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Improving the performance of SPME using trap-based preconcentration with enrichment

This study shows that enhancing conventional SPME by using trap-based preconcentration with enrichment of analytes from a single sample vial greatly improves the number of compounds identified, thus expanding the applicability of SPME in GC–MS analysis.

Solid-phase microextraction (SPME) is a solventless sampling technique that is easily automated and avoids the inconveniences of solvent-based methods such as liquid/liquid extraction. However, using a standard GC injector for the fiber desorption requires careful optimisation, using a dedicated liner (<1 mm i.d) in conjunction with a high gas flow, in order to obtain optimum chromatography. In MS systems where carrier gas flow is limited, an injector split flow is therefore often used, but this reduces the quantity of analytes reaching the detector, and therefore the sensitivity of the technique.

In this study we demonstrate how the performance of SPME can be enhanced by SPME–trap, whereby conventional SPME is powered by the unique **trap-based focusing** on the Centri sample extraction and enrichment platform. This focusing trap operates cryogen-free, can contain a variety of sorbents, and using temperatures ranging from ambient to –30°C can efficiently trap VOCs and SVOCs released from a sample. The trap is then ballistically heated (up to 100°C/s) to transfer the analytes to the GC in a small volume of carrier gas (~100 µL), resulting in a tight band of analytes at the head of the column and sharp chromatographic peaks, even using low-split or splitless modes.

An added feature of SPME–trap that is only available with Centri is **enrichment**: the use of a single SPME fiber to repeatedly extract analytes from a single sample vial, and then desorb them onto the same focusing trap. This, combined with a low-split or splitless injection, can increase both sensitivity and number of compounds detected. These can be crucial factors when dealing with 'unknowns', or when profiling samples containing important low-level components.

To demonstrate the advantages of SPME–trap with enrichment, a spiked tea sample was analysed using three sampling modes (Figure 1). Over twice as many compounds were detected by SPME–trap with enrichment, compared to

Background to Centri®

Markes International's Centri system for GC–MS is the first platform to offer high-sensitivity unattended extraction and enrichment of VOCs and SVOCs in solid, liquid and gaseous samples.

Centri allows full automation of immersive and headspace extraction using HiSorb™, high-capacity sorptive extraction probes. It also offers full automation of headspace, SPME and tube-based thermal desorption with enrichment. Leading robotics and analyte-trapping technologies are used to improve sample throughput and maximise sensitivity for a range of applications – including profiling of foods, beverages and fragranced products, environmental monitoring, clinical investigations and forensic analysis.

In addition, Centri allows samples from any injection mode to be split and re-collected onto clean sorbent tubes, avoiding the need to repeat lengthy sample extraction procedures and improving security for valuable samples, amongst many other benefits.

For more on Centri, visit www.markes.com.

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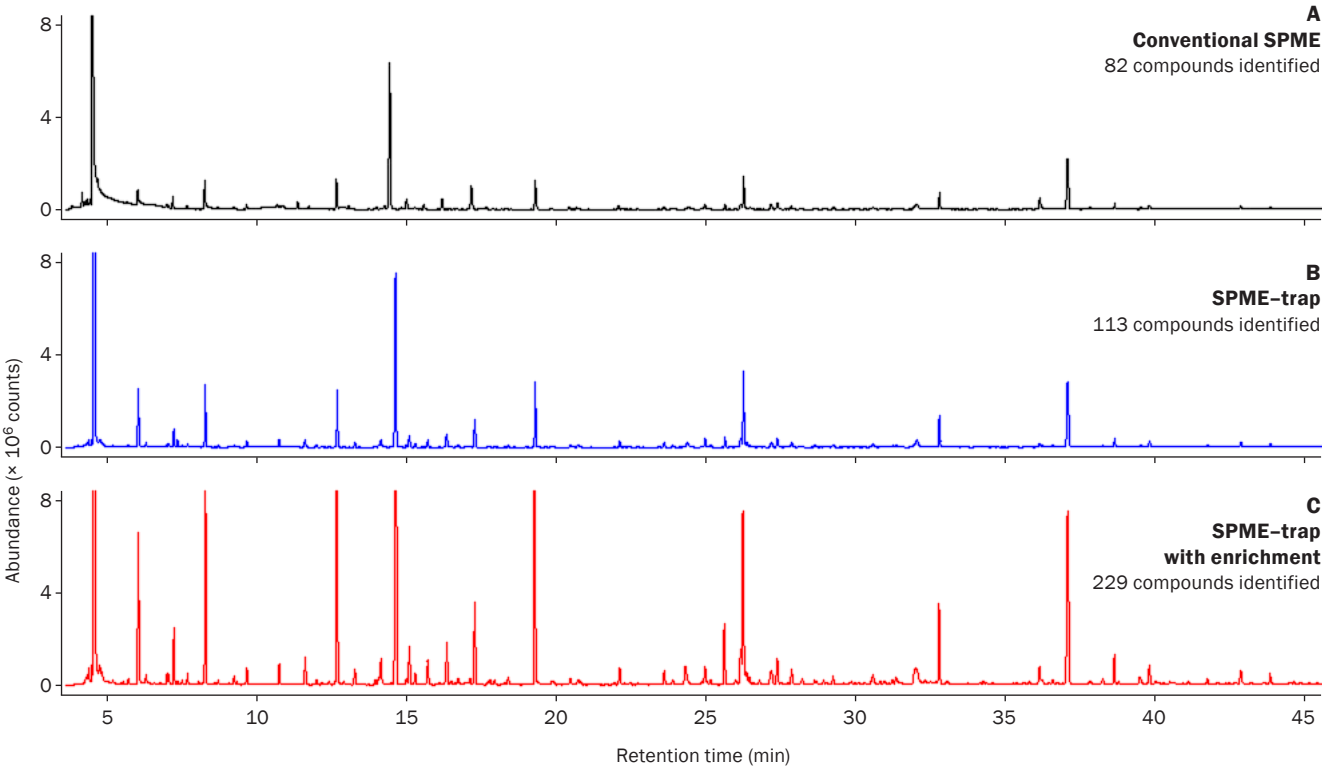

Figure 1: Analysis of the same spiked tea sample using (**A**) conventional SPME; (**B**) SPME–trap and (**C**) SPME–trap with enrichment.

conventional SPME. This could be critical for applications such as food and beverage profiling, where the detection of minor components is essential for accurate sample characterisation.

In conclusion, we have shown improved detection of trace components in a sample using SPME combined with trap-based focusing with enrichment, taking multiple extractions from a vial for analysis with a single GC run. A further advantage associated with trapping is the ability to re-collect split flows onto a thermal desorption tube, for repeat analysis or method validation. On Centri, all of these operations are automated, greatly improving efficiency for high-throughput laboratories.

Applications were performed under the stated analytical conditions. Operation under different conditions, or with incompatible sample matrices, may impact the performance shown.

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