LC-MS for Improved Analysis of Food and Water Contaminants

Essential Knowledge Briefings

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LC-MS for improved analysis of food and water contaminants

Wiley Essential Knowledge Briefing

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INTRODUCTION

Contaminants in our food and water are an unavoidable fact of modern life. Pesticides, pharmaceuticals, personal care products, industrial chemicals and even by-products from the disinfection of drinking water can all make their way into food and water. Some are applied directly, like pesticides on food crops, where residues can remain even after washing and processing. Others, such as pharmaceuticals and personal care products, inadvertently make their way into the water supply after being flushed down the drain.

These contaminants are usually only present in food and water at trace concentrations, however they can still have adverse effects in humans and research is still ongoing to identify any synergistic effects that may occur when we are exposed to a cocktail of them in food and water. Further, it is known that some of these contaminants are potentially toxic and carcinogenic, regulatory authorities around the world have set maximum permissible concentrations for them. This, in turn, calls for regular testing to ensure these concentrations aren't being exceeded.

Such testing is required to assess the concentration of contaminants in environmental waters. These contaminants not only have detrimental effects on the wildlife in these waters, but many contaminants are very persistent. Meaning, they can build up in the environment, including in animals in the food chain (biological amplification). If these animals are subsequently consumed by humans, as with fish, then this provides another route of exposure to these contaminants.

Whether food or water, this testing presents quite a challenge, due to the contaminants being present at trace concentrations in samples that are very complex. The analytical technique mainly employed to meet this challenge is liquid chromatography with mass spectrometry (LC-MS). This works by first separating complex samples of food and water into their individual components with liquid chromatography, and then detecting and identifying any contaminants among those components with mass spectrometry.

In this Essential Knowledge Briefing (EKB), you will discover just why LC-MS has become the 'gold standard' for detecting contaminants in food and water. It will begin by introducing the technique and the latest instruments, including ultrahigh performance LC and triple quadrupole MS, then detailing what benefits they offer for food and water analysis.

After a short introduction, it will go on to describe how an LC-MS analysis is conducted in practice and what factors need to be considered, including sample preparation and method optimization, and then outline the specific challenges involved in analyzing food and water samples and how to overcome them. This will be complemented by three case studies on scientists who are actually utilizing the latest LC-MS instruments in ensuring the safety and quality of food and water supplies.

Lastly, the EKB will finish by considering what the future holds for LC-MS in food and water analysis, including the development of instruments that are smaller, more automated and even more sensitive.

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HISTORY AND BACKGROUND

Food and water, both environmental surface water and drinking water, can contain a wide range of different contaminants from various sources. For example, both food and water can contain pesticide residues. In food, these are generally residues from pesticides directly applied to food crops, but in environmental water sources such as rivers and lakes they come from pesticide runoff accidentally washed in from surrounding fields.

Other chemical contaminants, such as phthalates and primary aromatic amines, can enter food from their packaging and from cooking utensils such as plastic containers and cutlery. Bottled water can also contain phthalates that have leached from the plastic bottle, while tap water can contain contaminants such as haloacetic acids and other disinfection byproducts, formed when disinfectants are added to the water and react with other organic chemicals.

In addition to pesticides, environmental surface water can contain many contaminants that get flushed down the drain, both by commercial industry and households. Many of these contaminants aren't completely removed by current water treatment processes, making way into the environment. They can include: personal care products such as shampoos and cosmetics; pharmaceuticals, both human and veterinary, and hormones which are excreted in urine; and industrial pollutants such as per/polyfluoroalkyl substances (PFASs), which are found in almost everything – from nonstick cookware to firefighting foam to marker pens. These contaminants can potentially have adverse effects on both humans and wildlife. Pesticides and drugs are obviously designed to have biological effects, which can unintentionally harm non-target organisms, including humans. But other contaminants, including phthalates, haloacetic acids, PFASs, are toxic to wildlife and are known or suspected carcinogens in humans.

Many of these contaminants are stable and persistent, meaning they aren't rapidly broken down in the environment, so tend to build up in the food chain. On top of this, foods can also become contaminated with

natural toxic compounds, such as mycotoxins, that can have adverse effects on human health at high enough concentrations.

Because of the potential risk to human health from these contaminants, both man-made and natural, regulatory authorities such as the US Food and Drug Administration, US Environmental Protection Agency and the European Commission define safe thresholds for them in food and water. Ensuring that these thresholds are not being exceeded requires regular testing, which can present quite a challenge.

For a start, the thresholds are very low because the contaminants are generally only present in food and water at trace concentrations; often at the low microgram or nanogram "per kilogam" or "per liter" levels. Food and water are also complex samples, containing a wide range of unrelated chemicals that can interfere with the detection and measurement of the contaminants.

Such testing requires an accurate, sensitive and versatile analytical technique that can reliably detect and measure trace concentrations of specific contaminants in complex samples, which currently means some form of LC-MS.

As its name suggests, LC works with liquid samples, which is fine when analyzing water samples but means that food samples need to be treated in some way to extract the contaminants into a liquid, such as dissolving it in an organic solvent. This liquid sample is then pumped through a chromatography column with a fluid known as the mobile phase, which is an organic solvent used to separate the contaminants based on their chemical properties.

The chromatography column is filled with a solid material known as the stationary phase, which can be made up of tiny porous particles or a single porous polymer known as a monolith. The idea is that, as the liquid sample is pumped through the column, the compounds of interest (analytes) within it will be attracted to both the stationary phase and the mobile phase to different extents. Thus, some analytes will bind to the stationary phase better than others, causing the complex sample to be separated into its individual components.

Over a period of time, the analytes will become separated as it flows through the column. As it elutes out of the other end, they will be converted into ions and directed into a mass spectrometer. As the analyte ions hurtle through the vacuum chamber in a mass spectrometer, they are exposed to electric and magnetic fields that deflect their path, with the amount of deflection depending on their mass and charge. By varying the electric and magnetic fields, analyte ions with different masses and charges can be directed towards a detector, allowing their mass-to-charge ratios (m/z) to be determined.

Specific analytes, such as contaminants of interest, can then be identified from both the time they took to travel through the LC column and mass spectrometer, known as the retention time, and their m/z values (Figure 1). Furthermore, the abundance of each analyte can be determined from the intensity of the signal produced by the detector. This is all reflected in a mass spectrum comprising an array of different sized mass spectral peaks, with each peak representing a different analyte ion detected at a specific point in time.

Because of its ability to separate a complex sample into its component parts, LC has been used for analyzing food and water samples since the late 1960s. This is when high-performance liquid chromatography (HPLC) was first developed, which increased the speed and sensitivity

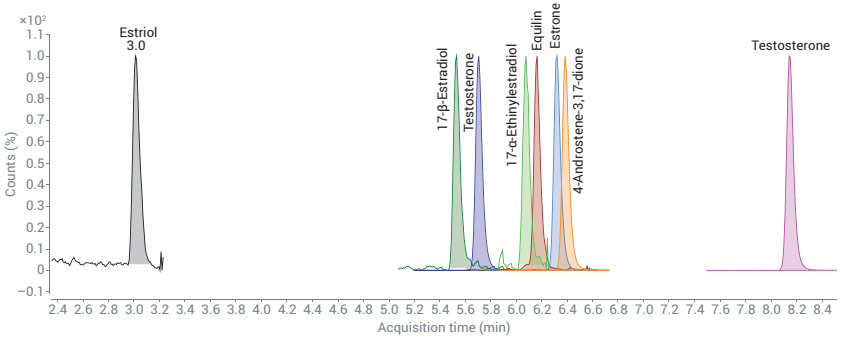


Figure 1. LC/MS Chromatograph for eight hormones in tap water. Each chromatographic peak represents each hormone's unique m/z, eluting at a unique retention time.

of LC by injecting liquid samples onto columns filled with tiny silica particles at high pressures. HPLC was first coupled with MS in the 1970s, but the technique initially struggled due to the difficulty of converting liquid samples into ions.

This was resolved in the 1980s with the development of electrospray ionization (ESI). In this technique, the liquid eluting from the LC column is passed through conductive capillary tubing and an electric voltage is applied to the open end of the capillary, transforming the liquid into a charged aerosol spray. The eluting analytes in the liquid are thus sprayed into the mass spectrometer – often with the assistance of an accompanying flow of nitrogen, known as sheath gas – becoming ionized as the droplets in the spray evaporate.

Since then, LC-MS systems have steadily become faster and more sensitive as the technology has advanced, such that they can now detect contaminants in complex food and water samples at sub-nanogram per liter (parts per trillion) concentrations. For LC, these advances include the development of ultra-high-performance LC (UHPLC), which uses porous silica particles below 2μm in size or porous monoliths to achieve better separations. So-called core-shell particles, in which the particles comprise a solid core surrounded by a porous shell, have also proved very effective.

For MS, the advances include the development of triple quadrupole MS – a highly selective and sensitive detector. A quadrupole is a set of four metal rods arranged parallel to one another around a central chamber. Direct current (DC) and oscillating radio frequency (RF) voltages applied to the rods create an electric field in the quadrupole for deflecting analyte ions. Analytes with the right m/z values (resonant ions) make it all the way through a quadrupole to the detector, while the others (non-resonant ions) collide with the rods before they can reach the detector. The quadrupole can be tuned to accept different m/z values by changing the applied DC and RF voltages.

A triple quadrupole comprises three of these quadrupoles in series, allowing the instrument to perform tandem MS. The first quadrupole is used to select an ion with a specific m/z value, which is then passed into

the second quadrupole, acting as a collision cell. Rather than a vacuum, this quadrupole is filled with gas molecules such as helium, nitrogen or argon, which the ion smashes into as it hurtles through, causing the ion to fragment. The resulting fragment ions are then analyzed in the third quadrupole. The *m*/*z* values of these fragment ions provide additional information for determining the identity of the original analyte ion and also allow its abundance to be calculated with great accuracy. Modern triple quadrupole MS systems can do this very quickly, as they have fast electronics that allow them to select and fragment up to 500 analytes in a single run, known as multiple reaction monitoring (MRM).

There is another mass spectrometer variant, known as Q-TOF, in which the third quadrupole is replaced with a time-of-flight (TOF) mass spectrometer. This measures *m*/*z* values by recording the length of time taken for ions to travel through the chamber when accelerated by a fixed electric field, as this time depends on the mass of the ions.

Because these two variants have slightly different abilities, they tend to be used for different types of food and water analysis. Triple quadrupoles are very effective at determining the abundance of different ions with very high sensitivity and selectivity, so are mainly used to test for the presence of *specific* contaminants and determine their concentrations. Q-TOFs, by contrast, have a high scanning speed and can measure masses very accurately, so tend to be used to screen samples to find out what kind of contaminants are present.

In practice

Before samples of food and water can be analyzed by LC-MS, they usually have to be prepared to get the contaminants of interest into a form that can be injected into an LC-MS. Food samples need to be converted into a liquid form that can be injected into the LC column without damaging or clogging it. This tends to involve crushing and homogenizing a few grams of the food sample into a pulp or slurry, mixing it with water or an organic solvent like methanol or acetonitrile to release any contaminants, then centrifuging this mixture to separate the solid particles from the contaminant-containing liquid.

This step obviously isn't required for water samples, which can be injected directly into the LC column. But because any contaminants are likely to be present at trace concentrations, both food and water samples are often treated to extract and concentrate any contaminants, thereby making them easier to detect and measure.

Many different methods have been developed for doing this, with perhaps the simplest being liquid-liquid extraction. This involves adding a liquid that is immiscible with the liquefied sample, meaning the liquids don't mix but instead form two distinct phases (see Figure 2). The most obvious example is water with an organic solvent, but many organic solvents are also immiscible with each other.

The idea is to choose a liquid that is not only immiscible with the liquid sample, but to which the contaminants being analyzed have a greater affinity. Thus, when the two liquids are mixed together, the contaminants will naturally transfer into the added liquid, while the components that aren't of interest, such as proteins, lipids and other biological molecules, will remain in the original liquid. The two liquids will then naturally separate into two phases, allowing the liquid that contains the contaminants to be used for analysis by LC-MS. The most common extraction procedure based on this principal to extract organic contaminants like pesticides, drugs and others is called QuEChERS. Additional details on this can be found in literature (see the Further Information section).

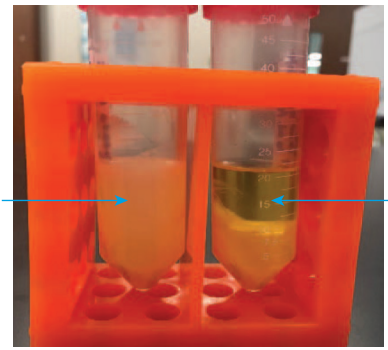


Figure 2. The organic solvent acetonitrile (ACN) is immiscible with high-salt water, causing them to form two distinct phases when mixed.

Other methods tend to be solid-phase extraction (SPE), in which the contaminants bind to an absorbent material, often a polymer. There are several ways in which this absorbent material can be exposed to the sample, which again is usually in a liquid form.

In classic SPE, the absorbent material fills some kind of container, such as a column or a glass vial, into which the liquid sample is poured through. In solid-phase microextraction, the absorbent material is coated onto a fiber, which is immersed in the liquid sample for a set period of time. In stir-bar sorptive extraction, the absorbent material is coated onto a bar that is stirred through the liquid sample; the bar can be magnetic, allowing it to be stirred continuously be simply applying a rotating magnetic field. Magnetism is also employed in magnetic solid-phase extraction, where the absorbent material is coated onto magnetic particles that are dispersed in the liquid sample for a set period of time, before being removed with a magnet.

In all these SPE methods, the contaminants are subsequently released from the absorbent material by exposing it to a liquid, often an organic solvent, with which the contaminants have a high affinity. The solvent is then usually injected directly into an LC column for analysis.

Traditionally, this extraction and concentration step was performed separately from the LC-MS analysis – and some methods still have to be – but increasingly this step is being directly linked with the LC-MS

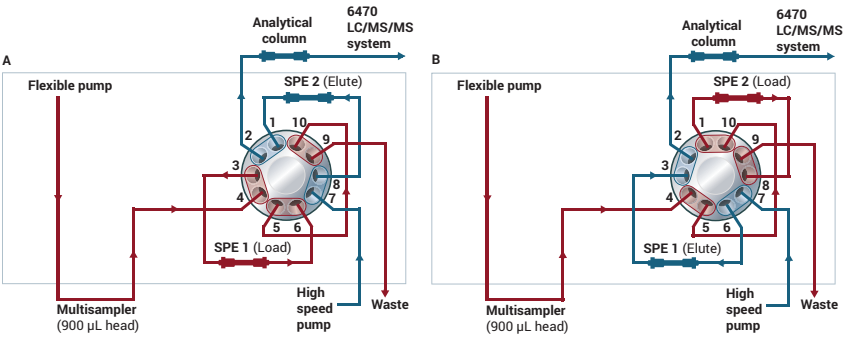


Figure 3. Diagrams showing how multi-way valves can be used to link SPE and LC-MS together. A: valve position for loading; B: valve position for eluting.

analysis, by using automation and multi-way valves to connect the SPE column with the LC-MS system (see Figure 3). This allows the whole process to be conducted automatically, with minimal human involvement, saving time, reducing human variability and reducing the risk of contamination as the sample moves between the different steps.

To get the best results from LC-MS – in terms of separating the contaminants with high resolution and then accurately detecting and identifying them – various experiment parameters need to be optimized for each specific analysis. For LC, these parameters include the type of stationary phase and mobile phase. Different stationary phases are designed to bind with different types of analytes, utilizing a range of interactions such as electrostatic, hydrophobic and van der Waals. Additionally, different mobile phases have affinities for different kinds of analytes. The partitioning of the analytes between the mobile and stationary phases dictates how long they take to travel through the column and influence how well they are separated.

Because analytes vary quite a bit in how strongly they bind to the stationary phase, the mobile phase is often varied over the course of an analytical run, ensuring that all the analytes elute from the column. This is usually done by using a mixture of two liquids as the mobile phase, gradually changing the ratio between the two over the course of the run, such that one dominates at the start of the run and the other dominates by the end. This is known as gradient elution.

Other parameters also affect how well the analytes are separated and thus need to be optimized. These include the sample volume, column temperature and mobile phase flow rate.

For MS, ion source temperature and electronic parameters such as polarity and ESI capillary voltage must be considered, because it influences how well ions are formed and transferred to the instrument. It is important to consider an analysis in positive or negative ionization mode, because some ions are more effectively formed in one mode or the other. Most modern LC-MS systems are able to rapidly switch between the two modes in a single run. Important ion source parameters include: the flow rate and temperature of the sheath gas; and the temperature at the entrance to the mass spectrometer.

Until recently, these parameters had to be optimized by a combination of previous experience and trial-and-error, but software in modern LC-MS systems can do it automatically when given information about the sample and target analytes (see Figure 4). This software can also automatically processes and display the resulting spectral data.

Contaminants are identified by matching the retention times and spectral data for both the precursor and fragment ions to that of known contaminants in databases, or by trying to use the spectral data to determine molecular formula and structure of potential contaminants from scratch. However, both approaches currently require a lot of human involvement. In triple quadrupole MS systems, the intensity of the signal from one or more of the fragment ions can also be used to accurately determine the concentration of the precursor ion.

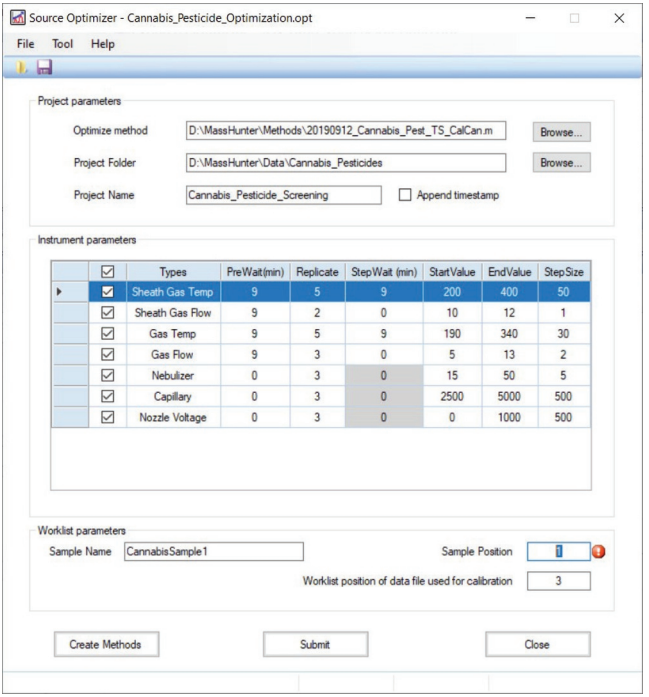


Figure 4. An example of the optimization software in modern LC-MS systems.

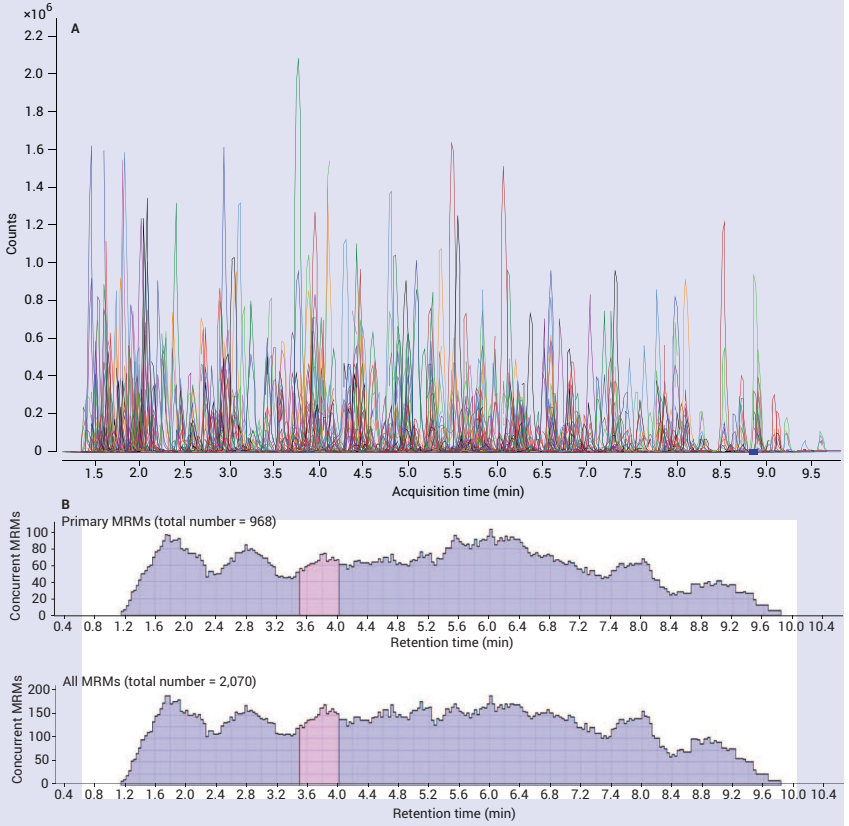
CASE STUDIES

Case study 1

Kate Mastovska, Covance Laboratories

As associate scientific director of Nutritional Chemistry and Food Safety at Covance Laboratories, a global contract research organization, Kate Mastovska and her team use triple quadrupole LC-MS to analyze contaminants, residues and adulterants in food.

"The analysis of food contaminants is challenging because you have to analyze many different compounds at low levels in complex matrices," says Mastovska. But the sensitivity and versatility of triple quadrupole LC-MS is allowing Mastovska and her team to detect more and more contaminants in an increasing



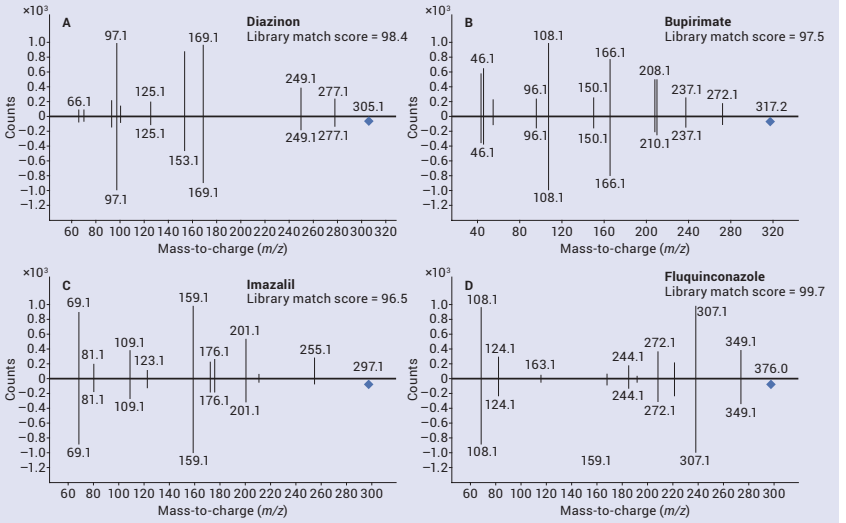
range of food products. "Using the sensitivity of the instrument to dilute the sample we can analyse even very difficult matrices," she says.

One focus of her team's work is pesticides. "[Triple quadrupole instruments] have allowed us to improve the selectivity of our detection and achieve very low detection limits." Recently, she and her team managed to implement analysis of over 500 pesticides in a single method.

"Our triple quad LC-MS systems allows the use of triggered MRMs, which means we can obtain more information per compound without sacrificing performance," she explains. "That helps us in identification, which is crucial in contaminant testing because we really need to be sure of what we find, especially if it is not supposed to be there. Expanding the method to over 500 pesticides helps us comply with global regulations and serve global clients."

Another focus is detecting mycotoxins in infant formula. "Regulatory limits are very low and the sensitivity of the instruments allows us to achieve those low quantification limits," she says. Again, she and her team use triggered MRM to obtain more information and thus obtain a high level of confidence in their results.

"We do testing throughout the supply chain. It's not easy. That's why identification is very important. Sometimes we get into disputes where we need to be almost 100% confident that what we found is the compound we identified."



(Examples of triggered MRM spectra and their matching against a reference library obtained using 10 MRMs for selected pesticides.)

CASE STUDY 2

Agustin Pierri, Weck Laboratories

As highly persistent chemicals that don't biodegrade, per- and polyfluoroalkyl substances (PFASs) have spread far and wide since they were first produced in the 1940s. Commonly used in non-stick cookware, stain resistant clothing, grease resistant paper, household cleaning products and even personal care products, PFASs can now be found everywhere, from drinking water and food, to living organisms, including fish, animals and people.

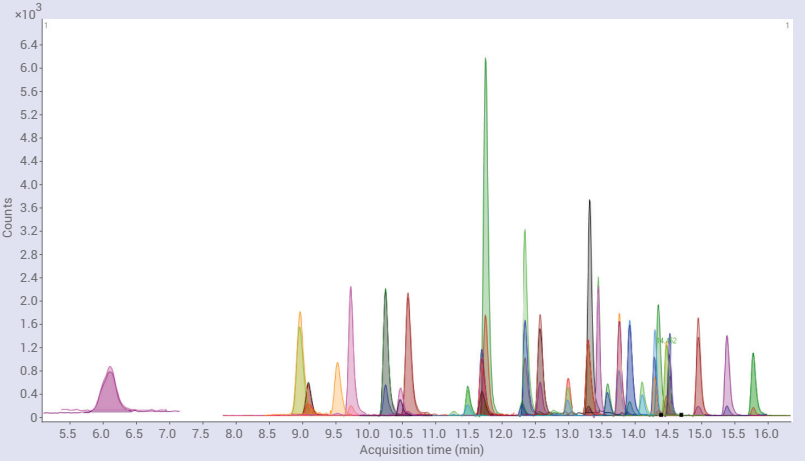
This ubiquity has raised concerns, because there is evidence that exposure to PFASs can damage reproductive systems, liver and kidney functions, and may even cause cancer. As a result, there have been calls for more regulatory guidance and tougher rules on detecting and monitoring PFASs, particularly in drinking water.

But according to Agustin Pierri, laboratory technical director at Weck Laboratories, an environmental services company in California, US, PFASs are actually quite difficult to detect in environmental samples. In part, this is because they are amphiphilic, comprising a long hydrocarbon tail, which is hydrophobic (water-hating), topped with a hydrophilic (water-loving) polar group. As a consequence, PFASs tend to congregate at the surface of water samples, with their polar groups immersed in the water and their tails lying across the surface, which makes sampling them quite difficult. "It requires a lot of steps to make sure that you get all the targets," says Pierri.

There are also issues with contamination. "These compounds really are everywhere, so you need certain measures in place to minimize the effects [of contamination so] you can accurately quantitate these compounds in [samples] without having to deal with environmental contamination in the lab," Pierri explains.

Once they have dealt with the sampling and contamination issues, Pierri and his team turn to triple quadrupole LC-MS. "PFAS work well with LC-MS," he says. "They are generally permanently negatively charged, so they have a huge response to negative mode in LC-MS. We have so much signal from these compounds."

Pierri and his team have used triple quadrupole LC-MS to detect PFAS in samples of drinking water and soil. "We get enough sensitivity down to the levels [required] in real world samples… normally down to the nanogram per liter concentrations. That's pretty low."



(Extracted ion chromatogram of a composite working standard of all analytes at 80 ng/L in 0.1 % acetic acid in 1:1 (v:v) ultrapure water:methanol.)

CASE STUDY 3

Michael Thurman, University of Colorado

Michael Thurman, co-director of the Laboratory for Environmental Mass Spectrometry with his research Partner, Dr. Imma Ferrer, at the University of Colorado in the US, heads a team that detects contaminants such as pharmaceuticals, pesticides and hormones in water. To do this, he employs several LC-MS systems, and pushes them to their limits.

"Ten years ago, we had to detect 1 part per billion and now it is 1 part per trillion, or even lower than that," Thurman says. "We also need to make information available in a very short turnaround time."

One of these systems is UHPLC with triple quadrupole MS, which Thurman says is fast, sensitive and able to detect compounds in water samples at very low concentrations.

"Where it used to take one hour to analyze a sample, now it takes five minutes. Triple quad also has the ability to do positive/negative switching on the fly. This allows us to condense all of the analysis into one run. The MassHunter software is an important component of this because it allows us to generate standard curves for as many as 25–30 compounds in an instant."

To screen water samples for novel contaminants, Thurman employs UHPLC with QTOF-MS. In a recent study, he and his team used it to analyze flowback – a mixture of water, dirt, sand and chemicals that flows to the surface after an oil and gas well has been injected with fluid during hydraulic fracturing (fracking).[1]

For the first time, Thurman and his team were able to detect polypropylene glycols (PPGs) and polyethylene glycols carboxylates (PEG-Cs) in this flowback. While PPGs and polyethylene glycols (PEGs) are known additives in fracturing fluid (used as clay stabilizers and surfactants), PEG-Cs are not. Thurman and his team suggest that PEG-Cs may be in the flowback as trace impurities or degradation products of PEGs.

1. Thurman EM, Ferrer I, Rosenblum J, *et al.* Identification of polypropylene glycols and polyethylene glycol carboxylates in flowback and produced water from hydraulic fracturing. ***Journal of Hazardous Materials*** 2017;323:11–17 (https://doi.org/10.1016/j.jhazmat.2016.02.041).

PROBLEMS AND SOLUTIONS

The latest UHPLC and triple quadrupole MS systems may be very sensitive, but they are still being asked to do some very challenging analyses when detecting and measuring contaminants in samples of food and water. As mentioned earlier, this is primarily because the contaminants are present at very low concentrations (often nanogram per liter or kilogram; parts per trillion) in very complex samples containing lots of other compounds that can interfere with the detection of the contaminants.

One of the ways they interfere is by eluting from the LC column at the same time as the contaminants, producing spectra containing lots of peaks that can be difficult to distinguish from each other. This is especially the case if the interfering compounds are present at higher concentrations than the contaminants, in which case the signal produced by the interfering compounds can completely obscure weaker signals produced by the contaminants of interest.

Co-eluting compounds can also interfere by suppressing ionization of the contaminants, which they do by taking up lots of space on the evaporating droplets. This reduces the intensity of the signal produced by the contaminants at the MS detector, making them more difficult to detect and artificially reducing the measured concentration.

Both these causes of interference can be addressed by performing an extraction and concentration step, in order to separate the contaminants from interfering compounds. But this step is usually only partially successful, especially for really complex samples such as food, because whatever extraction method is used will usually extract other compounds along with the contaminants. So, while this step will reduce the level of interfering compounds, it won't get rid of them entirely.

The whole sample treatment workflow prior to analysis by LC-MS, including preparing the sample and the extraction and concentration step, can also cause complications for determining the concentration of the contaminants. This is because reliably extracting all the contaminants from a complex food or water sample is almost impossible, meaning that any measurement is often an over- or underestimate.

This problem can be addressed through the use of internal standards. These are compounds that are almost identical to the contaminants but won't naturally be found in the sample, as they have been engineered to contain unusual isotopes of different mass. Because they're chemically similar to the contaminants, these compounds should respond in a similar way to the treatment process.

The idea is to add a known concentration of the internal standard to the sample, perform the normal sample treatment process and then measure the concentration of the internal standard by LC-MS. The percentage difference between the concentration added and the concentration measured can then be used to correct the concentrations measured for the contaminants.

A similar approach can be used to make allowances for ionization suppression. This involves analyzing a sample of food or water spiked with specific contaminants and then analyzing a specially prepared solution of those contaminants at similar concentrations as in the sample. Because the solution obviously doesn't contain any interfering compounds, any difference in the size of the signal generated by the detector for specific contaminants in the sample and solution must be due to ionization suppression, allowing the level of suppression for the contaminants to be determined.

Another option is to intentionally spike the sample with a known concentration of a specific contaminant. Not only can this help to confirm that a contaminant is present in a sample, by increasing the size of an existing peak rather than introducing a new one. But it can also help to determine the concentration of the contaminant, by comparing the size of the original peak with the peak produced by the spiked concentration.

The high sensitivity of UHPLC and triple quadrupole MS also permits the use of a very simple way for reducing the interference from the other compounds in complex samples. This is to dilute the sample with a

solvent such as water or acetonitrile, thereby reducing the concentrations of the interfering compounds. While this does nothing to make the mass spectra any simpler, as all the interfering compounds will still be present, it 'dilutes-away' the ion suppression caused by these compounds.

Obviously, this dilution also reduces the concentration of the contaminants, but usually not enough that they can't still be detected by the latest UHPLC-triple quadrupole MS systems, especially as ion suppression is less of an issue. In a recent application note on detecting 250 pesticides spiked into samples of black tea, Agilent showed that diluting the samples by a factor of 20 almost completely eliminated the ion suppression for 93% of the detectable pesticides, substantially enhancing their recovery rates.

What's next?

Despite all the challenges, the latest UHPLC and triple quadrupole MS systems are probably sensitive enough for most food and water testing applications, thanks to recent improvements in the ion optics, collision cell geometry and ion detector on triple quadrupole MS instruments. Increasing the sensitivity further may still offer advantages, such as reducing the need for sample preparation and allowing further dilution of the samples. But instrument developers such as Agilent are now focusing more on improving their systems in other ways, such as by making them smaller, more automated and easier to use.

With the increasing need for higher throughput with less available space in the lab, there is a large adoption of miniaturized instruments. Innovations have made it possible to obtain the same LC/MS performance in a smaller footprint. This allows labs to increase efficiency without utilizing costly lab space. In addition to their smaller form factor, these instruments often include smart technology to further enhance productivity. This smart technology can provide real-time feedback on system stability and maintenance needs, therefore minimizing downtime.

Additionally, developers of commercial UHPLC and triple quadrupole MS systems are focusing on making them more automated and easier to use. These two aims are clearly linked, because increased automation means the instrument can perform tasks on its own that used to require the involvement of an experienced operator, thus making it easier to use.

Some forms of automation have become increasingly common, such as the automatic transfer of samples and extraction solutions between different steps of the LC-MS process. Whereas sample preparation used to be conducted in a separate step involving several manual procedures, it is increasingly becoming entirely automated, with the treated sample passed directly to the LC-MS system. This means that an operator merely needs to inject the untreated sample at the start of the process and then collect the results at the end, with no further involvement required.

The optimization of parameters such as temperature and flow rate is also increasingly being automated, with the instrument's software suggesting the optimum settings for a specific sample and task. Even the interpretation of the results, which has always required an experienced scientist, is becoming automated, with machine learning tools now being used to determine likely identities for analytes from the spectral data. If the success of machine learning in areas such as face recognition is any guide, these tools are likely to be much faster and more accurate than experienced scientists.

It's not just the analysis that is becoming automated, but also the maintenance of the instruments. Rather than an operator needing to gauge whether an instrument needs cleaning, servicing or fixing from its performance, instruments now tend to tell operators when they need cleaning, servicing and fixing, and often exactly what needs to be done.

All these advances should help ensure that LC-MS remains the 'gold standard' for detecting contaminants in food and water samples for years to come.

Source: monticello/Shutterstock

FURTHER INFORMATION

Application notes for tripe quadrupole LC-MS on Agilent website (https://www.agilent.com/en/products/liquid-chromatography-mass-spectrometry-lc-ms/lc-ms-instruments/triple-quadrupole-lc-ms/6470a-triple-quadrupole-lc-ms)

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