Prof. Gauthier Eppe
Université de Liège, Belgium

Setting new standards in POP identification

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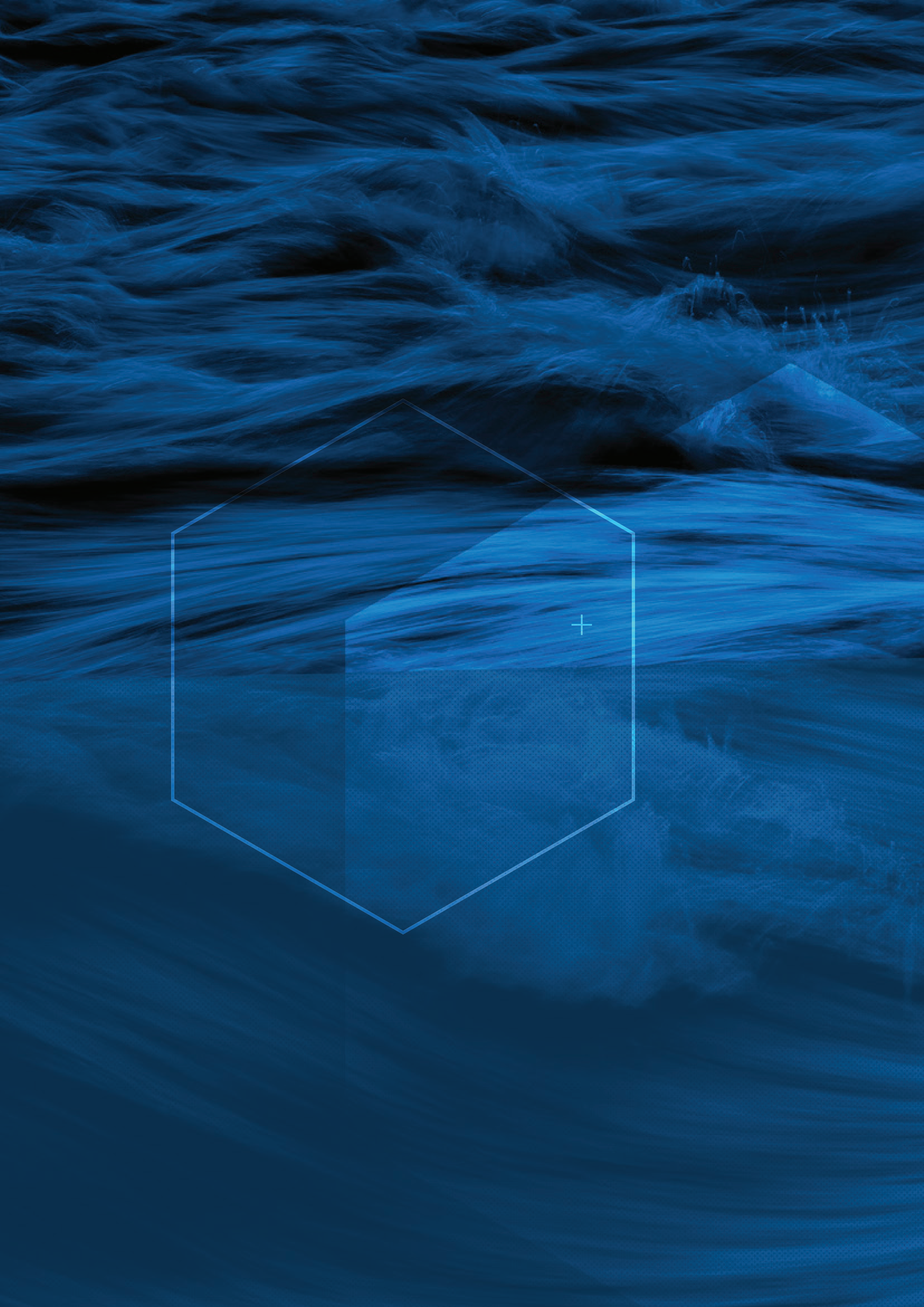

The timsTOF Pro 2 and our collaboration with Bruker AMS have been a real game changer for us, allowing us to significantly advance our research into the impact of POPs.

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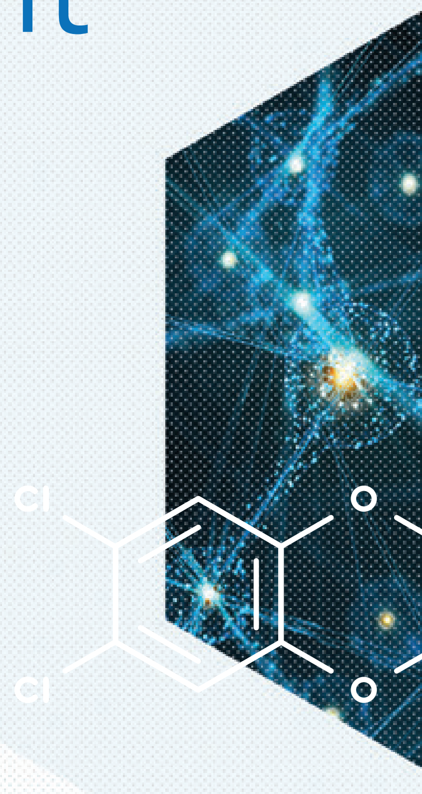
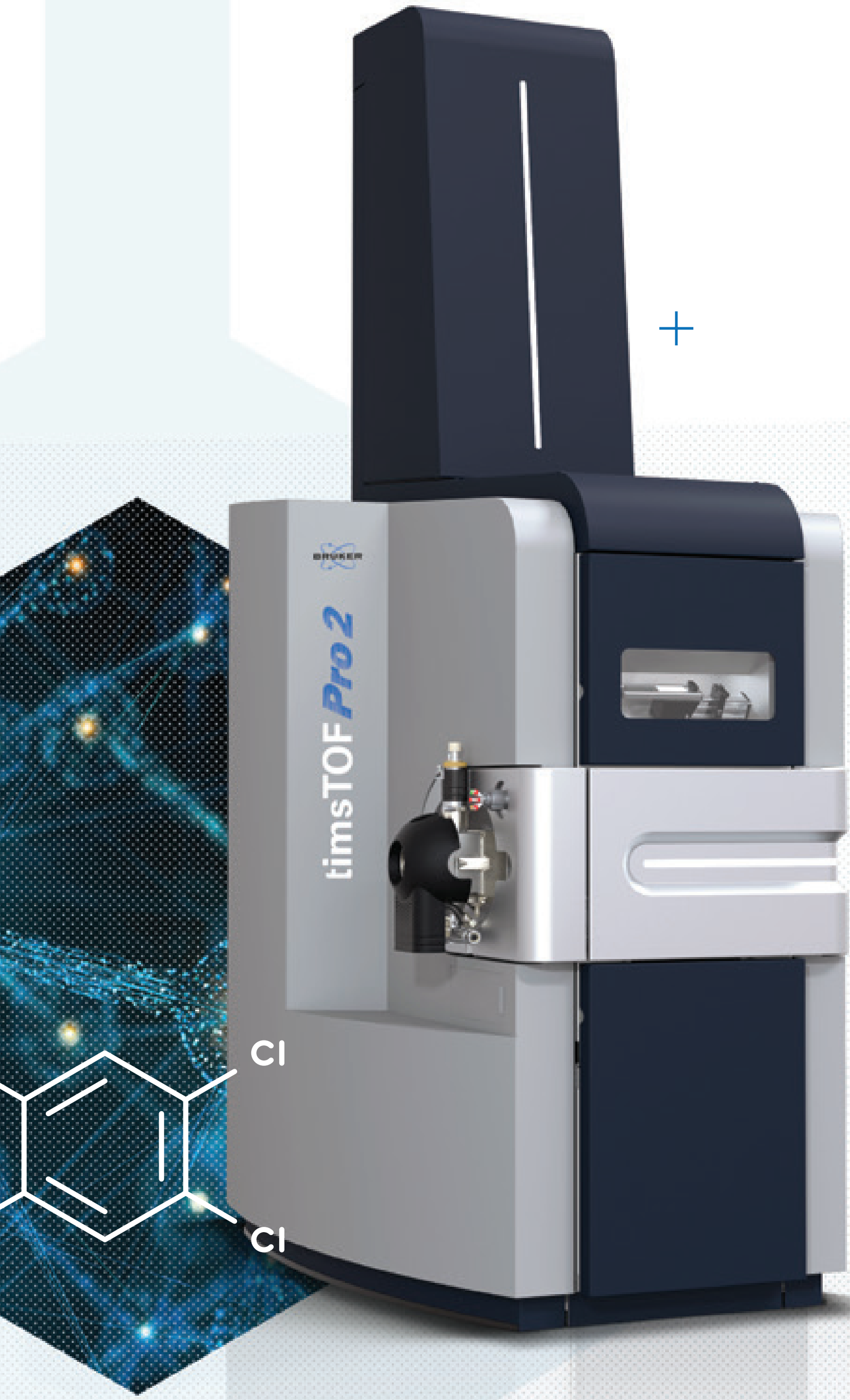
Latest mass spectrometry technology drives persistent organic pollutant analysis





Scientists from the Université de Liège are collaborating with Bruker's Applied Mass Spectrometry division to enhance persistent organic pollutant analysis in the food and environmental sectors using trapped ion mobility spectrometry (TIMS) on the timsTOF Pro 2.

Led by Professor Gauthier Eppe, the Mass Spectrometry Lab (MSLab) at the Université de Liège, Belgium, is a world leader in the field of mass spectrometry research. Recently, Prof. Eppe and his team joined forces with Bruker Applied Mass Spectrometry (Bruker AMS) to develop alternative methods for persistent organic pollutant (POP) analysis, including dioxins and per- and polyfluoroalkyl substances (PFAS), in food, water and the environment. Using Bruker's latest cutting-edge mass spectrometry technology, Prof. Eppe aims to better understand the impact of POPs and ultimately help limit their negative effects on humans and ecosystems.

Gauthier Eppe is a Full Professor at the Université de Liège where he also holds the positions of Director of the MSLab and Director of the Molecular Systems (MolSys) Research Unit. He says:

"I first started research in the field of contaminants over 20 years ago, at a time when there was a serious dioxin contamination crisis in the food chain in Belgium."

From this point, Prof. Eppe has continued to work in food safety, focusing on environmental contaminants' entry into the food chain. He has contributed to the development of various multi-residue analytical techniques, encompassing exposure characterization from agricultural supplies, food products and human biomonitoring in biological fluids.

"With the help of my fantastic team, we are now developing and applying global untargeted characterization approaches in biological and environmental samples, specifically focusing on emerging halogenated POPs but also in metabolomics and lipidomics, utilizing state-of-the-art mass spectrometry detection techniques."

The harmful effects of persistent organic pollutants

POPs are toxic chemicals that are resistant to degradation and can bioaccumulate and biomagnify in ecosystems, the food chain and the human body. These compounds, which include substances like dioxins and PFAS, pose a significant threat because they can cause serious health issues in humans, such as cancer, reproductive disorders, and developmental abnormalities, and also negatively impact wildlife and ecosystems.[i] Their long-range transport through air and water can lead to widespread contamination, making them a global concern that necessitates concerted international efforts for their reduction and elimination.

Recognizing the danger posed by POPs, international agreements like the Stockholm Convention on Persistent Organic Pollutants have been established to control and phase out the production and use of these hazardous chemicals.[ii] The Environmental Protection Agency (EPA) also recently proposed the first federal limits on PFAS chemicals in drinking water in the US.[iii] However, the legacy of past POP use as well as the ongoing emergence of new potential contaminants continues to challenge efforts to mitigate their impact, making ongoing monitoring and remediation efforts crucial to protecting both human health and the environment.

Limitations of current POP analytical methods

The gold standard method for POP analysis is gas chromatography coupled with high-resolution mass spectrometry (GC-HRMS). Whilst this is a powerful and effective analytical technique, it is typically only used to analyze and identify targeted compounds of interest.

Prof. Eppe comments:

"The health impacts, the fate and transport of many POPs are not fully understood because we currently only monitor regulated contaminants."

"This means there is insufficient information regarding the hundreds to thousands of other unregulated compounds synthesized by the chemical industry, including potential precursors and by-products that may give rise to even more harmful substances. With new contaminants emerging in the environment and the food chain all the time, it has become necessary to broaden the scope of analysis and develop new methods that not only consider legacy POPs, but also address the newly emerging ones that are not yet subject to regulatory monitoring. I believe there are potentially thousands of compounds that are going unmonitored – and we need to adapt testing accordingly."

Prof. Eppe continues: *"As the list of target compounds grows, however, each additional family of compounds requires a separate analysis using traditional GC-HRMS systems. This means that for an extensive list of compounds, multiple analyses are necessary to cover all the analytes of interest, which takes valuable time and resources. We therefore require new methodologies that can analyze all analytes more quickly, in a simplified workflow."*

Prof. Eppe explains that another major challenge in the analysis of POPs is the complexity of environmental, food and human blood samples. These matrices contain a multitude of other compounds that can interfere with the accurate detection and quantification of specific POPs. Furthermore, many contaminants exist in extremely small quantities, often in sub-parts per trillion. This means scientists are constantly searching for more advanced equipment that offers higher specificity and sensitivity, which is necessary to ensure accurate and reliable analysis.

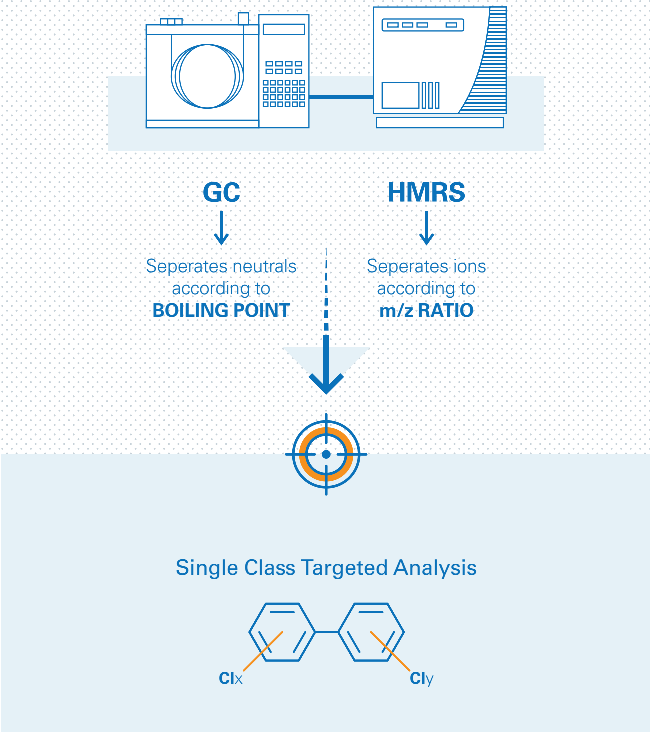
TIMS enables faster and more accurate POP analysis

Prof. Eppe and the Université de Liège have partnered with Bruker AMS to advance the field of POP analysis using the timsTOF Pro 2, a versatile solution tailored for environmental analysis. This solution combines the precision of mass spectrometry with world-class software, offering rapid and accurate analysis of a wide range of contaminants such as dioxins, PFAS, and pesticides.

Prof. Eppe shares his thoughts about this new technology:

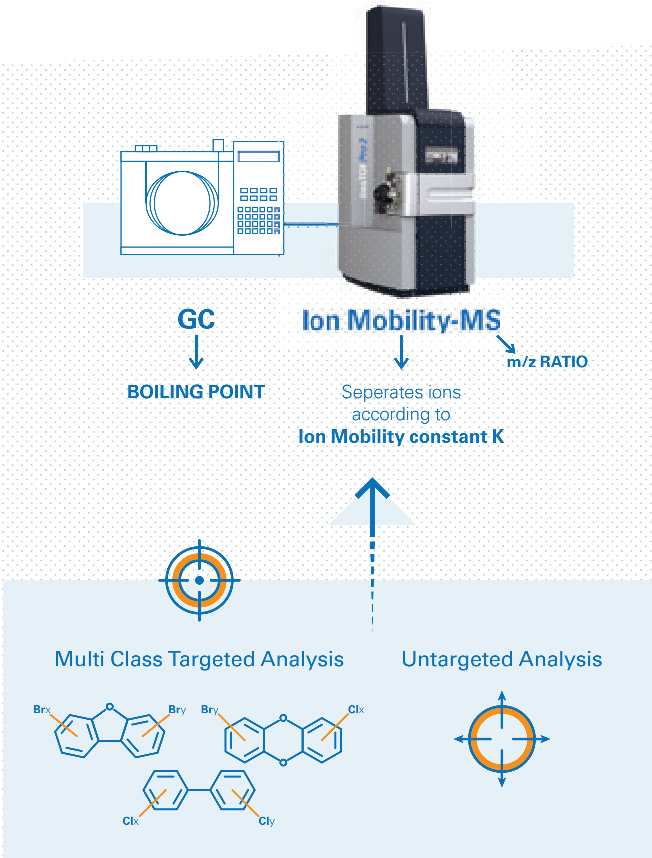
"What sets the timsTOF Pro 2 apart from other analytical methods is its unique combination of high-resolution mass spectrometry and TIMS, a gas-phase separation technique. This adds an additional dimension of separation to improve accuracy and confidence in compound characterization within complex samples. Plus, because the system also simultaneously accumulates and concentrates ions of a given mass and mobility, we are able to increase both the sensitivity and speed of our analysis. Through this unrivaled sensitivity, we can detect extremely low levels of contaminants."

Classical approach



✓ Selectivity / Specificity ✓ Sensitivity

Advanced approach



✓ Selectivity / Specificity ✓ Sensitivity

The timsTOF Pro 2 supports 4D omics analysis, encompassing lipidomics, proteomics and metabolomics, which allows Prof. Eppe and his team to investigate the intricate molecular responses and biomolecular alterations induced by POPs exposure. Prof. Eppe adds:

"This instrument not only facilitates the analysis of specified POPs but also enables us to detect novel, untargeted POPs, helping us obtain a more accurate and comprehensive assessment of the possible exposure to contaminants."

Integrated with state-of-the-art TASQ software, the timsTOF Pro 2 ensures seamless transitions between targeted and untargeted workflows and enhances automated screening and quantitation of POPs.

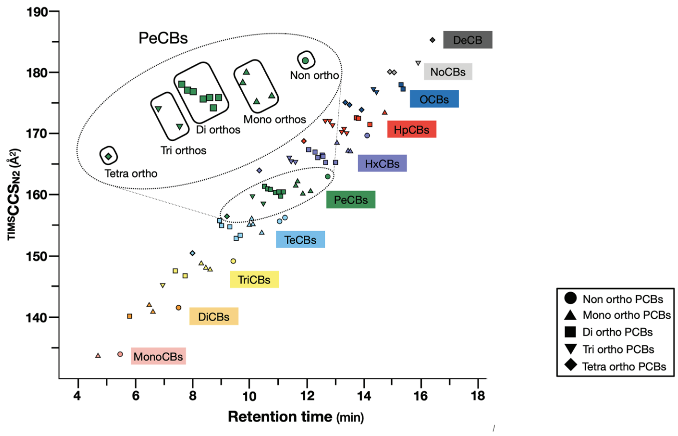


***Figure 1** – Retention time versus CCS of PCBs (n=82). Color and shape respectively refer to halogenation and ortho substitution degree of the PCBs. The inset is a close-up view of the data of penta chloro substituted biphenyls (PeCBs).[iv]*

What's more, Prof. Eppe explains this analysis can be done in a single run rather than requiring the multiple analyses needed in traditional GC-HRMS: ***"This is hugely beneficial as it saves a significant amount of time and improves workflow efficiency, enabling us to perform high throughput analysis for larger studies."***

The system further enhances efficiency by providing predeveloped workflows and an extensive reference database containing thousands of relevant analytes, including pesticides and environmental pollutants, which dramatically reduces laboratory setup time. ***"Overall, the timsTOF Pro 2 and our collaboration with Bruker AMS have been a real game changer for us, allowing us to significantly advance our research into the impact of POPs,"*** says Prof. Eppe.

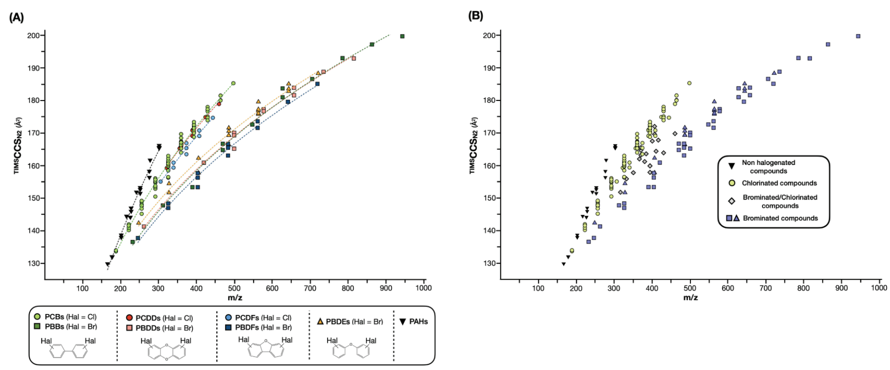


***Figure 2** – CCS vs. m/z plots of the different POP classes analysed in this work. Panel (A) represents the data as a function of the different classes of POPs. Data were fitted with power trendlines (except for the classes of mixed brominated-chlorinated POPs due to the lower number of data points). Panel (B) represents the same data as a function of halogenation type.[iv]*

The future of POP analysis in food safety

Prof. Eppe outlines that his next step is to collaborate with food manufacturers to monitor contamination in the food supply chain. POPs can enter the food chain through various environmental sources which contaminate food webs and aquatic ecosystems. Due to their persistence and ability to biomagnify as they move up the food chain, POPs can become concentrated in high trophic-level species, leading to elevated amounts in foods such as meat, dairy products, and fish.

Prof. Eppe says:

> *"Monitoring and managing POPs in the food supply chain is critical to ensure food safety and protect human health from the potential adverse effects of these toxic substances. By collaborating with industry leaders like Bruker AMS and leveraging the analytical capabilities of advanced technologies like the timsTOF Pro 2, we will be able to promptly identify and address potential contamination issues."*

The analysis of foodstuffs is particularly challenging as technology has to be sensitive enough to detect even the smallest trace amounts. *"The timsTOF Pro 2 system has proven to be highly sensitive for these types of analysis and we are very excited about what we might be able to achieve next with this technology, including detecting POP contamination in food products like eggs, milk and other dairy products,"* he explains.

Prof. Eppe concludes:

"Over time, my hope is that we can collect enough data on other harmful non-target compounds to advocate for their ban and incorporate them into European legislation. Although this process can take years, it is vital that we continue to monitor and advance POP analysis – particularly for unknown and emerging pollutants – in order to better safeguard our environment, food supply, and public health in the future."

Plots of CCS values versus degree of halogenation of compound classes

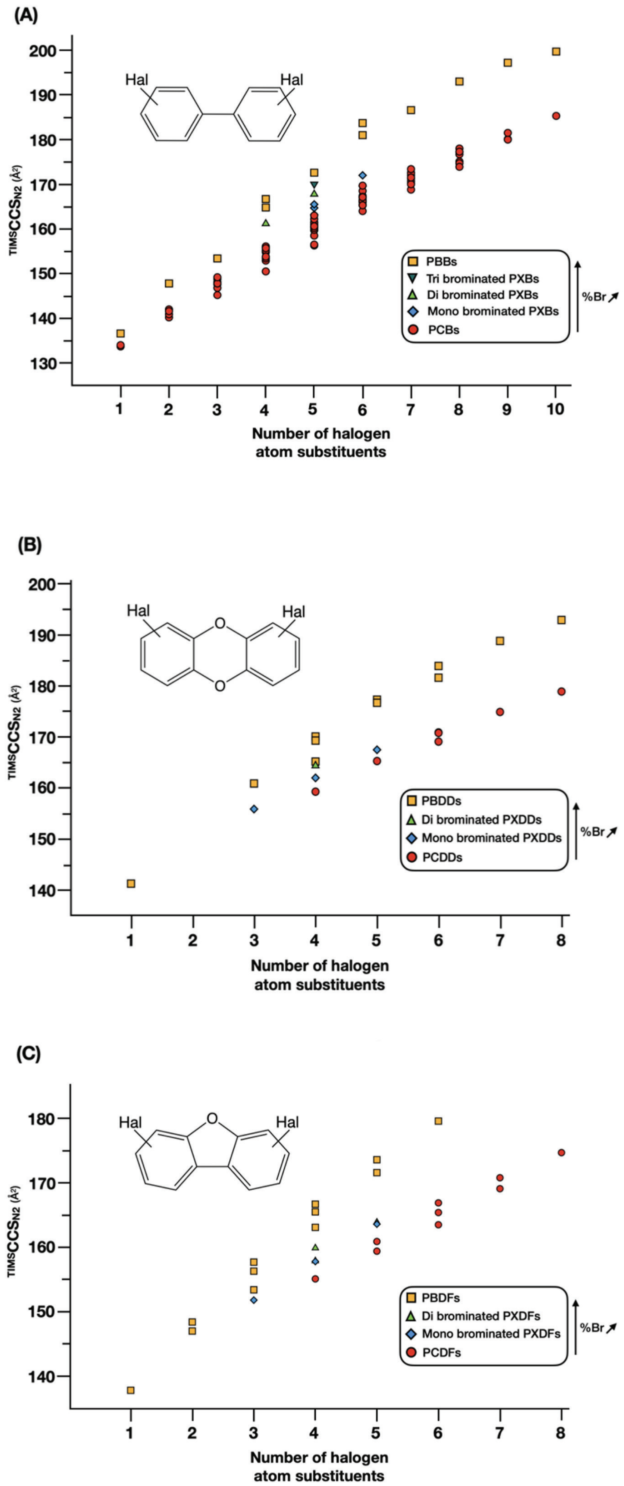
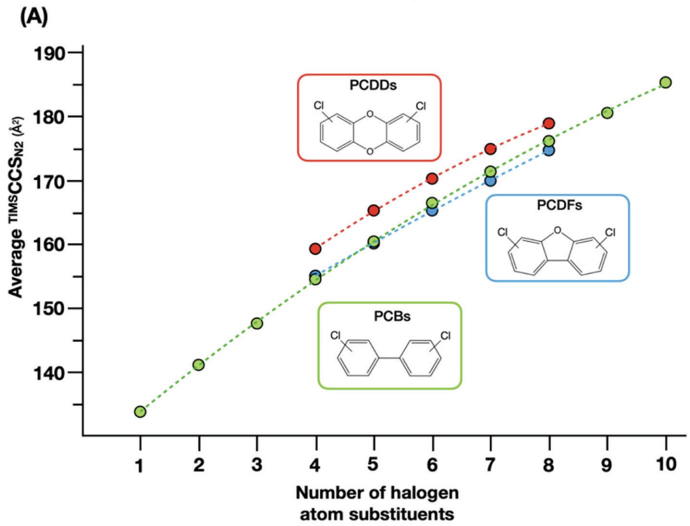


***Figure 3** – CCS vs. halogenation degree plots for the different classes of halogenated biphenyls **(A)**, halogenation dibenzo-p-dioxins **(B)** and halogenated dibenzofurans **(C)**.[iv]*



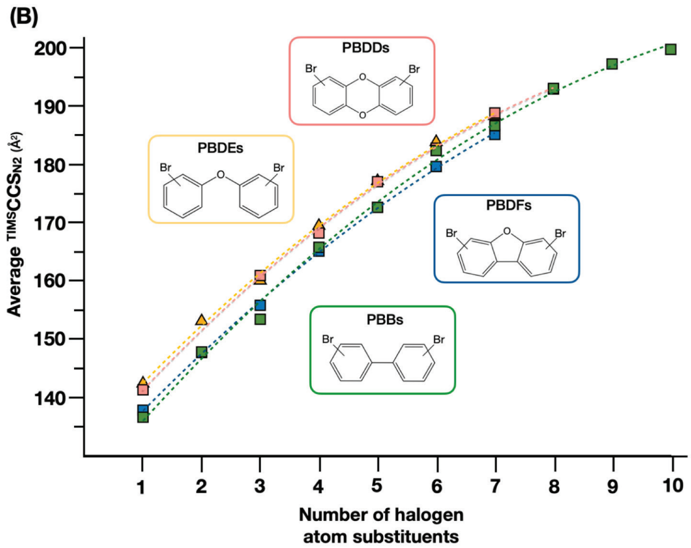


Figure 4 *– Average $^{TIMS}CCS_{N2}$ of chlorinated (A) and brominated (B) POPs classes per halogenation degree. Data were fitted with a second order polynomial regression (dotted curves).[iv]*

References

i Carpenter, David O. "Health effects of persistent organic pollutants: the challenge for the Pacific Basin and for the world." (2011): 61-69, https://doi.org/10.1515/reveh.2011.009

ii Lallas, Peter L. "The Stockholm Convention on persistent organic pollutants." American Journal of International Law 95.3 (2001): 692-708

iii Phillis, M. (2023). "EPA to limit toxic 'forever chemicals' in drinking water." AP News. Available at: https://apnews.com/article/epa-pfas-forever-chemicals-water-contamination-regulations-560d0ce3321e7fa8ed052f792c24f16f

iv Reprinted (adapted) with permission from Muller HB, Scholl G, Far J, De Pauw E, Eppe G. Sliding Windows in Ion Mobility (SWIM): A New Approach to Increase the Resolving Power in Trapped Ion Mobility-Mass Spectrometry Hyphenated with Chromatography. Anal Chem. Published online November 17, 2023. doi:10.1021/acs.analchem.3c03039. Copyright 2023 American Chemical Society.

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Bruker Switzerland AG

Fällanden · Switzerland
Phone +41 44 825 91 11

Bruker Scientific LLC

Billerica, MA · USA
Phone +1 (978) 663-3660

marketing.bams.emea@bruker.com

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