PFAS Testing: Is a Storm Brewing?

Are dark clouds on the horizon for the food and beverage industry as regulators ponder wholesale PFAS bans and consumer lawsuits multiply?

the Analytical Scientist x Agilent Technologies

On January 13, 2022, five EU member states submitted an official proposal, which, if implemented, would result in the most extensive chemical ban in European history.

Germany, The Netherlands, Denmark, Norway, and Sweden are seeking to ban all chemicals in the per- and poly-fluorinated alkyl substances (PFAS) group due to serious concerns about their impact on human health. Studies have linked PFAS exposure to reduced fertility and altered menstrual cycles, thyroid function abnormalities and hormone imbalances, altered liver profiles, and even cancer (see sidebar: PFAS Exposure and Health).

Such a ban would have wide-reaching implications for many industries that use PFAS for their non-stick, water-resistant, and heat-resistant properties – including the food and beverage industry. For food companies, it would mean a dramatic increase in testing to ensure PFAS – which can enter food through using water in the production and manufacturing process or via bioaccumulation in livestock, including crops, silage, and grass grown on sludge-fertilized fields – is below prescribed limits.

The proposal is currently being reviewed by the EU's scientific committees, so there's no guarantee that such a ban will come into force. But it should sound alarm bells for the industry that, sooner or later, will need to get to grips with PFAS.

"The PFAS problem is an incredible societal challenge," says Michele Suman, Food Safety & Authenticity Senior Scientist-Research Manager at Barilla SpA, and Adjunct Professor at Catholic University of Sacred Heart Milan. "The distribution of PFAS in the environment and food is almost ubiquitous, and the number of compounds that are progressively detected continues to grow – with the simultaneous problem that many of them seem to be already at levels that, although low, still exceed the presumed safety threshold. Food and drinking water are important vectors of human exposure; therefore, it is of utmost importance that analytical tools are available to uncover the problem."

Uncertainty abounds

The uncertainty around future PFAS testing requirements is a big challenge facing food manufacturers. Indeed, the current guidelines and requirements are far from the proposed blanket ban and hard limits.

"The EU has both regulations and guidelines for PFAS; the guidelines are more stringent than the ones currently in regulation," says Lorna De Leoz, Global Food Segment Director at Agilent Technologies. The existing regulations cover four PFAS compounds in egg, seafood, fish, and meat. But the acceptable levels differ in terms of the food matrix.

Suman walks us through the European regulations: "The European Food Safety Authority (EFSA) has set a Total Weekly Intake (TWI) at 4.4 ng/kg body weight for four main PFAS, namely perfluorooctanoic acid (PFOA), perfluorooctane sulfonic acid (PFOS), perfluorononanoic acid (PFNA), and perfluorohexane sulfonic acid (PFHxS). In correspondence, the European Commission issued recommendations and regulations ((EU) 2022/1431 and (EU) 2022/2388) and contextually requested Member States to monitor PFAS in several foodstuffs. The EU Recommendation also requests the monitoring of other 23 PFAS compounds (sulfonates and fluorotelomer alcohols)."

"It's a little odd that PFAS is the only chemical regulated in terms of weekly intake, rather than tolerable daily intake," says Karl Pettit, Technical Director at Veritas Laboratory Services, UK. Pettit has been involved in trace organic analysis since 1987 and PFAS for the past decade. "Is it safe to fast throughout the week and binge on PFAS at the weekend? I jest. But there's a serious point: whether a product exceeds the limit over the course of a week depends on how much of a product you are likely to eat – which is open to interpretation."

In the UK, regulatory limits are even harder to come by, suggests Pettit. "There are some in meats, but if we go to fresh products, there are some guidelines that suggest 'further investigation' – but no hard limits," he says. "The UK still has a long way to go."

In the US, there are no federal regulations for food. "But the FDA can issue recalls on imported and local food products if they deem that the PFAS levels are a health concern," says De Leoz. "And there is a state-level restriction in Maine for milk, beef and fish."

The key question is, will things change? "We expect the more stringent recommendations to convert to mandated regulation eventually," says De Leoz. "But as for the EU-wide ban, the picture is far from clear."

Nick Birse, Lecturer in Mass Spectrometry at the Institute for Global Food Security and the School of Biological Sciences, Queen's University, UK, is skeptical about the prospect of a wholesale ban in the EU. "Given what we've seen with other EU regulations – the delayed EU deforestation regulations, for example – I could easily see the PFAS ban getting suspended," he says.

Wave of litigation?

Amid the regulatory uncertainty, another dark cloud looms on the horizon for the food industry: the potential threat of litigation. Indeed, litigation is already happening – with several cases having already been filed. For example, Kerrygold was faced litigation for its Pure Irish Butter product, where it was alleged the foil packaging contains PFAS. In addition, Coca Cola, McDonald's, and other companies including microwave popcorn producers have also been sued.

PFAS Exposure and Health

A recent review into the impact of PFAS highlighted the following potential human health consequences.

Immunotoxicity
PFAS exposure suppresses the immune system, diminishing its ability to fight infections and respond to vaccines. Key findings include:

- Altered cytokine expression and activation of peroxisome proliferator-activated receptors (PPARs), which regulate lipid metabolism and inflammatory responses.
- A study showed a 50 percent drop in vaccine-induced antibody levels in children with increased PFAS concentrations in their blood. This highlights the risk of compromised immunity even at low exposure levels.

Carcinogenicity
The International Agency for Research on Cancer (IARC) classifies PFOA as a possible human carcinogen. Research has linked PFAS exposure to:

- Increased risks of kidney, testicular, prostate, and liver cancers.
- Mechanisms such as oxidative stress, endocrine disruption, and epigenetic modifications, with long-chain PFAS demonstrating the highest toxicity.

Endocrine and kidney disorders
PFAS disrupt endocrine function and impact kidney health through mechanisms such as:

- Competitive binding to thyroid hormone transport proteins, reducing circulating thyroid hormones and impairing thyroid homeostasis.
- Links to decreased kidney function, kidney cancer, and glomerular filtration rate changes, contributing to various kidney diseases.

Effects on fetal growth
PFAS exposure during pregnancy affects fetal development:

- Studies found that prenatal PFAS exposure correlates with low birth weights and growth variability in early childhood.
- PFAS transfer from mother to fetus, with higher concentrations of PFOS in maternal plasma than in placentas, raises concerns about prenatal exposure risks.

Source: Z Habib et al., Pollutants, 4 (1), 136–153 (2024). DOI: 10.3390/pollutants4010009.

However, the difficulty facing any litigation attempt is proving that PFAS contained within food or food packaging did indeed cause harm, which is no easy feat – especially from an analytical perspective.

A central problem is that we don't know exactly how many PFAS there are. Different agencies and organizations use varying definitions, leading to significant discrepancies in the estimated number of compounds. "I've heard estimates of 7,000, 14,000 – and more recently 1 and 2 million," says Pettit. Moreover, even if non-targeted analysis can be used to identify a compound that appears to be PFAS, confirming it is a significant challenge. Analytical standards are needed for confirmation, and as of now, there are few authentic standards available – and many of those that are available aren't pure enough to include in targeted analysis, or contain contaminants that interfere. In other words, it's not just about developing the methods, it's also about the accessibility and reliability of the analytical standards needed to make these methods robust and accurate. "I've hunted around for nearly every standard available – and found only about 100," says Pettit. "Another complication is that some companies will litigate against those making standards! Really, we're in a dire situation."

"I don't think anybody really has an idea what's going on," says Birse. "Part of the problem is that there's a lack of toxicological data. We genuinely don't know which PFAS compounds are safe and which are dangerous."

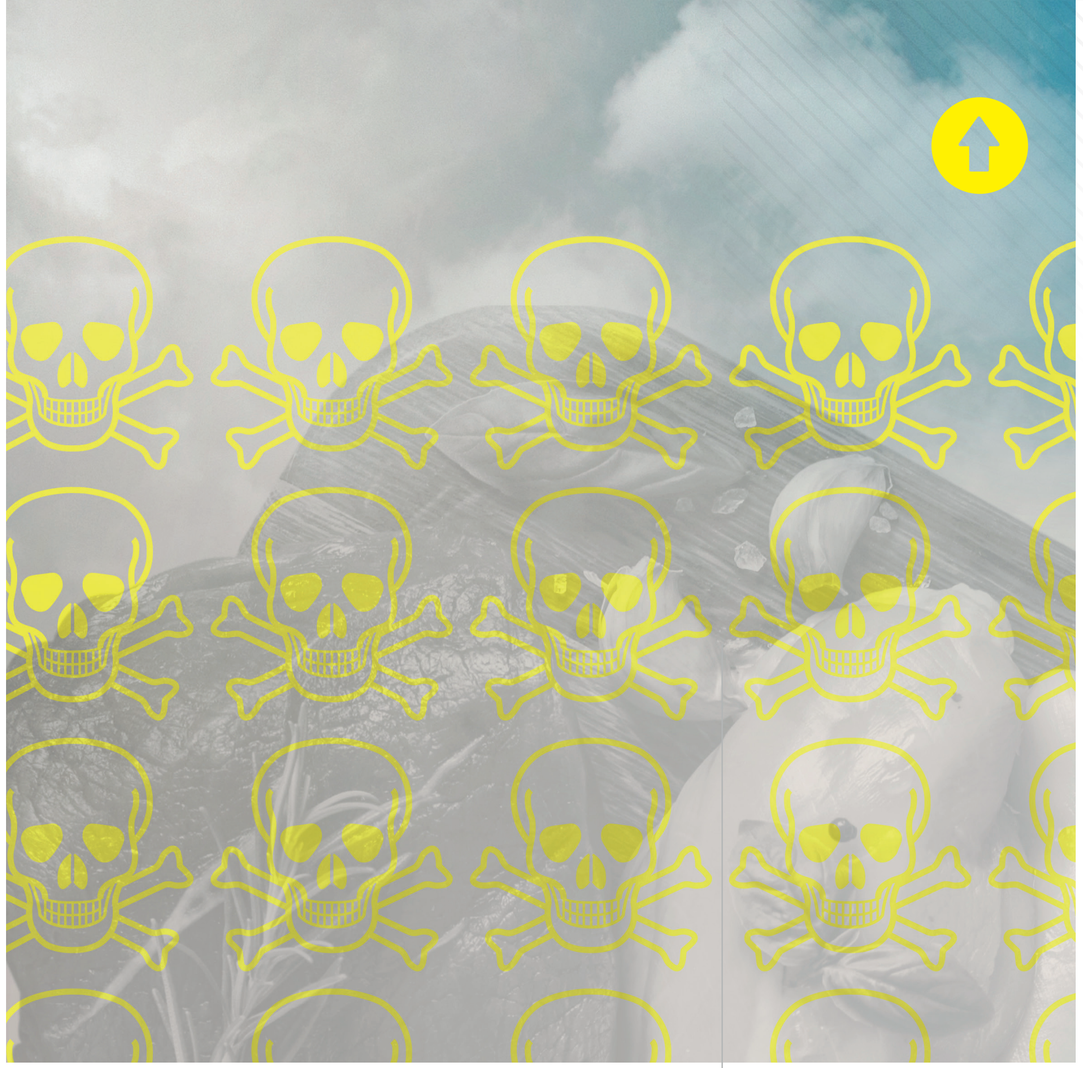
According to Birse, the analytical community is focused on long-chain PFAS compounds, which are easier to detect and analyze using mass spectrometry – but they're not the ones that are most likely to have a toxicological impact, he suggests. "It's the smaller compounds that are much harder to detect, particularly with LC-MS, that we need to be interested in," he says. The problem, Birse says, is that the longer compounds can fragment into shorter compounds. "We end up going around in circles about which ones we should be interested in. Are we genuinely seeing them in the sample or were the peaks generated in-source? Although the sensitivity of the instruments is improving all the time, I just don't think we're quite there yet.

"In my view, the evidence isn't quite there to pursue a class action lawsuit."

Homing in on Birse's comment about in-source fragmentation, recent research from Gary Siuzdak and Martin Giera showed that in-source fragmentation could account for many peaks in LC-MS datasets – potentially over 70 percent. "But looking at in-source fragmentation isn't easy to do – you need standards and known concentrations, then you have to do time-of-flight on the breakdown products," says Pettit. "Good luck finding a lawyer that understands this!"

However, De Leoz argues that a lawsuit doesn't necessarily have to succeed – or go all the way to proving damage – to have an effect on an industry. "Often companies facing a large number of lawsuits will settle to avoid going to court – even if the individual cases against them might not succeed. Sometimes, it's a question of brand protection, rather than the chemical realities."

Another important question is whether taking action early might increase liability further down the road. "If a manufacturer does the testing now and is unwilling or unable to take the necessary action to reduce risk to their consumers, would they be more likely to face legal action in the future than if they'd have simply thrown up their hands and said: 'we followed the guidelines at the time?'" asks Pettit. "Of course, you'd like to think a company would do the right thing…"



What – and where – to test

Despite the uncertainty concerning regulatory trends, potential litigation, and the chemical realities at play, Pettit believes that food manufacturers should prepare for more testing. "There's going to be lots of testing – I think that goes without saying," he says. "But it might take time to really take off."

Sue Bullock, head of chemical compliance, stewardship and sustainability at TSG Consulting, also sees more testing on the horizon for the food industry. "The EU PFAS REACH restriction, still under development, will almost certainly have implications for both packaging and food manufacturing equipment," she says. "PFAS is also now an emerging priority of public interest, with regular reporting regarding PFAS in the environment, food, and our bodies across mainstream media. We expect to see continued pressure from consumer groups, investors, and NGOs on this topic. Companies that demonstrate understanding and control on PFAS will be better positioned to manage these pressures."

Bullock also believes there will be a new focus on PFAS in packaging following the decision to prohibit PFAS above certain thresholds in food contact packaging under the EU Packaging & Packaging Waste Regulation (PPWR). "The PPWR is expected to be finalized imminently with the limits of PFAS coming into force 18 months later," she says. "These regulations require testing at raw material level. That is, contaminant limits for PFAS are set for meat, fish, and eggs as raw materials – not processed products. With this in mind, we expect a short-term increase in testing in Europe, driven by these regulations."

Learning from the Past

By Jacob de Boer, Professor in Environmental Chemistry and Toxicology at VU University, Amsterdam, The Netherlands

Having worked for almost 50 years on persistent organic pollutants (POPs), I have seen a lot of processes reoccurring. Many halogenated compounds have caused concern for human and environmental health. First produced without initial testing, analytical scientists detected them some years later in food and the environment. Once toxicologists assessed the effects and discussed them with authorities and industry, we started to see severe worldwide restrictions. I have also seen that the sensitivity of our instruments has improved by a factor of at least a million. This impressive achievement means we now have an early warning system: we can now detect compounds at a level before they are doing harm.

Today, the focus has shifted to PFAS; their strong immunotoxic effects demand that we push our detection systems to their limits – but it can be done. We have wonderful mass spectrometers and advanced chromatographic systems that enable us to tackle the PFAS problem, while new approaches and instruments are in development. Labeled standards have become available, but the complexity of PFAS mixtures requires more standards (native and labeled) to be made.

My hope is that commercial analytical standards producers, just like instrument companies, will reduce the prices of these standards; otherwise, research may stop simply for cost reasons. In addition, the production of certified reference materials should be encouraged worldwide because they are essential to maintaining good QA/QC with properly validated methods. It is important that the European Joint Research Centre and the National Institute of Standards and Technology in the US will continue their efforts in this regard.

Of course, it would be much better if we didn't have to focus our analytical work on detecting new persistent chemicals in the environment and our food, but so long as we continue to produce them – that's our job. That said, I believe that chemicals should be tested before production and market release, following a "benign by design" approach. Learning from past cases, including DDT, polychlorinated biphenyls, and now PFAS, it is clear that similar scares will return if we do not introduce such pre-production tests. In the short term, it is essential that we accept the European proposal to ban PFAS – as a group of chemicals – to prevent further global contamination.

"PFAS testing will certainly increase in the future," says Vikash Kumar, Program Lead, Polymers & Materials, at ChemBizR, a business research and consulting company for chemical companies. "Completely removing PFAS would be an expensive decision, and it requires investment, innovation, and time. But controlling PFAS can be immediate, which is the way forward for now – more testing to meet the PFAS limits set by regulatory authorities on a daily basis across the value chain."

"We really are going to have to start testing food," says Pettit. "I often speak with water companies that test for PFAS, where there are some guidelines with regard to acceptable limits. But that water is going into feed/food. We're spraying it on crops. And this will be taken up by food – which we're not even monitoring."

"I could see pesticides regulation – where they test in batches – being expanded into the PFAS area," he says. "But it comes down to the toxicology data – that's the key."

"It's unclear at this stage whether companies will need to focus on supply chain management and certification or fully assess their raw materials and finished products for PFAS contamination," says De Leoz. "And it might take a scare to make people really invest."

De Leoz alludes to the big question for companies aiming to mitigate the risk to consumers and themselves, while also readying themselves for future regulatory changes, namely, figuring out what – and where – to test.

"The priority has to be the end product, as it's presented to the consumer," says Pettit. "But if we're talking about looking at the source, then that's a different testing regime altogether."

"I think testing will need to take place across the whole value chain," says De Leoz. "This starts with raw materials received from food ingredient suppliers, testing after they are unloaded into the manufacturing plant, during production, and prior to the release of the finished and packaged food product."

"We've spoken to a lot of customers, and PFAS contamination is a very real issue," she says. "Contamination can occur through vials, solvents, air vents, carpets – even the clothing of lab technicians or scientists. One striking example involved a customer who traced PFAS contamination back to the mascara worn by one of their lab technicians."

"For a factory, the PFAS limit can be higher in some processes, so PFAS testing is not required in every process," says Kumar.. "However, the control should be made at the liquid and gas effluent. Also, to avoid last-minute warning (leakage to the environment), a factory should also have a SOP (standard operating process) to test the effluent 1-2 steps before exit. For the inlet materials, a factory should get a certificate/declaration from their vendors for the PFAS limit to avoid overtesting the PFAS limit. The solid PFAS waste can be managed carefully to avoid any future leakage to the groundwater or air."

Another final consideration is sourcing, given that regulations differ significantly by region. "What may be strictly regulated in the US or EU might not be regulated at all in other countries," says De Leoz.

Pettit agrees. "Yes, when dealing with pesticides, an assessor once said: 'Why test for 700 pesticides when you know the origin of the product and can narrow it down to the 10 or 20 pesticides likely in use?' This approach could be applied to PFAS as well."

Pettit believes that by understanding where the product comes from and knowing the common PFAS compounds in use in that region, companies could implement more targeted and efficient testing strategies.

Birse points to areas that are known to be PFAS hotspots – areas close to airports or PFAS production facilities, such as Northwest United States and the Amsterdam/Rotterdam area. "If companies are hearing of potential contamination sites and avoiding those, that might give them some cover in the event of litigation – because they've done their due diligence."

"Manufacturers need to prepare," says Bullock. "They should carry out risk assessments of the raw materials they buy and consider including PFAS testing in their goods in protocols. They should work closely with their packaging suppliers and manufacturers to ensure that their packaging will be future compliant based on a detailed breakdown of packaging constituents. Where food contact materials and other packaging rely on PFAS for functional purposes, considerable effort may be required to find suitable alternatives."

The Techniques at the Fore

Three experts explore the arsenal of analytical techniques at the forefront of PFAS detection and identification

Jochen Mueller (Professor, University of Queensland): There are so many methods that have a role in PFAS analysis. Of course, we routinely use liquid chromatography mass spectrometry (LC-MS/MS) for the main bulk of target PFAS analysis and liquid chromatography-high resolution MS for suspect screening and discovery. IC-MS is employed for short chain compounds like TFA and GC-HRMS is used for some volatiles. DESI-MS can be very useful for "2D" visualizing PFAS in niche applications, and DART-MS is used for surface desorption work. There is also space for FTICR and NMR for identifying new PFAS. And techniques such as PIGE, μ-X-ray Fluorescence, and Fluorine K-edge μ-X-ray absorption near-edge structure (XANES) spectroscopy are also used. Every technique has its relevance.

Several sample preparation methods, such as the total oxidizable precursor (TOP) assay, have become important to PFAS analysis. LC-MS/MS and possibly increasing LC-HRMS are likely to remain essential in this area for some time – depending on the specific needs of analysis, of course.

Mark Strynar (Senior Physical Scientist, US EPA): Both LC-MS and GC-MS have access to authentic analytical standards for method development and robust validation of each analyte in specific environmental media. Analytical methods that have been developed with these approaches perform very well for the intended purposes. However, the coverage of PFAS analysis is only for the compounds within the method and the media it was designed for.

A number of analytical techniques have sprung up to address this issue, such as non-targeted analysis (NTA), total oxidizable precursor (TOP) assay, total organic fluorine (TOF), extractable organic fluorine (EOF), adsorbable organic fluorine (AOF), and particle-induced gamma-ray emission (PIGE) spectroscopy. However, these techniques also have shortcomings and no one method answers all the questions being asked. For example, the TOP assay oxidizes precursors to terminal PFCAs with the assumption that all precursors will go to the monitored terminal PFCAs, which is not always the case; PIGE analysis shows 19F content in solid samples without knowledge of the source compound and generally reduced sensitivity; and while NTA shows new and emerging PFAS, it is only those analytes amenable to the chosen extraction techniques (for example, solid phase extraction) and analysis techniques (for example, negative mode MS) employed that are detected.

What remains lacking is a universal NTA method or ability to quantitate newly discovered PFAS – though progress is being made on both fronts. Additionally, GC-MS NTA applications are far behind the LC-MS applications currently in use. Though we consider many PFAS to be ionic (anionic or zwitterionic) and respond well to LC-MS, there are a host of volatile and semi volatile PFAS amenable only to GC-MS applications. Like any other method, NTA has its pros and cons, but it would be my preferred choice for PFAS analytical sampling by a wide margin. With the addition of other analytical techniques and workflow applications, I anticipate that NTA will gain additional ground – so long as they are supported by additional analysis techniques.

Stefan van Leeuwen (Senior Scientist, Wageningen University): I'd add that sensitive targeted analysis is important for dietary exposure assessment, but this can cause challenges with a high variety of matrices (such as fat, protein, and carbohydrate-rich samples) reaching lower (sub)ppt levels. To get an idea of the potentially hundreds or thousands of PFAS we have yet to detect and their relevance for our exposure, we also work with high resolution mass spectrometry (HRMS) identification and chemical and biological screening assays. These complementary approaches allow us to identify a response in a sample extract or identify compound, indicating a potential effect on the immune system or other effects. Chemical screening and HRMS identification can provide answers on the total amount and identity of individual PFAS present in a sample. In my opinion, a combination of these approaches can provide us with understanding of the levels, identities, and possible effects of PFAS in our food. There is no single analytical solution or one-stop-shop. The aim of research in this area dictates which analytical approach – or combinations thereof – to take.

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