

Interview: PFAS analysis

„Struggling with high blank values“

The planned EU regulation of all per- and polyfluorinated alkyl compounds (PFAS) requires analytical methods that detect small amounts of the substances in all kinds of samples. Susanne Sölter, application specialist LC/MS at Agilent in Waldbronn, knows the pitfalls of PFAS analysis and how to avoid them.

Analytica Pro: Your application laboratory is in contact with industrial users from various sectors. Do you sense an increased interest in PFAS analytics?

Susanne Sölter: It depends. Customers in the textile industry were some of the first to reach out to me and enquire about PFAS analysis throughout the whole garment-making process, from the fabrics they use to the process water and wastewater. Apart from that, I can't say too much about the wider requirements across all industries because most sectors keep a fairly low profile when it comes to PFAS. We see more questions from environmental and food analytical laboratories. However, we notice big investments are being made in analytics both from industrial customers and from contract laboratories.

There are already some standardised methods for PFAS analysis in drinking water and food. What about the analysis of clothing, cosmetics and other articles of daily use?

In principle, the same methods of liquid chromatography and mass spectrometry (LC-MS) can be used across different matrices. The range of analytes is also similar, as those PFAS are measured against commercially available analytical standards. However, the sample preparation, the type of extraction, differs depending on the matrix.

In the case of foodstuffs, for example, relatively complex purification or enrichment steps are necessary in order to reach the detection limits. In part, the detection limits are already specified by law, but some are still being discussed.



Susanne Sölter, application chemist at Agilent, specialising in PFAS. (Photo: S. Sölter)

How do I ensure that the analysis actually correctly detects all PFAS contained?

Every method must be validated. This can be done, for example, using certified reference materials with known contents. This makes it possible to check whether one's own extraction and processing steps lead to the correct result. Taking the textile industry as an example, the suppliers of reference materials have collected a large number of garments, created a sample from them, and ground it into a powder. This powder, which has a certified content, can be bought.

And if there are no reference materials for a particular product?

Then you can spike the samples with isotope-labelled standards before extraction and thus determine the recovery. If the sample matrix strengthens or weakens a signal, this can be recognised on the iso-

tope-labelled standards and thus compensated. These standards are especially crucial with PFAS.

Why is this more important with PFAS than with other substances?

PFAS are relatively unruly analytes. Many of these substances are surface-active. On clothing, for example, they are supposed to repel water and not be washed off. PFAS also adhere very strongly to other surfaces, such as the walls of vials. When I put an extract into a vial, I often don't see much at low concentrations. This loss can be compensated for by isotope-labelled standards.

What else is tricky in PFAS analysis?

The biggest problem is that PFAS are used in many production processes and are therefore found almost everywhere. This leads to relatively high blank values in the analysis. The limit values currently discussed in the EU, and some of which already exist, are often almost impossible to achieve because the blank value is already above or in the range. An example: We need solvents for analysis, such as methanol or acetonitrile – and PFAS are already present. Of course, in extremely small quantities, but they are still there. Pipettes and many other things used in the laboratory can also consist of fluorine-containing polymers that release PFAS or contain PFAS, contaminated at production.

Are materials containing PFAS also used in analytical instruments?

Yes, there are also PFAS in the LC-MS. This is an important point in the discussion with customers. If we don't take any precautions with the equipment, a

relatively high background already follows from the analytical system alone. A remedy for this is to replace all parts that contain PFAS for PFAS-free components. This is very important in the case of hoses and other large components. In liquid chromatography, delay columns ensure that contaminants from the analytical system have a different retention time than the actual analytes. This helps to get the problem under control.

How high are the blank values?

If I take everything into account, if I use high-purity solvents and so on, they are usually between 0.1 and 0.5 nanograms per litre, but sometimes even at 1 or 2 nanograms per litre. In principle, that's almost nothing, but it's still too much. The new EU Drinking Water Directive contains a sum parameter that sets a determination limit for individual PFAS of 1.5 nanograms per litre. That is not far from the blank value. As long as we are struggling with the high blank values, there is not

much we can do about the sensitivity of the measurement in terms of equipment. But our goal is accelerated analytics, robust detection with fast evaluation.

PFAS are relatively unruly analytes.

Susanne Sölter

Do you concentrate on methods that detect individual substances?

No, these are always multi-methods along the lines of pesticide analysis. Ideally, you detect 500 pesticides with one injection, each with its own individual peaks. In the case of PFAS, there are far fewer so far, as there are not as many standards available yet. However, up to 60 different PFAS can be determined in parallel currently. The trend is to look for more, and a good way

to do this is through a non-target approach: Which unknown PFAS are in my sample? This can also be investigated by fragmenting the substances in the mass spectrometer.

Is the analysis always LC-MS-based?

Not necessarily. LC-MS is the common method, but there are some fluorinated compounds that are more compatible with gas chromatography (GC) and cannot be detected with LC-MS. GC-MS is better suited for highly volatile, small PFAS in particular, and perhaps also for their degradation products. Alternatively, NMR spectroscopy or element-specific detections, for example via ICP-MS, can be used for PFAS analysis. You need a whole bundle of analytical techniques, as there are thousands of different PFAS.

The interview was conducted by Uta Neubauer.

**Agilent at Analytica
Hall A2, Booth 201**

30 Years of Passion & Innovation

Reagents • Standards • Services



ZYMO RESEARCH

The Beauty of Science is to Make Things Simple



Celebrate with us! Stay tuned for great birthday deals throughout the year!

Meet us at booth
A3 - 312 B