Special Series Environmental Analysis

Five pioneers explore new frontiers in the quest to protect our planet – as part of our special collection of recent environmental content

the Analytical Scientist x GL Sciences

Portable PFAS Profiling

Using nanopore technology, Chang Liu and Xiaojun Wei discuss their accessible and inexpensive new option for detecting "forever chemicals" PFAS

Researchers from the University of Massachusetts Amherst, USA, have designed a cost-effective device to identify per- and polyfluoroalkyl substances (PFAS) – the group of "forever chemicals" that resist environmental degradation and are associated with a wide range of health complications.

Using biological nanopores with a cyclodextrin adapter, the device is capable of identifying PFAS levels as low as 400 parts-per-trillion (ppt).

We reached out to co-authors Chang Liu and Xiaojun Wei to find out more about how the instrument was developed, and to get their perspectives on how the approach compares to current methods.

Could you describe how your device works?
We utilized single-molecule nanopore technology as a promising approach for developing portable detection devices, specifically employing α-hemolysin (α-HL) nanopores embedded in a liposome membrane to create a single-molecule channel. This setup enables the characterization of molecules within the pore by detecting changes in ionic current signals. To target PFAS molecules, we used γ-cyclodextrin (γ-CD) in a "host-guest" strategy, incubating it with PFAS molecules to form complexes. Due to its unique structure, γ-CD also functions as an "adapter" within the nanopore.

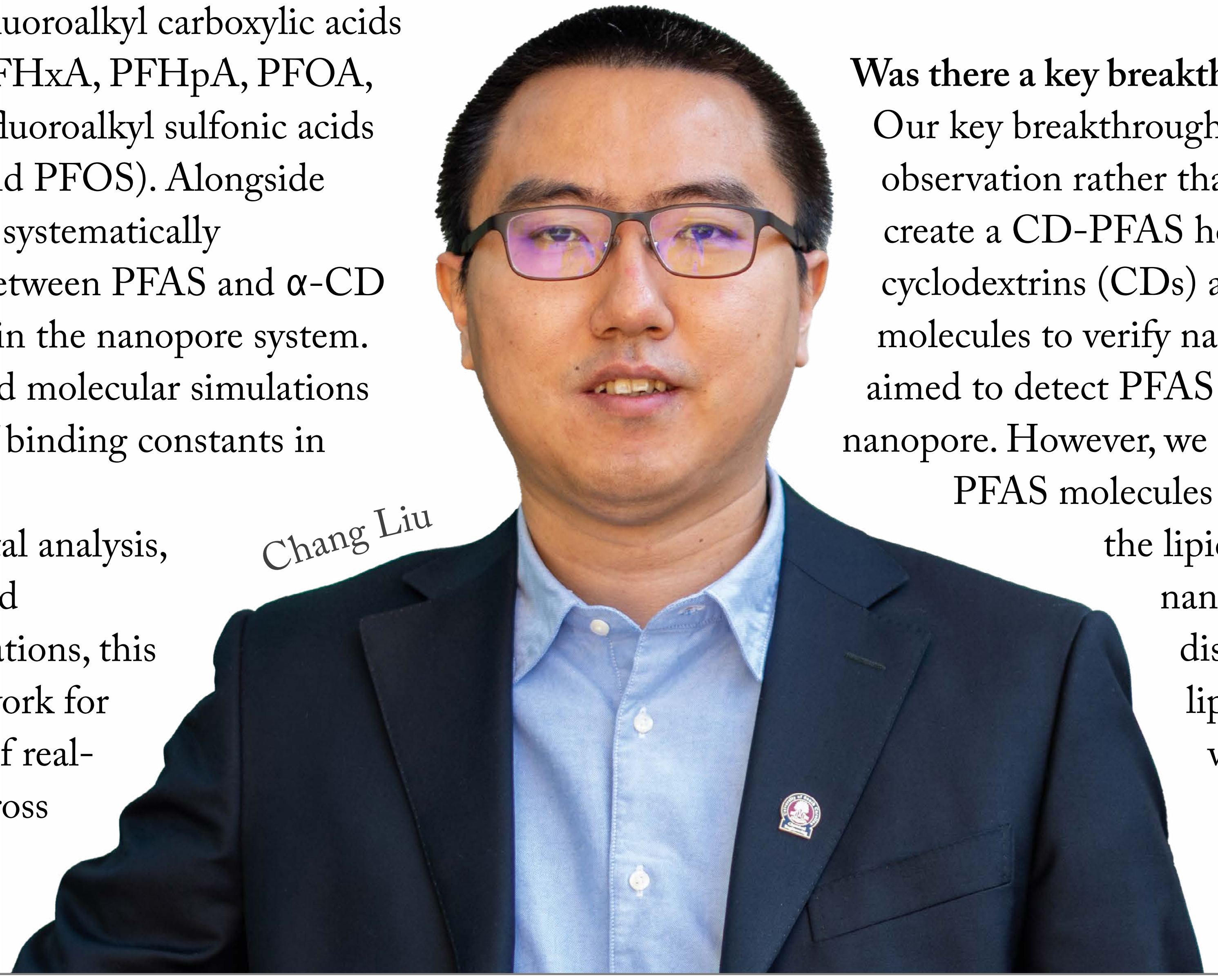
Through this design, interactions between PFAS and γ-CD in the nanopore system produce measurable current signal changes, facilitating the detection of perfluorooctanoic (PFOA) and perfluorooctanesulfonic (PFOS) acid molecules. By modifying the CD structure, specifically using 2-hydroxypropyl–CD (HP-γ-CD) as the host molecule, we achieved selective identification of PFOA and PFOS molecules, detecting PFOA, PFOS and mixed PFAS species in water directly at concentrations of 0.4-2 parts per million, which can be further reduced to 400 parts per trillion after pretreatment.

Additionally, the host-guest interaction between HP-γ-CD and PFAS molecules enabled us to identify broader PFAS families, including six perfluoroalkyl carboxylic acids (PFCAs: PFBA, PFPA, PFHxA, PFHpA, PFOA, and PFNA) and three perfluoroalkyl sulfonic acids (PFSAs: PFBS, PFHxS and PFOS). Alongside γ-CD and HP-γ-CD, we systematically investigated interactions between PFAS and α-CD and β-CD molecules within the nanopore system. Theoretical calculations and molecular simulations revealed the critical role of binding constants in these interactions.

Combining experimental analysis, ion transport modeling, and molecular dynamics simulations, this approach lays the groundwork for a portable device capable of real-time PFAS monitoring across diverse environments.

What was the biggest analytical challenge you faced during your research?
We were initially excited to detect PFAS signals after incubating with γ-CD and PFOA or PFOS; however, we quickly realized that using γ-CD as an "aptamer" did not yield differentiated signals for PFOA and PFOS. Our challenge was to identify a suitable cyclodextrin to serve as an aptamer. After further investigation we found that by optimizing the cyclodextrin structure through chemical modifications, we could enhance detection sensitivity. Through continuous exploration, we selected HP-γ-CD as a new adapter, achieving differentiation and recognition of various PFAS molecules.

Was there a key breakthrough during development?
Our key breakthrough came from an unexpected observation rather than a deliberate attempt to create a CD-PFAS host-guest structure. In our lab, cyclodextrins (CDs) are commonly used as reference molecules to verify nanopore functionality. Initially, we aimed to detect PFAS molecules directly through the nanopore. However, we quickly discovered that adding PFAS molecules directly to the system caused the lipid membrane supporting the nanopore to break down, due to the disruptive effects of PFAS on the liposome. During one experiment, we noticed that the membrane's stability improved significantly when β-CD and PFAS


Chang Liu

> *"Our method offers a unique approach to PFAS detection by using a nanopore system with cyclodextrin as an adapter for PFAS recognition, providing a potentially rapid and cost effective alternative to traditional techniques."*

molecules were both present in the system. This led us to consider that introducing a CD molecule could enable PFAS detection via the nanopore – a realization that was truly exciting! Our second "eureka moment" came when we replaced β-CD with γ-CD and observed distinct PFAS signals in the nanopore's current readings, indicating successful detection.

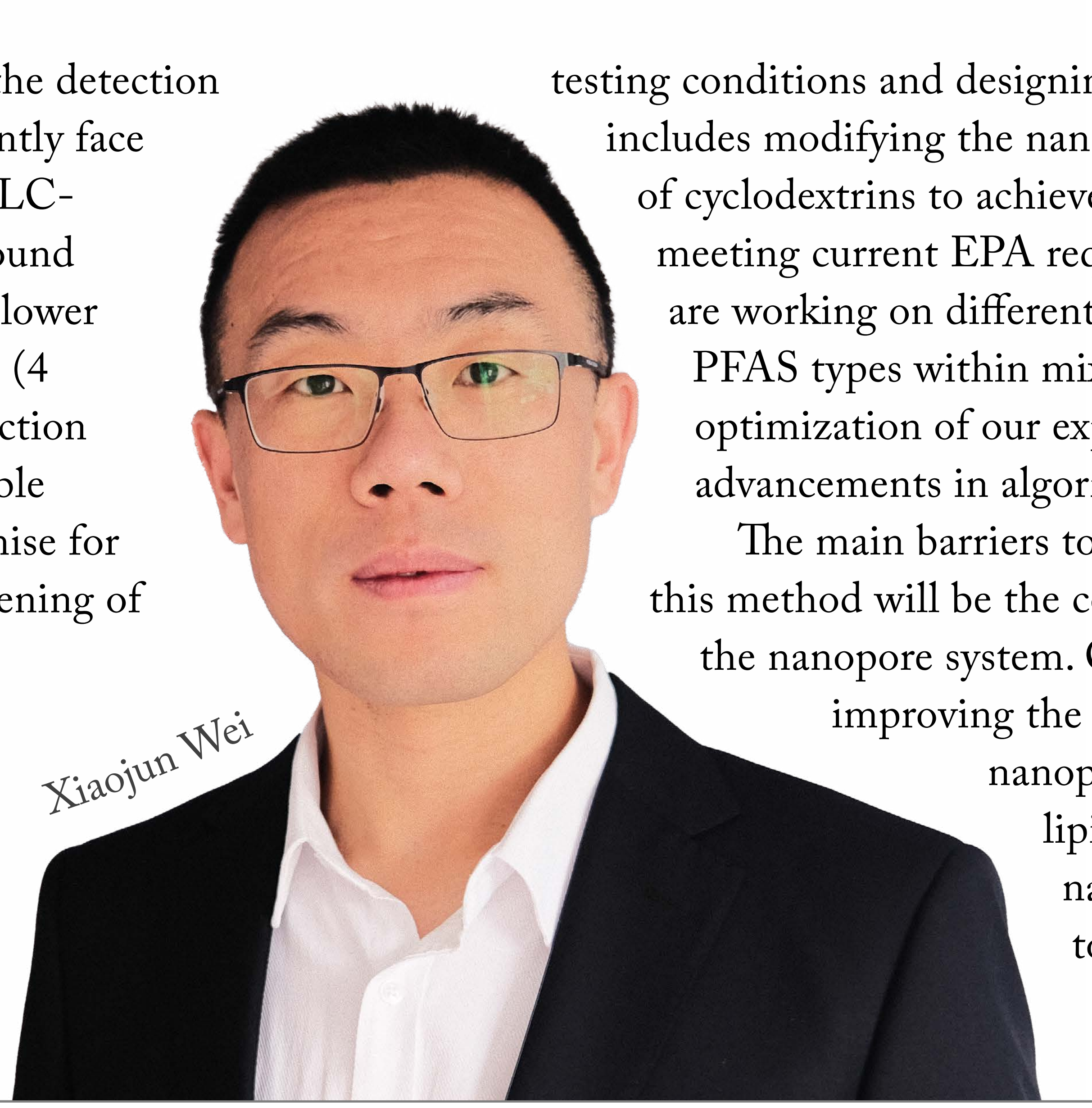
How does your method compare with the "gold standards" of PFAS testing?

Our method offers a unique approach to PFAS detection by using a nanopore system with cyclodextrin as an adapter for PFAS recognition, providing a potentially rapid and cost-effective alternative to traditional techniques. Unlike the "gold standard" approach using liquid chromatography-mass spectrometry (LC-MS), which is highly accurate but also time-intensive and costly, our nanopore approach requires smaller sample volumes and aims to simplify the detection process as a whole. However, we currently face limitations in sensitivity compared to LC-MS, as our detection limit remains around 400 ppt, whereas LC-MS can reach a lower level to meet the latest EPA standards (4 ppt). Further optimization in our detection strategy is needed to achieve comparable sensitivity, but our method holds promise for field-based and rapid preliminary screening of PFAS in environmental samples.

What are the next steps for your research?

Our next steps involve enhancing detection sensitivity by optimizing testing conditions and designing new detection channels. This includes modifying the nanopore and improving the structure of cyclodextrins to achieve lower limits of detection, ideally meeting current EPA requirements. Additionally, we are working on differentiating and quantifying various PFAS types within mixtures, which will require the optimization of our experimental design as well as advancements in algorithms and data analysis.

The main barriers to more widespread adoption of this method will be the controllability and stability of the nanopore system. Currently, challenges include improving the long-term storage of biological nanopores and potentially replacing the lipid membranes that support these nanopores with synthetic materials to increase robustness.



Xiaojun Wei

Worse Than the Sum of their Parts?

Chemical-by-chemical regulation won't cut it, Beate Escher argues, after her team discover that chemicals at thought-to-be-harmless concentrations can form neurotoxic mixtures in the body

Automated pipetting platforms for the preparation and measurement of plasma samples and chemical mixtures in high-throughput bioassays at the UFZ.

Credit: Bodo Tiedemann

Naturally ingested chemicals that are usually ineffectual at low concentrations have the potential to form mixtures with others in the human body to produce neurotoxic effects, according to a recent study involving pregnant women.

A team from the Helmholtz Centre for Environmental Research (UFZ) used high-resolution mass spectrometry (HRMS), in addition to an in vitro assay, to determine the neurotoxic effects when environmental chemicals are combined in the human body. They discovered that certain chemicals could partner with others to produce neurotoxic effects on analytes, despite having no effect on their own.

The team extracted plasma samples from 624 pregnant women from the Leipzig mother-child cohort LiNA, using a nonselective extraction method for organic chemicals. They then used HRMS to test for 1,000 different chemicals that can be ingested by humans, which are naturally present in the environment, of which they were able to quantify around 300. From there they analyzed the neurotoxic effects of the individual chemicals, as well as around 80 self-produced chemical mixtures in realistic concentration ratios, using a prediction model to quantify the degree of neurotoxicity and a cellular bioassay (based on human cells) to then test their predictions.

To better understand the inspiration and implications of the findings, we reached out to first author Beate Escher for her thoughts on the study.

What was your main inspiration for this work?

After working on complex mixtures in the environment for many years now, we asked ourselves how they might transfer to the human body. With this in mind, we took the target screening analytical methods for wastewater and surface water and exchanged a fraction of the chemicals by suspected neurotoxicants.

Our team has a lot of experience with high-throughput toxicity testing of environmental samples and chemicals – physically mixing chemicals in the concentration ratios they're detected in (environment or people), before testing in the same assay where the sample extracts and single chemicals are also assessed. Comparison of mixture effect models and effects allowed us to evaluate how chemicals act together in mixtures.

What was the biggest analytical challenge you faced during your research and how did you overcome it?

We encountered several challenges. First, we had to improve the extraction method in order to extract hydrophilic, hydrophobic, neutral, and charged chemicals. What you do not have in your extract you cannot detect, even with the finest method. As we combined chemical analysis with mixture effect assessment with bioassays, internal standards could not be added. In the end, we settled for a two-step extraction process that gave us the greatest recovery of 400 chemicals and also mixture effects. We then performed polymer extraction with silicone followed by solid-phase extraction.

Next we had to perform quantitative chemical analysis to measure and model mixture effects. Our goal was to capture up to 1000 chemicals, so we had to work with a target screening analysis using external standards. To make the chemical space as broad as possible, we used LC-HRMS and GC-HRMS in parallel and complemented the chemical analysis with the quantification of mixture effects using an in vitro bioassay. Finally, we had to develop and optimize an automatic script (data evaluation is a nightmare!).

We had many samples, which meant we needed to test the mixture effects with a bioassay that was simple, yet representative. Our bioassay is based on neuroblastoma cells that have been differentiated to form neurites and perform signal transduction – although it is an artificial test system, it mimics basic functions of neural cells. When using a more complex test system (or even animal studies), it isn't possible to perform toxicity tests that produce reliable and reproducible results with >1000 samples (e.g. blood extracts, single chemicals, mixtures). For this reason we have evaluative in vitro test systems. The assay we are using is well-established in research, we simply applied it to high throughput. The idea was not to test health effects directly, but to capture mixtures of chemicals that cause a specific effect (in this case neurotoxicity).

Can you sum up the significance of your findings for our readers?

We found more and more diverse chemicals in human blood, ranging from legacy chemicals such as persistent organic pollutants, to more modern chemicals. Despite being present at low concentrations where they wouldn't cause an effect on their own, all of them acted together in mixtures.

Ultimately, we were able to view the entire history of chemical production over the course of 100 years. From metabolites and long phased out persistent organic pollutants (POPs) such as dichlorodiphenyltrichloroethane (DDT), to modern chemicals, food contact material chemicals, personal care products, antioxidants, industrial chemicals, what's found in the environment and the kitchen cabinet; you can find it if it is sufficiently persistent. Perfluorooctanoic acid (PFOA) was found in almost every sample. We couldn't capture everything as our screening analysis was too coarse – our aim was to quantify new and old, persistent and degradable, to demonstrate the relevance of mixtures of seemingly unrelated compounds. We designed over 80 mixtures in concentration ratios as they were detected in individual humans, and in every case the in vitro effects added up.

What implications could your findings have for our understanding of chemical exposure risk, monitoring, and regulation?

Our findings clearly show that we cannot regulate on a chemical-by-chemical basis, and that complex mixtures must be addressed in risk assessment and regulation.

Democratizing Microplastics Analysis

How a portable fluorescence labeling tool detects micro- and nanoplastics released from disposable cups and water bottles

A low-cost, portable device developed by University of British Columbia researchers can quickly detect micro- and nanoplastics released from everyday items, such as disposable cups and water bottles. The tool uses luminescent metal-phenolic networks (L-MPNs) to fluorescently label and identify plastic particles as small as 50 nanometers, providing rapid results with minimal sample preparation.

L-MPNs, made from zirconium ions, tannic acid, and rhodamine B, enhance the fluorescence of microplastics, allowing the particles to glow under a green LED light in the device's integrated microscope – enclosed in a biodegradable 3D-printed box. The wireless digital microscope is connected to a smartphone app powered by customized MATLAB software that uses machine-learning algorithms. By capturing and analyzing images of plastic particles, the device is able to "count" the plastics in the sample.

In the study, the device successfully identified microplastics released from disposable polystyrene cups after exposure to hot water. The study found that these cups released millions of nanoplastic particles, highlighting the potential for everyday items to contribute to microplastic pollution. The device operates with less than a drop of liquid sample and delivers results in minutes, making it suitable for both laboratory and field applications.

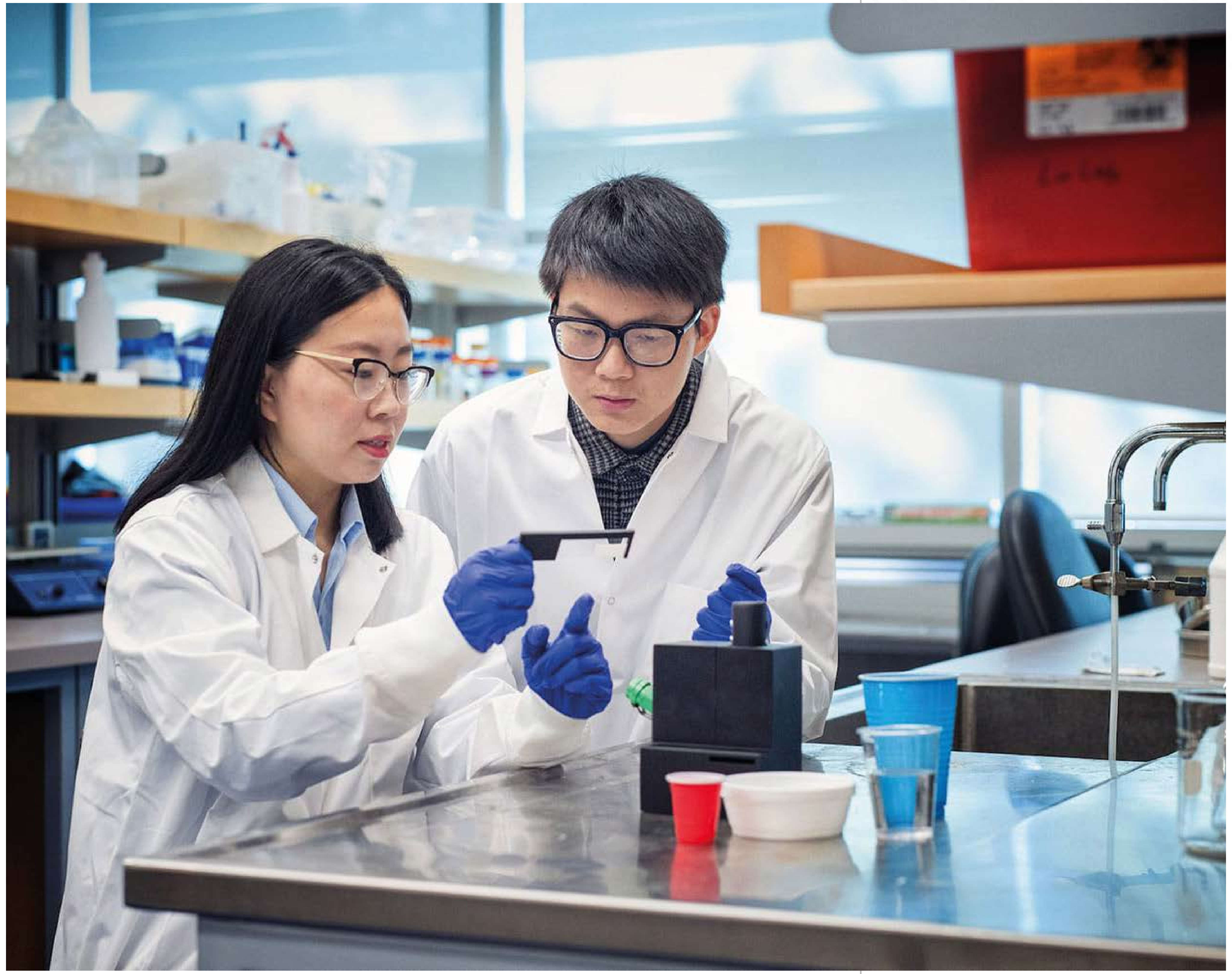
Each test costs about 1.5 cents, and the device's portability means it can be used in various settings, from food processing labs to environmental monitoring stations.

"My team aimed to create an accessible, low-cost detection tool that could be used by anyone, from scientists to everyday consumers, to identify microplastics and nanoplastics in liquids," says Tianxi Yang, an assistant professor in the faculty of land and food systems, and corresponding author of the study.

"One of the biggest challenges during the research was simplifying the complex process of microplastic detection, which traditionally requires expensive equipment and skilled personnel, such as using transmission electron microscopy (TEM)," says Yang. "TEM also can not achieve quantification. In contrast, our new device, although slightly less accurate regarding the size and shapes of plastic particles than TEM, achieves impressive results by using a wireless digital microscope, LED light, and machine-learning algorithms to identify plastic particles with ease, portability, and affordability."

"The technology could be employed in various sectors, including food processing labs, environmental monitoring, and even household use," says Yang. "We currently do not see challenges to commercializing devices that detect all single types of particles in water. However, challenges remain regarding adapting it to detect plastic particles in a complex matrix, such as soil."

The next steps for the researchers include commercializing the device. They also plan to adapt the device's algorithms to detect different types of plastics, such as polyethylene and polypropylene, broadening its applicability for various materials and environments.



Credit: Martin Dee

"We hope to push towards commercial applications that could help reduce the global health and environmental impacts of plastic pollution," says Yang.

Five Frontiers in Environmental Analysis

The world looks to the analytical community to guard against the harmful compounds in our environment. And in the furnace of environmental pressure, innovation sparkles. The community's imaginative application of increasingly sensitive technologies is helping to paint a more complete contamination picture; it's also driving genuine solutions to the myriad threats we face – from the removal of PFAS from our environment, to delivering a circular plastics economy. Here, five leaders in environmental analysis each highlight a different area where analytical innovation is helping to protect our planet.

Climate Change

Preventing carbon emissions is the ultimate goal, but analytical science offers key insights into carbon's behavior across systems, guiding breakthrough strategies for climate mitigation and adaptation

With Michael Gonsior, Professor, University of Maryland Center for Environmental Science, USA

What is the most pressing climate change issue right now, and how can analytical science help address it? The simplest solution in theory – and the hardest to achieve in practice – is to prevent carbon dioxide from entering the atmosphere in the first place. While we're making progress, including in alternative energy technologies and developing electric vehicles, the scale of removing CO_2 from the atmosphere is vastly different from preventing it from being emitted in the first place. That's where the real impact lies.

From an analytical science perspective, the biggest challenge is understanding the complexities of the carbon cycle. We're looking at tracking highly complex mixtures and pinpointing specific, indicative tracers. This helps us understand the activity and residence time of carbon in different pools, like the atmosphere and the ocean. Knowing how long a particular carbon molecule stays in a specific part of the carbon cycle helps us predict its feedback effects on CO_2 levels.

To put this in perspective, let's consider the ocean – a massive carbon reservoir. If just 1 percent more of the ocean's dissolved organic carbon were to mineralize annually, it would offset the entire annual anthropogenic CO_2 production. Understanding these large carbon pools and how changes within them contribute to positive or negative feedback loops is essential for predicting climate outcomes and making informed decisions about climate strategies. Analytical science plays a critical role here in uncovering these mechanisms and helping us see where we're heading.

One major area of advancement is in mass spectrometry for analyzing complex mixtures and unknown contaminants. In my lab, we're using an extremely sensitive triple quadrupole mass spectrometer, which is traditionally a targeted tool. But I'm actually using it in an unconventional way by systematically scanning all potential transitions across the entire mass range of organic matter

samples. In this case, we're focusing on deep ocean samples, and this approach allows us to pinpoint specific molecular transitions. It's a slow, meticulous process, but it's opening doors to new tracers and even potentially the first structural identifications of deep ocean molecules, especially in refractory organic matter.

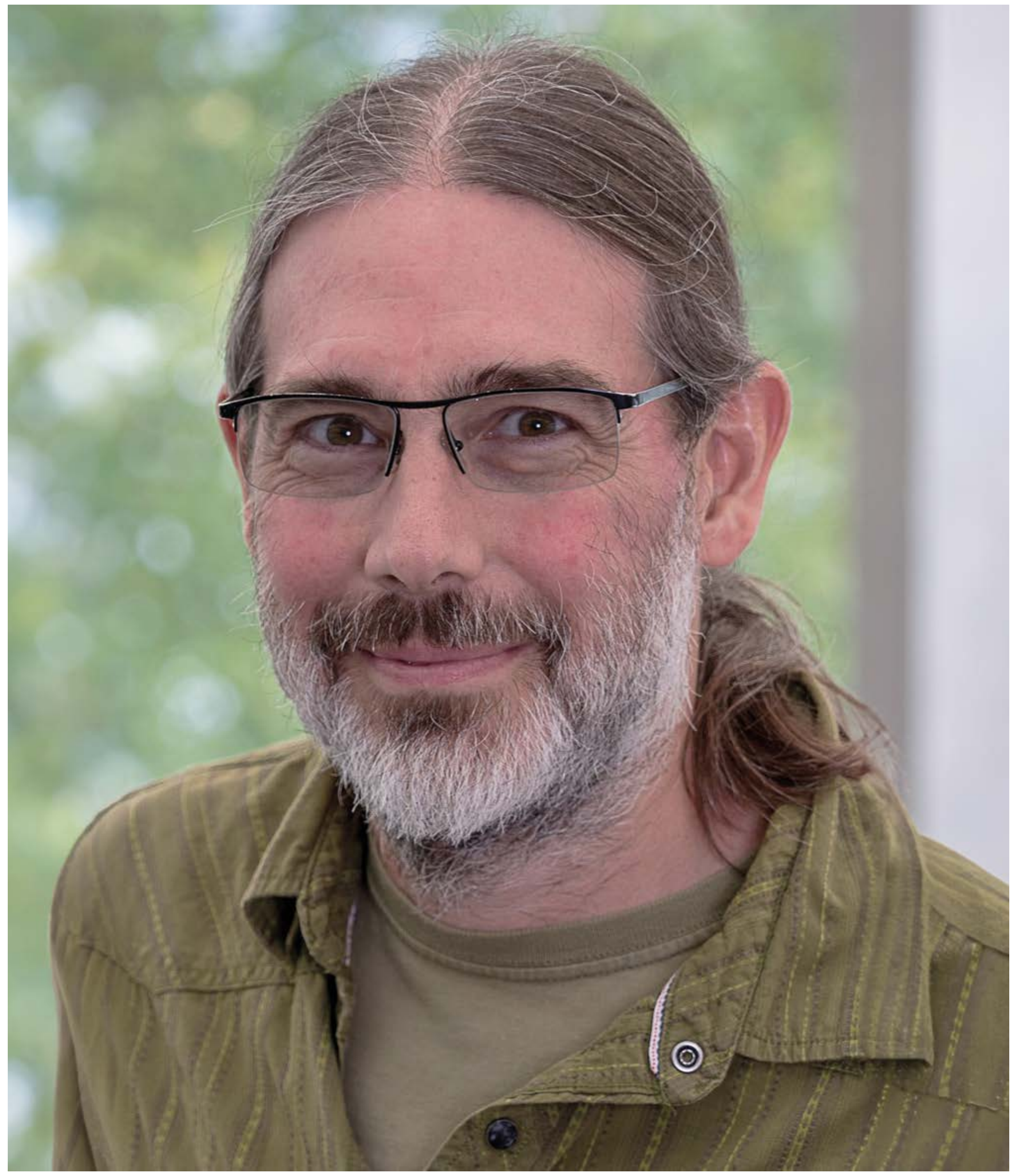
In the broader field, the trend has been toward high-resolution instruments, like the Orbitrap and FT-ICR-MS systems. However, a drawback with these high-resolution instruments is that they're typically less sensitive than triple quadrupole systems, which is why I turned to the latter. Sensitivity and speed are still major hurdles for high-resolution instruments.

Looking ahead, I'm excited about the development of hybrid mass spectrometers – systems that combine the strengths of different instrument types. For example, some researchers are pairing triple quadrupole instruments with time-of-flight mass spectrometers to get high resolution and high sensitivity together. These hybrid systems aren't widely commercialized yet, but I think they'll be game-changers, especially for complex environmental samples where precise molecular structures are essential for understanding the system. That's the direction I see making a real impact in Earth System Science.

Thinking about global efforts to address climate change, I could take the pessimistic route – but I'd rather share a different perspective that might seem a bit pessimistic but is actually hopeful in its own way. Often, we speak as though we're separate from the environment – as if we're somehow removed from nature. In reality, that's an illusion. We're probably the only species that sees itself as separate from nature, but that viewpoint doesn't make sense. Nature itself isn't under threat; it's we who are shaping our world, defining how we want it to look and feel. Nature will carry on; the Earth will still be here in a million years, no matter what we do. But what we're really impacting is our place within it, our quality of life, and the balance of ecosystems we depend on.

What gives me hope is that we're slowly – painfully slowly – starting to realize we're part of the environment, not separate from it. We're moving, though gradually, towards understanding our interconnectedness with nature. People are traveling more, and while you could argue that's counterproductive for the environment, it does broaden our perspectives. It lets us experience different environments, ecosystems, and communities firsthand, and that shifts mindsets in powerful ways. The fact that we have access to information more rapidly than ever is helping, too. Despite its challenges, the flow of knowledge allows us to see more of the world and our impact on it.

In the end, I think the biggest shift – and the one we're slowly seeing – is realizing that climate change is about us. We're not really "threatening nature"; we're shaping the conditions for our own survival and the future we want. That's a powerful motivator, and if we can fully internalize that, I believe it'll drive real change.



LINKS

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Part One

Part Two

PFAS Remediation

Analytical scientists are playing a crucial role in the development of technologies to remove PFAS from our environment

By Diana Aga, Professor of Chemistry at the University at Buffalo, USA

I first heard about PFAS in 2002, early in my academic career at the University at Buffalo. At that time, a visiting professor from the University of Toronto, Scott Mabury, introduced me to the complexity of analyzing these compounds and highlighted their widespread presence in air, water, and the environment. PFAS are notoriously difficult to detect, and this complexity initially deterred me from working on them. I was an assistant professor, focused on securing tenure, and I didn't want to risk developing a method that might not work. So, for years, I avoided studying PFAS.

Fast forward to about five years ago, PFAS resurfaced in my professional life when collaborators started asking if I had a method for detecting them. They needed it badly, and that pushed me to dive deeper into the field.

Detection remains a central challenge, but it also underpins our response to the PFAS problem, whether it be monitoring or regulation. My PFAS research focuses on potential solutions and detection techniques. In assessing remediation technologies, detection plays a crucial role.

When you're dealing with remediation, you really need to know where the PFAS are going and if they've been fully destroyed. For example, with biosolids or solids remediation techniques like incineration, pyrolysis, or gasification, even at high temperatures, you need to be sure whether you're breaking them down completely or just volatilizing them. The same goes for groundwater treatments using plasma – you blast the PFAS, but where do they go? Are they just broken down into smaller, potentially harmful compounds? You need accurate detection to know.

Though we've made progress in analytical capabilities, there's still a lot of room for improvement, especially when it comes to volatile PFAS and unknown degradation products that emerge from these remediation technologies. I'd say that analytical challenges remain a significant hurdle even today.

Can we remove or destroy PFAS?

There are several remediation technologies for PFAS in development; the choice of technology depends on what you're trying to remediate. Right now, biosolids are a big issue. Many wastewater treatment plants used to apply biosolids on agricultural fields, but they're now discovering that these biosolids often contain PFAS, especially if the treatment plant receives wastewater from an industrial source. There are farms in places like Maine that had to close due to high PFAS levels leaching from biosolids, which can contaminate surface water and groundwater, especially when it rains.

To address this issue, people are exploring pyrolysis or gasification to remove PFAS from biosolids. These processes essentially involve burning the material at very high temperatures, around 900–2,200 °F, to produce biochar or other byproducts (for example, syngas), depending on factors like oxygen levels and temperature. However, the challenge is that we don't always know if PFAS are being fully destroyed or just volatilized during the process.

Several strategies are being explored to remove PFAS from contaminated soil and water environments. For drinking water, activated carbon and anion exchange resin are the most widely used sorbents to capture PFAS. These materials work well, but after PFAS capture, you need to regenerate them or destroy the concentrated PFAS in the spent media. In addition, when using activated carbon filters, a key challenge is knowing when the filter is saturated and thus needs to be replaced. Breakthrough (where

> *“The PFAS that are already in the environment will persist – they’re called ‘forever chemicals’ for a reason. So, while regulatory measures are essential, we also need to focus on how to remediate what’s already there.”*

PFAS, especially short-chain compounds, start leaching back into the drinking water because the activated carbon has reached its capacity) is not acceptable for water utility companies.

Right now, the typical process is to sample the filtered water on a regular basis for LC-MS analysis at a certified lab. But if there is a backlog, results can take days, or even weeks. To avoid the risk of delayed result reporting, water utilities preemptively replace the carbon filters after a set volume of water has been treated, but this approach can be expensive. Hence, it would be incredibly helpful to have a faster method for detecting low levels of PFAS, ideally something like a PFAS-selective online sensor that could provide real-time data. Unfortunately, sensors for detecting PFAS at such low levels do not yet exist.

Searching for a complete solution

To truly address the PFAS problem, everyone needs to work together, including regulatory authorities. We are still quite far from a complete solution. The more we regulate PFAS, the less they will enter the environment. However, the PFAS that are already in the environment will persist – they’re called “forever chemicals” for a reason. So, while regulatory measures are essential, we also need to focus on how to remediate what’s already there.

A major hurdle linked to regulation is the lack of toxicity data for many PFAS. We have a good amount of data on PFOA and PFOS, the C8 compounds, but there’s limited toxicity information for many of the other PFAS. These compounds often occur in mixtures, so how do you regulate mixtures? It is a significant challenge.

I’d also like to see more progress on sustainable bioremediation technologies. For example, if we could find naturally occurring bacteria that are capable of breaking down PFAS into carbon, hydrogen, and fluorine, it would be the ultimate remediation technology. In this regard, there are some promising lab-scale isolates, but we still have a long way to go to scale-up these solutions to something that can be implemented in real-world environments. Investing more in bioremediation research would be a great step forward.

About 25 years ago, people were concerned about antibiotics and pharmaceuticals in discharges from wastewater treatment plants. Now, many cities have adopted advanced treatment processes following conventional activated sludge systems, such as ozonation and carbon filtration, resulting in very clean water. But we don’t have anything like that yet that works well for complete PFAS treatment in highly contaminated water. If we could develop something similar on a municipal scale, it would have a huge impact. If we could inoculate wastewater treatment plants with bacteria that can degrade PFAS, we could prevent these compounds from ending up in surface water, which is often used for drinking water. And if all PFAS are removed during wastewater treatment, the biosolids produced would also be free of PFAS, giving the agriculture industry a valuable and safe fertilizer. Right now, many farmers are hesitant to use biosolids, and they have been banned elsewhere.

As we continue to grapple with the complex issue of PFAS, it is clear that there is no single solution. Remediation technologies are advancing, but challenges in detection, degradation, and regulation remain.

To truly make strides, we need a multifaceted approach that includes stronger regulations, improved analytical techniques, innovative and cost-effective remediation technologies, sustainable substitutes, and greater interdisciplinary collaboration.

LINK

An expanded version of this article is available online

PFAS Remediation

Atmospheric Aerosols

How volatile organic compounds and aerosol particles shape atmospheric processes, influencing cloud behavior, and the interconnected dynamics of ecosystems and climate

With Markku Kulmala, Professor of Aerosol and Environmental Physics, University of Helsinki, Finland

We've established research stations in Finland and expanded to places like China, India, Estonia, South Africa, and beyond. We continuously collect data from these stations, analyze it – using e.g. atmospheric pressure interface mass spectrometry (APi-MS), especially time-of-flight (TOF) instruments. These instruments allow us to measure different compounds and clusters of gas molecules. We can generate a wide range of spectra, capturing the detailed composition of gasses and aerosols in the atmosphere.

We also collect data from the soil, including temperature and humidity profiles. We measure ecosystem functions, such as photosynthesis and gas exchange between tree needles and the atmosphere. We also track tree growth by monitoring diameter and other functional aspects. We measure aerosol concentrations in the air, and we record fluxes – meaning we monitor changes of CO_2 and other greenhouse gasses. We even track various types of radiation, including solar radiation, cosmic radiation, and radon emissions from the soil. As you can see, we cover a broad range of measurements.

What have we found? Let me give you a comparison between a major city like Beijing and our background station in Finland, at Hyytiälä. Hyytiälä isn't the cleanest place on the planet, but it is on the cleaner end of the spectrum, whereas Beijing is much more polluted. Interestingly, some atmospheric processes are quite similar between these locations. For instance, the growth rate of newly formed aerosol particles – about 4–6 nm per hour – is roughly the same in both places. However, the rate at which new particles (around 1.5 nm in size) are produced is about 100 times higher in Beijing than in Hyytiälä.

Moreover, measuring so many different variables with so many different instruments is throwing up a number of surprises. During the COVID-19 lockdown, for example, we observed that in Beijing, nitrogen oxides (NOx) and particle mass concentrations decreased, but ozone levels and particle numbers increased. Surprisingly, the overall particle mass only went slightly down, remaining almost the same before, during, and after the lockdown.

At our station in Hyytiälä, we've observed how increased CO_2 levels lead to more photosynthesis, which in turn emits volatile organic compounds (VOCs) like monoterpenes. These VOCs undergo chemical reactions in the atmosphere, producing low-volatile highly oxidized organic compounds that contribute to new aerosol particle formation. These particles can grow to become cloud condensation nuclei, which influence cloud droplets and, ultimately, precipitation. However, when there are more aerosol particles, clouds live longer, become thicker, and reflect more sunlight. On the other hand, the more aerosol particles we have the more the amount of diffuse solar radiation is enhanced, which penetrates radiation deeper into the ecosystem, further enhancing photosynthesis. It's a fascinating feedback loop with significant implications for both weather and climate.

Longer term, we've observed changes such as a decrease in SO_2 concentrations, both in Beijing and Hyytiälä. We also see how factors like population growth and land-use changes are impacting

air quality and climate; in turn, we see how climate changes are influencing processes like photosynthesis. Though our data doesn't span hundreds of years, the data we have – over decades – allows us to clearly see these changes at various time scales.

A central challenge is that many important atmospheric compounds, especially in the gas phase, are present at extremely low levels – often in the parts per quadrillion (PPQ) range, with as few as 1,000 or 10,000 molecules per cubic centimeter, sometimes even less. This makes it incredibly challenging to push the detection limits of techniques, such as API-MS, low enough. Additionally, we need analytical chemistry expertise to help us properly calibrate these measurements. Though we understand some of the basics, the need for better calibration methods and possibly developing standards for these measurements is crucial. And that requires specific expertise in analytical techniques.

That is to say, analytical chemists play a crucial role in efforts to improve atmospheric conditions!

LINK

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Atmospheric Aerosols

Water Analysis

PFAS demand attention, but natural and anthropogenic threats create opportunities for innovation in water analysis

With Torsten C. Schmidt, Professor, Instrumental Analytical Chemistry and Centre for Water and Environmental Research (ZWU), University of Duisburg-Essen, Germany

Water research, especially water quality, has been a topic of interest for me since my school days. I started with small projects at school, and I knew early on that I wanted to work on something that would help the environment. For a long time, I considered how best to approach this. I thought about more technically oriented study programs but ultimately settled on chemistry, which led me to analytical chemistry – a field I became passionate about right away.

Today, PFAS are certainly top of the list of emerging water contaminants, but there are several other compounds with the potential to become major environmental issues.

While not a single substance class, compounds that are persistent and mobile (sometimes also toxic), known as PMTs, are increasingly concerning. Some PFAS compounds fall into this category as well. Over the last decade or so, PMTs have gained attention because they have been largely overlooked in the past but are crucial, especially from a water cycle perspective. These compounds don't degrade easily, and are mobile. They can pass through natural barriers such as soil or bank filtration, and quickly travel through the water cycle. They are very difficult to contain.

Part of the reason they went undetected for so long is because of the limitations in analytical methods, particularly for detecting low concentrations in water. Historically, we relied heavily on gas chromatography (GC) for organic contaminants, which works well for volatile compounds. Later, liquid chromatography-mass spectrometry (LC-MS) became more widespread, but it was primarily based on reverse-phase LC, which doesn't sufficiently retain highly polar organic compounds. However, advances in recent years have introduced new separation methods that better capture these polar compounds. This progress has revealed that many of these compounds are indeed present, sometimes in high concentrations, in aquatic environments – levels we simply couldn't detect before.

The growing awareness has been somewhat of a success story, as these persistence and mobility characteristics are now being considered in the registration of new chemicals under regulations such as REACH. This is a positive shift, helping us avoid the pattern of focusing only on certain properties and then discovering new risks decades later.

We're also starting to recognize that it's not only anthropogenic contaminants we need to be concerned about – naturally occurring compounds can also have serious impacts. For instance, the Oder River disaster on the Polish-German border highlighted this, where higher salt concentrations and warmer temperatures enabled certain algae to grow explosively. The algae then released toxins that killed fish and other organisms. This wasn't directly due to a man-made contaminant but rather a natural toxin exacerbated by anthropogenic influences.

Cases like this emphasize the need for a comprehensive approach that combines chemical data with biological analysis. To address such incidents effectively, we should combine information on chemical composition with data from environmental omics, such as eDNA and RNA-based methods, to see how ecosystems respond to changes in both chemical and biological conditions. This combined approach could advance our understanding of how ecosystem dynamics are influenced by both anthropogenic and natural factors, which is key for developing more effective environmental protections.

While not every individual project or piece of research might have immediate or visible effects, I firmly believe that, as a whole, our work is essential. Environmental analysis provides the data that makes people care, and without it, many of these issues would remain unseen or unaddressed. So, yes, I do think we're having a positive impact, and we're doing work that's critical to advancing environmental protection.

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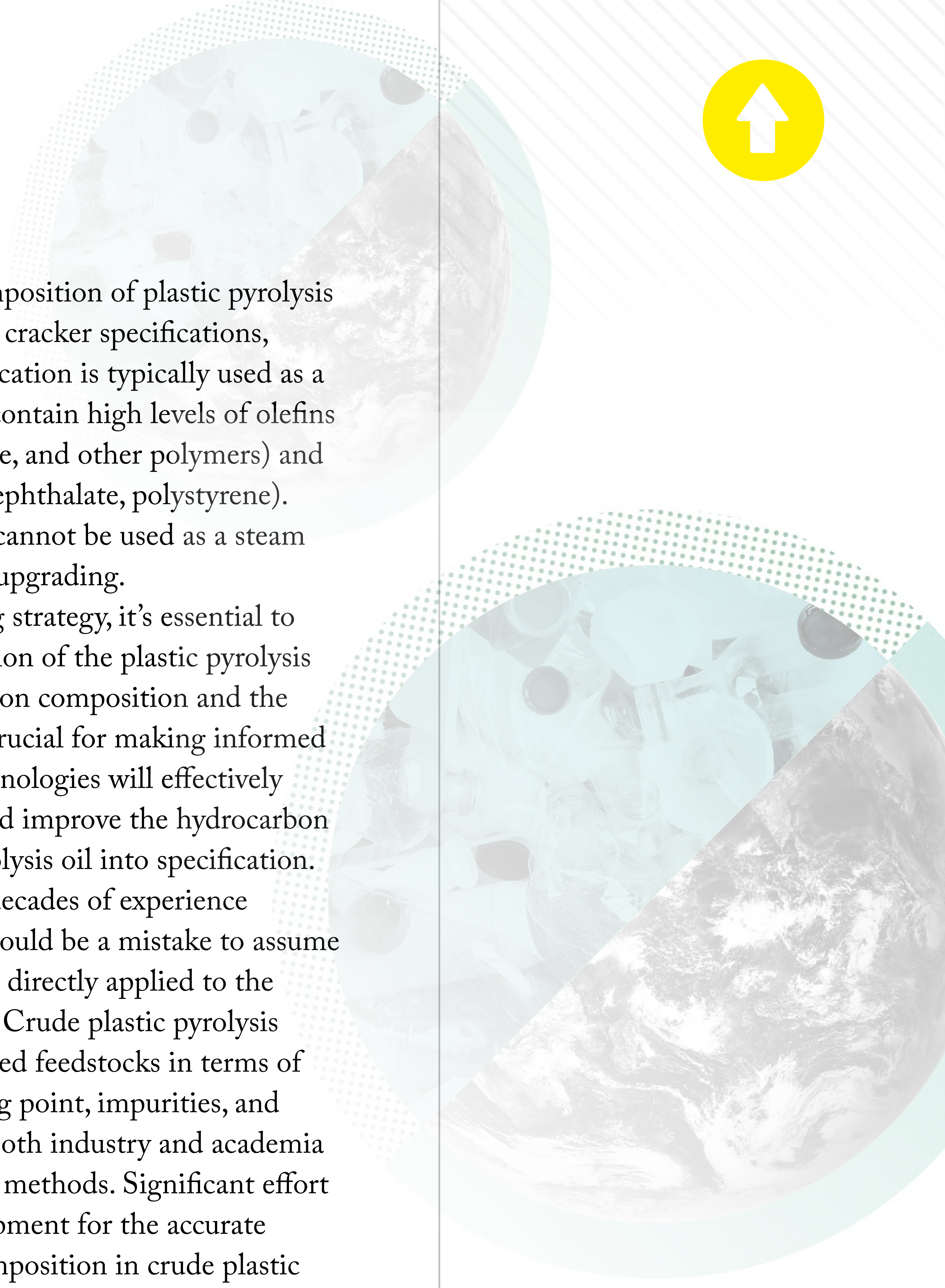
An expanded version of this article is available online

Part One

Part Two



The Circular Plastic Economy

Emerging analytical methods for more precise quantification of hydrocarbon composition and impurity detection may prove essential to realizing a circular plastic economy

By Melissa N. Dunkle, Senior Research Scientist,
Dow Benelux, The Netherlands

Plastics are an integral part of our daily lives, found in everything from synthetic fabrics used for clothing to food packaging, building materials, and more. As the global population has grown, so has the production of plastics, which reached 475 million tons in 2022. However, only about 12 percent of this plastic is collected for recycling. So where does the rest go? Approximately 80 percent of the remaining plastic is never recovered; it is either mismanaged, ending up in landfills, or incinerated, following the traditional linear economy model for plastics. A global shift is needed to transition from this linear economy to a circular economy for plastics, requiring action not just from governments and industries but also from consumers.

Governments are beginning to create legislation that mandates minimum recycled content in plastics. For example, the EU's Single-Use Plastics Directive requires that plastic bottles contain at least 25 percent recycled plastic by 2025, increasing to 30 percent by 2030. As we move towards a circular economy for plastics, similar legislation around minimum recycled content for other plastic materials is expected to be implemented by various countries worldwide.

Achieving a circular economy for plastics will require different recycling technologies to close the loop. While mechanical recycling for rigid plastic bottles is well-established, not all types of plastics can be easily recycled this way. For this reason, advanced recycling (also known as chemical recycling) is gaining attention as a viable alternative. In mechanical recycling, the polymer structure is maintained, but in advanced recycling, plastics are broken down into their original monomers. Several technology routes can be used for advanced recycling, including solvolysis, pyrolysis, and gasification.

The need for analytical data

Analytical evaluation and data processing are key components in achieving plastics circularity. While this example focuses on advanced recycling through pyrolysis, it's important to note that such evaluations and data are equally crucial when considering mechanical recycling or other advanced recycling technologies.

In advanced recycling via pyrolysis, the importance of analytical data becomes clear. Pyrolysis involves heating waste plastic to temperatures that thermally decompose it into three fractions: gas, oil, and char. For advanced recycling purposes, the oil fraction is considered a potential feedstock for steam cracking. However, depending on the composition of the waste plastic feed, the oil fraction may contain not only hydrocarbons but also undesired impurities. These impurities can include nitrogen species (e.g., from the degradation of polyamide in the plastic feed), oxygenates (e.g., from polyvinyl alcohol, polyethylene terephthalate, or additives), aluminum (e.g., from poly-Al packaging), chlorine (e.g., from polyvinylchloride or additives), and more.

Additionally, the hydrocarbon composition of plastic pyrolysis oils does not align with current steam cracker specifications, where the Platts open naphtha specification is typically used as a guideline. Plastic pyrolysis oils often contain high levels of olefins (e.g., from polyethylene, polypropylene, and other polymers) and aromatics (e.g., from polyethylene terephthalate, polystyrene). Therefore, crude plastic pyrolysis oils cannot be used as a steam cracker feedstock without significant upgrading.

To select an appropriate upgrading strategy, it's essential to conduct a thorough analytical evaluation of the plastic pyrolysis oils to understand both the hydrocarbon composition and the impurity profile. This information is crucial for making informed decisions about which upgrading technologies will effectively remove or eliminate the impurities and improve the hydrocarbon composition, bringing the plastic pyrolysis oil into specification.

While the chemical industry has decades of experience analyzing fossil-based feedstocks, it would be a mistake to assume that these established methods can be directly applied to the analysis of crude plastic pyrolysis oils. Crude plastic pyrolysis oils differ significantly from fossil-based feedstocks in terms of hydrocarbon composition, final boiling point, impurities, and more. As a result, research groups in both industry and academia are actively developing new analytical methods. Significant effort has been dedicated to method development for the accurate quantification of the hydrocarbon composition in crude plastic pyrolysis oils, as well as for the identification and quantification of impurity profiles.

For the analytical evaluation of crude plastic pyrolysis oils, much focus has been placed on gas chromatography (GC). In

> *"The evaluation of crude plastic pyrolysis oils is a burgeoning area of research, with each analysis yielding new and insightful information, making it an exciting time to be an analytical chemist."*

the works mentioned above, both one-dimensional GC and comprehensive GC (GC×GC) were utilized, and various detector technologies were exploited. One of the newer detectors being evaluated is the vacuum ultraviolet detector (VUV), which is still relatively new to the market. GC-VUV was introduced in 2014, and since then, the technique has shown promise for the characterization of the hydrocarbon composition of various materials, including crude plastic pyrolysis oils. While GC×GC can also provide insight into the hydrocarbon composition of materials, the co-elution of olefins and naphthenes in the same elution band of the 2D plot can complicate data processing when both compound classes are present. However, GC×GC is compatible with various GC detectors, including (but not limited to) mass spectrometry and element specific detectors, making this technique well suited for the characterization of the impurity profile of crude plastic pyrolysis oils.

The evaluation of crude plastic pyrolysis oils is a burgeoning area of research, with each analysis yielding new and insightful information, making it an exciting time to be an analytical chemist.

Moving forward

There are many challenges to overcome to make plastics circularity a reality. Change isn't easy, and achieving it on a global scale will require concerted efforts from governments, industry, and consumers.

One perspective not yet discussed in this article is the cost associated with transitioning from a linear plastics economy to a circular one. According to a recent McKinsey report, the plastics industry may need to invest as much as $100 billion to achieve 20–30 percent recycled content in materials. Cost is just one factor that will influence the speed at which plastics circularity is implemented. Other critical considerations include technology readiness and economic feasibility, among many others.

However, before moving forward, it may be necessary to take a step back. As mentioned earlier, only around 12 percent of produced plastic is currently being collected for recycling. Optimizing existing collection and sorting infrastructures and processes is essential to increase this percentage. This challenge presents an interesting opportunity for analytical scientists, as current automated optical sorters have limitations in detecting different types of plastics. Enhancing automated sorting capabilities and possibly integrating artificial intelligence are areas worth watching.

Transitioning from a linear to a circular plastics economy will take time, but the journey has already begun. McKinsey reports that chemical industry players have committed to achieving 7 million metric tons per year of advanced recycling capacity by 2030. It is challenging to predict what the landscape will look like beyond 2030, as this will depend heavily on legislation and the technological advancements that still need to occur.

In conclusion, change is happening, and progress is being made to transition from a linear economy to a circular plastics economy. For those working in this field, this represents a new and exciting area of research, where success means making a positive impact on the environment. Even if you're not directly involved in this field, you can still contribute; we are all consumers, and our choices have an impact.

LINK

An expanded version of this article is available online

The Circular Plastic Economy

Analyses of PFAS in Water Using Ion Exchange SPE

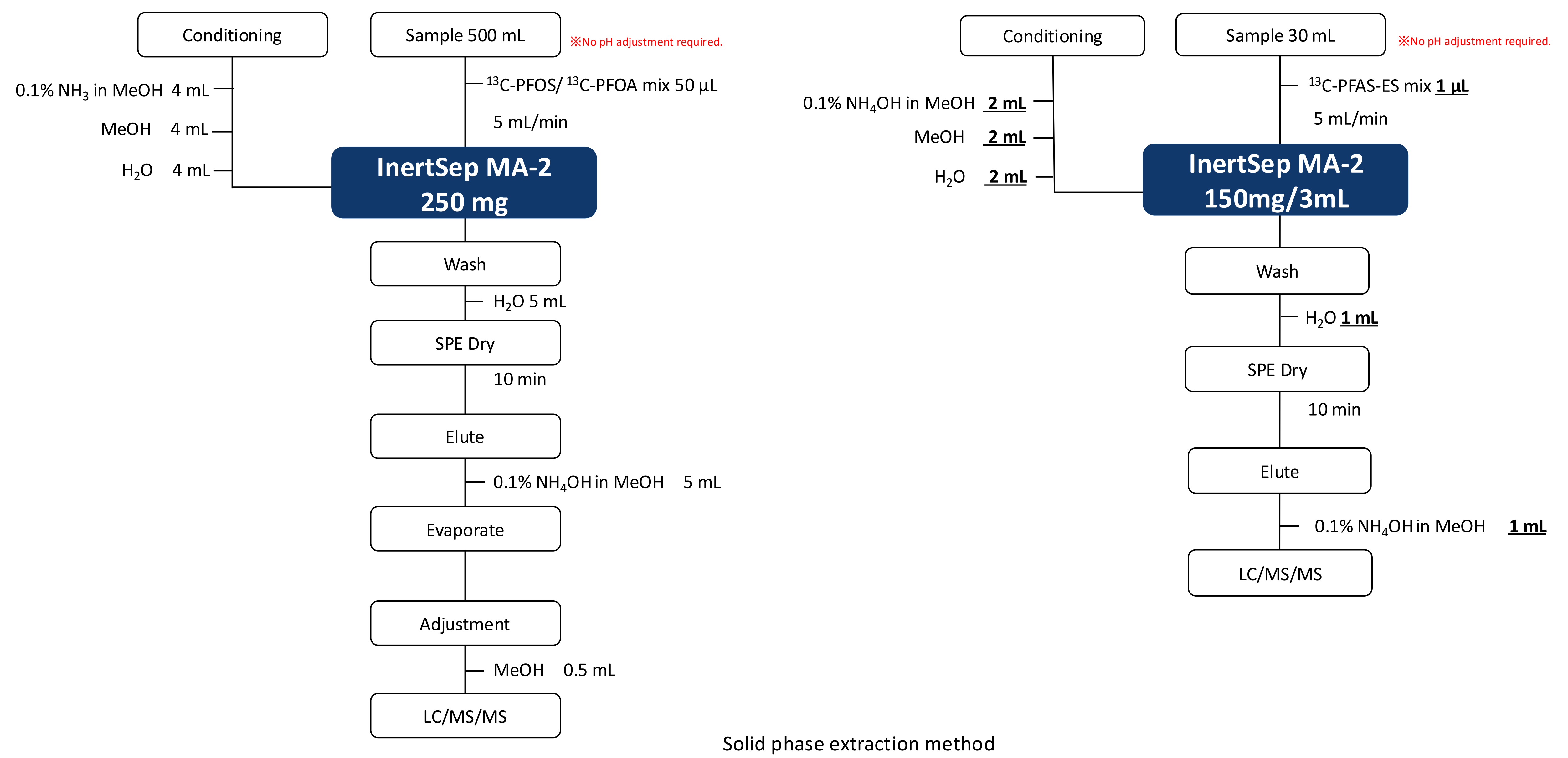
PFAS components in water can be effectively extracted using the small-scale SPE cartridge, InertSep MA-2.

The phrase per- and polyfluoroalkyl substances (PFAS) is a general term used for organofluorine compounds. These substances are known to degrade slowly; therefore, they persist in the environment for a long time. Their toxicity and the environmental pollution they cause have attracted worldwide attention, and research to mitigate these effects continues. The methods of solid-phase extraction (SPE) and liquid chromatography–tandem mass spectrometry (LC–MS/MS) have been used to analyze PFAS in drinking water under EPA methods 537.1 and 533.

Here, two SPE cartridges of different sizes, 250 mg and 150 mg, were used and reference standard addition recovery tests were conducted for each cartridge.

We confirmed that the 21 PFAS components in water can be effectively extracted using the SPE cartridge, InertSep MA-2. Recovery tests and analysis of extracted tap-water samples demonstrated excellent linearity ($R^2 \geq 0.99$) in the range of 1–20 ng/L and repeatability within 16% at 5 ng/L. Using a 150 mg SPE cartridge allowed for a reduction in both sample water volume and eluting solvent volume. Additionally, the evaporation step for the elution solvent was found to be unnecessary, resulting in a significant reduction in sample preparation time.

By switching to smaller-scale solid-phase extraction columns compared to conventional sizes, we confirmed that elution could be performed with less solvent. This efficiency may be attributed to the use of weak anion-exchange solid-phase extraction columns without a reversed-phase mode. These findings suggest that smaller-scale, faster processing is feasible depending on the sample concentration and instrument sensitivity.



Solid phase extraction method

LINK

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Planet Protectors Assemble!

We asked our 2024 Power Listers to tell us the most critical environmental issue that analytical science can help address over the next decade…

Michael Gonsior: Deciphering the marine organic carbon cycle is a missing link in truly understanding our oceans ability to store and sequester carbon. We need to find ways to predict the half-life of individual carbon molecules in the deep ocean to better define the marine carbon reactivity continuum and to give guidance. To date, not a single molecule is known in the rather vaguely defined refractory or recalcitrant deep-ocean DOM pool. This is critical to evaluate if marine carbon dioxide removal (marine CDR) ideas are effective or counterproductive. Unfortunately, it seems that numerous marine CDR projects are likely based on doubtful assumptions about the true age of deep ocean DOM.

Teresa Rocha Santos: Analytical science plays an important role in all environmental issues. For me, one critical current environmental issue is microplastic pollution. Dealing with particles from different polymers with different physicochemical properties (e.g. shape, color, size) and the lack of rapid screening devices make the fast determination of microplastics in environmental matrices difficult. Therefore, further developments in the analytical science field are needed to overcome this environmental issue.

Torsten Schmidt: Analytical science is a necessary component of all endeavors tackling environmental issues to help prioritize the many tasks on the agenda, identify and track trends over space and time, and monitor efficacy of measures. Some of the issues that require expertise and further development in analytical science are outlined below.

Juliane Hollander: Analytical scientists should support necessary changes due to climate change, such as the energy transition, so that they are efficient and do not create new problems.

Emma Schymanski: Interestingly, I was asked 5 years ago to ponder the future of non-target screening (NTS) in 100 or even 25 years' time and I remember thinking at the time "I hope by then the main challenges of NTS are solved and we can move onto other topics" (which would have been a very short review article so I wrote about something else instead). Reflecting on this now, perhaps I was a little optimistic. Today I feel we have made a lot of progress but we still have a number of issues to overcome before NTS moves into truly routine applications, but I certainly hope that this is now achievable within the next decade. Moving NTS into routine applications will enable (more broad) real-time monitoring of the environment and thus the establishment of early warning systems to better prevent or mitigate environmental catastrophes, or large-scale chemical contamination of human (or other) populations as seen in Dark Water and other films, through to earlier recognition and prevention of regrettable substitutions. I believe we have many of the "puzzle pieces" already available and the general awareness, willingness and know-how – a concerted community effort at harmonizing and transforming NTS into a routine effort could really revolutionize environmental but also personal health monitoring. I believe analytical science can help us achieve this in the next 10 years if we focus our efforts well. It will be interesting to revisit this in 10 years and see if we managed!

Stefan van Leeuwen: I believe society needs to re-think on what basis chemicals are allowed on the market. The speed of designing new chemicals is enormous, and environmental scientists cannot keep up with that. We're still investigating problems from the past (PCBs, BFRs, etc) while at the same time new pollutant issues rise. PFAS are an explicit example of that. With such challenges ahead, analytical scientists can develop smart and comprehensive approaches to address the ever expanding chemical space.

Janusz Pawliszyn: Pollution. Develop sustainable portable technologies to monitor pollution and its effect on human health (exposome).

Susan Richardson: Identifying chemicals in water that no one yet knows about could be impacting human and ecological health. Our work on DBPs in drinking water is an example of this, and another notable recent example is the work that Katherine Peter, Ed Kolodziej, Jen McIntyre, and Nat Scholz did in identifying 6ppd-quinone as the tire wear chemical killing coho salmon in the Northwest. There are many compounds left to discover!

Diana Aga: Identification and quantification of currently "unknown" contaminants and their transformation products that are toxic, even at trace levels, are highly challenging especially when they occur in complex mixtures. There are still many pollutants that remain unaccounted for in the environment, which are causing deleterious effects on humans and wildlife. Analytical science can help answer many of these environmental mysteries if we use complementary techniques so that we can detect both polar and nonpolar, volatile and non-volatile, organic and inorganic pollutants.

DBPs: an Underestimated Threat

Why Susan Richardson's recent discoveries about disinfection by-products are (even more) cause for concern

Back in 2019, Susan Richardson, Arthur Sease Williams Professor, Department of Chemistry & Biochemistry, University of South Carolina, USA, wrote a feature for The Analytical Scientist discussing her work on disinfection by-products (DBPs) – and the threat they pose to our health, wildlife, and environment.

We recently connected with Susan to see where this research has taken her over the past few years, what she's mostly concerned about today, and how environmental analysis can progress in the near future.

What is the biggest analytical challenge in environmental research today – and how do you believe we can overcome it?
Identifying unknown contaminants, whether in drinking water or environmental surface waters, remains as our biggest environmental analytical challenge. It is much harder to identify new contaminants than those we already understand, and they are more likely to be found at very low levels in a sea of contaminants – it's like looking for a needle in a haystack!

Effect-directed analysis (EDA) is a valuable method for pinpointing harmful contaminants causing toxicity effects. However, it takes a great deal of time and effort, which discourages many groups from using it. There are several examples of EDA's effectiveness, such as the discovery of 6ppd-quinone harming coho salmon in the Northwest US (1) and a new algal toxin affecting bald eagles in the Southeast US (2).

Which environmental contaminant are you most concerned about today?

Despite having worked in various now-mainstream areas of environmental analysis, from microplastics to PFAS, I'm still most concerned about DBPs. This is because they're typically found at ppb levels (1,000 times higher than PFAS) and a body of evidence states that they cause serious effects to human health – such as bladder cancer, miscarriage, and birth defects. For example, dibromoacetonitrile – one of the emerging, unregulated DBPs – is carcinogenic in two animal species and often seen in drinking water at ppb levels (3).

What updates can you share about your work with DBPs?

In 2022, we published our new discovery on an important class of DBPs that weren't known before: halocyclopentadienes. We found these DBPs in real chlorinated and chloraminated drinking water using a very sensitive GC-MS instrument (4). I was especially surprised by two aspects of this research. Firstly, this class is the first to be bioaccumulative; secondly, hexachlorocyclopentadiene is now the most cytotoxic DBP known. These six new DBPs were found completely by accident by a PhD student in my lab, Jiafu Li, who spotted the new peaks and figured out what they were. As always with analytical science, important discoveries seem to appear when you least expect them!

Additionally, in a recent Environmental Science & Technology publication, we applied EDA to assess different size fractions of DBPs and determine which are most important toxicologically (5). We discovered that DBPs over 5000 Da molecular weight are not toxic – validating a statement made by a prominent toxicologist at a conference I attended. The most toxic fraction was <1000 Da, challenging the common focus on larger fractions in previous research.

How important is mass spec to your work?

Hugely important! Mass spec is the most important tool in our arsenal thanks to its high sensitivity and ability to handle complex mixtures. For me, it's the most important tool in environmental research because we can use it to identify new contaminants, quantify contaminants, and so on. If NMR was this sensitive and could handle mixtures, maybe we'd veer away from mass spec. But as it stands, mass spec remains on top!

Is there anything missing from the analytical toolbox for environmental analysis?

Some people in the field have started using supercritical fluid chromatography (SFC)-MS. Only time will tell if this fills an important niche. One thing we're missing is an automatic process for testing toxicity of collected prep-LC-MS fractions. As other areas of analytical chemistry begin to use AI, maybe environmental research should take inspiration to push EDA into the future.

Any advice for the next generation of analytical scientists working in environmental analysis?

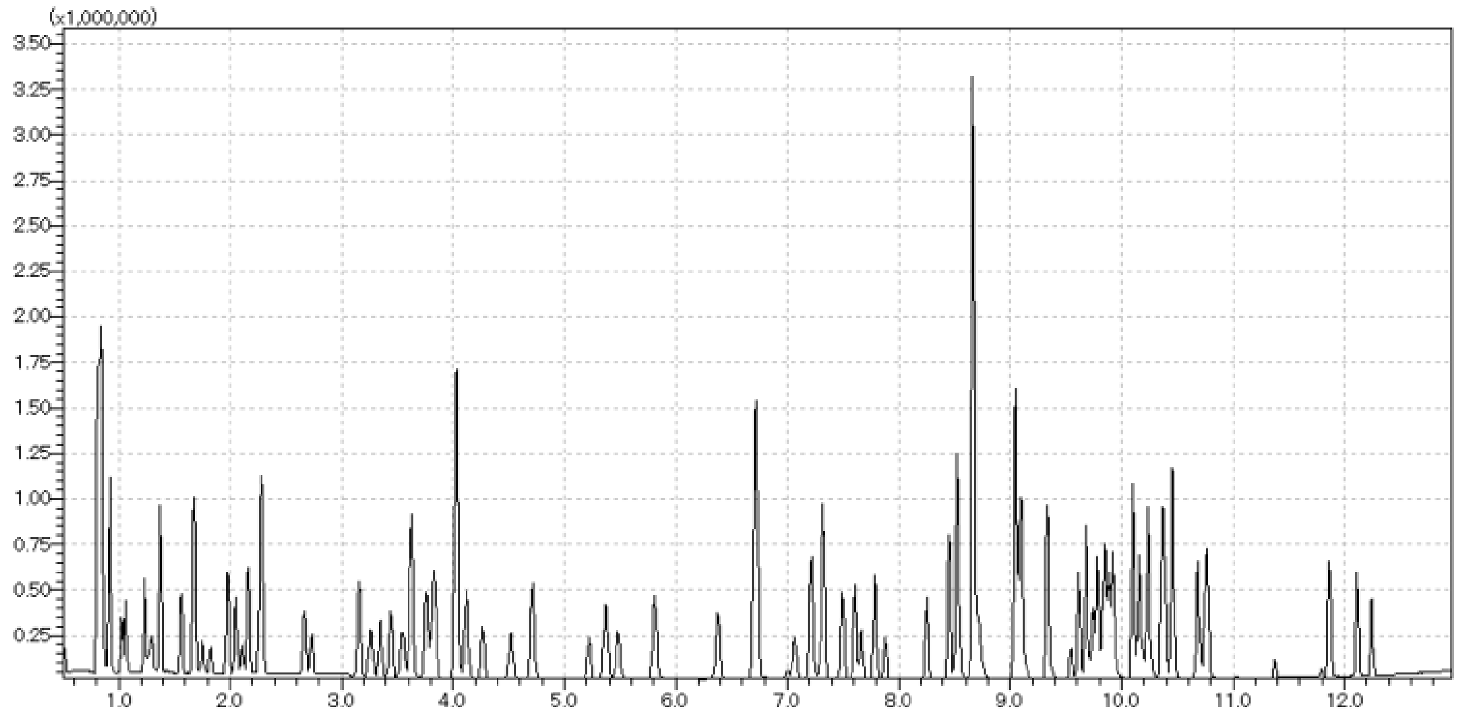
There's so much to discover and do – newly emerging analytical scientists have a wonderful opportunity to make a difference in human and environmental health. Alongside grasping every opportunity that presents itself, it's crucial that you don't give up! There's always pitfalls, but with collaboration and determination, you will achieve exciting results.

What are your hopes for the future?

I'm hopeful that as the field evolves, we will identify important new contaminants that allow us to draft solutions to minimize human and ecological exposure.

Simultaneous VOC Analysis in Water via Purge and Trap GC-MS

US EPA Method 524.2 specifies P&T GC-MS analysis of 83 VOCs in water using the PT7000 system with MCS.



Purge and Trap (P&T) gas chromatography-mass spectrometry (GC-MS) is a widely used method for analyzing volatile organic compounds (VOCs) in drinking water. This application demonstrates an example of VOC analysis conducted using the PT7000 purge and trap concentrator and GC-MS, in compliance with US EPA Method 524.2. The PT7000 system features a vial autosampler with Peltier-based electronic cooling and a concentrator with an advanced inert sample path, enabling high-precision VOC analysis. Furthermore, the high-performance moisture control system (MCS) effectively controls the amount of moisture introduced into the GC-MS, achieving both high sensitivity and accuracy.

The figure on the right shows the total ion current chromatogram (TICC) obtained under the above analytical conditions. Excellent separation and reproducibility were achieved for 83 VOCs. The relative standard deviation (RSD) for both internal standards and surrogates was below 5%. The full version of this application includes detailed data on retention times, linearity, method detection limits (MDLs) at 0.5 ppb, accuracy, and precision for each component. The RSD for all components was below 20%, excluding iodomethane, with most components achieving an RSD below 10%. Results for linearity, MDL, and precision were also highly satisfactory.

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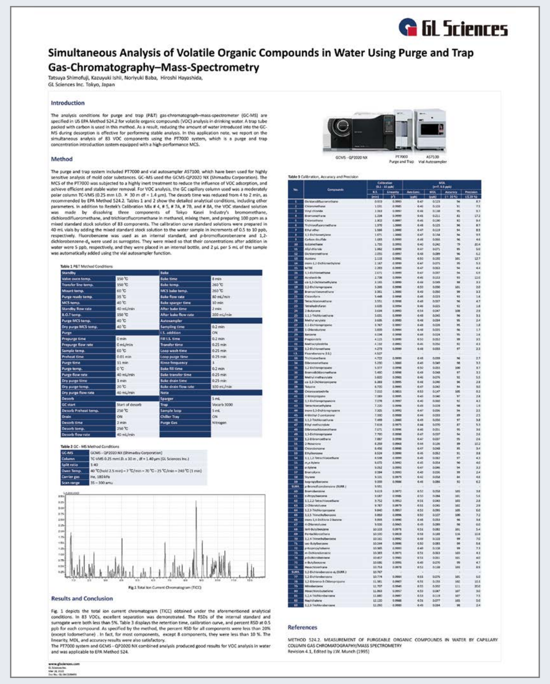

GL Sciences

Simultaneous Analysis of Volatile Organic Compounds in Water Using Purge and Trap Gas-Chromatography–Mass-Spectrometry

Tatsuya Shimofuji, Kazuyuki Ishii, Noriyuki Baba, Hiroshi Hayashida,
GL Sciences Inc. Tokyo, Japan

Introduction

The analysis conditions for purge and trap (P&T) gas-chromatograph–mass-spectrometer (GC-MS) are specified in US EPA Method 524.2 for volatile organic compounds (VOC) analysis in drinking water. A trap tube packed with carbon is used in this method. As a result, reducing the amount of water introduced into the GC-MS during desorption is effective for performing stable analysis. In this application note, we report on the simultaneous analysis of 83 VOC components using the PT7000 system, which is a purge and trap concentration introduction system equipped with a high-performance MCS.

Method

The purge and trap system included PT7000 and vial autosampler AS7100, which have been used for highly sensitive analysis of mold odor substances. GC–MS used the GCMS-QP2020 NX (Shimadzu Corporation). The MCS of the PT7000 was subjected to a highly inert treatment to reduce the influence of VOC adsorption, and achieve efficient and stable water removal. For VOC analysis, the GC capillary column used was a moderately polar column TC-VMS (0.25 mm I.D. × 30 m df = 1.4 µm). The desorb time was reduced from 4 to 2 min, as recommended by EPA Method 524.2. Tables 1 and 2 show the detailed analytical conditions, including other parameters. In addition to Restek's Calibration Mix # 4, # 5, # 7A, # 7B, and # 8A, the VOC standard solution was made by dissolving three components of Tokyo Kasei Industry's bromomethane, dichlorodifluoromethane, and trichlorofluoromethane in methanol, mixing them, and preparing 100 ppm as a mixed standard stock solution of 83 components. The calibration curve standard solutions were prepared in 40 mL vials by adding the mixed standard stock solution to the water sample in increments of 0.5 to 10 ppb, respectively. Fluorobenzene was used as an internal standard, and p-bromofluorobenzene and 1,2-dichlorobenzene-d_4 were used as surrogates. They were mixed so that their concentrations after addition in water were 5 ppb, respectively, and they were placed in an internal bottle, and 2 µL per 5 mL of the sample was automatically added using the vial autosampler function.

GCMS - QP2020 NX
PT7000 Purge and Trap
AS7100 Vial Autosampler

Table 1 P&T Method Conditions

Standby		Bake	
Valve oven temp.	150 ℃	Bake time	8 min
Transfer line temp.	150 ℃	Bake temp.	260 ℃
Mount temp.	60 ℃	MCS bake temp.	260 ℃
Purge ready temp.	35 ℃	Bake flow rate	80 mL/min
MCS temp.	40 ℃	Bake sparger time	10 min
Standby flow rate	40 mL/min	After bake time	2 min
B.O.T temp.	150 ℃	After bake flow rate	200 mL/min
Purge MCS temp.	40 ℃	Autosampler	
Dry purge MCS temp.	40 ℃	Sampling time	0.2 min
Purge		I.S. addition	ON
Prepurge time	0 min	Fill I.S. time	0.2 min
Prepurge flow rate	0 mL/min	Transfer time	0.25 min
Sample temp.	60 ℃	Loop wash time	0.25 min
Preheat time	0.01 min	Loop purge time	0.25 min
Purge time	11 min	Rinse frequency	3
Purge temp.	0 ℃	Bake fill time	0.2 min
Purge flow rate	40 mL/min	Bake transfer time	0.25 min
Dry purge time	1 min	Bake drain time	0.25 min
Dry purge temp.	20 ℃	Bake drain flow rate	100 mL/min
Dry purge flow rate	40 mL/min	Note	
Desorb		Sparger	5 mL
GC start	Start of desorb	Trap	Vocarb 3000
Desorb Preheat temp.	250 ℃	Sample loop	5 mL
Drain	ON	Chiller Tray	ON
Desorb time	2 min	Purge Gas	Nitrogen
Desorb temp.	250 ℃		
Desorb flow rate	40 mL/min		

Table 2 GC - MS Method Conditions

GC-MS	GCMS - QP2020 NX (Shimadzu Corporation)
Column	TC-VMS 0.25 mmI.D. x 30 m , df = 1.40 µm (GL Sciences Inc.)
Split ratio	1:40
Oven Temp.	40 ℃(hold 2.5 min) – 7 ℃/min – 70 ℃ - 25 ℃/min – 240 ℃ (1 min)
Carrier gas	He, 180 kPa
Scan range	35 – 300 amu

Fig.1 Total Ion Current Chromatogram (TICC)

Table 3 Calibration, Accuracy and Precision

Results and Conclusion

Fig. 1 depicts the total ion current chromatogram (TICC) obtained under the aforementioned analytical conditions. In 83 VOCs, excellent separation was demonstrated. The RSDs of the internal standard and surrogate were both less than 5%. Table 3 displays the retention time, calibration curve, and percent RSD at 0.5 ppb for each compound. As specified by the method, the percent RSD for all components were less than 20% (except Iodomethane) . In fact, for most components, except 8 components, they were less than 10 %. The linearity, MDL, and accuracy results were also satisfactory.
The PT7000 system and GCMS - QP2020 NX combined analysis produced good results for VOC analysis in water and was applicable to EPA Method 524.

References

METHOD 524.2. MEASUREMENT OF PURGEABLE ORGANIC COMPOUNDS IN WATER BY CAPILLARY COLUMN GAS CHROMATOGRAPHY/MASS SPECTROMETRY
Revision 4.1, Edited by J.W. Munch (1995)

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The Planet Protector

Sitting Down With… Damià Barceló Cullerès, Honorary Adjunct Professor, Chemistry and Physics Department, University of Almeria, Spain

Could you briefly introduce yourself, focusing on your background in analytical science?

I completed my chemistry degrees in Barcelona, finishing my PhD in 1984. After that, I spent a few years in Amsterdam at Vrije with Roland Frei and U. A. Th. Brinkman. Unfortunately, Professor Frei passed away at a young age, but Professor Brinkman is still with us and remains somewhat active in the field.

After returning to Spain, I joined the Research Council as a university faculty member, where I started my career in analytical chemistry with a focus on LC-MS, which was just emerging at the time. I was fortunate to learn these techniques at their origin in Amsterdam and apply them to environmental issues. My work then expanded to include a range of environmental matrices, including water, soil, sediments, and biota, and covered diverse pollutants, particularly what we now term "emerging contaminants," such as microplastics and nanomaterials.

Since then, I've supervised about 70 PhD students, and I still have new students joining, including two recent hires from China. Although I'm partly retired, my career has centered on fate analysis, transformation, and toxicity of emerging environmental contaminants. I primarily use mass spectrometry, as well as a range of sample preparation methods, spectroscopy, and other analytical techniques.

Tell us about your current activities…

My current activities focus on student support and mentoring. I'm semi-retired, so I dedicate much of my time to mentoring, with three ongoing PhD students – two finishing this year and another next year. Additionally, I have two new students joining from China, one focusing on microplastics and the other on wastewater, using proteomics. This area, which I started about five years ago, represents a newer direction in my work.

Beyond mentoring, I maintain collaborations globally, particularly in China, where I advise several labs primarily on microplastics in soil and agro-ecosystems. There, the emphasis is on agricultural production, as the demand for food and healthy soils is high. My role involves studying the impact of microplastics, along with antibiotics and other contaminants, on soil enzymes and crop productivity.

In Italy, I collaborate on microplastics' effects on biota and ecotoxicology. While in Lithuania, I mentor colleagues on ecosystem services and broader environmental policy issues. I also have a partnership in India, initiated during the COVID-19 pandemic, focusing on antibiotic resistance and SARS in wastewater.

Within Spain, I have two labs: one in Barcelona focused on environmental proteomics – analyzing peptides and proteins in wastewater – and another in Almería, working on microplastics in agriculture. Almería is an important region for food production, so we investigate microplastic contamination in tomatoes and other crops.

Additionally, a significant part of my work involves editorial roles across several journals, which is a major focus of my current activities.

> *“Microplastics absorb a range of harmful substances – metals, organic pollutants, and even pathogens – so they create a harmful system beyond the plastic particles themselves.”*

The main environmental issue that you’re working on is microplastics. Could you detail the dangers of microplastics and the actions we should take?
Microplastics are a very interesting area because, in many ways, they’re similar to nanomaterials. In water, they act as vectors for contaminants, somewhat like carbon-based nanomaterials. Microplastics absorb a range of harmful substances – metals, organic pollutants, and even pathogens – so they create a harmful system beyond the plastic particles themselves. This system has a destructive effect because it’s not just the microplastic that’s hazardous, but what it carries with it. That’s one aspect.

The other side is that “microplastic” encompasses many different types of polymers, such as polyethylene, polypropylene, PET and others. Each has its own properties, sizes, and ages, making the chemistry involved quite complex and, to me, very intriguing. For example, the age and degradation level of a microplastic influence its chemical interactions. A degraded microplastic, detectable by its spectral profile, might have different functional groups that interact in new ways with pollutants.

So, the real challenge and interest lie in this complex chemistry and in understanding how microplastics interact with conventional pollutants. I believe it’s essential to think of microplastics as part of a larger “cocktail” of pollutants, where microplastics act as the primary vector or transmitter. Due to their slow degradation, they’re present in the environment for extended periods and are everywhere.

And which types of contaminants are you referring to that are absorbed by microplastics?
My primary focus with microplastic interactions at the moment is on antibiotics, which I see as a major global issue. Antibiotics are overused worldwide; I recently read a paper estimating that between human and animal usage, we consume around 20 to 30 billion doses of antibiotics daily. With 8 billion people, that’s a massive amount. This excessive use leads to the development of antibiotic-resistant genes, which poses a significant environmental threat. Antibiotics like sulfamethoxazole or ciprofloxacin are present everywhere – in rivers, soils, and ecosystems. Their impact is vast, affecting not only farming but human health as well, especially over prolonged periods.

What is your personal driving force behind your research?
Well, I think it’s the sheer number of challenges still present in the field, especially as an analytical chemist. Take microplastics, for example – there are so many open questions. There aren’t standardized analytical methods for their analysis, and it’s incredibly complex. If you look at the literature, you’ll find significant variation in how measurements are conducted and reported. Some report particles per liter or particles per kilogram, but what kind of particles? What sizes? There are so many unresolved issues, and this makes the field incredibly challenging and exciting.

I recently gave a lecture to encourage young researchers. I told them there are ample opportunities for PhD theses in this area because almost everything is still uncharted – even basic things like inter-laboratory standards. Recent studies comparing standardized polymers, like polyethylene, across expert labs showed discrepancies in identification, indicating how far we are from consistent methodologies. Labs rely on different techniques, such as pyrolysis GC-MS, micro-FTIR, and micro-Raman spectroscopy, and no single lab has all of these techniques to cross-validate results. We still need international collaboration to standardize these methods and measure the impact on fish and biota accurately. If we can’t measure precisely, it’s tough to determine the environmental effects.

I don’t know how many years I have left to work on this – I’m 70 now – but I intend to do my best to make meaningful contributions in this area.

Analytical scientists are often viewed simply as data providers – the people who deliver results. What’s wrong with that?
Well, if analytical chemists only deliver results, they risk being limited in their role and impact. I believe that analytical chemists should work more interdisciplinarily, with fields like medicine, toxicology, and engineering. We should be involved not only in producing data but also in interpreting it – explaining what a

nanogram or microgram level actually means in practical terms. We need to take that extra step beyond data delivery. If we remain data providers only, we'll continue to be viewed as mere analysts.

However, if we collaborate with others and apply our skills to broader contexts, we can attract more interest and demonstrate the unique contributions analytical chemistry can make. Many in our field already work across disciplines – food safety, environmental health – but there's always more we can do to learn from others and expand our impact.

Are contaminants the biggest environmental problem we face today?
The environment is incredibly complex. We're facing climate change, and that makes it hard to interpret what's happening clearly. For instance, at the end of my microplastics lecture, one question I often pose to the audience is, "What's more important – microplastic contamination or the effects of climate change?" A few years ago, there was an incident in Swiss rivers where fishermen couldn't catch salmon. Initially, people thought contamination was to blame, but the real issue turned out to be an increase in river temperature.

This example shows how many factors are at play beyond contaminants. Climate change impacts everything, affecting lives significantly and influencing ecosystems in ways that may overshadow the effects of certain pollutants. While both microplastics and climate change are critical, the temperature increase alone can have a profound impact on living organisms.

Contaminant levels in rivers, thankfully, are somewhat better controlled than they were two decades ago, thanks to tighter regulations. But there are so many variables in play, and climate change now appears to be a leading driver affecting our environment. It's essential to consider climate change first in many cases, as it has such a wide-reaching impact.

And how can analytical scientists help with that issue?
Analytical scientists play a crucial role because our expertise is in measurement and delivering accurate, standardized data. This ability to provide reliable numbers is essential. In climate change work, for example, there are many modelers, and while modeling has value, it sometimes makes assumptions that don't fully reflect reality. I've seen this firsthand in the area of microplastics. A few years ago, we conducted a collaborative study on European rivers and compared our measured data with global modeling results. The models often predicted higher contamination levels than what we actually measured, sometimes by as much as 30–50 percent. That's why reliable measurements are essential – they offer concrete data we can trust for drawing conclusions.

Although modeling is less expensive than monitoring, it may not be as accurate. I once discussed this with an EU project officer who explained that funding often favors modeling due to lower costs, despite monitoring being the ideal. Good analytical chemistry does come with costs – our instruments can be hundreds of thousands of euros, making it an expensive field. But accurate measurements, which give us real-world data, are what should guide our environmental actions.

Models are useful, but they aren't always correct. And even within the modeling community, there's often debate: one modeler may not use another's work because they each believe their own model is superior. Analytical scientists help ground these debates in reality by delivering accurate, field-tested data that models alone cannot provide. This is our strength and contribution, and it's where we should continue to focus.

Finally, could you summarize where analytical scientists should go from here – or what direction should they choose?
We need to prioritize precise measurements and collaboration across various fields. Take ecotoxicology, for instance; they often model but still rely on us to measure actual pollutant levels in organisms like fish or crustaceans. This is where analytical chemistry becomes essential – our measurements provide the baseline data they need. The same applies to engineers, who might want to know how much of a contaminant is removed during wastewater treatment. But as I always have to explain – their methods can create new metabolites that might be harmful. Analytical chemists are crucial because we can identify these metabolites and look beyond the primary compound to see what else is produced in these processes.

There are strong points we bring to our colleagues in fields like toxicology and engineering; we can help explain what's actually happening in a plant, a fish, or a wastewater system. If, for example, a medication like carbamazepine breaks down, it can result in numerous secondary chemicals, each with its own potential impacts. Our work reveals this complexity, while other fields may focus only on the disappearance of the parent compound, assuming the issue is resolved.

This depth of insight is what makes analytical chemistry an indispensable discipline. Yes, it's expensive work, with advanced instruments sometimes costing millions, but it allows us to give highly accurate data and explain what's occurring in different environmental contexts. And this is our strength – to observe, detect, and interpret, providing clarity where other disciplines might miss critical details. From my experience, these strengths help foster respect and collaboration with engineers, ecotoxicologists, and soil scientists, and it reinforces the essential role of analytical chemistry in solving complex problems.

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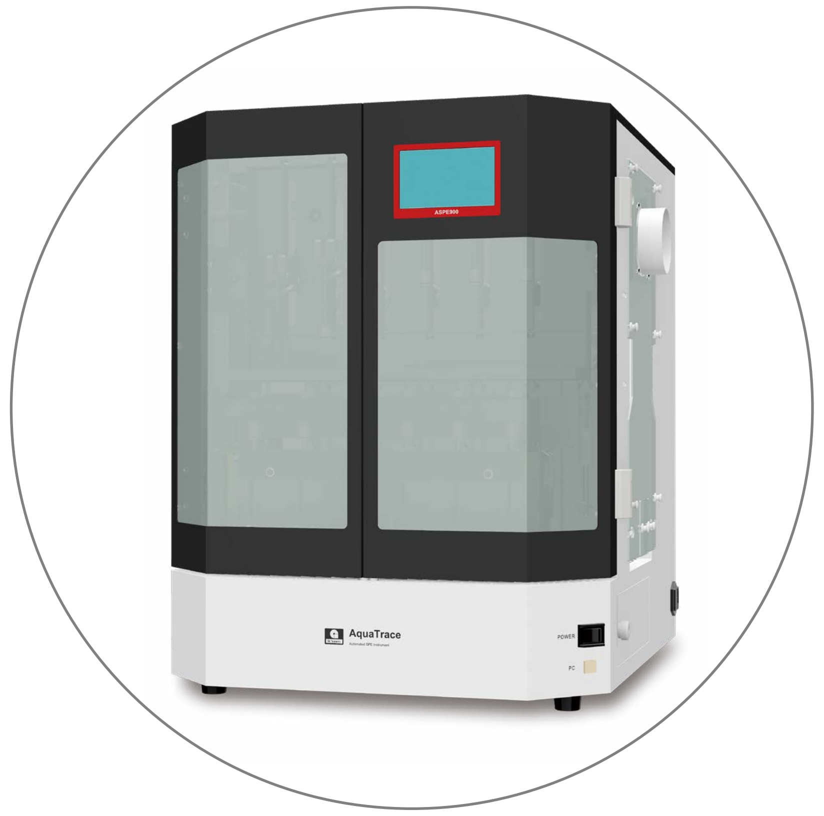

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