



# Ingenious News



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# PAL SPME Arrow - New Dimension for SPME Analysis

Since its development by Belardi and Pawliszyn in 1989, Solid-Phase Microextraction (SPME) has become one of the most popular extraction technologies for environmental, food and health analyses. However, the technique remained almost unchanged with some significant drawbacks, such as the limited mechanical stability and small phase volumes of the fibers. All attempts to overcome these limitations have until now been a trade-off between versatility and labor-intensive handling.

With the PAL SPME Arrow we present a new technology for micro-extraction, offering trace level sensitivity combined with high mechanical robustness. The PAL SPME Arrow features an outer diameter of max. 1.5mm, which allows the enclosure of a large sorption phase volumes with a highly resistant and stabilizing inner rod. The arrow-shaped tip allows smooth penetration of vial septa and injector. In contrast to traditional SPME fibers, the Arrow design fully protects the sorptive material, minimizing adverse influences and loss of analytes during transfer processes. With the PAL RTC and RSI the Arrow SPME microextraction is fully automated.

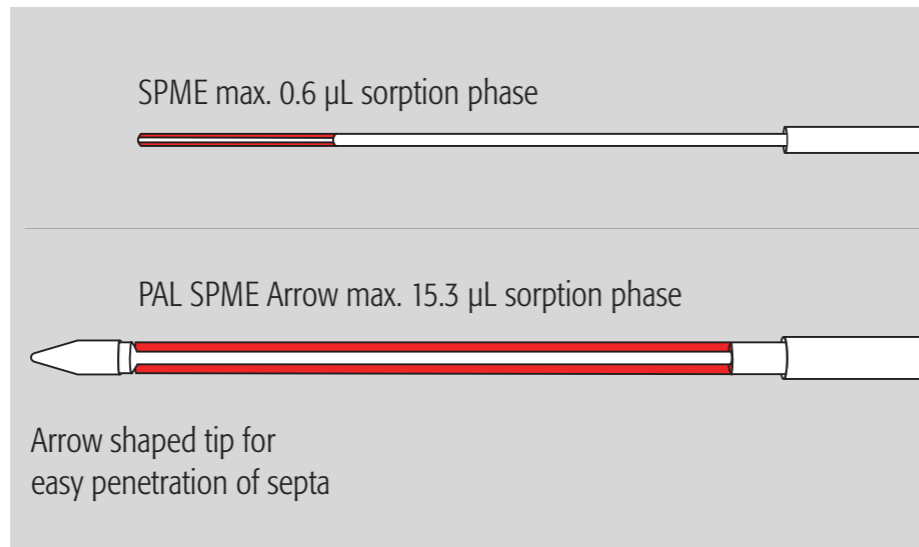


Figure 1: PAL SPME Arrow compared to a conventional SPME fiber: Size and position of the sorptive phases are shown in red.

## The PAL SPME Arrow outperforms conventional SPME Fibers

### SPME immersion extraction, detection limits

Limits of detection (DIN) (ng L<sup>-1</sup>)

	Naphthalene	Acenaphthylene	Acenaphthen	Anthracene	Pyrene	Benzo(ghi)perylene
Fiber 100/10	3.2	2.6	2.2	3.8	3.2	4.6
Arrow 250/15	0.03	0.04	0.05	0.02	0.02	0.5

Table 1: Limits of detection for six typical polyaromatic hydrocarbons (PAHs) in water, measured by immersion extraction with PAL SPME Arrow or a conventional SPME fiber. PAL SPME Arrow achieves a roughly 40 - 100 x better sensitivity compared to a conventional SPME fiber.

\*Conditions:  
PAL SPME Arrow, 250 µm PDMS, 15 mm length, 7.7 µL sorption phase  
Commercially available SPME fiber, 100 µm PDMS, 10 mm length, 0.6 µL sorption phase  
Determined according to German DIN 32645

## Aroma analysis of white wines

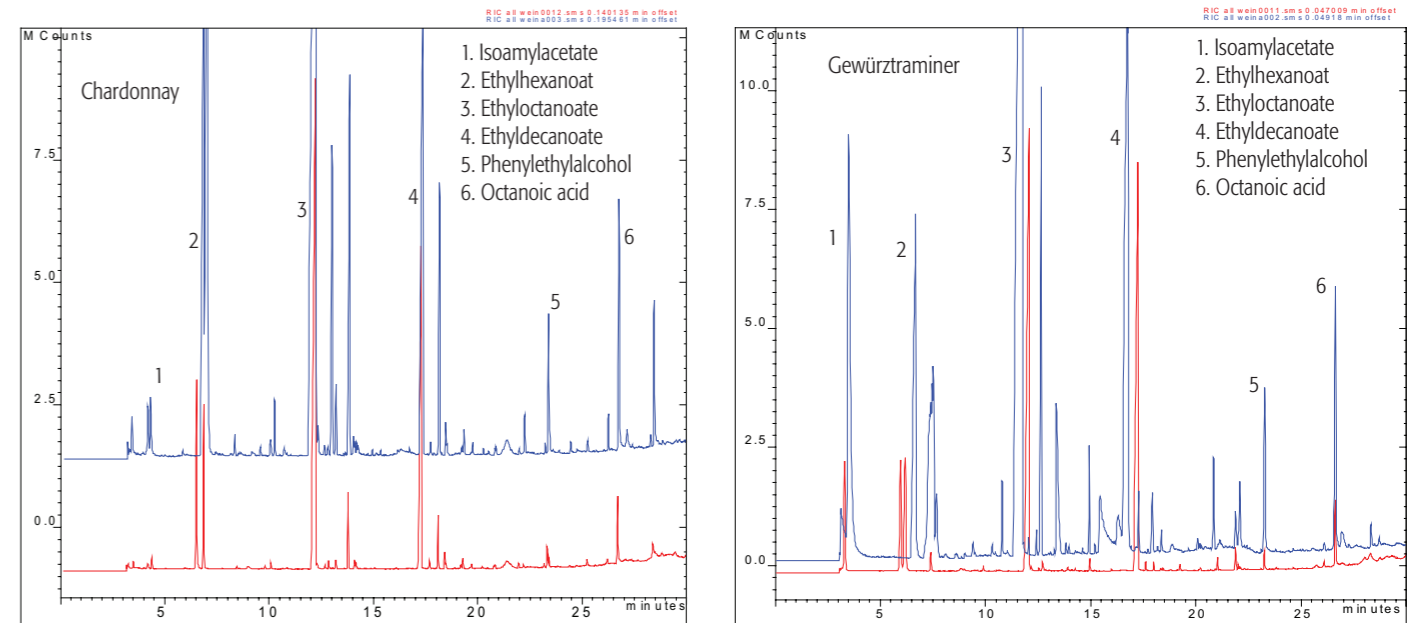


Figure 2: Analysis of Chardonnay aromas: PAL SPME Arrow (blue 100 µm PDMS 20 mm 0.80 mm OD) compared to a conventional SPME fiber (red 100 µm PDMS 10 mm 0.30 mm OD)

Figure 3: Analysis of Gewürztraminer aromas: PAL SPME Arrow (blue 100 µm PDMS 20 mm 0.80 mm OD) compared to a conventional SPME fiber (red 100 µm PDMS 10 mm 0.30 mm OD)

## Analysis of VOCs, EPA 502.2

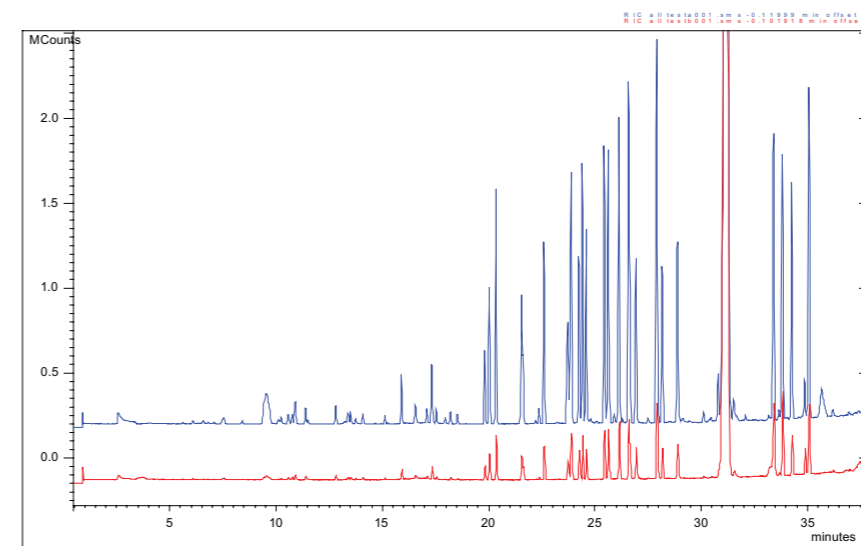


Figure 4: Analysis of VOCs in water, analogue to EPA 502: PAL SPME Arrow (blue 100 µm PDMS 20 mm 0.80 mm OD) compared to a conventional SPME fiber (red 100 µm PDMS 10 mm 0.30 mm OD)

## PAL SPME Arrow sorption materials

currently PDMS, Carbon WR (further materials under development)

## The PAL SPME Arrow offers

- Superior SPME sensitivity: typically a 5-fold increase compared to conventional SPME
- Excellent mechanical stability through patented design
- Full protection of sorption phase material
- Highest process safety due to a fully automated SPME process with PAL Systems

# PAL HeatEx Stirrer - New Mixing and Heating Technology for Sample Preparation and SPME

The efficient mixing of reagents is required for many operations in the laboratory, like sample homogenization, the dissolving of solids or liquid/liquid extraction. For the efficient headspace analysis of liquid samples a rapid exchange between the liquid and the gas phase (headspace) is required. Often magnetic stirrers are applied to achieve mixing. However, especially at higher speeds stir bars tend to loose contact to the magnet and stop turning. This problem is aggravated when solids are added to the sample liquid which is common practice, e.g. the addition of salts to the liquid to shift the equilibrium. Furthermore stir bars make auto-sampling cumbersome since a bar has to be added to every sample vial manually. Vortex mixers offer effective stirring, but cannot be used for SPME sampling.

The powerful PAL HeatEx Stirrer mixes samples rapidly applying cycloid shaped mixing pattern without the need for stir bars. For SPME headspace and immersion sampling the special design (pat. pending) ensures that the delicate fiber is not damaged (see fig. 3).

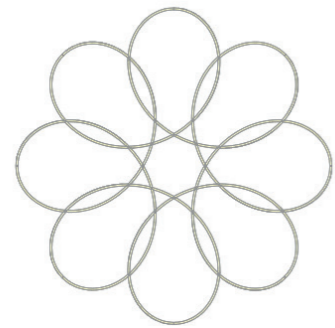


Figure 2: Flower power for stirring: cycloidal mixing patterns.

## Specifications of the PAL HeatEx Stirrer Module

- Temperature Range 40 - 150 °C
- Stirring Speed 0 - 160 rpm (0-1370 cycloidal loops)
- Dimensions (L x W x H) 190 mm x 85 mm x 160 mm

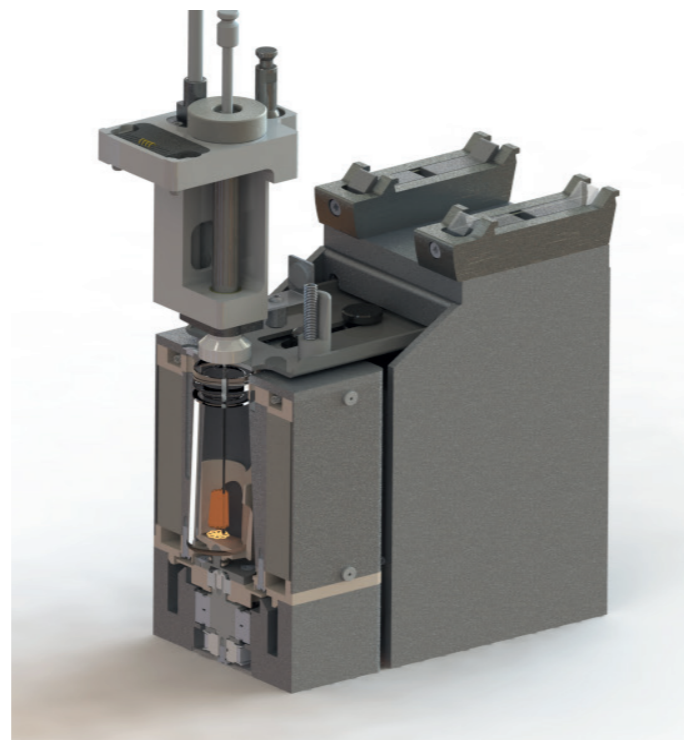


Figure 1: Cutaway view of the HeatEx Stirrer Module with SPME tool.

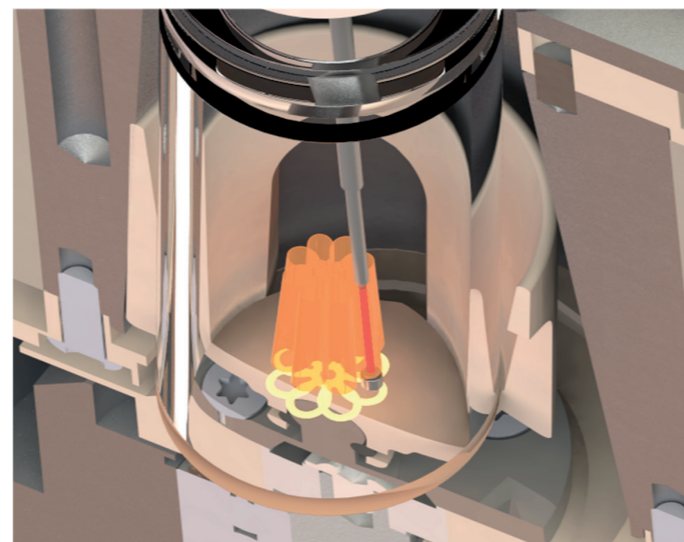


Figure 3: Motion of a liquid (orange) when applying a cycloidal mixing pattern (yellow). The entire volume of the liquid is mixed efficiently. The integrity of the SPME fiber (red) is not compromised.

## The PAL HeatEx Stirrer: Performance and unsurpassed handling

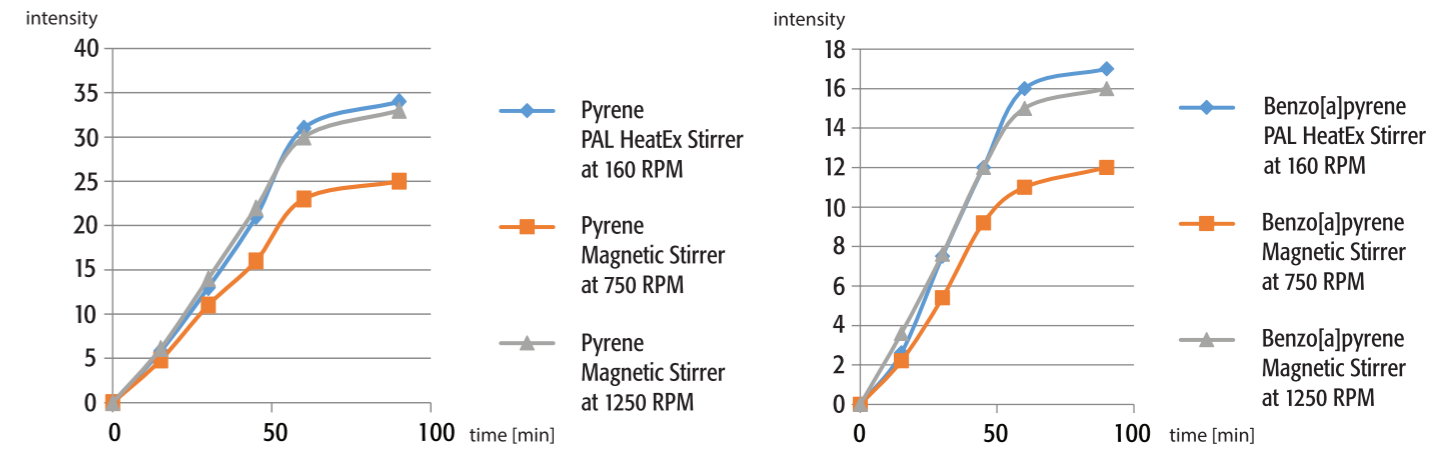


Diagram 1: Immersion SPME saturation curves of pyrene in water. The HeatEx Stirrer at 160 rpm (blue curve) is as efficient as the magnetic stirrer at 1250 rpm.

Diagram 2: Immersion SPME saturation curves of benzo[a]pyrene in water. The HeatEx Stirrer at 160 rpm (blue curve) is as efficient as the magnetic stirrer at 1250 rpm.



Figure 4: The PAL HeatEx Stirrer (left) gives superior results for liquid/liquid extractions, i.e. smaller droplets and hence more intense exchange between organic and aqueous layer when compared to liquid/liquid extraction with a magnetic stirrer at 1000 rpm (right).

With better performance at lower stirring speeds compared to conventional magnetic stirrers, the PAL HeatEx Stirrer offers full integration into the PAL3 System. There is no need for magnetic stir bars or heating bathes making automated stirring of samples easy and convenient.

## The PAL HeatEx Stirrer offers:

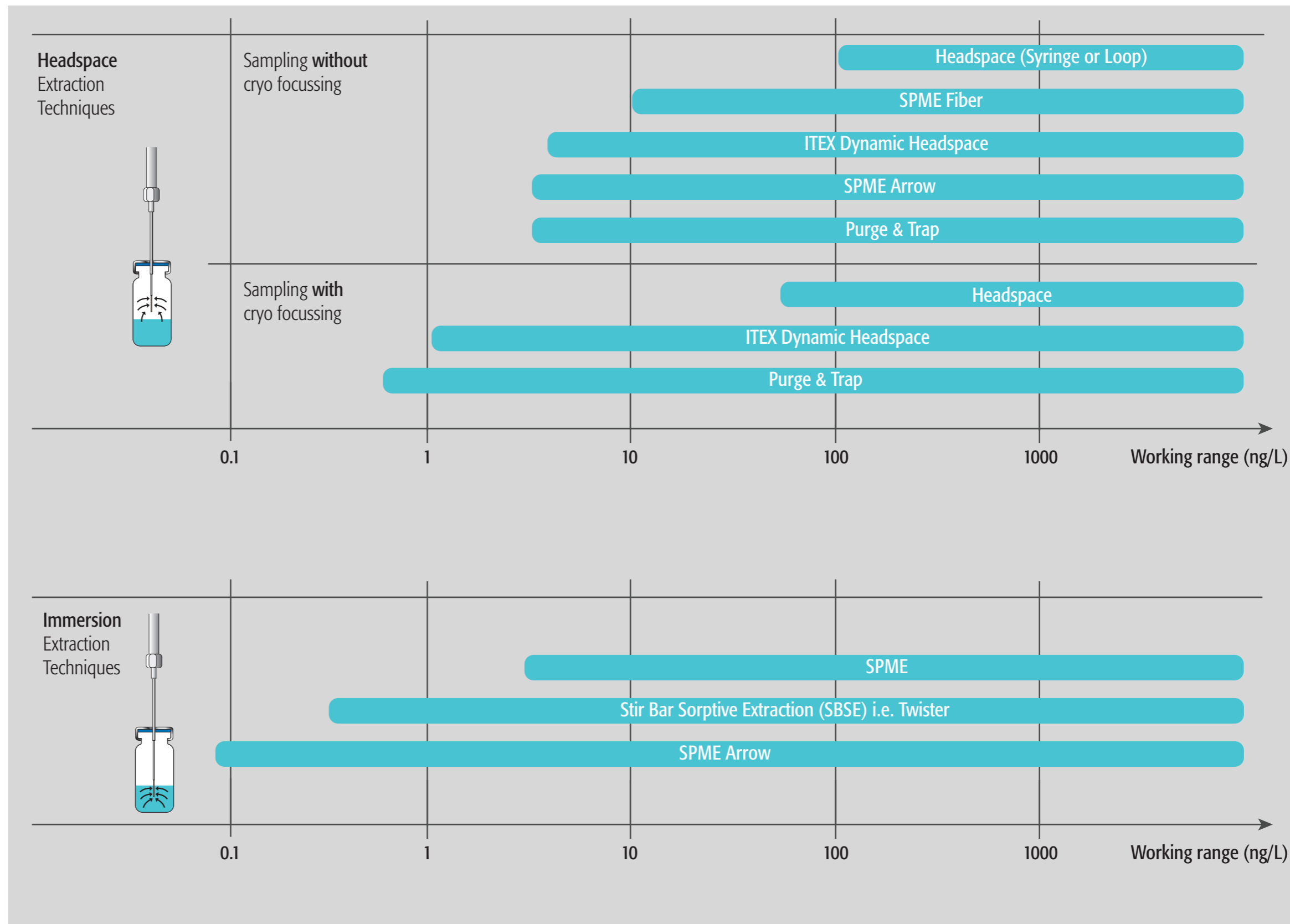
- Rapid equilibration through effective stirring for head-space and immersion SPME sampling while ensuring the integrity of the fiber
- Efficient dissolution of solids, temperature controlled
- Liquid/liquid extraction
- Stirring/heating for derivatization reactions
- No stir bar required, constant stirring also with samples containing solids
- Precise control of the equilibration temperature 40-150 °C
- Software controlled, temperature and stirring speed are logged
- Compact size

# How do different GC extraction techniques compare?

The two diagrams on the right outline the working ranges of the different techniques for GC headspace (Fig.1) or immersion extractions (Fig 2).

For extractions and sample preparation PAL System offers the necessary tools to achieve the required sensitivities:

- [PAL Headspace Tool](#)
- [PAL SPME Fiber](#)
- [PAL SPME Arrow](#)
- [PAL ITEX Dynamic Headspace Tool](#)



For more information on PAL System Tools visit:

<http://www.palsystem.com/index.php?id=284>



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