

Smarter Sample Preparation for Environmental Challenges



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An interview on solid-phase extraction with Dr. Claire Desbrow, Global Product Manager for Analytical Consumables & Applications at Biotage.

Today, solid-phase extraction (SPE) is a powerful analytical technique widely used for the extraction of semi-volatiles from water samples. However, there was a time when SPE products had something of a dim quality reputation—as did the quality of the results they gave. Fortunately, SPE cartridge technology has come a long way, and the advantages these products bring are now hard to disregard.

LCGC sat down for a master class with Dr. Claire Desbrow, Global Product Manager for Analytical Consumables & Applications at Biotage, on the types of SPE products available, their appropriate applications, and how to deploy these products in robust, reliable extractions that yield solid results every time.

LCGC: Can you give us the history behind Biotage SPE cartridge products?

Desbrow: Biotage's SPE product range originated about 30 years ago out of the UK company International Sorbent Technology, based in the South Wales Valleys. The company was started with one sole aim: to bring high-quality SPE products to global analytical scientists. At that time, SPE did not have a great reputation as a technique because of the less-than-desirable quality of the products on the market. But the founders of the company understood SPE's potential and wanted others to recognize the benefits it could bring. About 10 years ago, Biotage bought the company and has maintained the quality control and focus ever since.

LCGC: What are the advantages of extracting water samples with SPE cartridges?

Desbrow: There are several key benefits associated with SPE cartridges. When compared to liquid-liquid extraction, for example, the mode of use and ease of handling of the cartridge bring great advantages.

There is a reduction in the amount of labor needed, as liquid-liquid extraction is a

very manual process and cannot be easily automated, whereas SPE is much easier to do as a manual or automated process.

The design of the product means that the amount of solvent used can be reduced. With some liquid-liquid methods, there might be 100 mL of solvent to use and dispose of afterward. With SPE, there might be 20 mL for the equivalent extraction, which means savings in terms of solvent use.

Also, SPE does not leave users dependent on a partition between an immiscible organic solvent and an aqueous sample, so there are more types of analytes that can be extracted.

LCGC: What types of media are available in SPE cartridges from Biotage?

Desbrow: There is quite a wide range of media—from the typical C18s, C8s, and DVBs, which are ideal for the extraction of semi-volatiles from water samples—to cleanup columns such as Florisils, Aluminas, and Silicas, which are used for removal of unwanted interferences from extracted samples.

Biotage also has application-specific columns, such as the extractable petroleum hydrocarbons (EPH) column for aliphatic/

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aromatic hydrocarbon fractionation, as well as a range of ion-exchangers and mixed-mode media that includes WAX phases for poly- and perfluoroalkyl substances (PFAS) extractions.

Essentially, Biotage has a wide range of products that have been packed into many different column sizes. Although we typically look at 6-mL columns for environmental extractions, there is usually a solution for almost every circumstance, depending on what solid type of sample is being extracted.

LCGC: How do SPE cartridges complement Atlantic disks for environmental water extraction?

Desbrow: The SPE cartridge is best used for clean water samples (e.g. drinking-water samples). The bed geometry—which, compared to disks, is narrower but taller—means that less solvent can be used, reducing solvent costs compared to SPE disks.

However, that narrow bed is less equipped to handle samples such as wastewater or groundwater where there is a lot of particulate matter; that is where the disks come in. To load samples at high flow rates, or if samples have high particulate levels, disks work exceedingly well. Therefore, the two different formats complement each other for environmental extractions.

LCGC: How do Biotage SPE products differ from other manufacturers?

Desbrow: One key differentiator is the focus on the product quality that the company was founded on.

For example, product cleanliness is an important factor in SPE because when dealing with high-sensitivity environmental extractions, the goal is to get to low detection limits. To avoid SPE devices contaminating the sample or extract, Biotage puts a great deal of effort into quality control (QC) testing.

Not only do we make our own media, but we also mold cartridges from plasticizer-free, high-purity polypropylene. There are no release agents used in our molding process, as they are often a source of hydrocarbon contamination.

The frits are also important. Frits are the small filters that hold the media inside the SPE cartridge. They are very porous, which means they have the potential to contaminate the extracts because of their high surface area and contact with extraction solvents. As a result, each batch is cleaned and QC tested to ensure that there are no impurities, phthalates, or other contaminants.

Another important distinction associated with Biotage SPE products is media batch-to-batch reproducibility. We have been making media for about 30 years, and because we do not buy them from a third party, we have a lot of control over how media are made and their specifications. QC laboratories do a great deal of testing between media batches to ensure reproducibility, cleanliness, and suitability for the various applications the media will be used for.

Another aspect is the particle-size distribution (PSD) of the media. The actual particles not only need to be of a certain size, but in a tight range within that size; that makes a huge difference to the success of the SPE procedure. So again, because we make our own, we can easily control that.

One final aspect that sets us apart is application-specific media, (e.g., the ISOLUTE EPH column). With that product, every batch is tested in-house using a 'real-life' QC test in which we actually fractionate between the aliphatic and aromatic components. As a result, customers do not need to check when they receive the product, which removes some risk. In short, what makes Biotage different is our quality and deep understanding of SPE products.

LCGC: How can Biotage SPE cartridges be processed?

Desbrow: We use industry-standard dimensions for our cartridges, specifically the Luer tip outlet being the industry-standard size, which means they are compatible with all cartridge-processing devices. So from the manual systems using vacuum to process the samples—lower throughput methods—all the way up to fully automated systems for unattended sample processing.

LCGC: What SPE cartridges do you recommend for the US Environmental Protection Agency (EPA) methods 525.2 and 549.2?

Desbrow: Method 525.2 is for a wide suite of semi-volatiles in drinking water with a gas chromatography–mass spectrometry (GC–MS) endpoint, so we are looking for compounds with different characteristics and quite a sensitive endpoint and we need clean extracts to meet the detection limits. Biotage always recommends the ISOLUTE® C18 1-g, 6-mL cartridges for that application because they meet EPA cleanliness requirements, which means they are phthalate- and adipate-free. The cartridges' PSD allows them to meet the flow rate requirements to give higher recoveries across that wide suite of analytes.

EPA method 549.2 is a slightly different type of method, as it is for two polar herbicides that are highly charged and very water-soluble. The method uses an ion-pair type of approach, which means that a C8 SPE media is used. It is recommended to use the ISOLUTE® C8 500-mg, 6-mL cartridges for this method. They work well with this approach, and the quality of the sorbent results in high recoveries consistently from batch to batch within the required 4.5-mL elution volume. We recently launched an application note describing the automated use of these cartridges for that method using our Biotage® Horizon 5000 system.

LCGC: What about PFAS compounds, which are such a hot topic at the moment?

Desbrow: They really are. It is a fast-changing landscape, and these compounds are of immense concern in the environment.

Biotage has a range of products for these compounds, and there are two key approaches to their extraction. One is the nonpolar approach using DVB-type cartridges, which is the original method. And the newer methods are based on polymeric WAX-type cartridges—a combination of nonpolar and weak anion-exchange media—and they are used for the EPA 533 method. We also need to make sure that the processing platforms, be they manual or automated, do not contribute any background. And for these types of compounds, anything that is fluorinated (any PTFEs) can contaminate the extracts. Hence, it is crucial that our processing kit and products are fluorinated-compound-free for this type of analysis.