NOVEMBER 2020 # 92

the Analytical Scientist®

Holding a Mirror to Analytical Science

Racism is systemic in science – and analytical chemistry is no exception

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The Time for Change

Now is our chance to embrace a new era in analytical chemistry – one free of systemic racism

Systemic racism is the implementation of systems, procedures, and/or structures that disadvantage minority groups. These systems include, but are certainly not limited to, education, employment, housing, healthcare, politics, and the criminal justice system. Despite what some claim, science is not exempt from systemic racism. Scientists often bring their unconscious (or conscious) biases into their body of work and workplaces; even this year, we witnessed the controversy – and subsequent withdrawal – of Tomas Hudlicky's *Angewandte* article (1). Scientists of color should not be tasked with the additional burden of addressing microaggressions by senior professionals, remitting blatant acts of racism in the workplace, or steering around structures that encourage career stagnation.

The USA, Europe, and Australia are still grappling with histories entrenched in the oppression of minorities. The gut-wrenching, whirlwind of events during 2020 resulted in an uproar of protests that brought systemic racism – through police brutality and the disparate impact of COVID-19 on minorities – to the forefront. But a glimpse of hope shone through. People from all walks of life participated in the protests and engaged in social media movements, such as #BlackintheIvory, #ShutDownSTEM, and #ShutDownAcademia, demonstrating the level of support for such actions. We witnessed scientists and scientific organizations – who were once unaware or refused to acknowledge the existence of systemic racism – make statements in support of diversity initiatives and charges to be more inclusive. Institutions in the USA, UK, and the Netherlands have even begun implementing policies to rectify systems that were once created with racist intentions.

These efforts are appreciated – but they are only the initial steps to right previous wrongs. We need more organizations and institutions to become accountable for their actions. There is no quick fix to ending systemic racism, and there is still much to be done. We must call on all organizations to implement anti-racist initiatives and discontinue racist rhetoric and ideology. We must acknowledge and consider how those institutions that financially benefited from disadvantaged minorities can provide reparations. And we must also ensure we promote the equity of all people groups within the field of analytical chemistry – not just focus on gender equality.

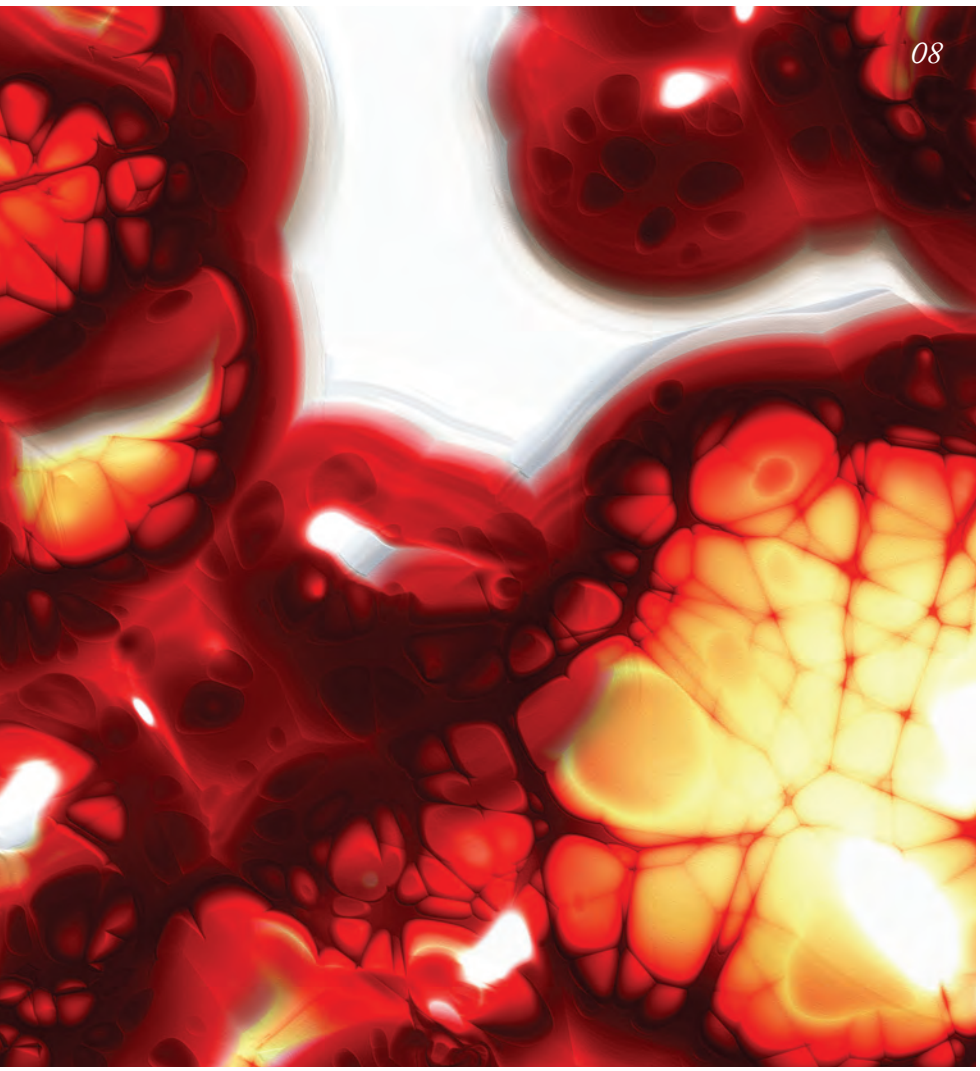
In honor of creating a more inclusive and equitable environment in the sciences, The Analytical Scientist provided a platform to vocalize the unique experiences of Black scientists, publicize our suggestions for change, and welcome this much-needed transition to a new era – one free of systemic racism.

Candice Z Ulmer
Christina M Jones
Michelle E Reid

Reference

1. *Chemistry World, "Angewandte essay calling diversity in chemistry harmful decried as 'abhorrent' and 'egregious,'" (2020). Available at: https://bit.ly/3dNME5A*

Contents



On The Cover

Candice Ulmer, Michelle Reid and Christina Jones take center stage for our November issue – leading the discussion around systemic racism in analytical science.

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Road to Pollution

Have we overlooked the role of asphalt as a source of air pollution precursors in urban environments?

As our urban air quality improves – thanks to decades of research and regulations – it's possible we have overlooked a significant source of pollution: our roads. In a recent study, researchers at Yale University suggest that asphalt could be a major contributor to non-combustion-related pollution in urban areas.

The team was already looking at the role of non-traditional sources of reactive gas-phase organic compounds on urban air quality, when they decided to focus their attention on larger organic compounds in the intermediate and semi-volatile range. These are important for secondary organic aerosol formation, but less is known about their emissions from non-combustion sources, such as asphalt.

The team used GC with high-resolution time-of-flight MS (with soft ionization) to resolve the complex mix of asphalt-related emissions. "The combination allowed us to observe the GC-separated mixtures and chemically speciate them to a degree that had not been previously possible," says Drew Gentner, co-author of the paper.

The results show that asphalt-related materials are a significant urban source of larger organic compounds that lead to the formation of secondary organic aerosols. These aerosols are a key component of PM2.5 particulate matter – one of the most detrimental forms of air pollution to human health.

Asphalt-related emissions were dependent on temperature and exposure to the sun – perhaps not unexpected; however, the researchers were surprised at just how significant the contribution was. "We saw large increases even after 46 hours of prolonged heating," says Gentner. "And that not only shows the potential for continued emissions well after initial application, but emphasizes the importance of looking at the entire life cycle of asphalt-based materials." This extends from storage to application and through the lifetime of its use.

But asphalt is just one part of the urban air quality puzzle. Personal care products, cleaning products, and paints are also major contributors of reactive emissions. "Our goal is to improve our understanding of the complete mix of urban sources of intermediate- and semi-volatile organic compounds," says Gentner. "Such knowledge is important as the field tries to constrain the full, diverse range of non-combustion-related sources that are increasingly affecting urban air quality."

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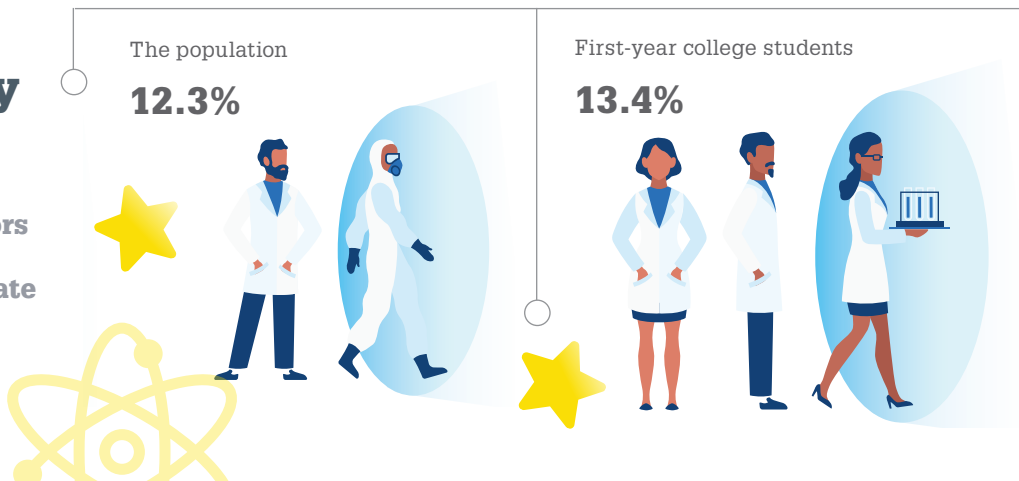
INFOGRAPHIC

Black Chemistry Progression

Very few chemistry professors at top US universities are Black – despite a proportionate number of Black undergrads



BUSINESS IN BRIEF

A roundup of the latest business news in analytical science, from COVID-19 commitments to instrumental innovations

- Knauer Wissenchaftliche Geräte GmbH and its new subsidiary, Das Labor GmbH, have started producing reliable COVID-19 antibody tests for sub-Saharan Africa in cooperation with Deutsche Gesellschaft für internationale Zusammenarbeit (GIZ) GmbH. It's hoped that 50 percent of the tests will be provided at cost price.
- J.A. Woollam is working with Linkam Scientific to bring precise temperature control to its ellipsometry instruments, enabling their use by thin-film researchers across applications.
- Thermo Fisher Scientific has expanded its COVID-19 testing portfolio with two further tests: the OmniPATH COVI-19 Total Antibody ELISA Test and the EliA SARS-CoV-2-Sp1 IgG Test. The first was granted emergency use authorization by the FDA; the second is available for commercial use.
- Bruker has announced its acquisition of Canopy Biosciences, LLC, a leader in multiplex biomarker imaging. The partnership aims to strengthen Bruker's capabilities in targeted multiomics and multidimensional immune profiling.
- Waters Corporation has introduced its ACQUITYTM PREMIER columns, a new family of sub-2µ columns for use with UHPLC systems. The columns are designed "for the analytical laboratory seeking to exercise greater control over chromatographic separations."

SARS-CoV-2 antibody test to be produced in Côte d'Ivoire. *Photo: Das Labor.*

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3. *ThermoFisher Scientific (2020). Available at: https://bit.ly/34dYnaa.*
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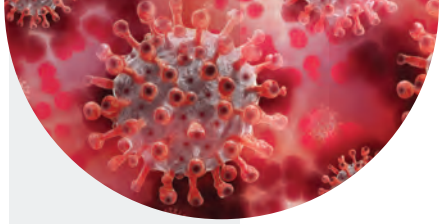

Grappling with Glycoproteins

Could an improved SARS-CoV-2 glycan analysis tool help us combat COVID-19?

Heavy glycosylation of the SARS-CoV-2 spike protein may play a role in immune evasion – partly owing to the presence of host glycan molecules. Thus, quick, simple and reliable methods to screen these glycans are needed. And that's exactly what Rod Chalk and colleagues set out to do…

"We analyzed short spike glycopeptides using LC-ToF-MS to ascertain both structural and positional information," says Rod. "LC-ToF has the advantage of generating signals from any ionizable species." The team identified 140 glycopeptides belonging to 13 glycoprotein sites, with a further six sites unassigned. Accurate mass and retention times were also determined for a further 306 glycopeptides. "Characterization of the spike protein is an essential contribution to our community's multi-pronged approach to fighting the pandemic," Rod explains.

Next up: taking the studies from duplicate to triplicate and extending the number of glycans detected.

Reference

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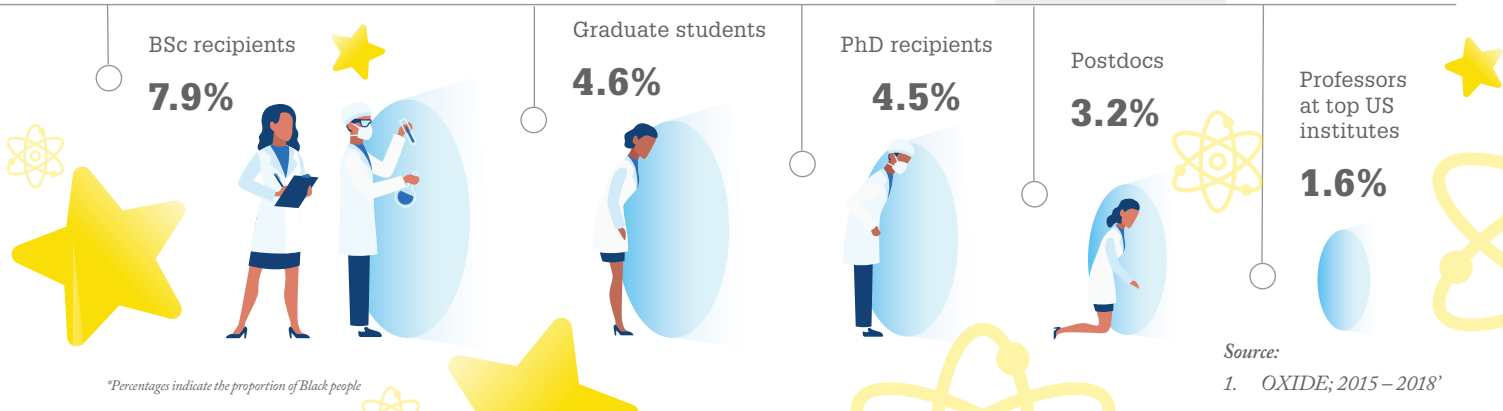
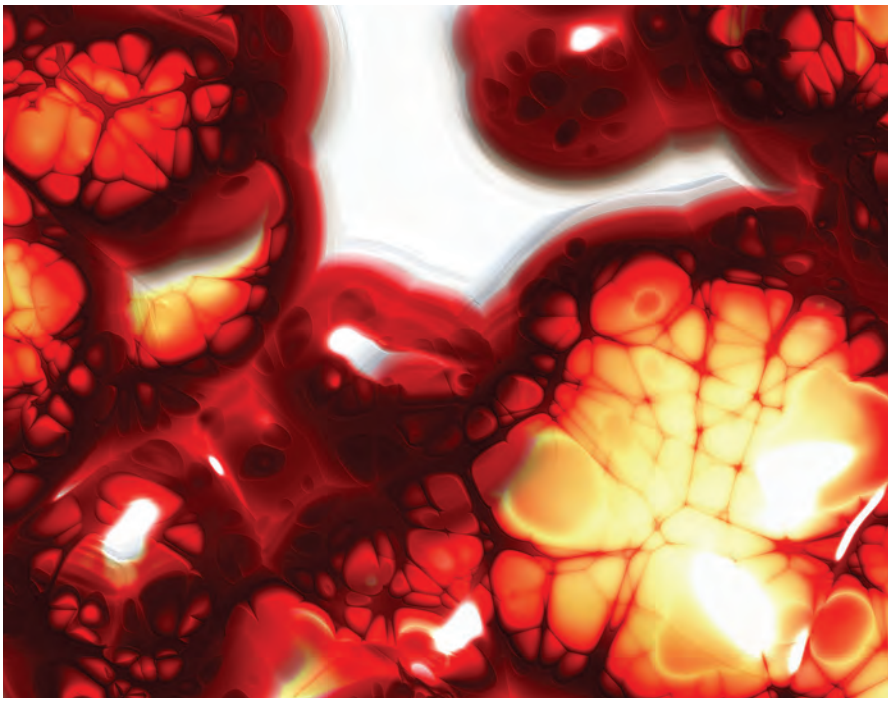


**Percentages indicate the proportion of Black people*

Source:

1. *OXIDE; 2015 – 2018'*



Biomarkers for Bacteremia

Using proteomics to predict mortality risk of Staphylococcus aureus bacteremia

Staphylococcus aureus bacteremia is a major cause of illness and death worldwide – and one that's tricky to diagnose early. The longer the infection goes untreated, the more a patient's risk of death increases. With this in mind, David Gonzalez and colleagues – proteomics specialists at the University of California San Diego School of Medicine – investigated whether proteomic and metabolomic readouts from a patient's blood sample could serve as predictive markers of response to infection – and, ultimately, risk of mortality.

"The faster we know what's going to happen to our patients, the better we can treat them," said George Sakoulas, a co-author of the study (1). Over the course of two years, the team worked to identify serum proteins and metabolites in patients' blood that could predict those most at risk of death from *S. aureus* bacteremia (2). By analyzing over 10,000 proteins and metabolites in more than 200 serum samples, they found that patients who died of *S. aureus* bacteremia exhibited a different pattern of proteins to those who survived.

The biomarkers most associated with death were lower levels of glycosylated fetuin A, unmodified fetuin B, and thyroxine, and higher levels of serum protein carbamylation. But an unanswered question remained: Do the differences actively cause increased mortality? Using a mouse model to investigate thyroxine levels, the team administered either hypo- or hyperthyroid treatment to infected mice and monitored survival rates. Compared with the control group, hyperthyroid mice had a survival rate four times higher 48 hours after infection, whereas hypothyroid mice exhibited decreased survival – suggesting that thyroxine levels directly affect disease outcome.

"This finding is a leap forward toward a point-of-care predictive tool for bacteremia risk," said Gonzalez (1). "It also opens up lots of new basic biological questions about how our immune systems respond to infections."

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Frontline Food Quality

Evaluating the feasibility of NIR spectroscopy for *in situ* analysis

Efficient analytical tools and control systems are vital to ensuring food integrity, but traditional methods are often slow and costly.

In a recent paper, researchers evaluated whether near-infrared spectroscopy could be used to determine the nitrate content of spinach plants in the field – and along the supply chain. "Our results confirmed that using this spectrophotometer offers an instantaneous, non-destructive, and cost-effective approach to quality and safety analysis of large numbers of samples throughout the supply chain," says Dolores Pérez Marín, co-author of the paper. "Such technology could transform the role of quality assurance from strictly conformance, to one that addresses a wide range of business critical concerns."

Though the paper focuses on its use in vegetable analysis, the authors suggest it could have wider applications.

Reference

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Spot the Fakes

In a recent exhibition on a taboo topic, the Museum Ludwig has revealed the results of a systematic investigation of over 100 paintings from the Russian avant-garde – a movement well known to be afflicted by forgeries. Jilleen Nadolny and her team at ArtDiscovery examined 14 paintings by prolific artist-couple Mikhail Larionov and Natalia Goncharova to build a detailed body of technical information against which forgeries can be compared. Their multi-analytical approach includes Raman microscopy, SEM-EDX, and polarized light microscopy. The image above shows a short-wave infrared photo of The Orange Seller by Goncharova, revealing the small pentimenti – or underdrawings – in the face.

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

QUOTE OF THE MONTH

"Follow your passion. Don't allow negative thoughts from yourself or others, your ethnicity, your location, environment or gender to be a limitation to what you can achieve."

Marian Asantewah Nkansah, Associate Professor, Department of Chemistry, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana.

Weeding Out Whiskey Wannabes

How spectroscopy helps authenticate whiskey – without wasting a precious drop

According to the European Union's Intellectual Property Office, counterfeit drinks cost the EU €1.3 billion in lost revenue each year. Kishan Dholakia and his team from the University of St Andrews in Fife, Scotland, set out to find a way to catch the crooks in a non-destructive manner – saving the liquor for the whiskey lovers.

They used fluorescence and Raman spectroscopy to collect the unique spectral signatures of different whiskeys. Importantly, the new method allows the spectra of the golden liquid to be directly collected from within the glass bottle.

"We generated a unique beam that looks like a ring on the glass surface and then focuses to a concentric set of rings with a small spot inside the liquid," says Dholakia. The approach is more straightforward and accurate compared with similar techniques. And it can be applied to other alcoholic beverages (including gin, vodka, and wine) or olive oil – another commonly counterfeited commodity. In the future, the team hopes to use the same principle for collecting Raman spectra at depth in biological tissues.

Reference

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In My View

Experts from across the world share a single strongly held opinion or key idea.

In My Skin

Analytical scientists from underrepresented groups do not have an easy route through academia – it is time we all understand their challenges so that we can establish positive, inclusive change

By Bhavik Patel, Professor of Clinical and Bioanalytical Chemistry, University of Brighton, UK

Recent coverage of the Black Lives Matter movement, and the events that have transpired as a result, have hit home with many analytical scientists from underrepresented groups – reminding them of the challenges they too have faced while progressing through academia.

Notably, these challenges have not surfaced overnight. There is clear evidence that Black, Asian and Minority Ethnic (BAME) scientists are less likely to be in leadership positions (1), less likely to be nominated or given prizes and/or awards, and have much lower success rates for grant proposals than their white colleagues (2). Furthermore, the ACS Division of Analytical Chemistry recently highlighted that, over the past decade, they received very few nominations from underrepresented groups (3).

This systemic racism should be enough to explain why many do not feel a sense of belonging within the wider STEM community, but, to add to this, many minority analytical scientists are also subjected to racism, tokenism, and microaggressions throughout their career. When I first started my career in analytical chemistry at the start of the millennium, I remember a strong feeling that there were seldom any role models that looked like me. And I think it's important to not underestimate the impact of this issue – it can leave many feeling like an outsider as soon as they commence a career in the field.

I was lucky – many of my mentors did not treat me any differently and have been highly supportive throughout my career. But I've also had to endure a sense that some have never really considered me as a "real" academic. I recall attending many analytical chemistry conferences in the UK and US, where attendees would think I was the audio visual technician, or speakers would ask me if I would be happy to upload their presentation. At the time, you laugh it off, but such interactions tend to stick with you.

Even now, as a senior academic, I

"I was lucky – many of my mentors did not treat me any differently and have been highly supportive throughout my career."

encounter people who might justify my achievements as tokenistic gestures to help support race equality. Imagine experiencing decades of this inappropriate behavior whilst striving to remain a professional at all times. And if minority analytical scientists raise these issues? They will often be dismissed or considered "troublemakers."

So what can the analytical community do? Changes are already being made; for example, The Analytical Scientist's 2020 Power List experimented with a new format to increase diversity. However, though this will certainly help with increasing the diversity of the list overall, many minority analytical scientists in the Western world are still likely to be overlooked. The challenges facing our field are vast and systemic; to overcome them first requires acknowledgement that these challenges exist and an acceptance that there is still a long way to go.

We cannot leave the responsibility of eradicating racism to BAME scientists – we need our white allies to stand up and call out any abhorrent behavior. The first step in being an active ally is to educate yourself about the lived experience of those from underrepresented groups. It is then critical that you stand up against any forms of racism and use your position of privilege to support and mentor minority analytical chemists, so they can be considered for the same opportunities.

Key figures in the field, such as those highlighted in the Power List, can also use their positions of leadership to help amplify those voices without a platform to be heard. In my view, it is key that our entire analytical science community come together and endorse the fact that diversity and inclusivity enriches our community. We must fight to build and then ensure we sustain this diversity and give hope to young scientists beginning their careers in analytical science.

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A Passion for Discovery

"Discovery research" has often led us to unprecedented findings – including the identification of unusual contaminants from wildfires in California. Here, we share our passion for this special approach to (instrumental) analysis.

By Mike Thurman, Director, and Imma Ferrer, Chief Analyst, the Laboratory for Environmental Mass Spectrometry, University of Colorado, USA

First off, it's worth explaining what we mean by "discovery research." To us, as analytical chemists, discovery research is about letting the instrument guide us to new and interesting research problems. The approach was first introduced to us by Nobel Laureate,

Linus Pauling, during a lecture Mike attended 35 years ago at the University of Denver. Pauling described his PhD research in humble terms:

"Bragg and his son had just received the Nobel Prize in 1915 using an X-ray diffractometer to determine the basics of crystalline structure. We had an X-ray instrument in our lab, so I went to the chemical storehouse on campus, asked what they had on the shelf, and set to work using X-ray diffraction to understand crystalline structure."

Pauling went on to say that discoveries simply "opened up in front of him," including the relationship between crystal structures and the chemical bond. This initial work eventually led to him writing one of the most important books in general chemistry of the 20th century: The Nature of the Chemical Bond. For us, as environmental chemists, that special instrument is the LC/ QToF-MS with accurate mass. And our current work on the nature of organic compounds in ash and water from wildfires is a good example of the discovery research approach.

Back in 2017, Mike was walking through the lab and saw a bright orange solution sitting on the countertop. Out of curiosity, he asked a colleague's postdoc what it was. She explained that she had just leached a burned soil with water and concentrated this solution on an XAD adsorbent. Having done his PhD thesis on natural organic matter on XAD resins, Mike knew yellow was common in such research, but Kool-Aid orange? This was unheard of. Though it was commonly accepted that humic substances from soil could not be

unequivocally identified – even by FT-MS – we decided to give it a shot.

The results were shocking. There were multiple, large peaks with low masses (less than m/z 250) and a low mass defect of 0.0050 – a typical humic-like compound has a mass defect greater than 0.1500. Within a few minutes we had identified several organic acids called benzene polycarboxylic acids (BPCAs). A quick check in the literature found that these compounds were used to identify black carbon, a name for the unknown dissolved organic matter resulting from wildfires. Earlier this year, we published our first paper (1) with these results – the first time BPCAs have been reported in water-extractable organic carbon from thermally altered soil – and we have a second paper on the topic in the pipeline. Overall, we've reported over 10 different classes of organic acids, the majority of which are original results.

The ramifications for water treatment are enormous. What happens with runoff to drinking water? What about the chlorination, ozone, UV, and peroxide treatment of these organic acids? On top of all these new areas for concern: are the substances actually toxic to wildlife and humans in water and ash? This question could not be more pertinent, as clouds of ash currently descend on our cities in California, including Los Angeles, San Francisco, and San Diego. A recent publication even suggests that benzene and naphthalene – possible carcinogens – are present in water from wildfires, most likely a continued decarboxylation of the BPCAs we found in our samples...

Out of that single curious experiment with some strangely colored water has emerged plenty of new research ideas for us to consider in the future. Our fire and ash study is just one example, but we've used the same approach to tackle many other unanswered questions, such as what is in flowback water from hydraulic fracturing (2)? Or what new pharmaceuticals are in wastewater (3)?

We think it's exciting to see the world through the eyes of a beautiful instrument, and that's why we're so passionate about discovery research. Occasionally, we might hear colleagues say that it's just a matter of measuring some compound in a water sample. In fact, the art of analysis (or an analytical scientist) is in turning a simple measurement into a whole new area of research – and we think that's a noble pursuit indeed.

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Taking MALDI to the Next Level

The rise of MALDI has historically been hampered by inherent challenges – fresh innovations could open the door to new application areas and opportunities

By Shannon Cornett, Global MS Imaging Market Manager, Bruker Daltonics, Billerica, MA, USA

I think it's fair to say that MALDI-MS imaging has been a transformative technique when it comes to understanding the molecular makeup and regional heterogeneity of biological tissues. Its label-free nature and ability to differentiate compounds by molecular weight make it suitable for many applications – from drug discovery to pathology and biomedical research.

And yet, it's also fair to argue that MALDI imaging faces inherent challenges – namely, ion suppression, sensitivity at higher spatial resolutions, and quantification dynamic range – which technological advances have failed to address. Until now.

Full disclosure up front: I work for Bruker Daltonics and – as its Global MS Imaging Market Manager – I have a clear interest in the continued rise of MALDI. However, I truly believe that new technology now on the market will be game changing when it comes to the applicability of MALDI. Take, for example, the lower sensitivity when increasing resolution, we adapted MALDI-2 – laser-based post-ionization (PI) that increases the sensitivity of MALDI imaging by up to three orders of magnitude (dependent, of course, on the sample, matrix, and analyte). The upside of a sensitivity boost? Opening the door to studying compounds typically inaccessible to traditional MALDI, including many smaller molecules, such as lipids, vitamins, and

glycans (1), as well as expanding the number of molecular channels available for untargeted tissue subtyping.

The downside? More ion signals result in more complex spectra. Here, a second recent innovation for MALDI – trapped ion mobility spectrometry (TIMS) – proves invaluable by adding the dimensionality of Collisional Cross Section (CCS) to separate the complex signals by CCS as well as m/z. Using TIMS we routinely observe dozens of near-isobaric ion signals from tissue cleanly separated by CCS and m/z that would otherwise not be resolved by m/z alone. An additional benefit for TIMS is that CCS is an intrinsic property of the molecule and therefore can be used in combination with m/z to more confidently identify the compound.

In my view, the powerful combination of MALDI-2 and TIMS could increase adoption of MS imaging in a number of application areas – but (bio)pharmaceutical research has much to gain in particular. By integrating the analysis of tissue, cells, and body fluids for a deeper understanding of disease mechanisms, researchers can develop better biomarkers, monitor more drug compounds and their metabolites, and work towards truly personalized medicine.

For example, the additional sensitivity of MALDI-2 is particularly significant for targeted drug and drug metabolite imaging for Drug Metabolism and Pharmacokinetics (DMPK) studies. As demonstrated in one study, a serial dilution of five pharmaceutical compounds – caffeine, chloroquine, rosuvastatin, reserpine and BI-YYY (a new drug compound) (2) were spotted onto control tissue and analysed using MALDI and MALDI-2. Results showed lower detection limits for MALDI-2 for all five compounds. In particular, the peak intensity of BI-YYY was enhanced by a factor of 300 by MALDI-2.

As part of the same study, liver and kidney tissue sections from rat dosed with chloroquine were imaged to compare MALDI vs MALDI-2. Chloroquine and metabolite images were more intense using MALDI-2. Given this degree of enhancement, MALDI-2 has the potential to deliver new distribution information from previously undetected metabolites as well as providing lower limits of detection for target compounds - both vitally important to DMPK studies. In addition, hundreds of other signals are available in the same datasets to explore pharmacodynamics.

In short, the adaptation of MALDI-2 onto the timsTOF technology yields a synergistic pairing. MALDI-2 produces ion signals in greater abundance and complexity, whilst TIMS has the capacity to resolve the additional signals with intrinsic CCS values that improve confidence of molecular identifications. Together, timsTOF fleX with MALDI-2 promise to broaden the scope of imaging studies of small (bio)molecules that were previously undetectable with traditional MALDI imaging.

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HOLDING A MIRROR TO ANALYTICAL SCIENCE

Racism is systemic in science – and analytical chemistry is no exception. Lauren Robertson spoke to Candice Ulmer, Christina Jones, Michelle Reid, Isiah Warner, Devin Swiner, and Renã Robinson about their own experiences in the field, their efforts to combat the problem, and what still needs to be done.

A Network FOR PROGRESS

The Coalition of Black Mass Spectrometrists (CBM) provides a much-needed space for discussion around racism, offering a platform to not only cultivate support, but also to drive positive change

TELL US ABOUT THE ORIGINS OF CBM…

Ulmer: A few years ago, Renã Robinson told me about her vision of Blacks organizing and networking at the ASMS conference given the increase in the number of minority groups present. Christina, Michelle, and I decided to formalize her vision at ASMS in 2018. We coined the name "Black People Meet at ASMS" with the goal of informally meeting other Black people at the conference and establishing rapport amongst each other. With ASMS going virtual for 2020, we were faced with the challenge of moving online as well. We knew it was important to still foster those relationships and provide a framework for networking, despite not being in the same place. Therefore, we decided to host our first virtual event during the ASMS virtual reboot conference – and it was fantastic. Our intention was really just to create a safe space to gauge the mental health of Black mass spectrometrists during this period of uncertainty, but we also played a few MS-themed games to help us get to know each other better.

We used our social media platforms to publicize the Black People Meet @ ASMS event and our "suggested resources" flyer, which subsequently attracted the attention of ASMS leadership. During the ASMS 2020 virtual reboot, we were asked to give a short presentation on systemic racism. There were obviously quite a few social movements at this time, so they asked us to discuss what systemic racism is and why it's important. CBM evolved from there!

WHAT ARE YOUR AIMS?

Ulmer: Our first virtual event really opened our eyes to the possibilities of CBM. Traveling is a hindrance to many people, but the virtual option allowed us to easily expand our scope. We want to connect people across the world and provide resources for as many people as possible. This goal was one of the reasons we changed our name from Black People Meet @ ASMS; we don't want to limit our efforts to one conference. The Coalition of Black Mass Spectrometrists better reflects the wider scope of our initiative in the future.

Even at the beginning, our hope was to eventually transition to a point where we could have more influence; for example, providing suggestions for more diverse speaker line-ups, giving talks, and highlighting each other's research.

Jones: In addition to this, we wanted to create a space to foster professional development opportunities and, more importantly, career opportunities. We want to interact with different companies looking for mass spectrometrists, and ensure Black people are aware of and being considered for these positions.

Reid: I'll just add that it's important not to underestimate the initial goal of CBM – to create a sense of comfort and belonging when you attend these events and might feel isolated as one of the only Black people there.

COULD YOU PLEASE SHARE YOUR OWN EXPERIENCES WITH SYSTEMIC RACISM IN ANALYTICAL CHEMISTRY?

Reid: I attended Spelman College, which is a historically Black college for women in Atlanta, Georgia. For this reason, I actually feel quite lucky because I obviously didn't experience racism there. But I recently moved to Switzerland and my experience living in Europe has been eye opening. America has its own issues with racism, but because there's not the same history in Europe, I've found the racism here to be a bit more nuanced. For example, I remember having a conversation with a colleague and trying to explain to them the concept of Historically Black Colleges and Universities (HBCUs). They just couldn't wrap their head around it, and suggested (albeit jokingly) that I was racist for attending

"I attended Spelman College, which is a historically Black college for women in Atlanta, Georgia. For this reason, I actually feel quite lucky because I obviously didn't experience racism there."

MEET THE COALITION CO-FOUNDERS

Candice Ulmer

Candice is a clinical research chemist at the Centers for Disease Control and Prevention, where she is the Project Lead and Acting Chief of the Clinical Reference Laboratory for Cancer, Kidney and Bone Disease Biomarkers. Her work focuses on the standardization of clinical measurements and the development of reference measurement procedures for chronic disease biomarkers. As an early career analytical scientist, Candice serves on various international committees, has published over 30 peer-reviewed papers, and is a co-founder of the Coalition of Black Mass Spectrometrists.

Michelle Reid

Michelle recently finished her postdoctoral fellowship at ETH Zürich, researching the systems-level view of Salmonella pathogenicity and quality assurance in MS. She is also a co-founder of the Coalition of Black Mass Spectrometrists, and is a co-chair of the Females in Mass Spectrometry Mentorship Committee.

Christina Jones

Christina is a research chemist at the National Institute for Standards and Technology, where she focuses on measurement harmonization and standardization for metabolomics. She has received numerous awards as an early career scientist and published over 25 peer-reviewed papers. As well as being a co-founder of the Coalition of Black Mass Spectrometrists, Christina is also co-founder of Facilitate 2 Motivate.

one of these colleges. I was shocked that someone could possibly interpret the situation like that. I believe this comes from a general lack of understanding about what it means to be Black in America and how deep-seated the racism actually is.

Ulmer: I had a similar experience when I was younger – I grew up in a city with two HBCUs, so I was introduced to Black scientists pretty early on. It wasn't until I attended college that I experienced a real culture shock – because there were no other African Americans in chemistry. I immediately felt isolated. On top of that, I also had to deal with actual blatant racism from certain peers or professors.

Some places are better than others, but in South Carolina, there's still strong racial tension and people are not afraid to show it. For example, when Obama was first elected, there were many Blacks celebrating – and some unhappy white people actually started throwing trash at us from their vehicles. Unfortunately, this sort of behavior is the norm in some areas.

Jones: I grew up in a predominantly Black area of Louisiana, and I had an HBCU around the corner. I wouldn't say I necessarily saw a lot of Black scientists, but from a young age I was exposed to viewing Black people as professionals and knowing that was an option. It was only once I got to a more diverse high school that I even learnt about mainstream white culture. At my predominantly white college, I experienced quite a lot of microaggression; for example, being excluded from certain study groups, my intelligence being questioned, or being asked to show someone a dance because "all Black people can dance."

WHAT ARE THE KEY THINGS THAT NEED TO CHANGE IN THE FIELD OF ANALYTICAL CHEMISTRY?

Ulmer: The key thing is representation. We need to be present on boards and we need to be given the opportunity to present our research and our perspectives.

Jones: More fundamentally, we need to have a better understanding of how to interact with people who don't necessarily look the same or share the same experiences, to engage more people in the sciences.

Reid: Overall, it's about helping people understand that bringing people together from diverse groups actually benefits everyone and creates a space to uncover novel scientific ideas. There are so many solvable diseases that are specific to a single population and not addressed until that group or population are engaged in the research question. And that's why initiatives like CBM are so vital – it's about bringing people together to encourage and prompt analytical science to really explore new and diverse facets of research that can benefit the community as a whole.

> *"The other thing I always say is to check the people in your life – start noticing if people are being biased and call them out on it! Educate them as well, because the people you surround yourself with become your biggest circle of influence."*

WHAT'S YOUR ADVICE TO WHITE COLLEAGUES OR "ALLIES"?

Reid: Engaging with the conversations going on in this area and being openly inquisitive is a good start. As a Black person, you have to teach yourself about your history – it's not taught to the extent it should be in history lessons in the US (or elsewhere). The onus should not be on Black people to educate you as well. It's really up to everyone as an individual to go out there, educate themselves, and truly engage in these discussions – not just on the surface level, but the root causes.

Jones: There's a lot you can do as an individual. When you're in a meeting and there's someone that's left out – whether they're Black or not – make sure they are involved and feel welcomed. Be an advocate for people – make sure Black people are considered for the same opportunities and speak up if someone is being overlooked. Some people will say these are small actions, but they can really make a difference. The other thing I always say is to check the people in your life – start noticing if people are being biased and call them out on it! Educate them as well, because the people you surround yourself with become your biggest circle of influence.

... AND LARGER ORGANIZATIONS?

Jones: They can go to NOBCChE and recruit people for their organizations to help build representation. All organizations should be striving for true representation – whether in your staff, your keynote speakers, or the research posters you present. Further to this, make sure you're creating these spaces for people to safely report any racist behavior and ensure they are able to talk about any issues when it comes to racial tension in the workplace.

Mastering
MENTORSHIP

Isiah Warner discusses the value of mentors to Black analytical chemists, and shares his own journey to the top of the field

HOW DID YOU INITIALLY GET INTO CHEMISTRY?

Funnily enough, the reason I got into chemistry was all due to my English teacher in high school. She was a great mentor, and one day she asked me what my major was going to be in college. I said I was going to major in science; I just had no idea what area. She asked, "How about chemistry?" And ended up contacting the chemistry chair at Southern University – an HBCU – and securing me a summer internship there. That experience changed my life, not only because I had a great time and learned a lot, but also because those of us that did well during this internship were offered the chance to skip the first year of chemistry if we chose to study at Southern – so that's what I did.

PLEASE TELL ME MORE ABOUT YOUR ROLE AS VP OF STRATEGIC INITIATIVES AT LSU...

The reason I was given this role is because we were starting to generate African American PhD students in chemistry at unprecedented levels! In fact, LSU Chemistry is now ranked number one in the country for the number of African Americans who receive PhDs, and number one for the percentage of women who receive PhDs. Because of these achievements, the upper administration asked if I could help implement similar strategies to help achieve this for the main campus as well. My role in Strategic Initiatives involves generating grant funding to get more underrepresented students into STEM, just as we did for chemistry. Since that time, we've generated close to $50 million in grants – funding that probably wouldn't have existed if my office did not exist.

WOULD YOU BE WILLING TO SHARE YOUR EXPERIENCES WITH RACISM THROUGHOUT YOUR CAREER?

I can't say that I've been held back in particular; I've worked hard and been lucky enough to win many awards throughout my career, I have close to 400 publications, and I'm at the highest professorial level at my university. But despite these credentials, I still have to deal with racist behavior.

I can think of times when I experienced more serious prejudice in my earlier days. When I first joined LSU, there was one faculty member who actually believed I was only offered the position because I was Black – and told me so. I said to him, "There are some people in this world who may very well give me certain privileges because I'm Black. However, I can assure you there are many more, like you, who will deny me privileges because of it. I will not gain anything from being Black in this country."

I've had to deal with this particular type of behavior quite a lot – people thinking I'm only as successful as I am because I'm Black. One of my international postdocs once had his former advisor come and look round our lab, and he was quite impressed. While I was not around at the time, he saw a picture of me and realized I was the one running the lab – and he made a comment like, "Oh, so that's why he's so successful." Before he knew I was Black, I was just successful. But as soon as he saw a picture of me, there was this whole other line of thought going on - I must only be successful because I am Black.

A more recent and nuanced example: I've recently been researching a new area of bioanalytical science – focusing on solid-state forms of ionic liquids, including the development of "GUMBOS" (Group of Uniform Materials Based on Organic Salts). Many people may recognize the word gumbo as being a famous soup in Louisiana. However, the word is actually African in origin and means "okra." I actually had papers rejected because people thought the name was some kind of joke and suggested that I change it. When I refused

Isiah Warner

An internationally recognized researcher, Isiah holds a Boyd Professorship in chemistry in the Louisiana State University (LSU) system and is also the Vice President of Strategic Initiatives at LSU. Isiah has over 380 publications to his name, has received numerous awards, and has served as Chair of the ACS Division of Analytical Chemistry. His research is focused on fluorescence spectroscopy and, more recently, the applications of ionic liquid chemistry to materials chemistry.

for one particular journal, the reviewer came back with untenable different critiques of my manuscript. I appealed to the Editor who refused to listen to my arguments. I decided to simply publish in a better journal and have not submitted to that journal since.

IN YOUR OPINION, HOW IMPORTANT IS MENTORSHIP?

It's very important to have those advocates in your life who will make sure you have opportunities available to you. I already mentioned how important my high school English teacher was to my pursuing chemistry. I also had a significant mentor in college: Wilbur Clark. This was back at the height of the Vietnam War, and there was a program called the Reserve Officer Training Corps, which was mandatory for two years. I was debating whether to do the extra two years, which would have meant going to the military after college and then to Vietnam. However, Dr Clark convinced me to pursue research instead. He had a great impact on my whole life, and he was probably my greatest mentor in college.

YOU'VE BEEN IN THE FIELD A LONG TIME. WHAT HAS CHANGED – AND WHAT STILL NEEDS TO CHANGE?

That's a good question. A lot has certainly changed since I've been in the field. I see a few more Black faces than when I started, and there seems to be less in-your-face racism. However, there are obviously still individuals with certain attitudes towards Black people, and that needs to change. It's difficult for some people to accept that Black people in this country can be as successful as or more successful than they are, based on merit alone. As I mentioned before, I've clearly been quite successful in analytical science – and yet, with one exception, I've never been on the editorial board of any major journals in this field. Why is that?

It's important to make sure everyone feels welcomed at all times, because this is the most conducive environment for success. Things have changed, but there's also been a lot of retrogression recently. This is particularly true in the US, which I see as a backlash to us having a Black president for the first time in history; there's been ever-increasing racial tension and turmoil under the current president. So clearly, both within analytical science and society as a whole, there's still a long way to go.

WHERE SHOULD PEOPLE IN THE FIELD BE FOCUSING THEIR EFFORTS?

My own focus is on the importance of mentoring. There are still people in this field that are not getting the same opportunities as others based on whether they are from a minority group or not. It's vital that these people have mentors that can help them navigate the system and reach their goals. These mentors don't need to be Black or from the same background as you – they just need to be able to empathize. I am writing a book on mentoring for this reason.

"There are still people in this field that are not getting the same opportunities as others based on whether they are from a minority group or not. It's vital that these people have mentors that can help them navigate the system and reach their goals."

I actually found an amazing mentor in my boss while I was working in industry. He was Japanese – and a lot of other Asians have figured prominently in my life in terms of mentorship. He knew I didn't want to continue working in industry and he asked me why I didn't go back and get a PhD. I said I wasn't sure I was capable, but he told me how he'd never worked with someone who was so quickly able to grasp difficult concepts on the job and he guaranteed me that I was capable of getting a PhD. That interaction really built my confidence and I started to apply to PhD programs. It's absolutely vital that you have someone who empathizes with you and is able to direct you towards your goals, particularly when you need to build confidence. I ended up completing my PhD in three and a half years in a program where the average was five and a half years.

WHAT'S YOUR ADVICE FOR IMPLEMENTING SUCCESSFUL MENTORSHIP PROGRAMS?

Make sure you're not making arbitrary assignments of mentors to students. Imagine if the faculty member I spoke about earlier, who thought the only reason I got the job was because I was Black, was assigned as my mentor… It's vital that you think about how you assign people, but you also need to offer training; being a great mentor doesn't just happen overnight. In addition to being a teacher, you have to be a counsellor, you have to be an intervener, and you have to be an advocate for that person. I often say that you need to invest in that person the same way you would invest in your own children. If my mentees are successful, I'm just as proud of them as I would be of my own children. That's really what it takes to truly help people progress in the field.

A Social (Media) Movement for Change

Devin Swiner, co-founder of the #BlackInChem campaign, explores the power of social media and describes the aims of the initiative

COULD YOU PLEASE SHARE YOUR EXPERIENCES WITH RACISM?

I'm sure everyone has similar experiences in terms of microaggression and feeling slightly isolated. But I think something else that people don't necessarily realize is that as a Black graduate student, not only are you at university to learn – you have to teach too. Universities tend to love showcasing the smart Black kids in any department. And that can lead to Black students being tokenized – they are expected to essentially work as an "inclusion and diversity representative." But there's no extra credit or pay for this work – it's just expected. For someone like me, that's fine – I'm outgoing and it's something I'm happy to speak about. But others may not want to play this role – an issue that actually came up in one of the discussions during #BlackInChem week. And it's absolutely fine if people don't want to do any of this stuff. The onus should be on other people to educate themselves – please don't just make it the knee jerk reaction to ask your Black students.

WHAT INSPIRED YOU TO START #BLACKINCHEM?

To give some context, there were a lot of campaigns about Black scientists all over summer this year – like #BlackInNeuro and #BlackInGenetics. So I and the other co-founders decided to do something similar for chemistry. Did I think we'd be getting celebrity endorsements like MC Hammer and Michael B Jordan tweeting about it? Absolutely not. I was completely surprised by the scope the campaign reached. But it was just a great way to highlight the amazing Black chemists in these areas, and to also give people a place to network and connect.

> *"The main motivation of all the campaigns was really to increase the visibility of Black scientists and showcase their talents – and to allow them to connect."*

COULD YOU EXPLAIN THE AIMS OF THE CAMPAIGN?

The main motivation of all the campaigns was really to increase the visibility of Black scientists and showcase their talents – and to allow them to connect. But we also had a range of events during the week to help develop not only the social side of things, but also the more practical skills. We had an undergraduate 101 session, we had an elevator pitch competition to see how quickly they could present their research, and we networked more generally. I think we were really successful at creating a space where people felt comfortable and could ask any questions.

Social media is a great way of connecting with people across the world. It's also a fantastic resource for archiving content – the hashtags in themselves make it easy for everyone to follow what's being posted and it means you don't miss anything.

ANY HIGHLIGHTS FROM THE WEEK?

Each day of the week was based on a subdivision of chemistry; for example, on the Monday it was #BlackInAnalytical. Every day was full of introductions, research pitches and then something a bit more fun for people to tweet about. Monday was a highlight for me, because we got everyone to tweet about their favorite technique. I'm a mass spectrometrist at heart, so for me, it's obviously MS! We also had these other skills-building events outside of the daily bits as well, and we really tried to get a mix of academia and industry representation.

WHAT ARE THE PLANS FOR #BLACKINCHEM IN THE FUTURE?

It was great to see how much people appreciated the campaign and how many people said they'd love it to continue in the future. We're going to try to continue some of these initiatives, perhaps organizing some lectures and having a monthly meet and greet style event. We've also set up a website and are working on uploading profiles to make it easier for people to connect and ultimately recruit Black scientists. People have also been asking about merchandise, so that might be something we look at in the future...

Devin Swiner

Co-founder of the Twitter campaign #BlackInChem, Devin is currently completing a PhD in MS at Ohio State University. Her research focuses on small molecule analysis, with applications geared towards clinical diagnostics, drug screening, and disease biomarkers. She is also Ohio State Chapter President for the National Organization for the Professional Advancement of Black Chemists and Chemical Engineers (NOBCChE).

Renã Robinson

Renã is an Associate Professor of chemistry and neurology at Vanderbilt University and a Dorothy J Wingfield Phillips Chancellor's Faculty Fellow. Her research primarily focuses on Alzheimer's disease pathogenesis and disparities, and she is a pioneer in combining sample multiplexing and MS for large scale analysis of proteins. She recently became the President Elect of NOBCChE.

Advocate
AND ADVANCE

Renã Robinson tells us about her role as President Elect of the National Organization for the Professional Advancement of Black Chemists and Chemical Engineers (NOBCChE), and why advocates are vital to ensuring true representation in the field of analytical chemistry

WHAT DOES THE ROLE OF PRESIDENT ELECT OF NOBCCHE INVOLVE?

My role at the moment is to support and help our current President carry forth the mission of NOBCChE. And that means focusing on developing strong leaders and having a dynamic board that will help make sure all of the organizational programs that bring together academic institutions and industry or government agencies thrive. We're also constantly working to grow our membership and support the needs of our members.

COULD YOU PLEASE SHARE YOUR OWN EXPERIENCES OF SYSTEMIC RACISM?

Many experiences throughout my career can be considered evidence of systemic racism – whether they were more subtle, like not getting the attention I was due or being discredited for my opinions, or more blatant, like inappropriate comments. I'd like to share one specific incident to give a flavor of what Black scientists have to constantly deal with; many years ago, I was applying for a student fellowship where one of the requirements was that you had to be from an underrepresented minority group. It was based on merit, but that was one of the criteria. I asked a faculty member to write a letter of recommendation for me and they refused because they wanted to validate me against the level of my entire class – despite the fact I was doing really well anyway. I remember being really hurt and confused – I was the only one in the whole class who was eligible!

It didn't make sense to me at the time, but you learn to deal with these things. It's really helped build my capacity to truly understand just how many barriers there are to Black students advancing in the field. The systems are broken, and even when someone does make it into this space they might merely be tolerated rather than respected based on their value as scientists. Organizations like NOBCChE offer the chance to combat such issues.

WHO WERE YOUR ROLE MODELS GROWING UP?

In terms of role models, the first to spring to mind is actually my mom. She's not a scientist, but she really showed me what it looks like to work hard and be persistent in whatever you do – to keep pursuing your goals no matter what comes your way. As I've grown in my career, I've also come into contact with many other individuals who've acted as role models; for example, when I thought about becoming a faculty member, Isiah Warner was a massive role model to me – as well as a mentor.

HOW IMPORTANT IS MENTORSHIP IN GENERAL?

Greatly. But I think it's important to first distinguish between mentorship and sponsorship or advocacy. Mentorship is vital for a number of reasons; to help individuals know they belong in a particular field, to give them the right tools to navigate the field, to understand how to excel in the field, and to give them an example of someone who has pursued that same path. Sponsors (or advocates) have a different role. They take it a step further and actually help promote those individuals and create opportunities for them that allow them to progress. I believe it's important that people have both mentors and sponsors in their network.

WHAT'S YOUR ADVICE TO WHITE COLLEAGUES IN FACULTY POSITIONS?

The first thing is to become informed and aware of systemic racism – and then look for individuals who are experiencing it. Try to be sensitive and empathize with what it feels like to be in these sorts of fields and institutions, where people may experience systemic racism on a daily basis. Use your influence and your voice to help create opportunities and drive change in your own spheres. If that's in your own group, then make sure there are opportunities that are equitable for everyone. Or maybe take it a step further and create change at a departmental level – creating new programs or seminar series to recognize scientists from diverse backgrounds.

Keeping Calm, Carrying On

The COVID-19 pandemic has put many aspects of our lives on hold, and many of us are still adapting to the "new normal." But science has persevered throughout. Such resilience will prove crucial in forging a path back to normality – and ensuring public safety.

Here, we share the stories of companies operating in the age of COVID-19. How have their leading minds risen to the challenge? And how are they contributing to the fight against SARS-CoV-2? Let's hear what they had to say...

A Powerful Alliance

With Nigel Skinner, Head of Marketing, Andrew Alliance, Geneva, Switzerland

How have your ways of working changed this year?

Like most companies, we have worked remotely to ensure compliance with guidelines and restrictions, as well as ensuring the safety of our employees, partners and customers. However, many of our users have had to continue working due to the critical nature of their employment. Their roles range from the development of diagnostics and therapeutics – including tests and treatments for COVID-19 – to food safety. The situation has of course created additional challenges for us in terms of manufacturing, marketing (due to canceled trade shows), and on-site customer demonstrations.

But it's not all doom and gloom. We have benefited from some of our existing practices, including an increasing use of "e-demos" to reduce travel and our carbon footprint (we are a carbon-neutral company!), as well as increased emphasis on digital marketing. Experience in these areas has helped us more readily adapt to our "virtual world" in lieu of customer visits and trade shows. A great example is our award-winning lab automation, which employs cloud-native software and can be demonstrated, set up, and operated remotely! We launched this in February 2019.

Have any aspects of the "virtual world" been particularly challenging to navigate?

The lack of trade shows has made it more challenging to strike up conversations with new customers. Major events in our calendar – Analytica for one – were postponed until mid-October and made entirely virtual. In addition to the e-demos I mentioned above, we're also fortunate that we have a number of products that are visually appealing, such as the Andrew+ pipetting robot and the Pipette+ smart pipetting system.

We have also been able to develop engaging videos for these products that highlight their important technical benefits for companies needing to accelerate their research and development in the age of remote working. For example, we launched a series of "deminars" to demonstrate the ability to set up and operate these robots remotely, as well as a series of webinars exploring how customers are already using these products remotely!

What's your proudest achievement in these troubling times?

Our products have been used by companies engaged in cutting-edge development of new diagnostics, tests, and life-saving therapeutics. Here are some amazing examples:

- Mammoth Biosciences is using an Andrew+ to continue developing their COVID-19 test in partnership with GSK. Their research leader is able to set up protocols at home, launch them remotely and watch the progress via Zoom.
- Bioside Diagnostics was able to quickly upscale its SARS-CoV-2 RT-PCR test during lockdown in Italy. Our robot was set up at its lab "remotely" and supplied them with the many benefits of our platform.
- HG Pharma is using our Pipette+ system in its mobile testing trucks. This is happening in Austria now, but is due to be expanded to other countries in Europe, including Germany and the UK. Why? It ensures absolute reproducibility in sample and reagent pipetting – in this case for a RT-PCR-based test – as well as full traceability. This last quality is particularly important for COVID-19 testing.

We've also been informed by Frost & Sullivan that we will be awarded the 2020 Product Leadership Award for North America to honor our Pipetting Robot for Life Sciences. The reasoning behind the decision: "The unique capability of providing remote data access has enabled Andrew Alliance to match market and customer needs that are increasingly moving toward working either remotely or in adherence to social distancing norms amid the COVID-19 pandemic."

And what about new business relationships?

Andrew Alliance was acquired by Waters Corp in January as part of its (and our) collective vision to bring the benefit of the fully "connected lab" to LC-MS users, as well as users in adjacent workflows, such as qPCR, next-gen sequencing, and so on. The move is incredibly exciting as it promises to bring improved repeatability and productivity to all aspects of the workflow, from samples to results – a clear, ever-present need that has only been highlighted by COVID-19.

Not only are we accelerating user workflows without compromising data quality, but we are laying the foundations for an exhilarating journey together in the post-COVID world. We will be more digitally aware, more agile, and better placed to support labs operating in a broad range of analytical techniques. These labs span from those conducting LC-MS proteomics and metabolomics to translational research, where automation for sample handling that supports various omics workflows is crucial.

www. andrewalliance.com

Andrew Alliance
A WATERS COMPANY

A Ray of Light

With Alexandra Knauer, CEO, KNAUER Wissenschaftliche Geräte GmbH, Berlin, Germany

How has the pandemic affected life in Berlin?

COVID-19 has led to drastic changes for Germany, and these are visible across all industries and social groups. Vibrant Berlin has not been doing much better than other major cities in the world. Our universities have stopped offering normal in-class courses, and this will remain mostly the case during the winter semester.

The financial situation for students is also very difficult because many of their classic temporary jobs are not available at the moment. And it would seem that more students than expected do not have a computer with sufficient performance for online conferences.

Donations were needed, so a fund was started to counteract the problem. KNAUER has itself donated to provide appropriate equipment or improved internet access to those students in need in Berlin. I think this is the spirit we must all embody this year. As long as societies and partners stick together and show that they care, there will always be a ray of light in these troubling times.

Sounds like a lovely initiative – have you been involved in any others?

Yes, we have! A number of contacts from institutions in the region – including the head of a women's shelter, the director of a local school, a kindergarten director, and employees of a neighboring refugee home – have all got in touch because they needed disinfectant.

Given the scarcity of disinfectant back in March, KNAUER employees suggested that we could produce it ourselves. After some investigation and approval, we mixed and bottled "Dr. KNAUER's disinfectant for hands" in line with a WHO recipe in our central laboratory. From May, we then provided this disinfectant (for free!) to suppliers, schools and so on, sometimes along with masks, gloves, and 3D-printed protective visors.

What were your main concerns as a business when the pandemic began?

I have a team of more than 150 employees and was of course concerned about how we could ensure their safety while maintaining business. We immediately set up a crisis management team that has planned, and is still planning, all pandemic-related measures.

We also feared that our supply chains and the availability of parts would cause us problems; such issues would have directly compromised our ability to deliver products. But we make three quarters of our purchases in Germany; many of them in Berlin and the surrounding area. Though it can be a financial disadvantage at times, we are motivated by the close cooperation it drives with our suppliers – as well as rapid delivery. It also means we can ensure fair and environmentally-friendly production conditions according to German and EU standards.

Our regional focus has proven to be a great advantage during the current crisis, allowing us to avoid many of the bottlenecks that could have arisen when trying to acquire parts from distant geographies in various states of disruption.

And how has business been in general?

We have actually been very relieved regarding business development. LC is an important tool in the research of vaccines and active ingredients, including those related to SARS-CoV-2. Our many years in the field of LC have allowed us to provide excellent support to such ventures.

Accordingly, we have been able to acquire major customers both last year and this year. And that success has resulted in a need to increase production, which we have already planned. But now comes the implementation; we have integrated many precautions to reduce the risk of COVID-19 interfering with our plans – and we hope things will go as smoothly as possible.

One existing customer, a Berlin-based manufacturer of oligonucleotides, was also able to introduce a test for SARS-CoV-2 very early on in the pandemic. In this particular case, it is very rewarding to know that we are making a contribution to the fight against the virus with our FPLC systems. Our other products will likely be used for COVID-19 vaccine production, but that's all I can say for now!

Have you had to make any difficult decisions in these trying times?

KNAUER is a family business, established 58 years ago. The founders – that is, my parents, Roswitha and Herbert – still feel very attached to the company and play an active role. Having to tell them in April, at the peak of the pandemic, that they were no longer allowed to visit the company was not easy. They didn't like being sent to work from home for a number of weeks, and they quickly returned when the infection figures dropped in May!

www.knauer.net

KNAUER

The Adaptation Game

With Dr. Daniela Held, Managing Director of Marketing & Sales, PSS GmbH, Mainz, Germany

How prepared were you for the disruption to business in 2020?

Though the pandemic has definitely been disruptive, we were fortunate to have an emergency plan in place that involved working in shifts from early in 2020. When the time was right, we acted on that plan. The benefit for us: we never had a total shutdown, just slightly reduced working capacity.

We also benefited from our existing experience with remote customer support, which we have employed for several years already. As a result, we were already equipped and familiar with the tools that were needed to adapt. All we needed to do was apply these tools to internal communications, such as meetings with colleagues. On this topic, the sharing of lab results with colleagues while maintaining social distancing took some getting used to…

The lab work and production also continued largely unaffected. Our stock was sufficient to serve all of our customers throughout, but I must confess that we were happy when supply chain management began to return to normality – something that only happened towards the end of August. I'm happy to say that our usual operations are now back in full swing – with the appropriate safety measures, of course.

Have any aspects of the pandemic been particularly challenging?

Our Training Academy was a challenge; we had to postpone all of our classroom-style training. Instead, we offered complimentary educational webinars so that our clients – who would normally be in the lab – could make good use of their time at home.

We have moved much of our education to a virtual format, including our three-day GPC/SEC Theory and Practice course. Engaging attendees is the main concern. Fortunately, we seem to have accomplished this! All attendees stayed until the course's end, and even participated in the optional sessions late Friday afternoon, resulting in very positive feedback. Younger participants also mentioned the environmental sustainability of such approaches. In future, we will conduct training both in the classroom and virtually.

Fulfilling on-site services requirements also hasn't been easy, particularly installations and qualifications. But we have developed ways to conduct these operations and have successfully provided these services in different countries.

Though it was difficult to abandon our usual ways of working – perfected over many years – we felt a duty to our customers, many of whom work in the pharmaceutical industry. It forced us to look at new and improved ways of doing things utilizing the modern tools available. We have remained a reliable partner for our customers, and we also now find ourselves in a better position to support them in the future.

How have the interactions with your clients been?

They have been fantastic. In fact, I'd like to say thanks to all of our partners and clients around the world for sharing their daily experiences in emails and video meetings. It was comforting to share experiences in this way, and we learned things about each other we might never have learned otherwise! At the end of the day, we're all in the same boat, so it's nice to feel that we have become closer despite social distancing being in place!

It sounds like you've done incredibly well...

It's a great feeling to say we were able to support our clients around the world – even with these harsh conditions. And I'm extremely proud of how my colleagues and coworkers were able to manage this crisis. Everybody (and I really mean everybody) has worked harder than ever, offering creative and proactive solutions, and was fully supportive. I really appreciate that, knowing that everyone has their own private concerns with schools closed and vulnerable or sick relatives.

It's amazing how well we have adapted and I'm optimistic that we can continue going forward even stronger. It is a difficult situation to navigate, requiring a massive amount of patience from everyone involved. I guess the one good thing about disruption is that it triggers new developments; in our case, it has certainly accelerated the finding of new solutions.

What advice would you give to yourself at the start of 2020 knowing what's ahead?

On a personal note, I would have told myself to meet friends and family more often – no excuses like "too much to do" or "I will do it next week." I have many friends in other countries and I miss them dearly. But I'm sure I'll see them one day (relatively) soon. Until then, we need to make do as best we can!

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PSS

An Innovative Response (Team)

With Steve Martin, Vice President of Global Research, Waters Corporation, Milford, MA, USA

Like most of you, I was overwhelmed by a feeling of helplessness as the COVID-19 pandemic became a reality. Those who work with me know that, when we find ourselves backed into any corner, my exhortation to the team is often: "If not us, who? If not now, when?"

A rapid response

Even before the World Health Organization officially declared a pandemic, we had begun working on an action plan to support scientists racing to understand the virus and develop vaccines and treatments. Thus, the Waters COVID-19 Innovation Response Team (IRT; see more at https://bit.ly/3owTPE2) was born. The goal: to remove the red tape and protracted discussions common to developing collaborations, by shifting to "thin" agreements that enabled direct support for researchers in the form of rapid access to critical supplies and equipment.

We were well equipped to do so, with expertise across instrumentation, chemistry and separation sciences at our disposal, as well as a global supply chain and field support. And, armed with Microsoft Teams, we were ready to support customers anywhere in the world. The dedicated IRT team rapidly grew to over 300 members supporting over 40 collaborations, in addition to providing columns and methods to more than 20 clinical laboratories. Urgency was central to the IRT's mission.

Forging friendships

The response to our outreach was immediate and overwhelmingly positive. Projects ranged from helping to develop a method for characterizing recombinant SARS-CoV-2 spike protein glycans to providing analytical columns and LC and MS methods to help clinical researchers accelerate critical assay development for the DISCOVERY clinical trial. The Waters IRT has also contributed to Maarten Dhaenens' consortium at Ghent University (1), where the goal is to develop a protein-based LC-MS assay to rival qPCR approaches.

We have also collaborated with Zoltan Takats at Imperial College London, which led to the development of a rapid screening test for COVID-19 infection. Use of DESI ambient ionization technology, coupled with our MS expertise, allowed us to build and test a prototype instrument capable of analyzing a dry nasal swab in less than 30 seconds, with promising results in distinguishing infected and non-infected samples (2).

Oligonucleotide purifications and mRNA-based vaccines have also attracted interest. We have supported this work by providing early access to our new ACQUITY PREMIER columns (https://bit.ly/3jfFcRB).

Several of our collaborators will take part in a global COVID-19 symposium (https://bit.ly/31OeZ6T) and panel discussion on November 18, 2020 – we hope you will join us (virtually, of course!).

Immerse Cambridge: an open innovation platform

Open innovation and engagement will be key to quelling the pandemic. Early in 2019, Waters planned an open innovation platform to put our scientists, engineers, and emerging technologies at the center of ecosystems that catalyze innovation in the life and material sciences. This concept culminated in the opening of Immerse Cambridge (https://bit.ly/2ISsuvR) – an open innovation laboratory to foster collaboration and drive analytical sciences forward.

One of the first projects for Immerse Cambridge is investigating the glycan structures of influenza, which change over time and enable immune system evasion, with Joe Zaia of Boston University. This work will use a Waters SELECT SERIES Cylic IMS System (http://www.waters.com/cyclicims), which is uniquely capable of revealing fundamental viral features and guiding vaccine design strategies.

Looking ahead

The pandemic has united researchers, removing geographic boundaries and energizing the Waters IRT team with exciting new partnerships. Building on all that we have learned during the pandemic, we aim to confront analytical challenges with urgency and innovation. I am proud of the foundational role analytical science has played thus far and I'm humbled by how much more needs to be done. As a community, we are collectively demonstrating a great way forward.

If not us, who?

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Embracing the Silver Linings

With Maria Dullaert, Total Support Manager and Applications Engineer, Ocean Insight, Duiven, the Netherlands; Yvette Mattley, Lab Services Manager; Amy Bauer, Principal Applications Scientist; and Derek Guenther, Senior Application Scientist, Ocean Insight, Largo, Florida, USA

How have you – and the community – adapted to working in 2020?

Amy: I'd been employed by Ocean Insight for six weeks or so before I was quarantined. I began working from my home in Minnesota as a result. Out of the lab, I find myself guiding others – largely by conducting data analysis and helping with business development. I am very fond of the people I'm working with, but also intensely jealous of their continued involvement in delicious measurements and hardware development.

Yvette: Scientists and researchers are very resilient – it's an essential quality in their line of work. As the pandemic swept the globe, researchers found themselves unable to access their labs (like Amy!), but knew their research must continue.

The stories of researchers finding new ways to conduct their work remotely has been an inspiration. Dining room tables, kitchens, and backyards became makeshift labs – and family members became research assistants. These remote research efforts were enabled by the availability of compact, portable instruments; the ability to make measurements anywhere and everywhere has never been more important.

Operating a remote lab sounds like no small feat.

Yvette: Remote working requires careful planning to ensure everything that might be needed is available. It's not like we can run to the lab down the hall to collect any missing equipment. As wonderful as our home labs are, they can never be as well-equipped as their traditional counterparts, of course. We only hope that the intense preparation involved in remote lab setup means the results are just as robust as they might have been otherwise – if not more so!

And what other challenges have you faced?

Maria: Working from home has been a challenge in its own respect. Maintaining momentum can be difficult, but the real problem is remembering to take a rest because, in a way, we are always at work.

Derek: We've also been hit with a huge demand when it comes to our Lab Services Team. We have been working hard to keep up with this demand – especially at the start of the pandemic, when many labs in California and the northeastern US experienced total shutdowns. Scientists couldn't continue with their work, but our team could act as their eyes and hands in the lab.

The increase in volume and frequent push for rapid turnarounds was a unique yet welcome challenge. Organizational tools like Microsoft Teams helped to manage all the juggling we've had to do – and, along with some extra hours, kept things moving smoothly! We're incredibly excited to help researchers take their next steps in their work, even when they don't have typical access to their labs and equipment.

Have you achieved anything you're particularly proud of amidst the madness?

Derek: Ocean Insight has been working with several teams developing rapid COVID-19 tests for the general public. It's an honor to be tightly connected to groups fighting to bring normalcy back to the world. It's also amazing to note that these groups will use different optical approaches to get to the same answer – and Ocean Insight technologies can accommodate and evolve to support these optical methods.

From rapid PCR using trace-level fluorescence to direct detection using SERS, these teams are improving testing times, giving results in just minutes – and, ultimately, seconds. All this effort will facilitate public screening in real time at key loci like airports, schools, and nursing homes. Rapid detection will be critical in reducing numbers and returning to our normal lives. Ocean Insight is very proud to be a part of that groundbreaking process.

Yvette: The continuation of research under these trying and unprecedented conditions is such an accomplishment. As we rush to understand the SARS-CoV-2 virus and develop rapid screening tests and vaccines, research must continue. The innovative approaches to continuing this research that our teams have come up with have been outstanding.

In hindsight, what advice would you have given yourself at the start of 2020?

Maria: Hang in there and do what you can!

Yvette: Think outside the box. Be innovative. Don't despair. Focus on the silver linings and keep researching.

Derek: Invest in toilet paper.

Amy: I'm with Derek – start hoarding toilet paper ASAP!

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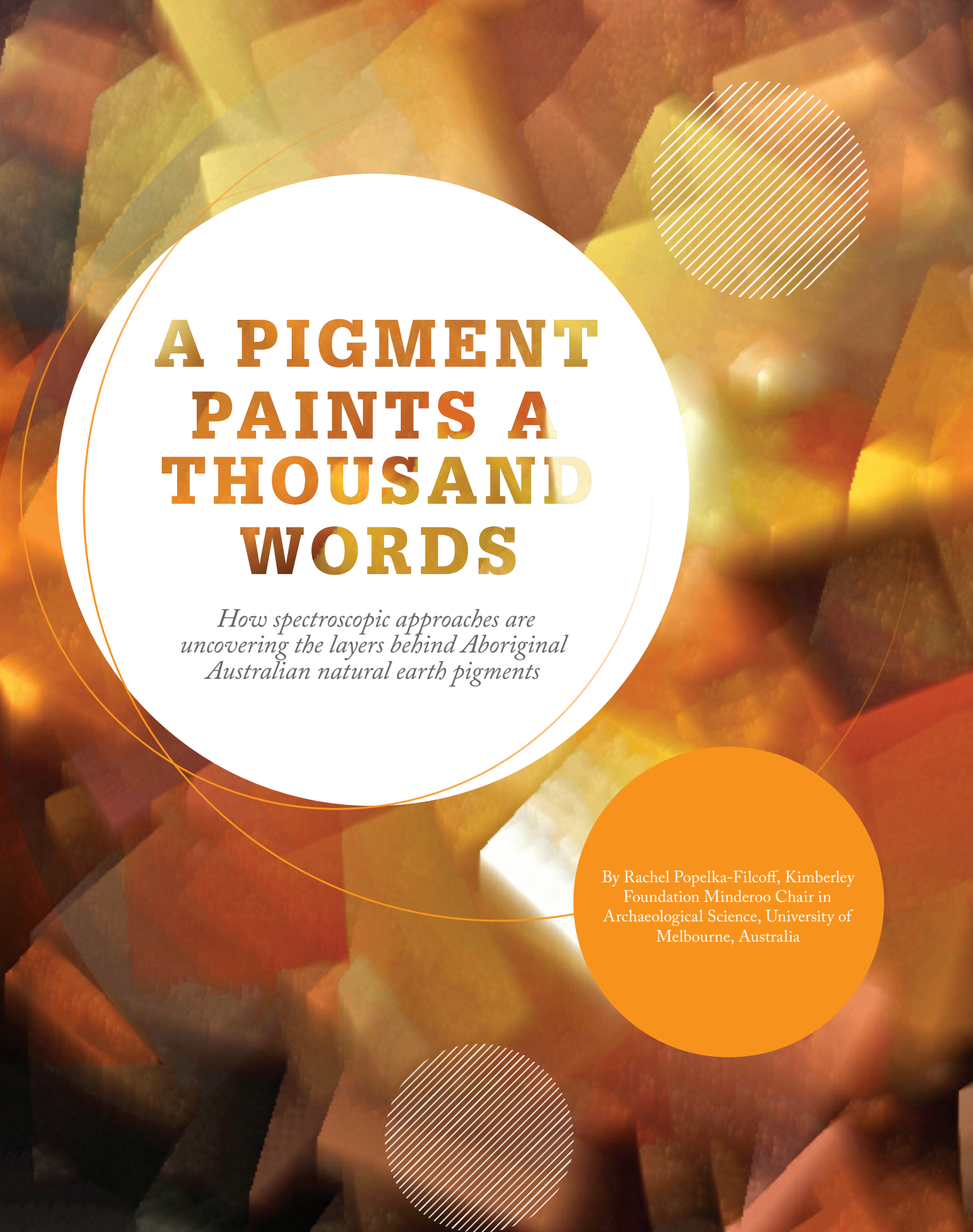


A PIGMENT PAINTS A THOUSAND WORDS

How spectroscopic approaches are uncovering the layers behind Aboriginal Australian natural earth pigments

By Rachel Popelka-Filcoff, Kimberley Foundation Minderoo Chair in Archaeological Science, University of Melbourne, Australia

Take a moment to reflect on the objects that are in your immediate vicinity. Depending on their composition, some of these objects may communicate cultural information that transcends generations. Those objects that are fundamental to our cultural expression often also reflect traditions that go back decades, if not centuries. Some of these may end up in museums - others may remain treasured family heirlooms.

Unfortunately, history often displaces communities or disconnects them from these culturally significant objects, meaning our own connection to the past is lost for generations as well. But chemical characterization offers an insight into these lost connections and our collective humanity. Despite differing environments and contexts, communities around the world often use the same materials for the same purposes – for example, the natural mineral earth pigments used in rock art. Modern analytical approaches – combined with fundamental questions about the exchange of materials, movement of populations, and uses of materials – help us to not only understand our past, but to inform our use of these same materials in today's society.

UNIQUE CONSIDERATIONS

Though the context (such as archeological stratigraphy) or documentation (such as historical writings) on an object are useful in providing framework, they are often not available. Often, the only way to reconstruct cultural interactions lost to time is through analytical chemistry. Chemical analyses help us answer questions regarding the materials used to make objects, the use of the object itself, and whether it was made locally or exchanged from a distance.

Cultural heritage objects are a complex mix of materials and technologies. And, unlike materials produced in the lab, unique considerations must be taken into account when analyzing them. Though some of the analyses are based on established methods, the process is not as straightforward as an industrial or routine analysis. For example, mineral pigments mixed in with plant-based binders are mixtures of inorganic and organic components. For these multimedia samples, a single analytical approach may not be optimized or sufficient to fully understand the composition

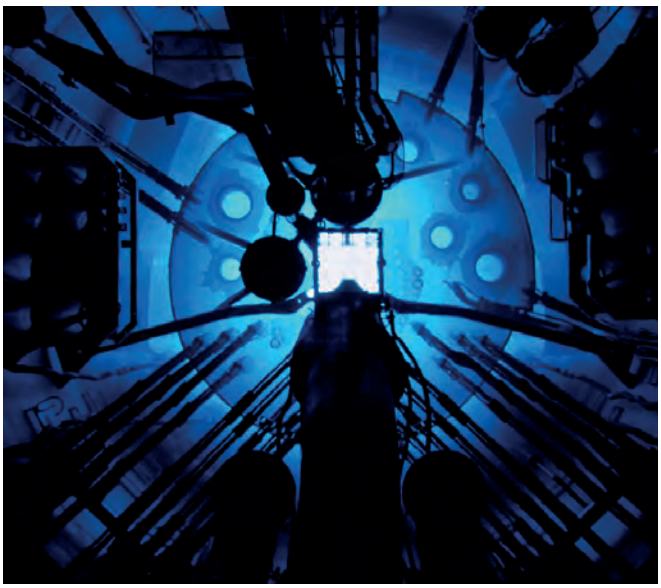
of both materials – or the interactions between the two. Therefore, it is imperative to build a fundamental understanding of the materials alongside any analytical approaches employed.

An additional layer of complexity also surrounds the approach to analysis. Unlike lab-synthesized samples, cultural heritage materials are often limited by access and permissions. On top of this, materials may have continuing cultural significance to communities today, or be in very limited supply. This means additional considerations must be taken to ensure analysis is non-destructive. Alternatively, where possible, microsampling is the way to go.

THE WORLD THROUGH PIGMENT - TINTED LENSES

I have studied several different categories of cultural materials across my career, including pigments, ceramics, metals, glass and stone tools. In my recent appointment as the Kimberley Foundation Minderoo Chair in Archeological Science at the University of Melbourne, I am establishing a new teaching program and laboratory, as well as leading new initiatives in archeological science focused on geologically based materials. This program builds on the strengths of the existing collaborations with Rock Art Australia and other partners towards developing novel methods of analysis and dating of rock art. For the past 20 years, my work alongside colleagues in both the natural and social sciences at universities and museums has focused on natural mineral pigments – specifically, examining Indigenous materials in North and South America, as well as Australia. However, the use of these pigments is universal across cultures and communities the world over, and these traditions have continued today through both cultural uses and modern engineering applications.

I have been investigating the provenance and composition of ochre-based pigments. These pigments are common worldwide but are of significant importance in Aboriginal Australian cultural tradition – in the past and through to the present day. The pigments applied to objects or rock art reflect the natural variation of the original source in terms of mineral and elemental distribution of particle shapes and sizes. Our group and collaborators have demonstrated that elemental and genomic analysis of ochre pigments can be used to uncover the provenance of a material right back to its geological source – as well as understand its cultural technology. And that means chemical analysis can enable researchers to reconstruct ancient exchange routes, and even offer useful information for future conservation studies.



The OPAL nuclear reactor core at the Australian Nuclear Science and Technology Organisation (ANSTO). Photo credit: ANSTO.

"NAA is a useful technique as it requires 50 milligram amounts of sample for each bulk analysis."

ALL EYES ON OCHRE

Ochre is a collective term for iron-based oxide pigments, primarily iron oxides (hematite and red pigments) and iron hydroxides (goethite and yellow pigments), but ochre materials ultimately cover a spectrum of mineral composition mixtures and colors. Rather than focusing on the mineralogical composition, we decided to focus on the characterization of

the "elemental fingerprint" of Australian ochre to allow us to differentiate between sources. We started by using k_0-neutron activation analysis (k_0-NAA) – at the OPAL nuclear research reactor of the Australian Nuclear Science and Technology Organisation (ANSTO) – to study ochre samples from several sites around South Australia to characterize the elemental profile (1, 2).

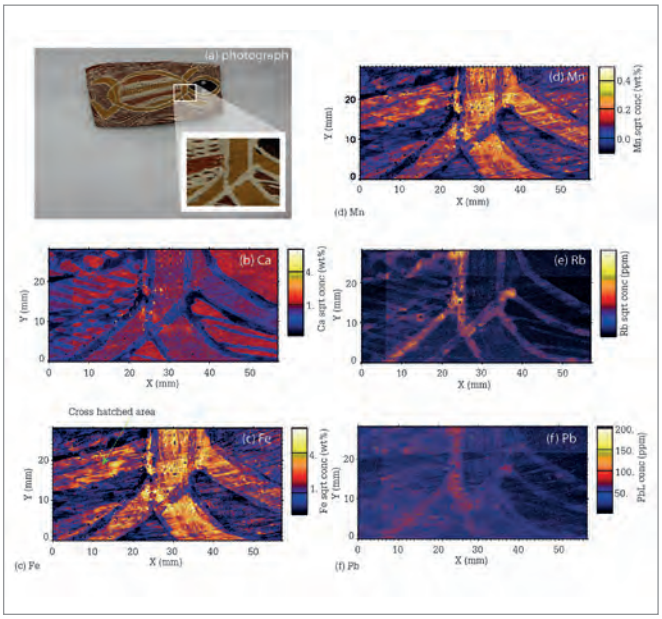
NAA is a useful technique as it requires 50 milligram amounts of sample for each bulk analysis. Yet, it still provides excellent precision and accuracy on different elements from percent down to the ppb level simultaneously. NAA has lower detection limits and higher precision than techniques like X-ray fluorescence spectroscopy (XRF) because NAA utilizes fundamental nuclear processes. Therefore, results are less affected by matrix interferences – often a limiting factor for XRF.

Thus, we were able to precisely and accurately quantify the bulk elemental concentrations of both the major and trace components of rare earth elements, transition elements and major composition elements in ochre. Using a log ratio method, we corrected for the majority iron concentration and subsequently removed elements that statistically correlated with iron. We then used principal component analysis and canonical discriminant analysis to differentiate ochre from different source locations across large geographic distances within a 90 percent confidence interval. These efforts established an important comparison database for understanding the provenance of ochre pigments without relying solely on contextual cultural information.

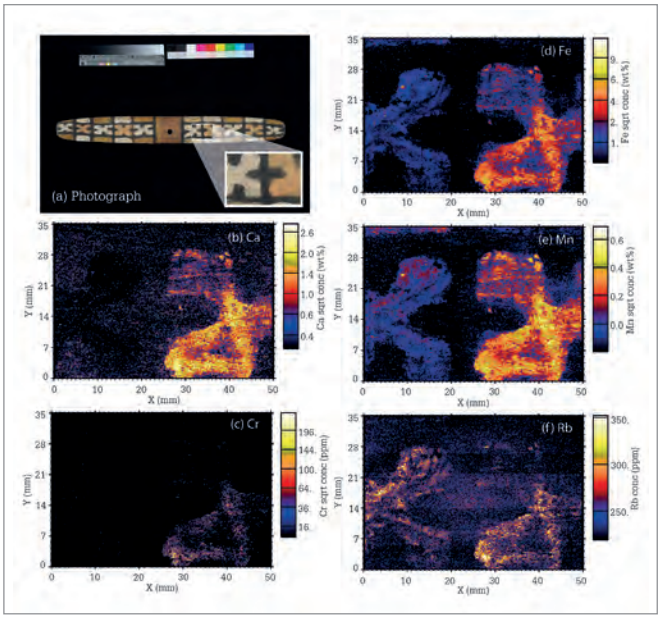
A CLOSER LOOK

A bulk analysis of ochre sources captures the average of any sample, but as this is an inherently complex material of multiple minerals and phases, it does not capture the contributions from individual grains. In addition, higher spatial resolution analysis is required to analyze the fine lines or thin layers of ochre often applied to substrates to create cultural heritage objects like boomerangs and bark paintings. Removing samples of these thin layers is also potentially disfiguring, so a non-destructive technique is required.

We therefore used the XFM beamline at the Australian Synchrotron to analyze both the source ochre and some ochre-treated objects (3, 4). This non-destructive analysis allows high-resolution (10-50 micron spot size) and semiquantitative elemental mapping of the applied mineral pigments. Using GeoPIXE software and modeling, we produced "heat maps" of elemental composition across the painted object based on spectral data. Creating heat maps of three elements in unison allowed us to identify a diversity of particle shapes, sizes and compositions, and identified that samples from the same geological source can still demonstrate variation on the micron scale.



Elemental maps showing the relative elemental concentration distributions for the pigments on a bark painting (3).



Elemental maps showing the relative elemental concentration distributions for the pigments on a boomerang (3).

Using these approaches, we visualized the application of pigments, including solid painted areas, fine lines and cross-hatching to an Aboriginal Australian boomerang and bark painting. Not only did we identify the major composition of

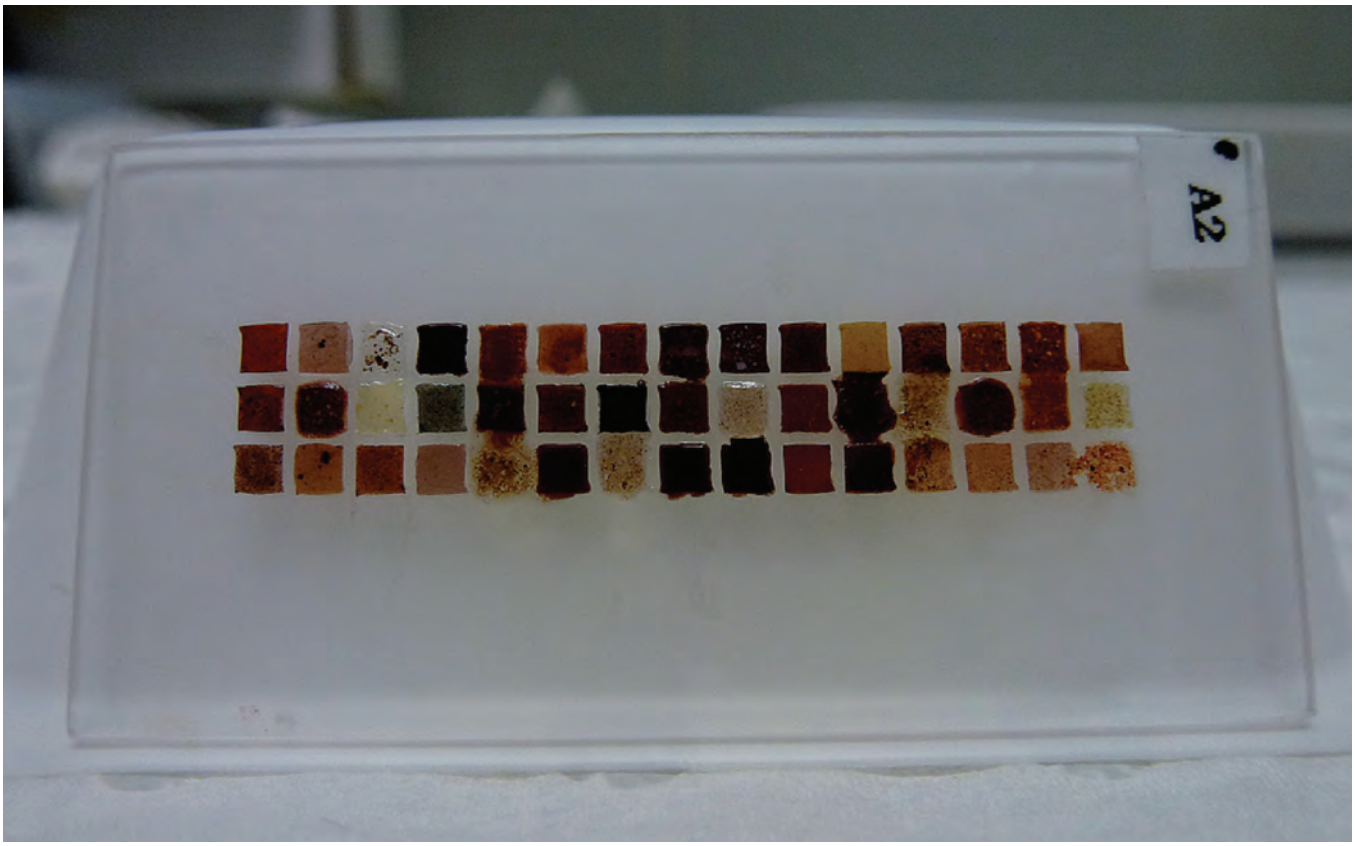
Image of a shield on a Hylogger™ instrument (7).

FROM MINING TO MINERAL ANALYSIS

By adapting a technique used in the mining industry – the HyLogger™ spectrometer – we developed an approach to non-destructively analyze and identify mineral pigments directly on Aboriginal Australian objects, regardless of the substrate or binder. This rapid spectroscopic logging and imaging system enables objects to be viewed in the shortwave-infrared and near infrared wavelengths – between 400 to 2500nm. As the technique uses two different segments of the electromagnetic spectrum, it allows us to identify other minerals in the pigment that are not visually apparent by color alone. The spot size in this technique is around 0.8 cm^2, so does not allow analysis of very fine pigment applications. However, this point-by-point analysis works very well on objects with larger areas of pigment application.

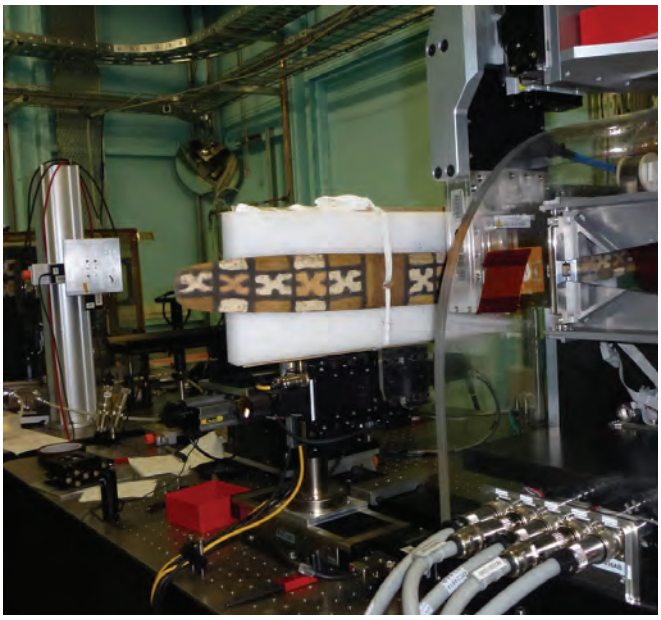
Using this technique, we identified previously unknown minerals in the pigments. The instrument scans at a rate of 1 cm per second, meaning artworks are analyzed within minutes (depending on the size of the object). For each "spectral pixel" analyzed, the instrument provides data on the individual spectrum, color, primary and secondary mineral interpretation, normalized Hull quotient data, and location on the image of the object (in cm). This setup is optimized for a range of minerals that are part of many common earth pigments. In the NIR, minerals containing Fe^{2+} and Fe^{3+} can be differentiated by their vibrational properties and the charge transfer between ligands and iron.

In the SWIR, identification of various aluminosilicates and other related minerals are possible. We discovered that the majority of pigments included kaolin, which agrees with cultural information, and that the crystallinity of the kaolin can be determined. However, we also identified gypsum, whewellite and other minerals as pigments or mixtures that had previously not been identified in cultural earth pigments.



A Petropoxy resin slide with 45 individual ochre samples. Each square is approximately 2 mm^2, with 0.5 mm between the squares.

Rachel with Attila Stopic at the NAA facility at the Australian Nuclear Science and Technology Organisation (ANSTO).



Aboriginal Australian boomerang mounted on the Australian Synchrotron XFM beamline, awaiting analysis (3).

the ochre pigments, including the differences between red and yellow pigments, but we also identified trace amounts of other elements such as rubidium. Thanks to the resolution of this technique, it was possible to visualize the fibers of the bark substrate beneath the pigment layer, too.

We were also able to model and estimate the thickness of pigments – about 40 microns – and gain a better understanding of the composition of pigments versus their color. Surprisingly, some of the yellow pigments were more iron-heavy than the red pigments. And, in particular areas, we could actually surmise the application order of layered pigments – deciphering the artist's application process. These elemental results agreed with our previous studies on the mineral composition of various red, yellow, black and white mineral pigments using an instrument adapted from the mining industry (see page 37).

But that's not all. With colleagues at Flinders University, we are also expanding the archeological science field further by investigating the role of microbial communities in the provenance of ochre (5,6). This method is established in forensic soil analysis and provides a unique perspective into the provenance of geological materials through a better understanding of their more recent environments, rather than those in the geological past.

To our knowledge, this is the first time DNA profiling has been used to determine the archaeological provenance of geological materials. We have previously demonstrated in a proof-of-concept paper that we can use 16SrRNA along with canonical analysis of principal coordinates to genetically profile ochre sources and distinguish between four ochre sites through their microbe profiles. These large, genetic data networks lend even more power to our investigations.

THE POWER OF PIGMENTS

Pigments remain a visually captivating medium, and one central to the expression of cultures across the world. In addition to appreciating their appearance, archeological science offers additional layers of interpretation about their technology, composition, and origins. Collaboration with cultural heritage professionals and community partners is essential to understand and design archeological science questions and discuss the use of cultural materials for analysis. Rather than being a potential afterthought on an archeological excavation or museum exhibit, archeological science will become an essential aspect of cultural research.

Through exhibits, programs and media, I hope the general public develops a better understanding of not only the cultural implications of these studies, but also the analytical chemistry methods behind them. No matter where we are in the world, current cultures are interlinked to those of our collective past. The power of analytical science lies in its ability to help us better understand our fundamental, universal connections with each other.

The author acknowledges the diversity of collaborations and publications with researchers at Flinders University, University of Melbourne, ANSTO, Australian Synchrotron, National Virtual Core Library-AuScope, South Australian Museum, Artlab, University of Missouri and Communities, and acknowledges funding from the following: AINSE Research Fellowship, Australian Research Council, University of Melbourne, Rock Art Australia and the Minderoo Foundation.

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Solutions

Real analytical problems
Collaborative expertise
Novel applications

Copycat Chemistry: Uncovering Counterfeit Drugs

As fraudsters get more sophisticated, it's vital that counterfeit analysis keeps pace

By Ravi Kalyanaraman, Director, and Scott Huffman, Associate Director, Global Quality Analytical Science and Technology group with Global Product Development and Supply, Bristol Myers Squibb Company, US

The US Food and Drug Administration (FDA) defines counterfeit medicine as a fake medicine that is contaminated, contains the wrong or no active ingredient, or contains the right active ingredient at the wrong dose (1). These "knock-offs" have caused significant harm to public health, and though many steps have been taken to deter fraudsters, fake pharmaceuticals remain a serious issue in both developing countries and the Western world. In fact, the Pharmaceutical Security Institute reported 5,081 crime incidents in 2019 – an all-time high – and the COVID-19 pandemic has only exacerbated the problem (2). Earlier this year, Interpol's annual Operation Pangea reported an increase of around 18 percent in seizures of unauthorized antiviral medication, and a more than 100 percent increase in seizures of unauthorized chloroquine (3) – a direct response to the coronavirus outbreak.

It is clear that counterfeiters will stop at nothing to make a profit, so what can be done to combat fraudulent pharmaceuticals? There are a number of measures in place around the world to detect counterfeits, including track and trace packaging, blockchain technology, and monitoring of online pharmacies. But with the global market for counterfeit medicines continuing to expand, it will become increasingly difficult to keep our supply chains safe. Here, we discuss the growing need for sophisticated chemical analysis of the drugs themselves, and our approach to telling the real products from the fakes.

Identifying a fake

The analytical methods used to detect counterfeits vary widely in the equipment, training, and preparation involved. For more detailed characterization needs, NMR, GC, HPLC, and MS are the more popular techniques, but they demand more extensive training and preparation of samples. On the other hand, spectroscopic techniques offer a faster and less labor-intensive route to counterfeit identification. Some of the more popular techniques include Raman, near-infrared spectroscopy, and mid-infrared spectroscopy. In addition, having access to energy dispersive X-ray and UV-Vis spectroscopy is useful when additional information is needed on a specific product; for example, to uncover provenance or link multiple cases together.

Typically, there is a progression of techniques that any counterfeit screening lab will run through to confirm or deny the authenticity of a product. Each and every case is different but, as a general rule, our team will start with a morphological examination of the sample, which involves examining the size, shape, and color of the suspect product, and looking for any inscriptions or engravings. We often use microscopy in this work, so we can take high-resolution images of the samples if we need to – especially if we start to see any evidence of foreign matter, or visual clues indicating inauthentic manufacturing processes or materials.

"There are a number of measures in place around the world to detect counterfeits, including track and trace packaging, blockchain technology, and monitoring of online pharmacies."

We'll then move on to a more detailed analysis of the sample. We usually rely on spectroscopic techniques – primarily because spectroscopy provides very specific data for what we consider to be a minimal investment of time and resources. In contrast to chromatography, where you spend maybe 90 percent of the time preparing and running your samples, and 10 percent interpreting the data, with spectroscopy it's almost the reverse. Moreover, it is non-destructive in nature – a key benefit when you're working with a limited amount of sample, which can go on to become part of evidence in court proceedings.

Raman spectroscopy is our go-to technique, but the same principle applies across all our vibrational spectroscopic approaches. Suspect drugs are scanned using a laser, which results in a change in the energy state of the scattered light

One Solution: Drop Coat Deposition Raman

With a growing number of counterfeit biologics cropping up worldwide, drop coat deposition Raman offers quick and accurate fingerprint analysis for proteins

The nature of biologics makes them difficult to analyze using traditional spectroscopy methods. The non-specific nature of certain bioanalytical techniques can throw up problems, and further issues can arise because of the sample volume required or because of the need for spectral subtraction (mid-IR and circular dichroism). However, it is possible to gather a wealth of information on a particular protein structure, and its environment, using a Raman spectral fingerprint – it's just a matter of getting the sample preparation right.

Drop coat deposition (DCD) uses microvolumes of a sample, which are manually dropped onto a hydrophobic substrate – a stainless steel plate with a thin layer of Teflon coating. Following solvent evaporation, the protein is pre-concentrated to form a "coffee ring." This high concentration of protein enables a spectral measurement to be taken with a much higher signal-to-noise ratio, without compromising the native structural form of the protein when in solution. A confocal Raman microscope can then be used to image the "coffee ring" and produce a spectral fingerprint of the protein.

We've previously shown that it's possible to accurately predict the secondary structure of proteins using the Raman Amide I vibrational band (4, 5). This band is dependent on the secondary structure of a protein, and can therefore be used as a fingerprint for a biologics drug. DCD Raman (DCDR) therefore offers a route to fast detection of counterfeit biologics, requiring little sample preparation, low volumes, and no spectral subtraction. Combined with peak fitting and other data analysis, this technique could also offer a way to distinguish between biologics and their generic versions (biosimilars).

by the different chemical functional groups present in the suspect product. The resulting spectral fingerprint can then be compared against the fingerprints of known drugs. Notably, our lab ensures that spectral fingerprints are consistent across different batches of the same Bristol Myers Squibb product – we measure the degree of fingerprint variation across batches, validating with products from other manufacturers. In this way, we can ensure that fingerprints are unique for each and every type of medicine we produce.

Knowing your limits

Our approach to counterfeit detection depends on the product we are testing and the limitations of certain techniques. For example, highly colored samples are problematic for Raman spectroscopy – with the more powerful lasers of a benchtop instrument, you can end up burning your sample. With limitations in mind, it's also important to have a complete toolbox of complementary techniques at your disposal. This way, you can piece different data together to get a complete picture of a drug.

One of our major limitations, until about 4 years ago, was the inability to routinely analyze biologics spectroscopically because of their size and complexity – and their low concentration in the aqueous solutions in which they are often formulated. That meant running more traditional, labor-intensive tests using MS or NMR that also consume the precious sample. To overcome the problem, our team at Bristol Myers Squibb developed a benchtop method for Raman analysis of biologics, using

"It's going to become harder for us to tell the difference between authentic and fake drugs."

a special sample preparation technique called drop coat deposition (DCD) (see sidebar). So far, the method has enabled us to stay ahead of biologics counterfeiters

Smarter, faster, more productive

The bad news is that counterfeiters are getting smarter and they are learning from their mistakes; over the years, we've seen counterfeit products get better in quality. As this continues, it's going to become harder for us to tell the difference between authentic and fake drugs. All manufacturers must continue to keep pace with emerging trends by employing the most cutting-edge technology in their counterfeit screening labs.

We're not only on the hunt for the latest technology, but we are also increasingly looking into portable technology. In fact, we're currently working on a handheld version for our biologics analysis using a portable Raman spectrometer. Miniaturization boosts flexibility and coverage by enabling any time, any place testing – but it does tend to come with a trade-off in terms of performance. That said, we've typically found that the benefits outweigh any drawbacks; for example, portable instrumentation allows us to roll out analytical technology in manufacturing sites across the world, and even in the hands of non-scientists, which improves efficiency and empowers the user.

As well as miniaturization, we also see an increasing need for user-friendliness. As counterfeiters get more sophisticated and widespread, it's inevitable that we're going to have to increase testing, which means more people outside of the lab environment conducting analyses. Whether it's the patient, the pharmacist, or someone else along the supply chain – these tools must be accessible to those without professional training. The software and sensor might increase in sophistication, but the usability must be streamlined and simple.

It's true that counterfeiters are getting increasingly intelligent in their approach. At some point, they will likely find a way to fool our packaging and tracking authentication systems. But being able to get the exact same chemical composition as a regulated product? Unlikely – and that's why counterfeit analysis labs need to stay ahead of the game.

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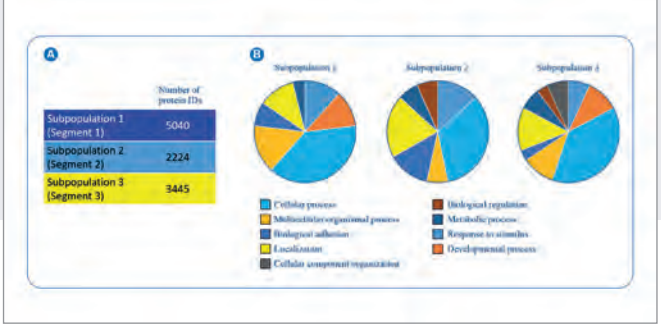

Figure 1. Proteomics of tumor subpopulations and biological process characterization. Proteins from microdissected tissue (approx 160 ng) were extracted, digested with trypsin and peptide extracts were run on the timsTOF fleX using PASEF. Number of protein IDs per tumor subpopulation segment A and biological process characterization per segment as revealed by PANTHER B.

MALDI Guided SpatialOMx® Reveals Proteomic Heterogeneity in Tumor Subpopulations of Breast Cancer

MALDI Guided SpatialOMx® provides an excellent tool for discovering deep proteomics insights into heterogeneous tumor subpopulations. The timsTOF fleX system bridges a current gap by providing regiospecific targeting of a multi-molecular imaging technique with 4D-Proteomics™ analysis in just one instrument. The instrument offers all benefits of a timsTOF Pro for time-efficient and sensitive 4D-proteomics, combined with high-resolution, label-free molecular imaging. Novel PASEF technology enables high protein ID rates with very small sample amounts typical of microextracted tumor subpopulations. Key findings: 1. SpatialOMx is a new workflow for in situ characterization of tissue subtypes based on molecular expression; 2. locations of selected subtypes guide laser-capture microdissection to cells of a specific molecular phenotype; 3. timsTOF fleX uses 4D-proteomic analysis of small microdissected tissue pieces to explore differences related to molecular phenotype; and 4. SpatialOMx, combining MALDI imaging and LC-MS/MS, with timsTOF fleX provides deeper proteomics profiling correlated to cell phenotype.

Learn more about new SpatialOMx workflow and its powerful approach in identifying distinct proteomic profiles for different tumor subpopulations in breast cancer: https://www.bruker.com/products/mass-spectrometry-and-separations/lc-ms/o-tof/timstof-flex.html

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Phenyl2: Reversed-phase material in which π-π and hydrophobic interactions take place. This phase is similar in hydrophobicity to a C4 but given the different interaction of the phenyl groups, it displays a high degree of orthogonality to C18 and C4.

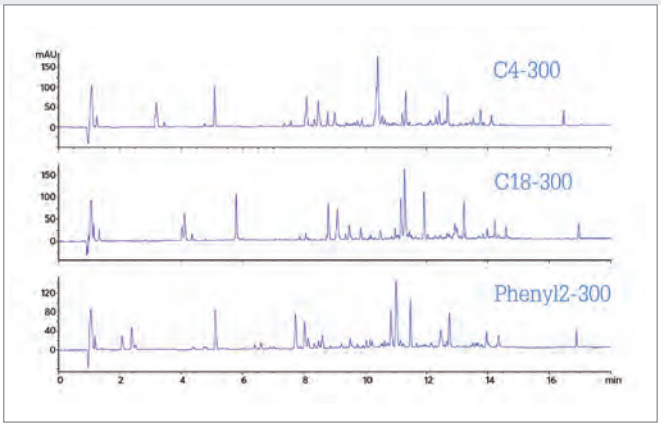

Figure 1. Tryptic digest of lysozyme on C4, C18 and phenyl2 wide-pore columns.

Spotlight on... Applications

An LC/MS Solution to Study Water Pollution

Per- and polyfluoroalkyl pollution is a serious concern that must be addressed by analytical techniques at the trace level. This Application Note describes a sensitive and reliable method for simultaneous quantitation of 53 legacy and emerging PFAS compounds using an Agilent 1290 Infinity II LC coupled to an Agilent 6495B triple quadrupole LC/MS. *https://bit.ly/3jPv5Ud*

Correlative Raman Imaging of Polymeric Materials

Knowledge of the morphology and composition of polymers is crucial to advancing their development. Raman imaging correlated with atomic force microscopy and scanning near-field optical microscopy is an especially versatile tool for their investigation. This application note describes the combined technique and provides examples of its use. *https://www.witec.de/assets/Literature/Files/WITec-AppNote-PolymersCorrelativeRamanMicroscopy-WebVersion.pdf*

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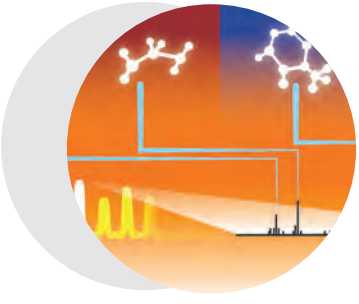

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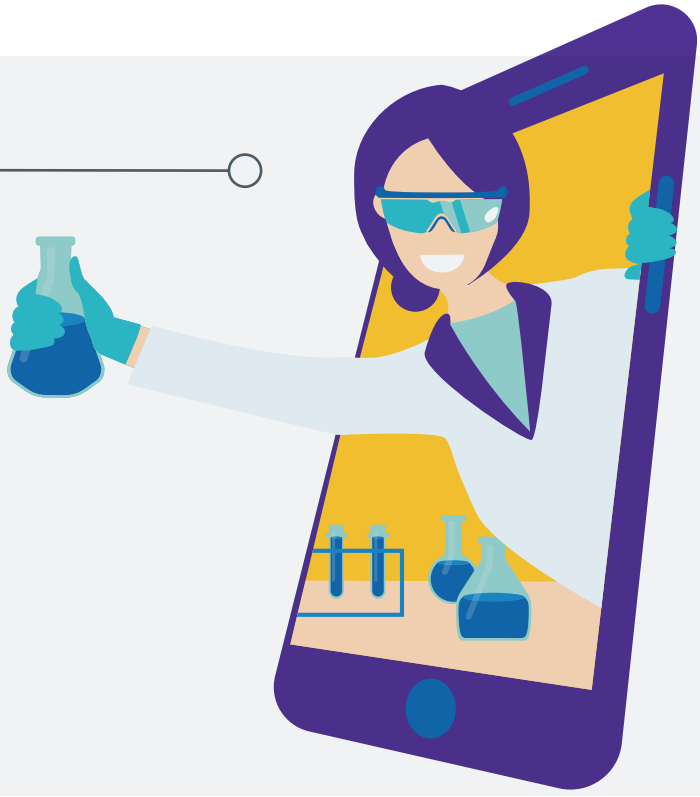

Veterinary Drugs in the Meat we Eat

This app note describes the development of a comprehensive LC/MS/MS workflow to quantify 210 veterinary drug residues in meat. Performance was evaluated across chicken, pork and beef using two different Agilent triple quadrupole LC/MS models (an Agilent 6470 and a 6495C triple quadrupole LC/MS). The aim: to accelerate and simplify routine testing.

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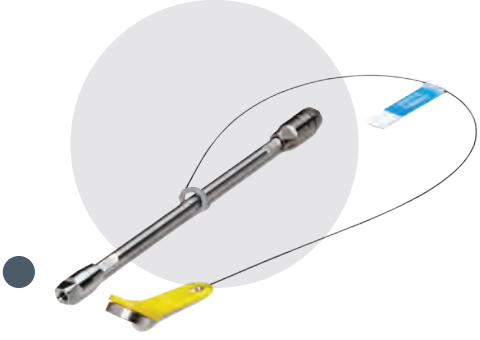

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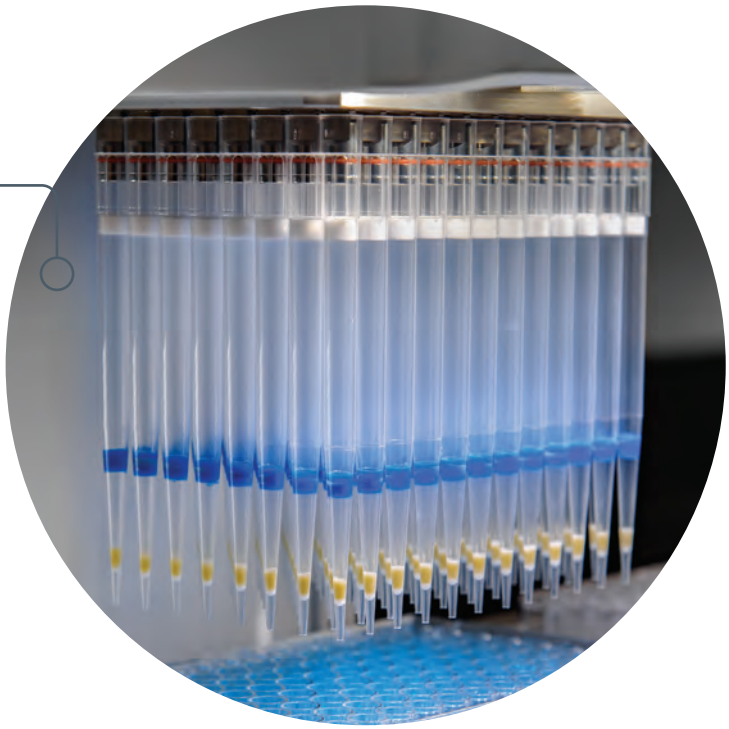

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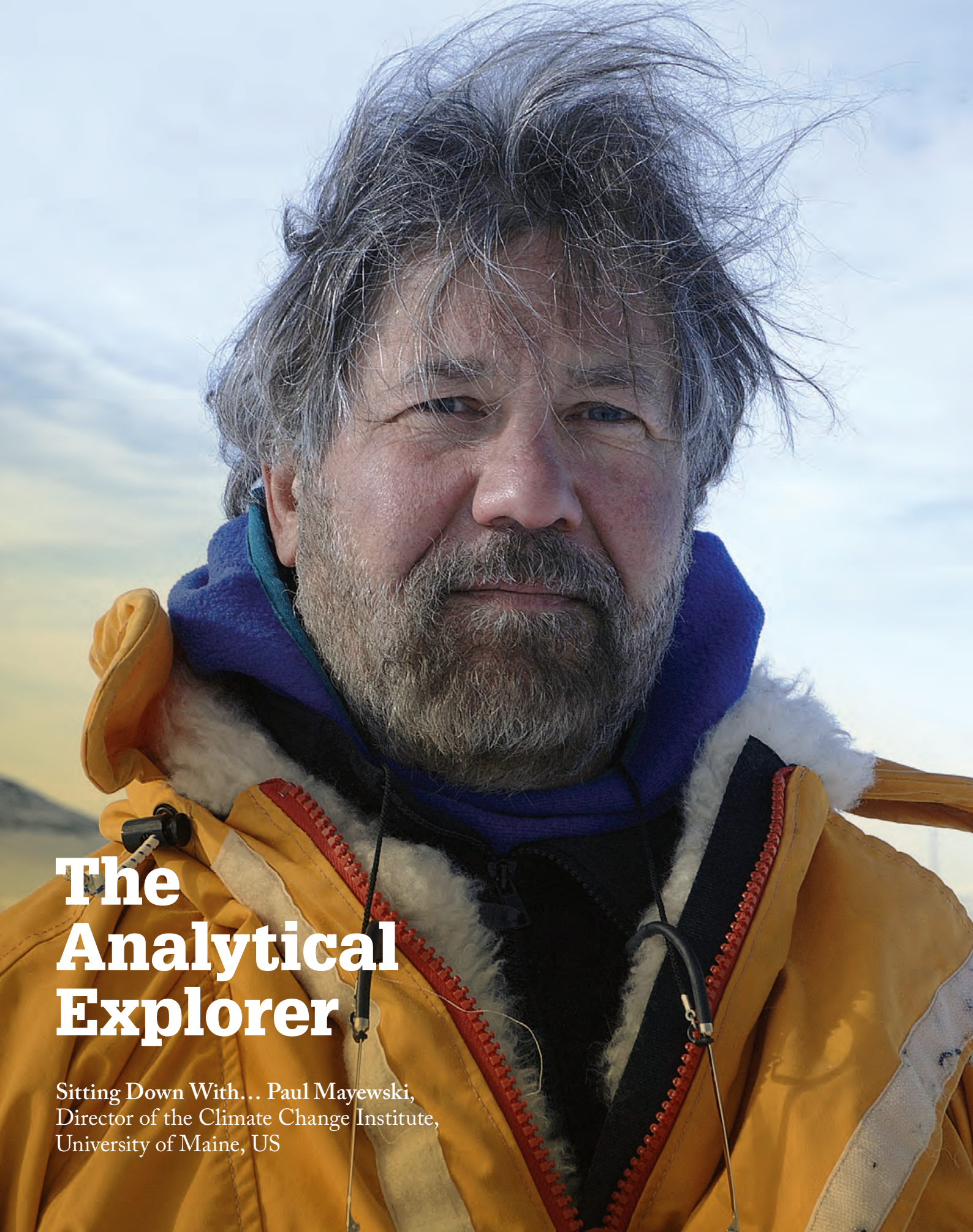

The Analytical Explorer

Sitting Down With… Paul Mayewski,
Director of the Climate Change Institute,
University of Maine, US

You've led more than 60 expeditions to some of the most remote polar and high altitude regions of the planet, what drew you to becoming a polar explorer in the first place?

I've always wanted to be an explorer. I spent much of my early school days in New York City, where I frequently went to the Museum of Natural History – and it got me thinking about distant places. By the time I went to college, my goal was to try to go to the Antarctic. My second year college professor showed a picture of Antarctica in one of his lectures and I asked him if he would take me there! He did, and I've been exploring these remote regions ever since.

When did you get involved in climate science?

I've always been driven by the desire to go to remote places and simply explore, but it occurred to me as the years went on that there were other types of exploration – namely, scientific exploration – and that these could really add significant value to people's lives. It's part of the reason I started going to Asia – I thought if I was really going to do something that made a difference, I needed to visit the areas where people were living.

The reason I got involved in ice cores specifically is all down to one student. By the late 1970s, I was running a course on glaciers in Canada. I asked each of them to write a proposal about what they wanted to do, and we'd figure out how to do it. One of these students said she wanted to work on ice cores – it turned out to be a brilliant suggestion because it's what I've been doing ever since!

How did the idea of chemically fingerprinting ice cores develop?

We certainly weren't the first to do chemical fingerprinting of air masses – but we were the first to demonstrate how it could be applied to ice cores. I led an expedition in 1980 to an extremely remote part of the Himalayas in Ladakh, India. We weren't allowed maps, or any form of communication, and it took us over six weeks just to reach the site from the US. In any case, we got there in the end, and we managed to drill this ice core. I was looking around one day with the team and we saw snow coming from the Tibetan plateau in one direction, and snow coming from the Indo-Gangetic Plain in the other. Intriguingly, they came together and precipitated down the mountain, so we decided to take samples. Sure enough, we could see different chemical fingerprints for the different air masses. The Tibetan was colder as it comes from a higher source, and it had a lot of dust in it. The Indo-Gangetic one had a marine chemical signal, an agricultural signal, and signs of pollution – there were a variety of things there to distinguish them. So that's really when we decided to apply chemical fingerprinting to ice cores as well.

What sort of information can you gain from ice cores?

Well, chemically fingerprinting ice cores allowed us to prove that acid rain was actually something produced by human activity. Back in the 1980s, people thought acid rain was natural and humans weren't to blame. We could show that, in fact, the emissions from both North America and Europe absolutely tracked the levels of sulfuric and nitric acid that we found in the ice cores from Greenland.

But that's just one (admittedly impressive) example – we've also shown that open pit mining in Australia over a 35-year period increased the levels of uranium in the Antarctic Peninsula and that the Chernobyl nuclear accident made its way to the South Pole. Years ago, an Egyptologist approached me and wondered whether there was any reason why some dynasties worshipped the Sun more than others. Hieroglyphs showed that at some times the people wore more clothing while worshipping the sun, and at other times they wore less and seemed less interested in the sun. Our ice core data showed that there were warmer and cooler periods in Egypt that correlated with the hieroglyphs.

What's the most challenging experience you've had in your career?

Obviously, as an explorer, there are many risks when traveling to these remote areas. Crevasses are a serious danger – friends have been lost on other expeditions. I once spent 17 days pinned down on a mountain because of wind storms. Access to food can often be an issue. Frostbite... Lightning! When you're on top of a mountain, no matter what you do you're going to be one of the highest points out there. So that can be pretty scary. I've also found myself in war zones because of my work. Of course, I do get scared, but I love the job.

How has the field changed since you started out?

It's become a lot more popular! There are so many applications now, and the younger generation are becoming increasingly interested in climate and the environment – it's great. It's also become a lot more diverse, at least in terms of gender – when I started working in Antarctica there were very few female graduates in the field and women were not even allowed to visit the US or British bases in the Antarctic! Now, in our institute, slightly more than half of all our graduate students are women.

Any advice for budding scientists (or explorers) out there?

Look at what you love doing, combine your interests, and seek opportunities. Today, most scientists understand that climate change is anthropogenic – but it seems many people think that's the end of the story. It's not. We're still learning so much about our climate system, and there are going to be surprises in the future. It's vital that the younger generation are inspired to tell this constantly evolving story about our climate – because there's tonnes more to explore!

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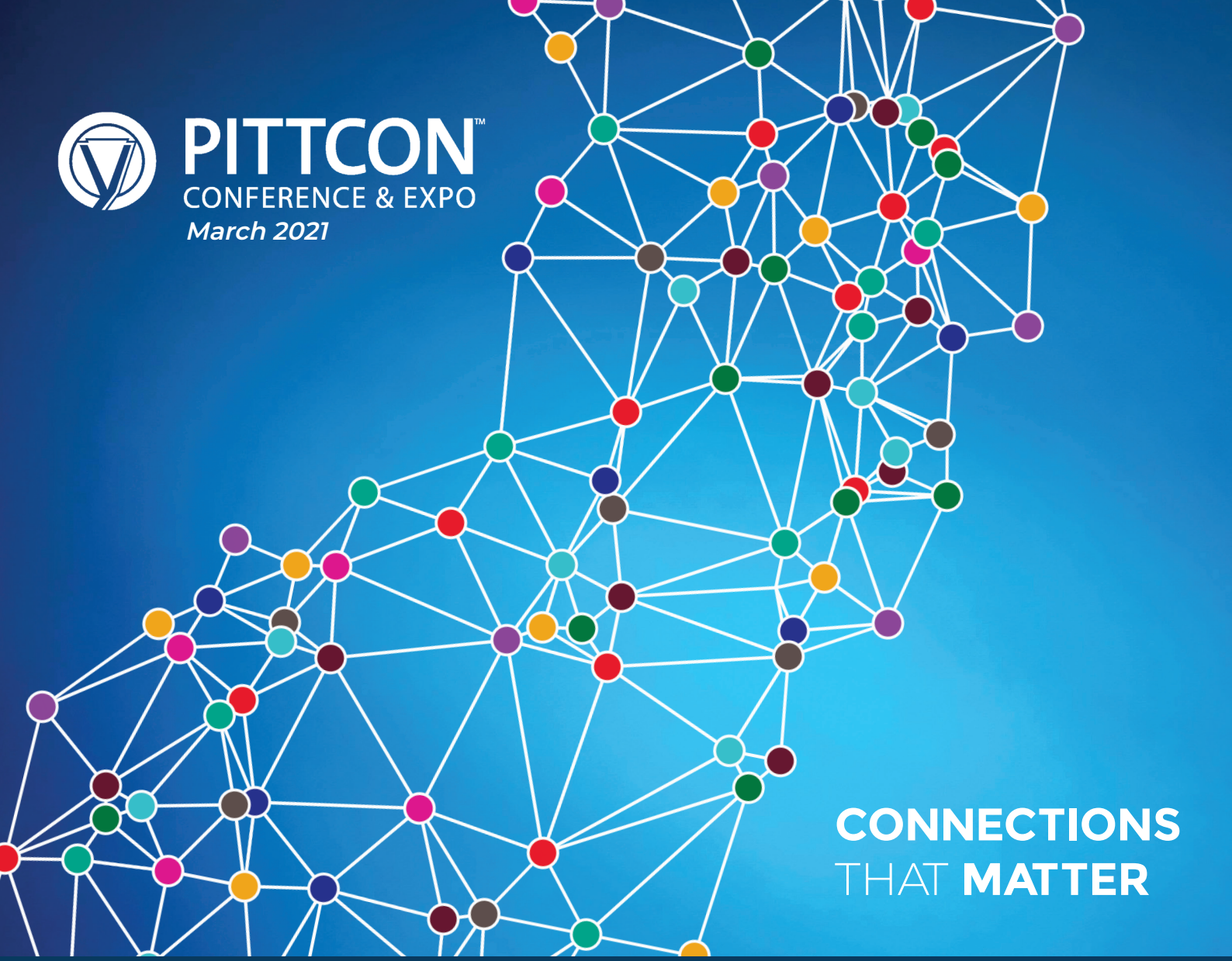

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