets out of their way and lets them discover things on their own – nudging, pushing or pulling where necessary.

Encouraging students' curiosity and freedom helps you reinvent yourself, too. In my own case, my crazy idea of doing something with a quadrupole mass spectrometer completely changed my PhD supervisor's career – he became a full-time mass spectrometrist and went on to be President of the American Society for Mass Spectrometry, something that would never have happened if this student hadn't wandered through his life. Not that I knew much about mass spec at the time – but I certainly was curious!

"Curiosity is the desire **– or passion –** to know something. I would argue that **curiosity has driven scientific research** since before Archimedes jumped out of the bath, yelling, **'Eureka!'"**

CULTIVATING CONFIDENCE

An interview with Suraj Dhungana

Suraj Dhungana is Market Development Manager for Biomedical Research at Waters Corporation in Milford, Massachusetts, USA. His varied career has taken in both environmental and biomedical research, and he has worked in government, academia, nonprofit, commercial labs and now a major instrument vendor.

"Curiosity is what makes us wonder. **Curiosity allows us** – consciously or subconsciously – to detect things that are different and ask why. **It is a knack for asking questions."**

I'm a chemist by training because of my curiosity. It started in high school; the teacher would give us data and say, "Experiment A generated these results. B generated these results. Why do you think they are different?" If we didn't have an answer right away, she would wait until we came up with hypotheses. And that is when I realized how important it is to be curious without limits.

The educational systems we are exposed to really drive how we think. I've studied in Nepal, Italy and the USA – and I've seen a startling difference in the way curiosity is encouraged (or not). In Nepal, you had to memorize and spit things out; you were examined based on what you could remember, not on your understanding. In Italy, the style of learning was the biggest adjustment I had to make; I soon realized that some kind of preparation was needed before class, if I was to come up with answers. Being curious about the subject matter drove you. Here in the US it was a combination of both.

I believe curiosity is in all of us, but it has to be nurtured – and it's easy to stifle it. My children are truly curious to learn – they enjoy finding things out. As we get older, we too often lose that. It's the same early in a science career, when you are figuring out how things work; taking your time to find the right balance, and the right topic is really important in driving curiosity. If we were to stifle curiosity in either setting, we would not be doing those students justice.

Nurturing curiosity takes patience because we work in such a fast-paced, result-oriented environment. With summer internship students, you can be so driven to help them to get a project done, that you forget to figure out where their curiosity lies. I used to sit with the student on their first day and talk about what they have to deliver. Crucially, I would tell them I don't really care how they get there. They could ask as many questions as they wanted and, as long as they come up with something, I would be happy.

Having worked in academia, government and industry, I think curiosity is appreciated across the sectors – but how they are supported differs. Academia used to be the place for the curious – you could devote your time to following your ideas – but now, if you don't publish enough papers or get enough grants, your curiosity doesn't mean anything. Working in the National Institute of Environmental Health Sciences (NIEHS) was a great time for me; I worked closely with two great guys who had probably never written a grant but were super curious – there I had time to try new experiments, design new instruments, and ask questions.

In industry, we do side projects to satisfy our curiosity – you need to manufacture those opportunities. In a CRO, I worked on revenue-generating and non-revenue-generating projects. The latter meant working on things we were curious about, that we could potentially develop into a technology in future and offer a new service to the commercial business. Every time curiosity drives something, we're learning something new, figuring out something that we can implement in the workplace. Of course, time management is important, and I figured out a way to carve out my own time and align that with the longer-term goals of the company, which was important to keep curiosity alive.

If you have an idea, the science should not be limited by what's available to you. What's available to you should be dictated by your science. That's the way scientific curiosity advances. In my case, I was not afraid. I probably did many things I wasn't supposed to do in the lab – my PhD mentor would agree – but without that curiosity, without making the choices I did, I wouldn't have gone anywhere. In fact, three of my papers wouldn't have been published!

CREATIVITY IN CONTEXT

An interview with Zoltan Takats

Zoltan Takats is Professor of Analytical Chemistry in the Faculty of Medicine, Department of Surgery & Cancer, Imperial College London, UK. He is well known for inventing desorption electrospray ionization (DESI) and the iKnife surgical instrument.

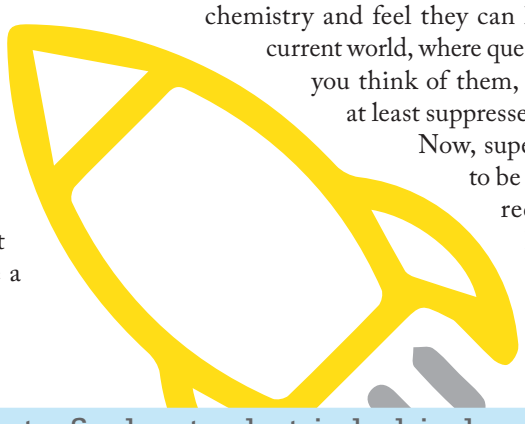
Curiosity is very important in instrument development – and it has a lot to do with creativity. Some people say that science is an art, and that, by doing research, scientists are creating the equivalent of an artist's painting. I disagree. There is a fine line between art and science, but the key difference is that science is systematic – whereas you could argue that good art isn't! With art, you need inspiration, and you don't care about the context – that helps your brain create something new. The inspiration for the novel One Hundred Years of Solitude appeared in Gabriel Garcia Marquez's mind in a single second, and all he had to do was scribble it down. Science doesn't always work that way – in most cases, it's more like building a Lego house. Sometimes you can have a huge impact on how the finished building will look – though having been a scientist for 20 years I can say that rarely happens! To continue the building metaphor; if you are a curious person, you take a step back and look at the building as a whole, try to have an understanding of the system behind it and create a hypothesis for yourself: why does it look like that? Non-curious people might see a rectangular gap and merely attempt to fill it.

Of course, you can be systematically creative but still pursue projects that are driven by passion; inventors often claim that their biggest ideas are driven by passion. I tell people that being a postdoc is the best time in your life – you have a young brain, you have lots of good ideas, and you're spending someone else's money. DESI was my hobby in the lab as a post doc – my supervisor, Graham Cooks, tried to convince me to go back and focus on my final project, instead of spending my time and his funding on something else! But while that was a passion project, it was practical at its root.

At the moment, I have dozens of questions in my mind that I desperately want to seek answers to – but I don't have funding to do them and, unfortunately, I can't go out to funders and say, "Last night a question popped into my mind – will you give me half a million dollars to pursue it?" If there was an infinite amount of resources, all research would be around passion projects – but, as it is, they are much more about opportunity.

Dogma says that there are creative people and non-creative people; curious and non-curious people. That is just not true; deep inside, everybody has curiosity and creativity, and everyone is able to enjoy creating something new. As I see it, there are two problems: first, there is enormous cultural suppression of curiosity and creativity in the 21st century; and second, people don't find their own interests, so sometimes pick the wrong subject, instead of pursuing something where they can exercise their creativity.

It's the lack of curiosity that then forms the bottleneck for many students. They might be interested in analytical chemistry and feel they can help other people, but in the current world, where questions are answered even before you think of them, all the curiosity is killed – or at least suppressed – at a shockingly young age. Now, supervisors have to train students to be curious. We have to help them rediscover the curiosity they had as children and point it towards scientific problems.



"Curiosity is the urge to find out what is behind something. **A simple example: when crossing a meadow, you see a beautiful flower.** Some people would sit and look at it, some would upload a picture onto Facebook. **A few people might Google the name of it.** And an even smaller group might want to know its Latin name, whether it's a native species, whether it's rare, and so on... **They are the scientists!"**

CURIOSITY APPLIED

An interview with Timothy Garrett

Timothy Garrett is Associate Professor at the Department of Pathology, Immunology and Laboratory Medicine, University of Florida, USA. His research is focused on the application and development of mass spectrometry techniques and instruments for clinical research, particularly small molecule quantitation or characterization.

As a scientist, you are starved without curiosity – asking a question that other people don't ask is what makes you a scientist. That's a bold statement, but it's based on my experience moving from a lab where we had total creative license (super innovation), to a lab where we had to answer questions quickly, but not necessarily innovatively. The transition was a struggle, until I figured out how to bring the curiosity back into my work.

I got curious about chemistry as an undergrad. The more time I devoted to learning about chemistry, the more I enjoyed it. I could visualize the chemical structures in my head, and I became fascinated about what happens at the molecular level. I was lucky to have a PI who gave me both the opportunity and the trust to run a mass spec instrument by myself as part of a project (once he knew I wouldn't damage it!).

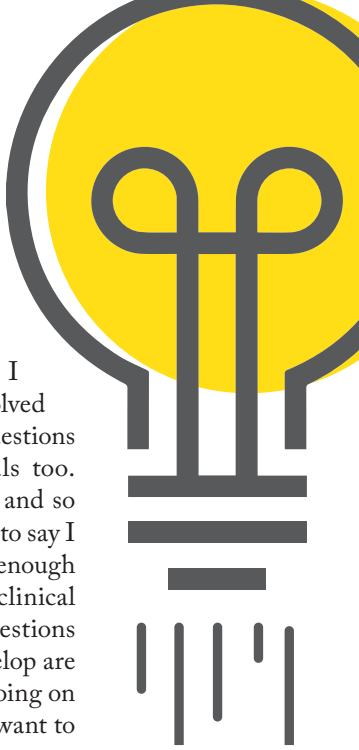
Now I'm a mentor, I think curiosity is probably one of the most important things a student can have. Research takes a long time and it can get frustrating when things don't work the way you expected, so you have to keep asking questions and be able to think through problems creatively. I also think curiosity helps you relax – when you have a new idea, I'm quite certain it changes your brain chemistry, and gives you a boost!

You also need a boss who will keep you engaged in the actual science of discovery, someone who will nurture that curiosity; one, by asking questions, and two, by helping you keep the focus and by trying to remove distractions. Presenting a talk is a really good exercise, because organizing your research helps you see it in a different light and spot any gaps.

For curiosity to truly flourish, you must have the tools and training necessary to answer the question; sometimes a big investment or the right collaboration is required, but there is no guarantee of success. For example, I have been using airbrushing to spray into the ion source – I noticed that an electric current can impart too much energy, causing fragmentation, and wanted to create clusters that weren't ionized by electric current. Airbrushing doesn't have any electricity, but it still produces fine droplets, and I thought that could be a unique way to do it. So far it hasn't worked – that's when passion or curiosity, rather than funding, keeps you focused.

Having said that, one of the dangerous parts of science is that sometimes you don't know when to give up! As a scientist, at some point you have to be prepared to say, "I can't do that." That's the danger in analytical chemistry – some people believe there's a definitive answer every time. When I first got into imaging, my collaborator, who had done some work on nerve damage, wanted to look for certain compounds. We were looking at very small molecules in tissue, hoping to see what their distribution was and how they were interacting. We and tried every which way to do it. Using MALDI, we were able to look for them but could never figure out if they were there. It was frustrating, but we had to move on to focus on other important aspects, such as trying to understand how the lipids are being altered as a result of a drug, even if you couldn't see the drug itself.

In the clinical field in particular, it's difficult to pursue projects purely



"To me, **curiosity is being pulled into something you weren't necessarily aware of** – and then asking questions **that take you down a different path.** Essentially, it's making the science **'real'"**

driven by curiosity and without a direct application. We often have to work with applied technology, but I think it's key that the students involved go beyond looking at the applied questions and think about the fundamentals too. Finding the time requires balance, and so it's up to me to devote the time to it, to say I think it's important and interesting enough to me. Also, because I work in the clinical arena, I want to make sure the questions we answer and the methods we develop are reliable enough to measure what's going on inside a patient. The last thing you want to do is give the wrong result.

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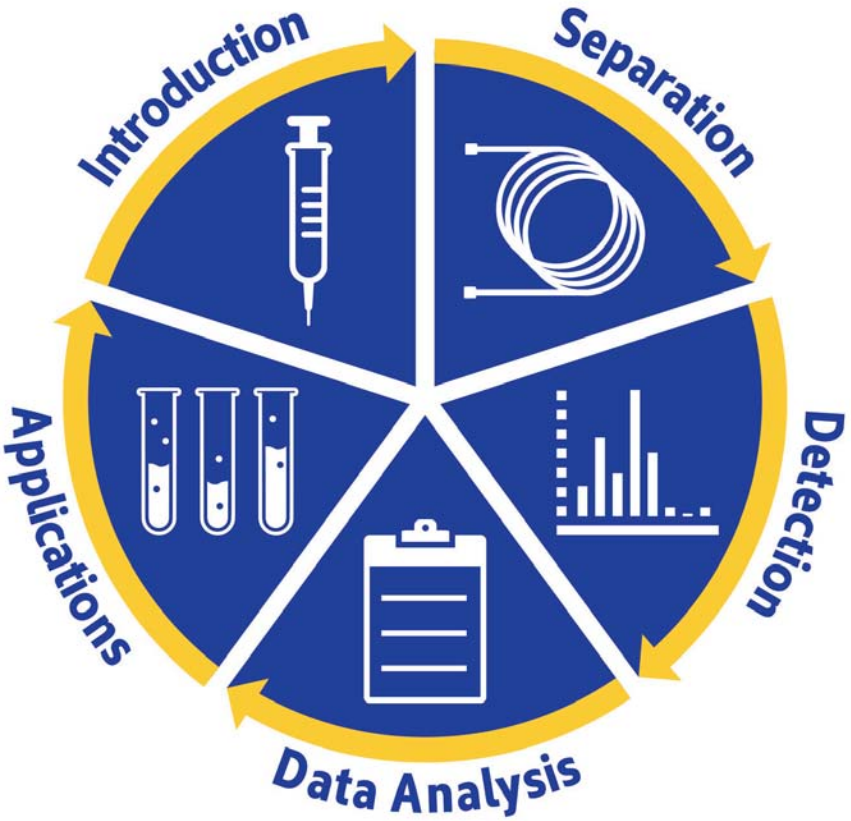

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A CLASS ACT

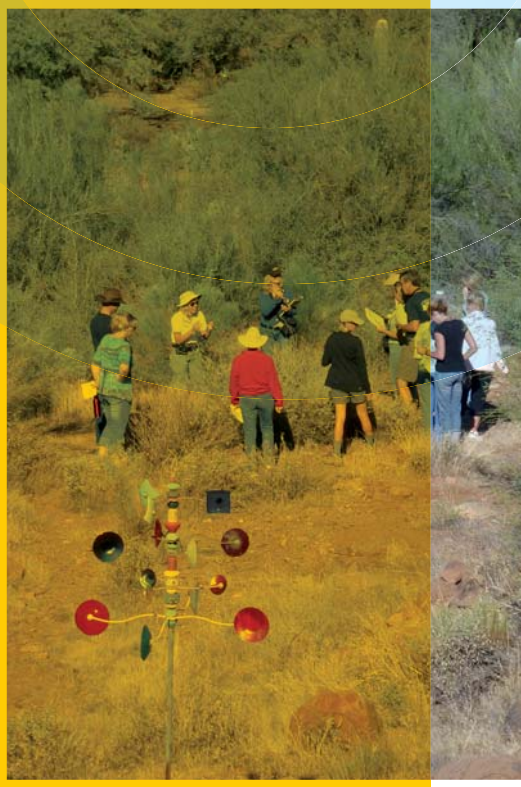
What is the secret to successful – and exciting – learning in the classroom? The answer is curiosity.

By Diane A. Vaszily, Science & Environmental Education Specialist, Desert Eye Education, Arizona, USA.

The youngest people are usually the most curious. Those who work with preschool children know that they have insatiable curiosity which provides unlimited opportunities to share. Toys for young children provide manipulatives that allow students to solve puzzles and indulge in their natural curiosity. However, as they get older, the 'hands-on' apparatus and techniques, especially in classrooms, are often replaced with book reading and lectures. As a teacher, the goal is to keep older students interested in seeking out knowledge, not forcing it onto them. If they are driven by to their own curiosity, they follow their own lead rather than yours.

Demonstrations – showing something happening, discussing it briefly, then allowing students to create a variation of the event – offer a chance to foster curiosity. Enabling the students to engage in their own experiments by making bubbles with various amounts and/or brands of soap solution, for example, then varying the sizes and shapes of the bubble holders, is an activity that covers a range of topics from surface tension to surfactants. And the enjoyment from blowing bubbles has no age limit!

As the students increase in age, making the topic relevant to the learner becomes more critical. Curiosity seems to decline



between the ages of 11 and 14 simply because students that age do not see the relevance of what they are learning. Relating the bubble topic to shampoos and conditioners will bring their heads up out of their notebooks. Pigmentation can be addressed through make-up and hair dye. Rocks and minerals have endless possibilities when associated with gemstones.

I speak from experience. As a biologist teaching earth science, analyzing gemstones by comparing birthstones brought eighth graders to attention, piquing their curiosity (and mine) so that we all wanted to know how, where and when they were formed. This led to analyzing the elements that contribute to the color of the gems as well as every other element found in the earth. I have found that this strategy of starting with a gemstone and working backwards to its formation, strata, parent rock associations and locations has aroused insatiable curiosity in people aged from 6–92.

How does one keep curiosity alive as an educator? I believe it is always in the presentation, the first contact with your students on any given day. Not only is a teacher tasked with reaching benchmarks, (also known as knowledge), but with tapping into the curiosity that lies right beneath the surface. Creating a "what is that?" or "how do you do that?" moment is surely the first thing an instructor needs to consider – teachers need to be entertainers as well as educators.

Having come from a professional rather than an educational background after graduation, most of what I did with students in the classroom was based on the way I learned – experientially, not via lectures or textbooks, important and necessary as more formal learning is. My goal was to get them so curious that they actively pursued the answers. To this day, former students always remark on the curiosity that was awakened and kept alive in them throughout their time in my classes. What they don't realize is that they taught themselves, because of the curiosity we developed together.

Science *in the* Courts!

With a passion for accuracy and reproducibility, analytical scientists are prime candidates for the witness stand. Presenting (often complex) scientific concepts to a jury comprised of laypeople is tough enough – but expert witnesses also have to keep their cool during intense cross examination by hostile lawyers. We speak to a psychologist and a forensic mass spec expert about the challenges of putting analytical science 'on trial' – and get a fascinating real-life story of a very unusual court case...

The Human Factor

William Thompson, Committee Chair of the OSAC Human Factors Committee, explains why the 'human element' is an important factor in forensic science testimony.

I study the underlying psychology or psychological dynamics of human decision making. For a long time, I've been interested in how experts – and especially forensic science experts – evaluate evidence and reach conclusions.

The human factor comes into forensics at two levels. In part, it's the psychology of the expert; how they make decisions (and sometimes make mistakes). The second part is the psychology of communicating scientific findings – particularly, to a jury or to lawyers who may not have any expertise – in a way that allows them to understand and draw appropriate conclusions.

Setting standards

I work with the OSAC (Organization of Scientific Area Committees for Forensic Science), an organization designed to help foster and create standards and practice guidelines in forensic science. After a 2009 report from the US National Academy of Sciences identified the human element in forensics as needing additional study, I was invited as a lawyer and a psychologist to take part in the OSAC's human factors sub-committee.

The Human Factors Committee is not empowered to create standards on its own, but we are expected to provide advice to other units within OSAC, comprising forensic scientists, industry professionals, and researchers. We also provide advice directly to the Forensic Science Standards Board, which ultimately reviews and approves those standards.

The three areas that we're most interested in are: i) how to minimize bias and interpretation – particularly contextual bias and the possibility that scientific conclusions are influenced by irrelevant investigative facts; ii) effective communication; and iii) assessment and testing of individual examiner performance.

We have to learn a great deal about a diverse range of forensic science disciplines; on the one hand, we're dealing with DNA analysis and analytical chemistry, and on the other, we're looking at pattern matching tasks, debris analysis, and forensic pathology. However, they all involve humans, and so they are all susceptible to human error.

Justice is blind

The Human Factors Committee has been identifying possible sources of bias, and how we minimize those effects. We need to make sure that the decisions made by experts are as good as they can be. Surprisingly, despite forensic science having been around in one form or another for over 100 years, there has been little discussion of the proper basis for a forensic scientist's opinion. When it comes to a particular analytical task, there's a surprising amount of debate over what is relevant. To avoid bias, we are constantly pressing forensic scientists to identify what kinds of evidence are relevant (and irrelevant) to particular tasks. Is it just the physical evidence in front of them, or is it necessary to take context into account? Should the fingerprint examiner, for example, be drawing scientific conclusions solely from the fingerprints, or should they be influenced by other investigative facts in the case?

Forensic scientist Michael Taylor and his colleagues did a series of studies (1) looking at whether experts who examine blood stain patterns are influenced by something other than the physical characteristics of the patterns themselves. They created spatter patterns for the study so that their actual origin would be known, and experts were asked to see how accurately they could characterize these patterns. They were given some contextual information in the form of police reports – and this information was found to influence the conclusion. If the police report mentioned somebody had heard a gunshot, the pattern was more likely to be interpreted as high velocity splatter, whereas if the report mentioned that someone had been heard coughing, the same pattern was more likely to be interpreted as expiration. Studies have documented similar effects in latent print analysis, forensic anthropology, document examination, crime scene analysis, and even DNA analysis. The fact that the analytical conclusions of the forensic scientist can be unconsciously influenced by contextual factors that seem irrelevant to the scientific task is an important concern.

Based on those findings, we have considered different ways

of sequencing the workflow to 'blind' forensic investigators to unnecessary information; for example, the blood splatter pattern expert doesn't necessarily need to read the police report until after they have interpreted the pattern. Ultimately, it may be impossible to eliminate all bias from our justice system, given the ubiquity and variety of bias we're confronted with. (There are even studies showing that judges become harsher in their sentencing right before lunch!) Nevertheless, we want to help forensic scientists gather and present the best possible evidence.

Present (and correct)

The big debate in forensic science right now, and a good example of the issues the OSAC committee tackles, is how best to present source conclusions. Source conclusions involve an examiner comparing two items and trying to reach a conclusion about whether the items come from the same or a different source. Were these two shell casings fired by the same gun? Were these two fingerprints from the same person?

How should a forensic scientist state their conclusions to a lay jury if they wish them to be understood and interpreted appropriately? In days past, many forensic experts would simply make an identification; they'd say, "I've examined these two prints and I've determined that they came from the same shoe." There's been a lot of criticism of that approach, because it implies that decisions can be reached with certainty, when we know that they can't.

With that in mind, how can you convey those findings in a way that makes the uncertainty more transparent or understandable to the lay audience? Do you give the conclusion and include data – for example, true and false positive rates? Or do you explain that the conclusion is merely 'likely' and try to compute some probability? There is currently a lot of debate about this.

I think we need to avoid giving categorical conclusions – it seems wrong to me to state that things are certain when they are not. To state: "I think there's a 99 percent chance these two items come from the same source," is also problematic, because it's difficult to reach conclusions of that type based on scientific evidence alone.

Research has found that people tend to misunderstand the probabilities they are given. They often mistake 'random match probability' as 'source probability'; in other words, an expert might state the probability of a match coming from a random person – and laypeople often think of it as probability it didn't come from the accused. In some instances, that can be a serious error.

For a long time, it was thought that people would give very little weight to likelihood ratios because they wouldn't understand them, but we've debunked that through our studies (2, 3).

The question of how best to communicate findings is a difficult one and I'm not sure that I know the right answer – the more research I do, the less certain I am! What is certain is that we need testing and validation of different approaches.

Before the court

A lot of human factor issues also become legal issues – so within OSAC, we have a lot of interaction with the Legal Resource Committee. Ascertaining the proper basis on which a forensic scientist should form his or her opinion, or what we want them to try to take into account (or not) are ultimately legal concerns.

We have had cases in the United States where forensic pathologists have taken everything into account – not just forensic findings, but all the background information, the police reports and so on. They have testified on that basis, and the cases have been overturned on appeal by courts – they felt that the experts have encroached on the jury's role by considering issues beyond the scientific. So, the legal questions are very much intertwined with psychological and scientific issues.

I am a lawyer and I used to practice law, so I have been involved in cases both as a lawyer and as an expert witness. I have to say, I like being a lawyer more than being an expert. It's more fun to ask the questions than to have to answer them! To scientists testifying, I would say the following: it can be quite grueling, but it can be a very positive experience. And it's really beneficial for the legal system and for society in general if good scientists are willing to come to court and explain what they know. One of the big problems in criminal justice is the difficulty of finding experts who are willing to give their opinion, so I would encourage anybody who is interested to give it a try. Once you are on the stand, be very clear, speak to the lawyers in advance, be very firm about what you can and cannot say… And stick to your guns. Don't allow lawyers to talk you into anything.

It's a complex field and my OSAC role is a challenging one, but it continues to fascinate me. It's absolutely necessary that we have lawyers who are scientifically literate and capable of understanding the nuances of forensic science evidence. I have enjoyed working as a lawyer, but I feel at this point in my life and career my time is better devoted to working with forensic scientists to help them get it right, rather than trying to challenge what they're doing in court.

William C Thompson is Professor Emeritus of Criminology, Law, and Society and Psychology and Social Behavior and Law at the University of California Irvine, Committee Chair of the Human Factors Committee of OSAC, USA.

References

1. *1. MC Taylor et al., J Forensic Sci, 61, 1461-1466, (2016).*
2. *2. WC Thompson & EJ Newman, Law Hum Behav, 39, 332-349 (2015).*
3. *3. WC Thompson et al., Law, Prob & Ris, 17, 133-155 (2018).*

Stand and Deliver

Kenyon Evans-Nguyen discusses the unique and challenging experience of presenting your science in court

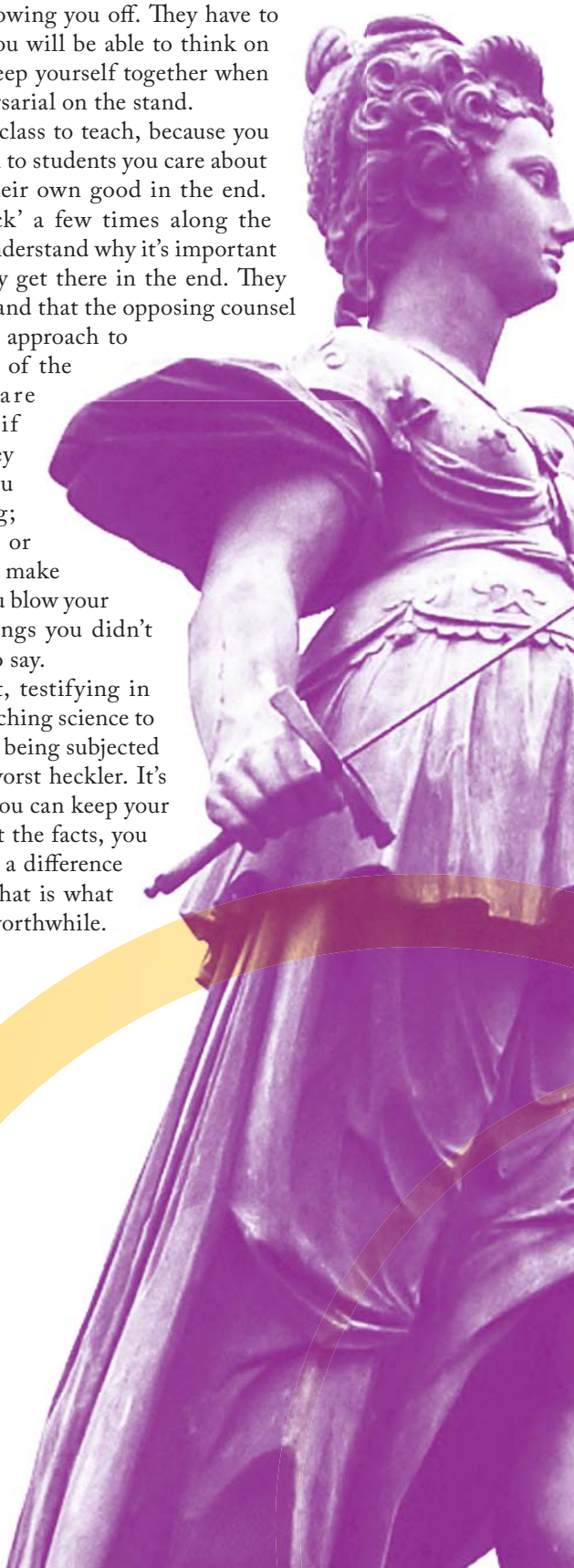
I've been on the stand many times, back when I was a practitioner – and it can be terrifying! It's a unique experience. Some practitioners are almost like survivors of trauma; they've been up on the stand and had to endure these stressful experiences where the object of the opposing attorney is to tear down everything that they're saying – and not necessarily based on science.

When your paper gets peer reviewed, you may face harsh criticism, but their arguments have to be scientific; their goal is not to invalidate your science, but to make sure it is rigorous. When the FDA examines your new drug, they go through everything with a fine-tooth comb, but their goal is to make sure the science is good. The goal of a defense attorney can be to cast doubt; if they get you to speak in terms that are inaccessible to the jury (referred to as 'muddying the water'), then they've won. If the science, or your defense of it, confuses the jury, your science goes down the drain.

I train students on how to take the stand, and I warn them beforehand that it's going to be weird and uncomfortable, and it's probably going to make them angry. It's part of the interview process in forensics – they want to know how good your science is, but then they will intentionally mess with you. They may give you a tour, and then at the end of the interview a couple of hours later, they'll test you on obscure details. They'll ask you bizarre questions you wouldn't be asked in a typical interview and stare at you to see how you respond, deliberately throwing you off. They have to establish that you will be able to think on your feet and keep yourself together when things get adversarial on the stand.

It's not a fun class to teach, because you have to be harsh to students you care about – but it's for their own good in the end. Most will 'crack' a few times along the way, but they understand why it's important and they usually get there in the end. They need to understand that the opposing counsel will adapt their approach to the personality of the person they are questioning: if you're timid, they try to bully you into conceding; if you're brash or bold, they try to make you angry so you blow your top and say things you didn't mean or want to say.

As a scientist, testifying in court means teaching science to the jury – while being subjected to the world's worst heckler. It's intense. But if you can keep your cool and present the facts, you can really make a difference to a case, and that is what makes it truly worthwhile.



"I train students on how to take the stand, and I warn them beforehand that it's going to be weird and uncomfortable, and it's probably going to make them angry."

Where There's a Will…

While providing scientific testimony for a high profile case, Harold McNair had several grueling encounters on the stand…

I've been involved in several cases that have been important, but a major one was the will of Howard Hughes.

I entered the scene when I was teaching an undergrad class at Virginia Tech. A man walked in, packing a weapon, with a bag handcuffed to his wrist. He said, "Are you Professor McNair? Can we talk alone?" If somebody has a gun, I tend to do as they say...

"I'm a federal marshal and I have the Howard Hughes will right here," he said. "It's worth $400 million. Do you have a secure place to keep this?"

I said, "Yes, but why would I want to?"

"I have a request from the FBI on behalf of the Hughes family. They would like you to do an analysis of the ink and see if it could be the ink that Hughes regularly used."

It turned out to be a very interesting case. An attendant in a gas station close to Las Vegas claims that early one morning he saw Howard Hughes in pajamas wandering around, looking for things related to nuclear bombs. (He was paranoid about those things.) The gas station attendant, Melvin Earl Dummar, claimed Hughes befriended him and that Dummar took him back to his hotel.

When Howard Hughes died, he left behind maybe half a million dollars – probably $4b today. People thought he would leave most of the money to Stanford University and the Hughes Medical Center – maybe a few friends and family. However, this handwritten will (dated before Hughes' death) left $156m to the Mormon church, and $156m to Dummar. It became known as the Mormon will.

Of course, the whole story is bogus. The will had been forged at a much later date. Howard Hughes would never have been wandering around in his pajamas in the desert. He had a chute in his hotel room, and he would climb into a special temperature-controlled "coffin" to go down to his car in the basement.

So it was my job to analyze the ink and clear up any doubt. Howard Hughes typically bought blue Paper Mate pens, maybe 1,000 for the year. Paper Mate originally bought the ink from DuPont – but just after the will had been written, Paper Mate decided to make its own. Using HPLC, I discovered the Paper Mate ink had two pigments plus two impurities – impurities that did not appear until about 1971 – and that this was the ink in which the will was written. The ink on the document said 1968 – and so it could not be valid. The

judge and jury didn't accept HPLC because it was too novel a method (I was one of the pioneers that introduced it), so they told me to analyze again by thin layer chromatography. I returned to Virginia Tech, performed the analysis, and the results were the same. The Mormon will had been written in 1971 or later.

When I went to testify, the plaintiff was extremely aggressive.

He said, "You wear glasses. You have a physical handicap – you don't see very well – and you didn't see very well on this case. You made a big mistake, didn't you?"

He continued, "Tell the audience how much money you make consulting and fabricating these lies."

I said, "I don't receive anything. I had to get permission from the governor of Virginia even to come here. The FBI asked me to come and testify."

"Why would you do that?" he scoffed.

I said, "Becausee I am, and have been the FBI's expert on inks and dyes for many years." We won the case.

It was the first time I'd been to trial, the first time I'd had someone eat me alive verbally – I really enjoyed the challenge of it!

"It was the first time I'd been to trial, the first time I'd had someone eat me alive verbally – and I really enjoyed the challenge!"

When the federal marshal escorted me back to the airport (there'd been a threat along the lines of "shooting the guy from Holland because he was questioning the validity of the will"), I was walking across the street and the plaintiff lawyer called out to me, "Sorry, Dr McNair, just trying to win the case – no hard feelings!"

Profession

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Top 40 Under 40: 4 Years On

With a new Top 40 Under 40 Power List released this year, we catch up with some of the finalists from the last Top 40 to find out where they are now, and how the experience has influenced their career. If you would like to see a student, friend or colleague star in this year's list, you can nominate at tas.txp.to/powerlist2018.

Rachel Louise Gomes
Associate Professor in Chemical & Environmental Engineering, Nottingham University, UK.

What's changed in the past four years?

I'm now an Associate Professor and my group has grown, as has the interdisciplinary nature of our work. I get to work with wonderful and passionate people spanning academia and industry, from all across the globe. My research has presented many opportunities, most recently travelling to Ghana to carry out research on water and food security.

Have any of your previous answers changed?

Looking at the question on which scientists I most respect, I would now include the late Anike Igunnu (née Akinrinlade), who cared passionately about research delivering value to society. Her doctoral research reflected this, leading to a posthumous PhD award. She inspired and continues to inspire many, including me.

What advice would you give to yourself four years ago, if you could?

Understand and appreciate what matters most to you at that point in time.

Did being in the Top 40 Under 40 Power List impact on your career?

When I received reviewer comments for my first new investigator grant (an interdisciplinary proposal between water process engineering and analytical chemistry) one of the reviewers queried my analytical science credentials, asking for evidence beyond my publication record. Responding that I was on the Power List was extremely timely... and I got the grant!

Sergio C. Nanita

Principal Investigator, Analytical R&D, DuPont Industrial Biosciences, Wilmington, Delaware, USA.

What's changed in the past four years?

A lot has changed in my career over the past four years, including my role within DuPont and the focus of my analytical chemistry research. In 2014, I was part of DuPont Crop Protection R&D. Today, I am a principal investigator within the Analytical R&D department at DuPont Industrial Biosciences. I am still a mass spectrometrist but my work now focuses on developing/applying state-of-the-art analytical methods to address research and business needs of DuPont Industrial Biosciences. My current role contributes to product discovery, development, and support of existing products from various businesses, including Biomaterials, Animal Nutrition, and Fabric & Home Care.

Other aspects of my career have also progressed, including my service through the American Chemical Society (ACS), where I now serve on the International Activities Committee. The appointment has given me the opportunity to contribute to science education in disadvantaged communities while representing the ACS abroad, among other projects.

Have any of your previous answers changed?

As well as the update to my research focus and objectives that I already described, I would expand my prediction for the future, to include the development and application of autonomous technology in analytical sciences. This trend will continue to replace human/subjective decisions with sets of objective (mathematically driven) instructions; for example, to decide when the next sample should be taken and from what location, and what dilution factor should be applied. This will become particularly important for in situ and in vitro analysis.

What advice would you give to yourself four years ago?

Two things:

- Teach and mentor more; it has been increasingly rewarding in my career to help others excel.
- A vision or a planned path to follow is important, and so is the ability to change it.

What did being on the Power List mean to you?

I remember reading the first Power List ("Top 100") published in 2013. It was inspiring to learn about the stellar careers and contributions of those at the top of the field. So being included in the "Top 40 under 40" edition in 2014 was motivating for me. It put me in the spotlight, and gave me opportunities to establish connections with other researchers and professionals, which may not have happened otherwise.

Sebastiaan Eeltink

Professor, Chemical Engineering, Vrije Universiteit Brussel, Brussels, Belgium.

What's changed in the past four years?

I have built my excellent research group – this year four new PhD students will join my group and there are still two PhD positions available. Also, I was awarded the position of full professor in 2018. Although I do not feel like I am "established" yet, the future looks bright.

Have any of your previous answers changed?

The research themes of my group have expanded over the last four years. Besides the development of novel technologies, we are focusing now on realizing super cool applications that we hope will have impact on the field of life science and biotechnology research.

Did being in the Top 40 Under 40 impact on your career?

At the time I was obviously very honored, but I did not realize the full impact that the article would have. Years later, people still refer to the Top 40 Under 40 list, and I have worked with several visiting students/scientists who I believe contacted me partly due to the article. Also, people from industry saw my name on this list, which helped to initiate new collaborative projects (and ultimately acquire new funding).

Helen G. Gika

Assistant Professor, Laboratory of Forensic Medicine & Toxicology, Department of Medicine, Aristotle University of Thessaloniki, Greece.

What's changed in the past four years?
I've moved to another department (Medicine) of the same institution, where I am an Assistant Professor of Bioanalysis. We continue to grow as a group, attract funding and collaborate with top scientists from the clinical sector.

What advice would you give to yourself four years ago?
Be focused and selective in collaborations.

Have any of your previous answers changed?
As well as Alexander Makarov and Janusz Pawliszyn, I would acknowledge my respect for two further wonderful scientists, Ian Wilson and Matthias Mann.

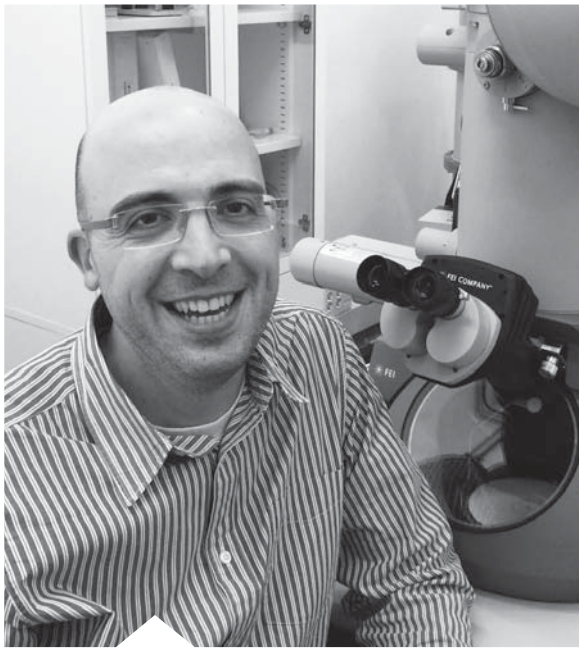
Did being in the Top 40 Under 40 have any impact on your career?
I believe it generated publicity and recognition, and therefore helped my proposals for grants to be successful.

Kevin Schug

Shimadzu Distinguished Professor of Analytical Chemistry, University of Texas Arlington, USA.

What's changed in the past four years?
Personally, I have been promoted to Full Professor and done a short stint in administration as an Interim Dean for Research. In terms of my group's research, we continue to evolve – some of the things we are doing now weren't even on our radar four years ago. For one, we have successfully demonstrated the potential for triple quadrupole-based determination of intact proteins. Many people told us that would not work, but it does. Plus, our environmental analysis research has transitioned from being solely chemically oriented and focused on environmental monitoring, to include biological species determination and methods for improved recycling of oilfield waste waters.

What did being in the Power List mean to you?
The Analytical Scientist has done a great job of promoting various aspects of the analytical community, and recognition of your efforts and accomplishments is always nice. Certainly, having more people know who you are and what you are doing makes this job that much more enjoyable.



Jordi Arbiol

ICREA Research Professor and Group Leader, Catalan Institute of Nanoscience and Nanotechnology, Spain.

What's changed in the past four years?
Four years ago I was working to consolidate my group. Now, we are recognized as one of the leading groups on advanced electron microscopy analysis of nanomaterials (such as nanowires and free-standing nanostructures). I have also become President of the Spanish Microscopy Society (SME), and scientific supervisor of the MET-CELLS Project to equip the ALBA Synchrotron with state-of-the-art transmission electron microscopes.

Did being in the Top 40 Under 40 impact on your career?
Together with other awards I received around that time, I believe being part of the Power List in 2014 helped me to obtain my current group leader position at the Catalan Institute of Nanoscience and Nanotechnology, one of the top Institutes of Excellence in Spain

Giorgia Purcaro

Research Scientist, Thayer School of Engineering, Dartmouth College, New Hampshire, USA.

What's changed in the past four years?

Much has changed! I left my academic position at University of Udine (Italy) to become the Scientific Director of Chromaleont srl, a private company spin-off of the University of Messina (Italy). That experience helped me realize that I wanted to explore new fields; in particular, I wanted to learn more about chemometrics, to extract useful information from the huge volume of data that modern techniques provide. Thus, I moved from Italy to Dartmouth College in the US, where I have applied my analytical skills to metabolomics applications. Recently, I won an academic position at University of Liege (Belgium), in the Gembloux AgriBioTech Department. I am excited to start my own research group, where I can merge and exploit all my previous experience and create new collaborations – and share my enthusiasm and passion for research with colleagues and younger scientists.

What advice would you give to yourself four years ago?

Be focused, but never stop exploring other fields.

What did being in the Power List mean to you?

It was a great honor for me to be in the Power List. Being a scientist is not always easy, but recognition like this helps you to keep going and focus on your goals.

Koen Sandra

Scientific Director, Research Institute for Chromatography, Kortrijk, Belgium; co-founder and R&D Director anaRIC biologics, Ghent, Belgium.

What's changed in the past four years?

Here at RIC, we have experienced a substantial growth in our omics and biopharma activities which, to my surprise, resulted in my being listed in the Analytical Scientist's "10 Top 10s" Power List 2017.

What advice would you give to yourself four years ago, if you could?

Dare to say "No" and stay focused.

Did being in the Top 40 Under 40 impact on your career?

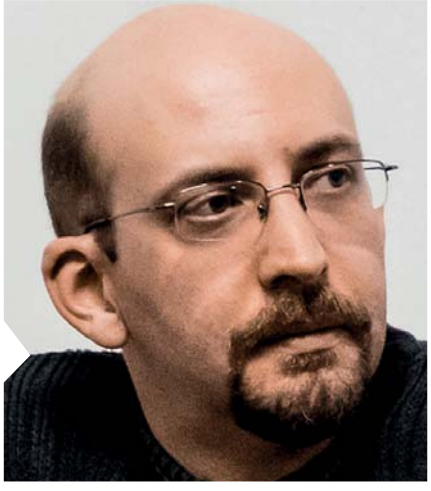
It definitely gave motivation and visibility to me personally, and to RICs as a whole – and it's always nice to have your hard work appreciated!

Alexander Leitner

Principal Investigator, Institute of Molecular Systems Biology, Department of Biology, ETH Zurich, Switzerland.

What's changed in the past four years?

I have moved to a permanent PI position at ETH Zurich and recruited some talented students and postdocs to my team. My research focus has shifted further from the analytical sciences – I am now mostly working on proteins and protein–RNA complexes and even a bit of personalized health, but chromatography and mass spectrometry still remain the core technologies in our work.



What did being on the Power List mean to you?

It was nice to receive recognition from analytical scientists, especially since I have chosen a somewhat unconventional career path that led me towards structural biology.

Profession

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Industrial Revelations: Michele Suman, Barilla

In a new series, industry scientists are in the spotlight – starting with a champion of food analysis.

Scientists working for companies have made huge contributions across the analytical sciences. Compared with their academic counterparts, however, industry scientists are more apt to stay out of the spotlight. In this new interview series, we will highlight some of those contributions, by talking to industry scientists about the joys (and pitfalls) of doing science in the "real world."

Michele Suman, a Research Manager at Barilla SpA Research Labs, is a true collaborator – a member of working groups in the European Committee for Standardization (CEN), Vice Chair of the ILSI Process Related Compounds & Natural Toxins Task Force, and member of the Board of Mass Spectrometry Division – Italian Chemistry Society. He is also a leader of major EU projects, such as the EU-FP7 FoodIntegrity Project and the EU-H2020 MyToolBox projects. His impressive publication list includes five book chapters, 115 contributions at national and international conferences and 70 papers in international journals.

Here, Michele shares how he successfully crosses the academia–industry divide – and his interdisciplinary vision for the future.

How did you get into food analysis?

I studied analytical chemistry at the University of Ferrara, and my first research project was dealing with new plastic materials development, so taking a role in food contact material research at Barilla was a natural progression.

"My professional life has been always characterized by this swinging between academic and industrial research poles"

My work at Barilla, studying sensors and electronic noses for detection of "off-notes" in food packaging, eventually brought me back in contact with academia, and prompted me to develop my skills further by returning to study for a PhD in Innovative Materials Science at the University of Parma. I had enjoyed my time in industry though, and happily returned to Barilla after my PhD as Head of the Food Safety & Authenticity Research department.

My professional life has been always characterized by this swinging between academic and industrial research poles, trying to extract the best from both worlds, and establish a dialogue between them to achieve more challenging goals.

Why is food analysis so fascinating to you?

Food is a subject that is central to the entire history of the human race (and indeed all life on Earth), and for me studying food is a wonderful combination of passion, health, energy and sustainability, along with innovation and technology.

Describe your current work in a sentence

I work with international public and private research organizations on projects within the field of food chemistry, food safety, quality and authenticity, food contact materials, sensing and mass spectrometry applications for food products.

What projects are you working on now?

Over the past few years, I have been devoting substantial time to EU-funded projects dealing with both food safety and authenticity. In particular:


Barilla
Thermo
Q EXACTIVE
UltiMate 3000
0.000
R-A8 10.00
25.0
UHPLC

i. MyToolBox, which aims to mobilize the wealth of knowledge gained from international mycotoxin research conducted over the past 25–30 years (and perform cutting-edge research where knowledge gaps still exist) to create affordable and practical tools for farmers and processors along the food chain and so reduce the risk of mycotoxin contamination of crops, feed and food.
ii. FoodIntegrity, which aims to meet the need for new harmonized methods and reference materials, consolidation of expertise, sharing of data, and improved understanding of consumer behavior for earlier detection of food fraud worldwide.

What has been your biggest success – and biggest disappointment?

I think that life (and work) is always a climb to conquer something bigger and more beautiful. Over the years, I have collected many successes and rewards – but there have been many critical and challenging moments too. Organizing and chairing the last international conference of the FoodIntegity project in Parma last year was a particular career highlight. Conversely, my most disappointing moments are when I am unable to convince the company to hire brilliant young scientists who I have had the pleasure of supervising.

What are the pros and cons of working in industry?

Working in industry is very different to academia in some respects. What I really appreciate about industrial research is the concreteness of the targets, the meritocracy of the career path, the interdisciplinary team and the chance to disseminate the results to a huge variety of stakeholders.

Clearly, there are some limitations related to confidential/strategic know-how that have to be preserved and/or patented before being divulged to an external audience. But I feel that by working to improve food products eaten every day by millions of people, I am, in my own small way, making the world a better place.

How can we transfer knowledge between industry and academia?

Effective knowledge transfer needs a new generation of scientists that is used to moving between the two environments during the course of their professional life – and able to take the best from each.

Is it a challenge to balance applied research with more forward-looking work?

A big difficulty is the time pressure in industrial research – we are always trying to do projects in 1–2 years that should take 3–5 years, especially those that need basic development in academia before fine-tuning by industry. Globalization and social media is accelerating changes in consumer habits, so we are constantly challenged to keep up. The consequent need for flexibility is not always understood or accepted in the more rigid world of academia.

How do you you find the right compromise between confidentiality and sharing information?

The answer to this question is heavily dependent on the type of food research we are doing; confidentiality is highly relevant for scientists developing an innovative new recipe or food process. On the other hand, in the field of food safety and authenticity, sharing knowledge and information between different stakeholders allows us to act together to face emerging threats in a coordinated fashion.

What's next for you?

I love working to bring people better food every day. I get to combine the pleasure of discovering and implementing new research with the satisfaction of doing something useful for others. My plans for the future? I'd like to increase the number of young, interdisciplinary scientists in my research team, ideally creating a "hybrid" environment where academic and industrial scientists can come together and share ideas.

Emerging Analyses in Clinical and Food Industries Using Electrochemical Detection

Recently, new applications have been developed based on Electrochemical Detection (ECD) in high-performance liquid chromatography (HPLC). Here, we will explore two applications: 1. Fluorodeoxyglucose analysis for the clinical/diagnostic market 2. Lactose measurement in lactose-free products for the food/dairy industry

J-P. Chervet[1], H-J Brouwer, L. van Heerwaarden and N. Santiago[2] [1]Antec Scientific, Zoeterwoude, Netherlands [2]Antec Scientific (USA), Boston, MA, USA

1.Fluorodeoxyglucose (FDG) tracer for PET scan imaging

In PET imaging, the radio-labelled tracer 2-[18F]fluoro-2-deoxy-D-glucose ([18F]FDG) can be used for the assessment of glucose metabolism in the heart, lungs, and the brain as well as for imaging tumors in oncology. The 109.8 minute half-life of 18F makes rapid and automated chemistry necessary; therefore [18F]FDG is produced in a cyclotron in close vicinity of the PET facility. Prior to injection of [18F]FDG into a patient, it is necessary to perform a purity check and determine the actual concentration of the unwanted by-products: 2-fluoro-2-deoxy-D-mannose (FDM) and 2-chloro-2-deoxy-D-glucose (CDG). HPLC-ECD is the industry standard for this important test due to its ability to selectively detect [18F]FDG, FDM and CDG at very low concentration levels. Compendial methods based on ECD are described in both the U.S Pharmacopeia (USP) and European Pharmacopoeia (EP). These EP and USP methods are to a large extent similar and based on High Performance Anion-Exchange Chromatography (HPAEC) in combination with Pulsed Amperometric Detection (PAD).

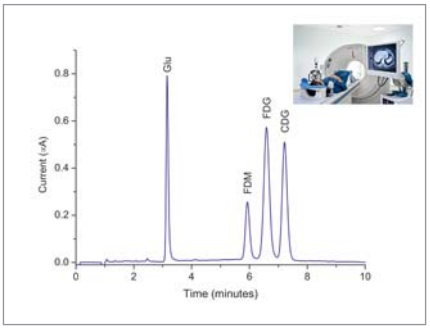

Figure 1. Chromatogram of a standard mixture consisting of 25 µg/mL FDG, FDM, CDG & 2.5 µg/mL Glucose (Glu) in water (20 µL injection). HPAEC-PAD using the ALEXYS™ Carbohydrate Analyzer equipped with SenCell™ (Antec Scientific).

2. Lactose content in lactose-free labelled products (lactose intolerance)

Lactose is a disaccharide that occurs naturally in the milk of mammals and is generally found to be around 5 percent (w/w). Other dairy products (like yoghurt and cream), and processed foods like sausages and cookies often contain lactose in detectable amounts. Lactose intolerance is a condition where a person cannot digest the normal levels of lactose present in the dairy/food products due to low levels of lactase in their intestine. Lactase deficiency results in various degrees of abdominal discomfort after consuming the products, depending on the amount of intake and lactase levels. As a result, the food industry has started producing various 'lactose-free' labeled products which contain decreased levels of lactose for consumers who would otherwise suffer from their intolerance.

In the past 'lactose-free' labeled products had levels of lactose below 100 mg/100 g product (0.1 percent), but nowadays it more generally indicates a lactose level below 10 mg/100 g product. These low levels of lactose in 'lactose-free' products require analytical methods with sufficient sensitivity. Current methods to detect lactose as described by the standardization agencies ISO (method 22662:2007; HPLC-RI) and AOAC (method 984.15; enzymatic/VIS) are not suited to test for such low levels; also here, the high sensitivity and selectivity of electrochemical detection makes HPAEC-PAD the technique of choice at lowered costs of operation and ownership (unlike LC/MS/MS).

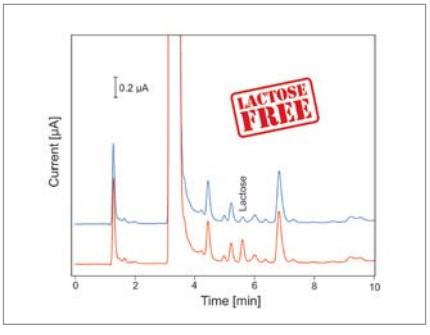

Figure 2. Top: Chromatogram of a 2.5 µL injection of a lactose-free milk sample (blue curve). Bottom: Chromatogram of lactose-free milk spiked with 3.3 µM Lactose (red curve). HPAEC-PAD using the DECADE™ Elite Electrochemical detector equipped with SenCell™ (Antec Scientific).

Analysis of Genuine, Recycled and Artificial Leather by Pyrolysis

Pyrolysis reveals components and differences between leather products

By Karen Sam

Pyrolysis-GC/MS allows for the analysis of organic materials that are too large to be compatible with GC alone. Material is heated in a controlled and reproducible way, facilitating breakdown into volatile compounds that can be studied by GC/MS. Results are polymer specific, making pyrolysis the perfect way to analyze all types of polymers, including natural and synthetic textiles, such as leather and artificial leather.

Genuine leather is created by tanning rawhides from cattle. The main constituent of animal skin is a protein called collagen. The building blocks of protein are amino acids, so the pyrolysis of collagen results in many ring structures such as pyrroles and indoles. Even though genuine leathers are all made from similar raw material, pyrolysis could still unveil subtle difference between different manufacturers by comparing the additives and contaminants. Figure 1 shows a collagen standard and two genuine leather samples pyrolyzed under the same condition. Comparing to the standard, one leather sample uses terephthalate plasticizer, and the other one has a phthalate plasticizer.

Recycled leather is generally considered the lowest grade of genuine leather and looks identical to genuine leather. When pyrolyzing recycled leather, a large amount of polyisoprene's monomer and dimer is observed in Figure 2. This indicates the recycled leather was made from scraps of leather and glued together with a polyisoprene adhesive.

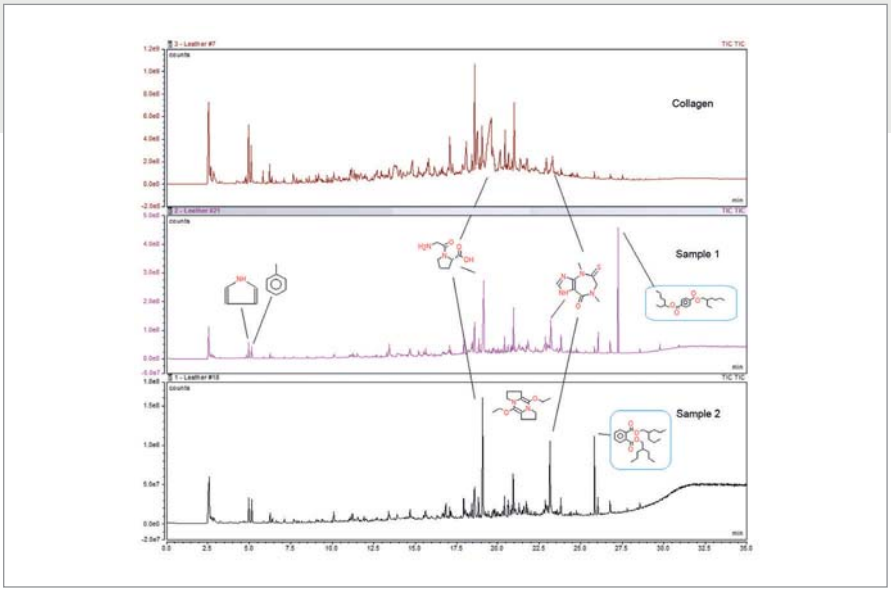

Figure 1. Pyrograms of collagen standard (top), genuine leather sample 1 (middle), genuine leather sample 2 (bottom). Sample 1 has terephthalate plasticizer, and sample 2 has a phthalate plasticizer (plasticizers circled in blue).

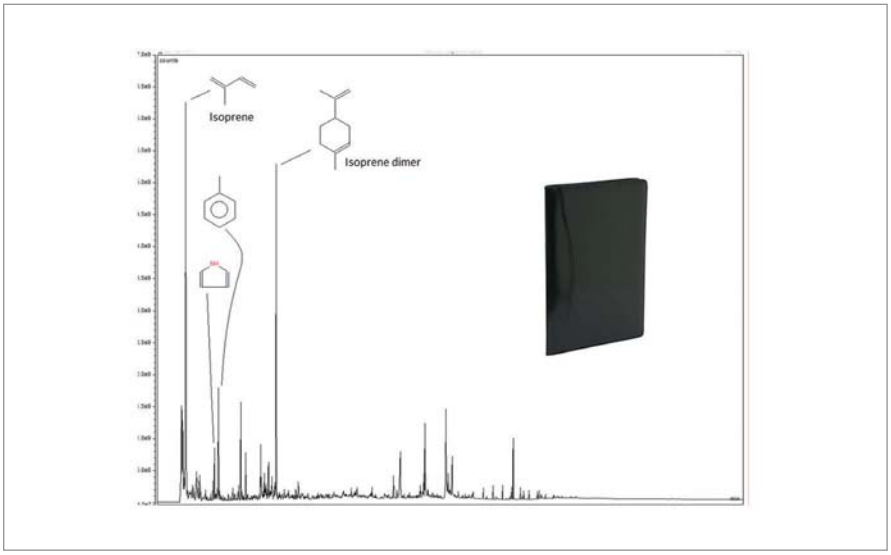

Figure 2. Recycled leather sample shows Isoprene monomer and dimer.

Finally, two artificial (faux) leather samples are analyzed. These samples are cut from two wallets that differ only in color (red versus orange). Pyrolysis reveals that they are both acrylic as shown in Figure 3. The red wallet contains butyl methacrylate, and the orange wallet has butyl acrylate.

In summary, pyrolysis-GC/MS, carried out in a CDS 6000 Pyroprobe with Autosampler, is proven to be powerful tool in screening different leather products.

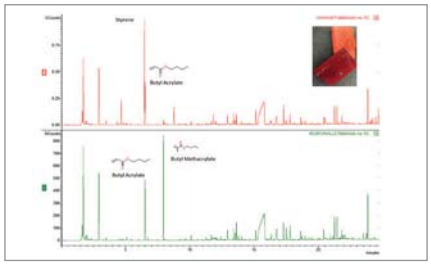

Figure 3. Two artificial leather samples that are different in color. Top is orange and the bottom is the red.

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Rapid Analysis of Volatile Compounds in Paperboard Using Direct MS

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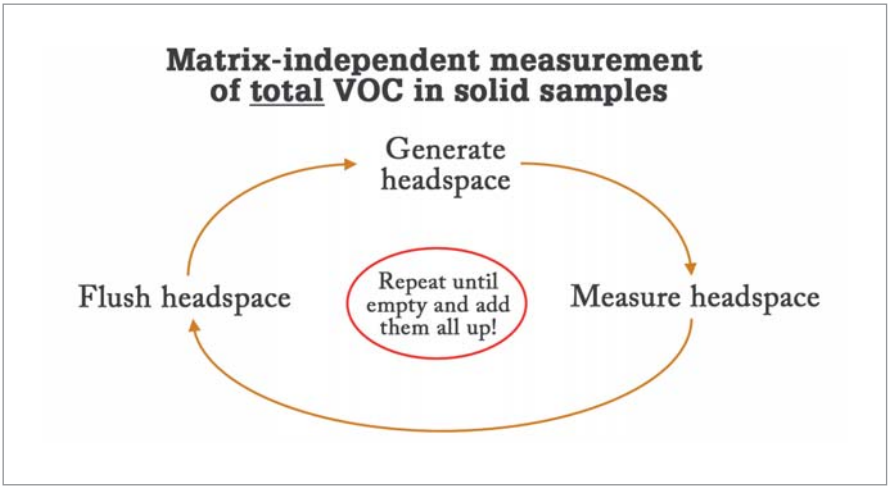


Figure 1. Schematic representation of the MHE technique.

Pharmaceutical and food products are susceptible to contamination from packaging volatiles – whether from polymeric materials, printing inks, or paperboard. VOCs can also migrate through multiple layers of packaging, so it is critical to analyze materials regularly.

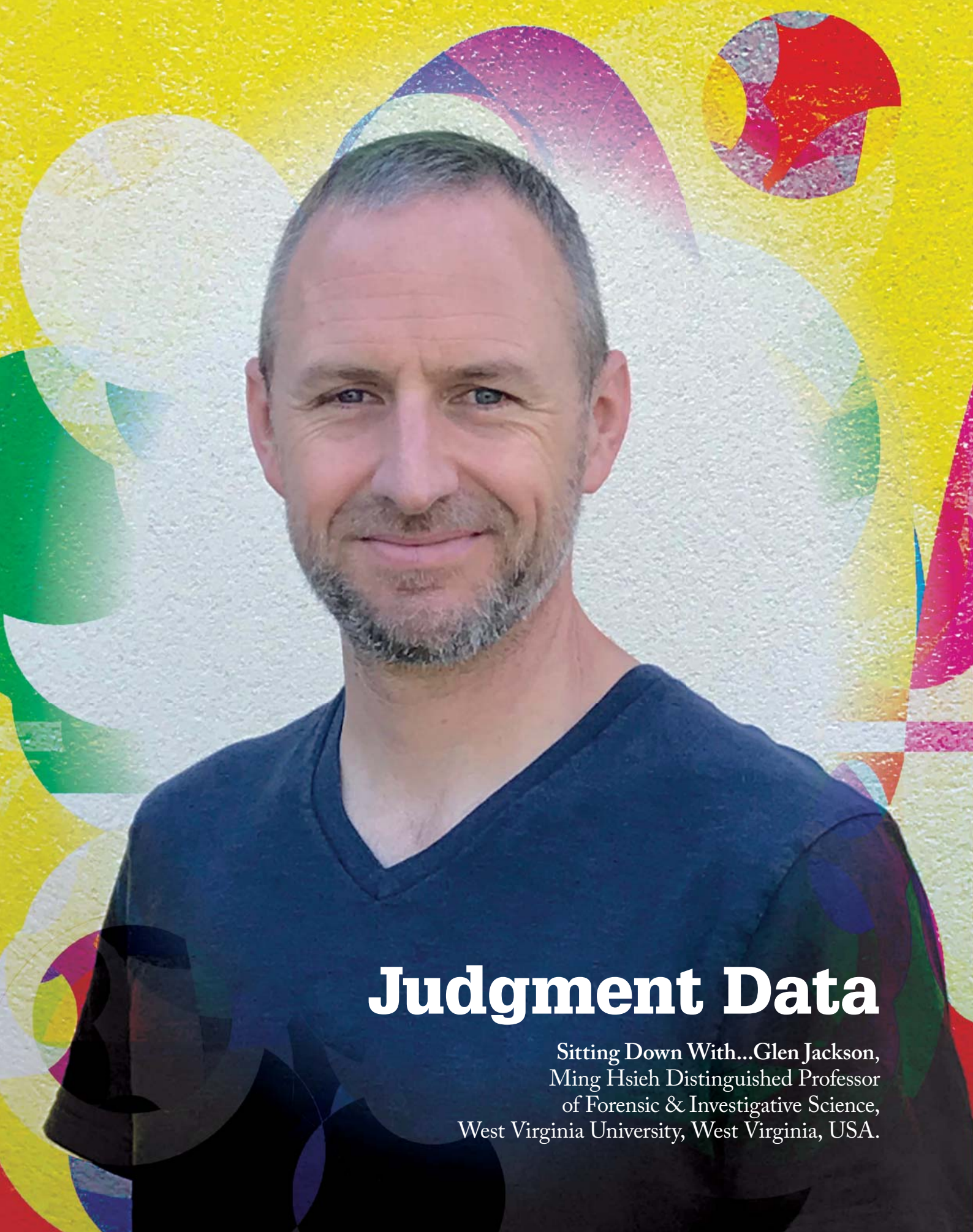
Traditional approaches utilize static headspace analysis, but it is difficult to relate these results to actual quantities of volatiles in the packaging due to matrix-dependent interactions. The MHE technique circumvents this issue by calculating the total concentration in the product from a limited number of consecutive headspace analyses (Figure 1). Typically six cycles are utilized in complete analysis of one sample, which makes it a very costly technique when coupled with gas chromatography-mass spectrometry (GC-MS). By utilizing rapid SIFT-MS measurement instead, headspace regeneration becomes the rate-limiting step and multiple samples can be analyzed in parallel.

The data shown here (Table 1) were obtained using a Syft Technologies Voice200*ultra* SIFT-MS instrument integrated with a GERSTEL Multipurpose Sampler (MPS) (GERSTEL, Mülheim an der Ruhr, Germany) equipped with a GERSTEL agitator/incubator and headspace vial racks. Replicate paperboard samples (linear dimensions 210 x 40 mm; mass 1.3 grams) were placed in four 20-mL headspace vials and incubated at 75 °C for 20 minutes, followed by a 3-minute post-measurement flush. Headspace was sampled with a 2.5-mL headspace syringe heated to 150 °C, and injected at a flow rate of 50 µL s^{-1} into the SIFT-MS instrument's inlet (total flow rate of ca. 420 µL s^{-1}).

This study demonstrates that MHE-SIFT-MS is a very powerful new technique for rapid determination of volatile compounds in paperboard. Not only does SIFT-MS provide a four-fold increase in sample throughput compared to MHE-GC-MS, but it also broadens the range of compounds detectable in a single analysis, quantifying polar species such as the small aldehydes without any need for derivatization or pre-concentration.

Parameter	*Formaldehyde*	*Acetaldehyde*	*Propanal*	*Butanal*	*Pentanal*	*Hexanal*	*Heptanal*	*Octanal*	*Nonanal*	*Decanal*	*Methanol*	*Ethanol*	*Acetone*
Rep 1	0.088	0.164	0.043	0.088	0.218	1.338	0.102	0.108	0.100	0.035	1.954	0.079	0.328
Rep 2	0.107	0.182	0.050	0.088	0.237	1.429	0.113	0.115	0.108	0.037	2.012	0.082	0.345
Rep 3	0.078	0.154	0.045	0.083	0.226	1.397	0.102	0.111	0.095	0.028	1.893	0.074	0.341
Rep 4	0.093	0.164	0.048	0.093	0.227	1.361	0.102	0.112	0.100	0.030	2.267	0.086	0.362
Mean	0.091	0.166	0.047	0.088	0.227	1.381	0.105	0.111	0.101	0.032	2.032	0.080	0.344
SD	0.011	0.010	0.002	0.003	0.007	0.035	0.005	0.003	0.005	0.004	0.142	0.004	0.012
%RSD	11.8	6.1	5.2	4.0	2.9	2.5	4.3	2.3	4.7	11.4	7.0	5.6	3.5

Table 1. Concentrations of volatiles (in µg g^{-1}) found in a paperboard sample using MHE-SIFT-MS. Concentration data, the mean, standard deviation and RSDs for the four replicate analyses are shown.



Judgment Data

Sitting Down With...Glen Jackson,
Ming Hsieh Distinguished Professor
of Forensic & Investigative Science,
West Virginia University, West Virginia, USA.

How did your interest in analytical chemistry begin?

I always loved science, but it was as an undergrad that I got hooked on mass spectrometry. I found it incredible that you could measure a property so precisely there could only be one substance that matched. After that, I looked for any and all opportunities to get involved with MS. For my PhD, I worked with Fred King, an expert on pulsed glow discharges. He did both optical spectroscopy and MS, and I knew I would get a great experience with him. I spent the first two years studying pulsed glow discharge plasmas using optical spectroscopy, and then I did a two-year internship at Oak Ridge National Laboratory studying pulse glow discharges with MS.

When did you go down the forensics route?

I was hired by Ohio University to be involved with their Forensic Chemistry program – to do research, direct PhDs, and teach the undergraduate classes. It was a new area for me, so I did everything I could to become an expert: I went to conferences, visited crime labs, taught GC-MS workshops to get one-on-one time with forensic practitioners... I really became embedded within the community, and from there, started to develop research projects related to forensic chemistry. Being able to teach techniques I enjoy alongside an application that everyone finds appealing is a joy.

You once described the forensics field as "conservative"…

It has to be conservative because the stakes are so high. Having said that, as part of high-level committees like NIST OSAC (National Institute of Standards and Technology - The Organization of Scientific Area Committees for Forensic Science), I get to communicate with many people trying to drag the discipline into the future. To move from the current safe zone will require the implementation of new methods, technologies and capabilities. In the future, I see more advanced chemometrics or statistics coming into chemistry – we need to get more intelligent about what to do with the data we have and how to interpret it.

How does that translate to the courtroom?

Historically, neither prosecution nor defense lawyers have known what kind of forensic evidence to ask for in court, or whether it was good science. Bad science is used over and over in the courtroom just because it has precedent. We need documents that explain each type of forensic evidence in a way that non-scientists can understand – the science behind it, how it meets Daubert/Frye criteria, why it ought to be admissible, how it should be applied properly, and what conclusions one can and cannot draw.

Does your role as Editor-in-Chief of Forensic Chemistry help move things forward?

I hope so. When we established the journal, we wanted to give authors academic freedom. We cover pretty much anything that could be used in a crime lab – but that doesn't mean that it ever will (or should) be used. For example, submissions might cover something fundamental, such as understanding the kinetics of pyrolysis of a material – it is several steps away from being useful now, but ultimately could improve fire debris analysis. Or a unique instrument that we know won't make it to a crime lab for 20-plus years. We're trying to inspire people with what's possible. At the other extreme, we accept very practical and implementable research like inter-laboratory method validations. Such research is not necessarily novel, but it's extremely important for the community.

What projects are you working on?

We are working on a really cool experiment – an interesting mix of fundamental and applied chemistry – on the evaporation of gasoline, which is important for fire debris analysis. We're using first principles and fundamental knowledge about volatility and evaporation rates of substances to understand the way gasoline will behave in a real fire scene at extremely elevated temperatures – and what we're finding is really interesting. We're pursuing other forensic research; for example, chemically determining the food source of certain blowflies, which is important in terms of forensic entomology. We're also trying to understand the mass-spectral fragmentation patterns of cathinones, certain synthetic cannabinoids, and opiates like fentanyl to see if we can help practitioners interpret their spectra. Our work will help predict the types of spectra that are likely to come from drugs in the future.

What drives you?

Micro-epiphanies! For example, I've believed for more than a decade that the way we analyze mass spectra doesn't do them justice – our algorithms are inadequate. I finally had a 'Eureka!' moment earlier this year, and we're now working on a better way to interpret spectra. It is tremendously intellectually exciting to have an idea that you know ought to work, then figure out how to use the math to match your intuition. Of course, the ultimate goal (and the hardest part) is to share that new knowledge in such a way that you inspire other people to implement it. I live for that totally engrossing opportunity to advance science. It's amazing to get paid to have an idea and then pursue it.

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