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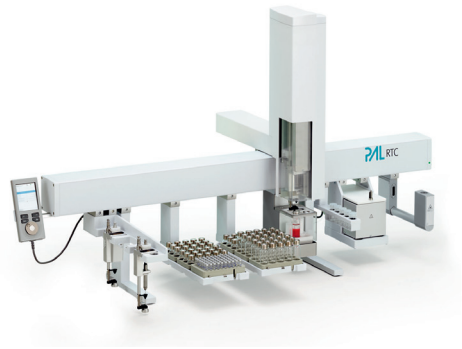

Comparison of PAL SPME Fibers with established Fibers

Abstract

The performance of SPME fibers is an essential aspect of an analytical method. This study shows the comparison of three different SPME fibers of a conventional brand (referred to in the following as 'Brand X') and the new PAL SPME Fibers. The tests involve the EPA Method 8310 for PAH standards using three dedicated Brand X fibers, and the PAL SPME Fibers PDMS 7 µm, PDMS 30 µm, and Acrylate 85 µm. Furthermore the EPA Method 502.2 was tested for a mixture of 64 VOCs (Mega Mix 502.2) using two dedicated Brand X fibers, and the PAL SPME Fibers PDMS 100 µm and Carbon WR 95 µm. The new PAL fibers (PDMS fibers 7 µm, 30 µm, and 100 µm and the Acrylate fibers) yield identical results when compared with the corresponding Brand X fibers. For medium and high boiling compounds the PAL Carbon WR Fibers show a significantly better performance than the respective Brand X fibers.

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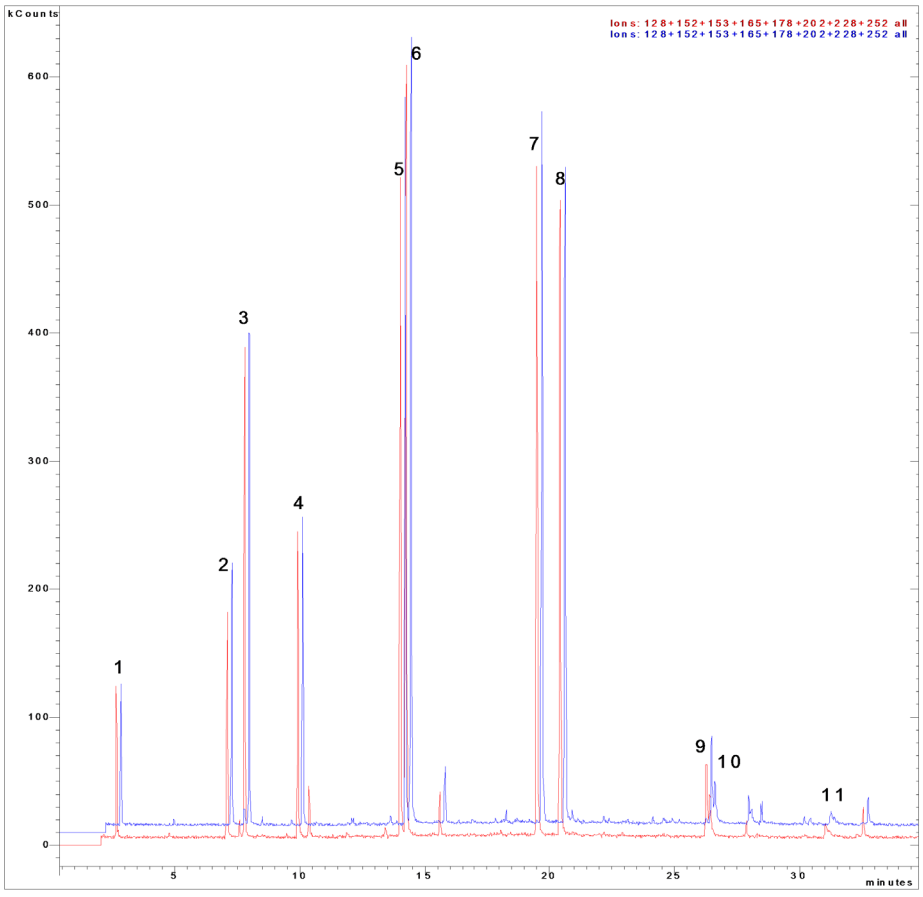
Performance of different SPME Fibers for PAH-Samples (EPA Method 8310)

7 µm PDMS Fiber

Test Procedure	
Sample:	10 mL Water + PAH Standard (20 ng/Compound) (EPA Method 8310, PAH Mixture)
Extraction:	30 min Immersion at 40 °C, Agitating at 250 rpm
Thermal Desorption:	1 min. at 250 °C in SPI Liner Directly Connected to the Column
Column:	Inertcap 5MS/Sil (GL-Science) 15 m x 0.25 mm ID, Film Thickness 0.25 µm
Temp. Progr. GC Oven:	90 °C hold 1 min, 5 °C/min up to 320 °C
Autosampler:	Combi PAL
GC MS:	Varian 3400 with Varian Saturn Ion Trap

Chromatographic Comparison

Measurements with the PAL 7 µm PDMS fiber were compared to the corresponding Brand X fiber. For each type of fiber a series of 12 analytical runs were performed. Figure 1 shows the overlay of a chromatogram from the PAL 7 µm PDMS Fiber (blue), and a corresponding Brand X fiber (red).



1 Naphthalene
2 Acenaphthylene
3 Acenaphthene
4 Fluorene
5 Phenanthrene
6 Anthracene
7 Fluoranthene
8 Pyrene
9 Benzo(a)anthracene
10 Chrysene
11 Benzo(a)pyrene

Figure 1: Chromatographic Comparison of PAL Fiber PDMS 7 µm (blue) and Brand X Fiber (red).

Quantitative Comparison

The diagram shows the signal areas of three analytical runs for five selected target molecules with the same fiber compared to the corresponding Brand X fiber. No significant difference between the Brand X and the PAL PDMS 7 µm fibers is observable. The two brands of fibers show identical results.

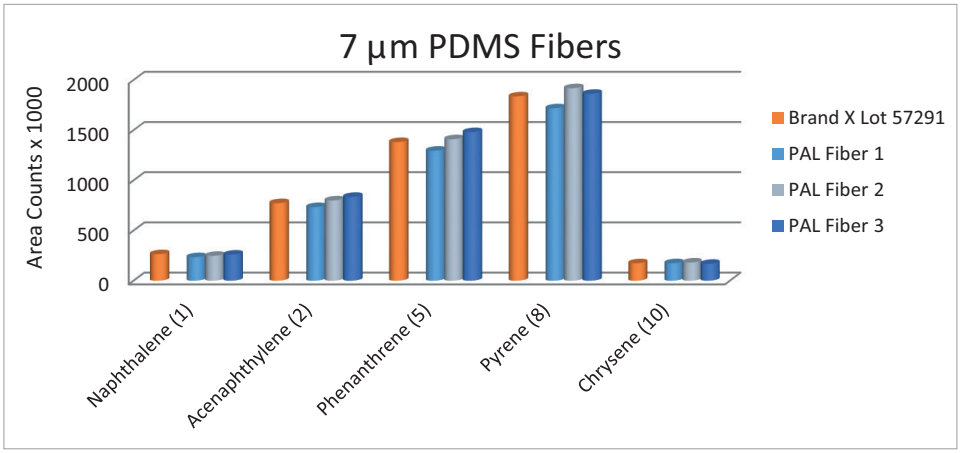


Figure 2: Quantitative Comparison of PAL 7 µm PDMS Fiber and Brand X Fiber for Five selected Target Molecules.

30 µm PDMS Fiber

Test Procedure	
Sample:	10 mL Water + PAH Standard (20 ng/Compound) (EPA Method 8310, PAH Mixture)
Extraction:	30 min Immersion at 40 °C, Agitating at 250 rpm
Thermal Desorption:	1 min. at 250 °C in SPI Liner Directly Connected to the Column
Column:	Inertcap 5MS/Sil (GL-Science) 15 m x 0.25 mm ID, Film Thickness 0.25 µm
Temp. Progr. GC Oven:	90 °C hold 1 min, 5 °C/min up to 320 °C
Autosampler:	Combi PAL
GC MS:	Varian 3400 with Varian Saturn Ion Trap

Quantitative Comparison

The diagram shows the signal areas of three analytical runs for five selected target molecules with the same fiber compared to the corresponding Brand X fiber. No significant difference between the Brand X and the PAL PDMS 30 µm fibers is observable. The two brands of fibers show identical results.

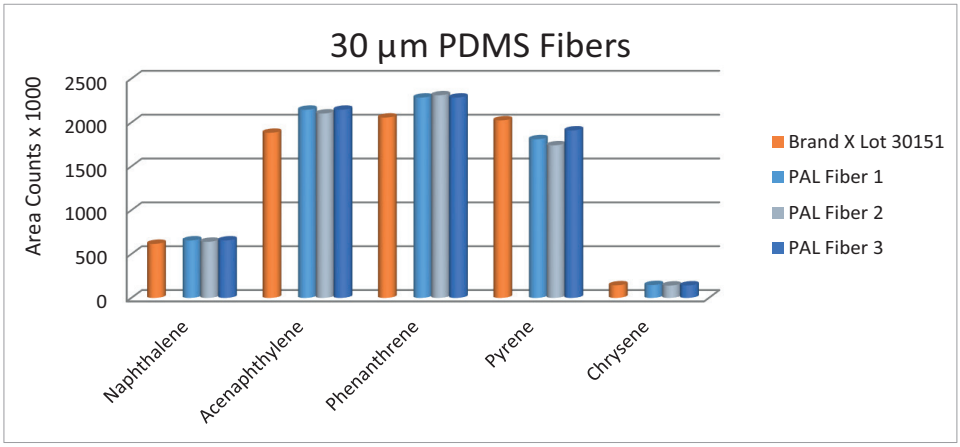


Figure 3: Quantitative Comparison of PAL 30 µm PDMS Fiber and Brand X Fiber for Five selected Target Molecules.

85 µm Acrylate Fiber

Test Procedure	
Sample:	10 mL Water + PAH Standard (20 ng/Compound) (EPA Method 8310, PAH Mixture)
Extraction:	30 min Immersion at 40 °C, Agitating at 250 rpm
Thermal Desorption:	1 min. at 250 °C in SPI Liner Directly Connected to the Column
Column:	Inertcap 5MS/Sil (GL-Science) 15 m x 0.25 mm ID, Film Thickness 0.25 µm
Temp. Progr. GC Oven:	90 °C hold 1 min, 5 °C/min up to 320 °C
Autosampler:	Combi PAL
GC MS:	Varian 3400 with Varian Saturn Ion Trap

Quantitative Comparison

The diagram shows the signal areas of three analytical runs for five selected target molecules with the same fiber compared to the corresponding Brand X fiber. No significant difference between the Brand X and the PAL PDMS 85 µm fibers is observable. The two brands of fibers show identical results.

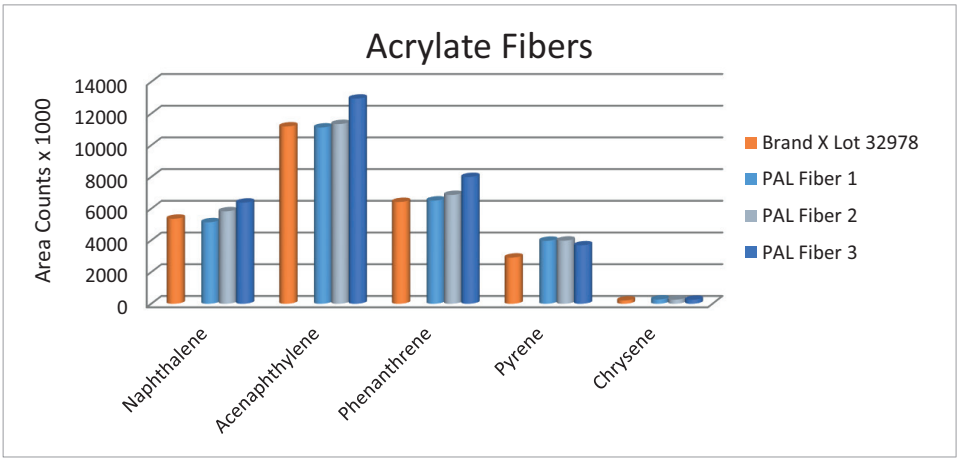


Figure 4: Quantitative Comparison of PAL 85 µm Acrylate Fiber and Brand X Fiber for Five selected Target Molecules.

Performance of different SPME Fibers for VOC-Samples (EPA Method 502.2)

100 µm PDMS Fiber

Test Procedure	
Sample:	10 mL Water + 3 g NaCl + 502.2 MegaMix (20 ng/Compound) (EPA Method 502.2 MegaMix Mixture Restek PNo 30431)
Extraction:	15 min Equilibration Time in Headspace at 50 °C, Agitating at 250 rpm
Thermal Desorption:	1 min at 250 °C in SPI Liner directly connected to the Column
Column:	BGB-624; 30 m x 0.32 mm, Film Thickness 1.8 µm
Temp. Progr. GC Oven:	50 °C hold 1 min; Afterwards 5 °C/min up to 220 °C
Autosampler:	Combi PAL
GC MS:	Varian 3400 with Varian Saturn Ion Trap

Quantitative Comparison

The diagram shows the signal areas of three analytical runs for six selected target molecules using PDMS 100 µm fibers. No significant difference between the Brand X and the PAL fibers is observable.

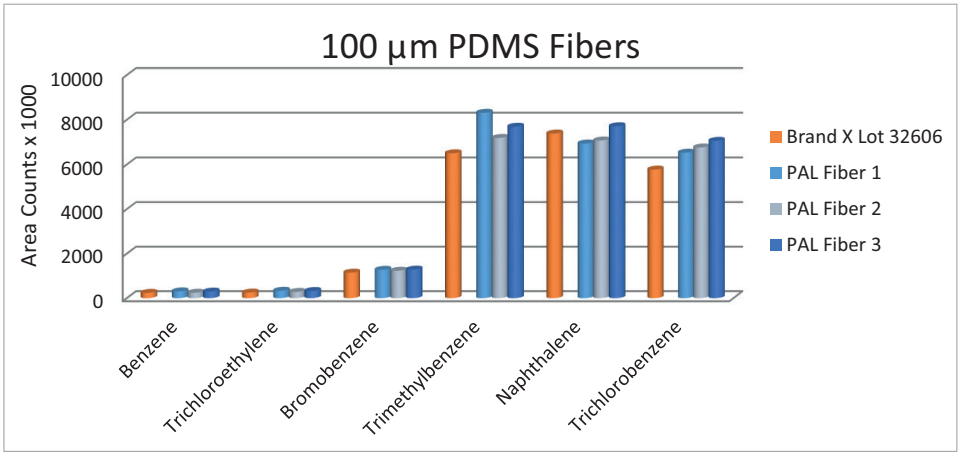


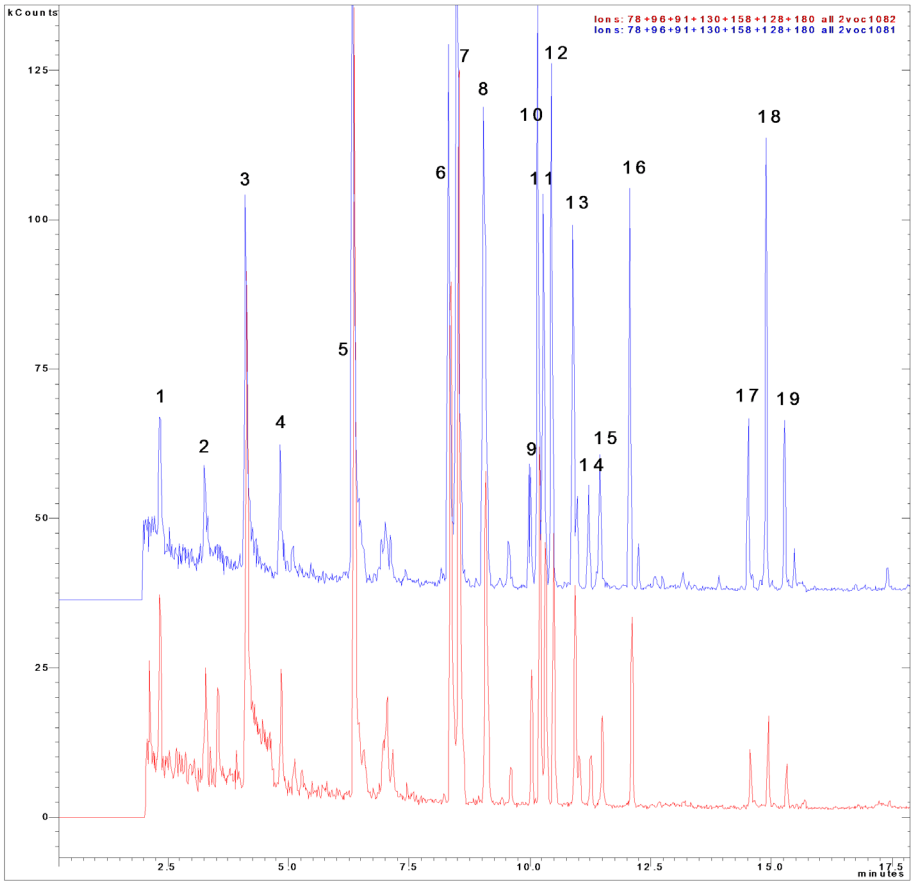
Figure 5: Quantitative Comparison of PAL 100 µm PDMS Fiber and Brand X Fiber for Six selected Target Molecules.

95 µm Carbon WR

Test Procedure	
Sample:	10 mL Water + 3g NaCl + 502.2 MegaMix (20 ng/Compound) (EPA Method 502.2 MegaMix Mixture Restek PNo 30431)
Extraction:	15 min Equilibration Time in Headspace at 50 °C, Agitating at 250 rpm
Thermal Desorption:	1 min 250 °C in SPI Liner directly connected to the Column
Column:	BGB-624; 30 m x 0.32 mm ID, Film Thickness 1.8 µm
Temp. Progr. GC Oven:	50 °C hold 1 min; Afterwards 5 °C/min up to 220 °C
Autosampler:	Combi PAL
GC MS:	Varian 3400 with Varian Saturn Ion Trap

Chromatographic Comparison

Measurements with the PAL 95 µm Carbon WR fiber were compared to the corresponding Brand X fiber. For each type of fiber a series of 12 analytical runs was performed. Figure 1 shows the overlay of a chromatogram from a PAL 95 µm Carbon WR Fiber (blue), and a Brand X Carboxen® fiber (red).



1 1,1-Dichloroethene
2 cis-1,2-Dichloroethene
3 Benzene
4 Trichloroethylene
5 Toluene
6 Ethylbenzene
7 m-,p- Xylene
8 o-Xylene
9 Bromobenzene
10 2-Chlorotoluene
11 1,3,5-Trimethylbenzene
12 4 Chlorotoluene
13 tert-Butylbenzene
14 1,2,4-Trimethylbenzene
15 sec-Butylbenzene
16 n-Butylbenzene
17 1,2,4-Trichlorobenzene
18 Naphthalene
19 1,2,3-Trichlorobenzene

Figure 6: Chromatographic Comparison of PAL 95 µm Carbon WR Fiber (blue) and Brand X Carboxen® fiber (red).

Quantitative Comparison

The diagram shows the signal areas of three analytical runs for six selected target molecules with the same fiber compared to the Brand X fiber. The Brand X Carboxen® and the PAL Carbon WR fibers show similar results for low boiling compounds. However, signal areas of higher boiling-point compounds such as Naphthalene show significantly higher performance with the PAL Carbon WR fibers (Figure 7).

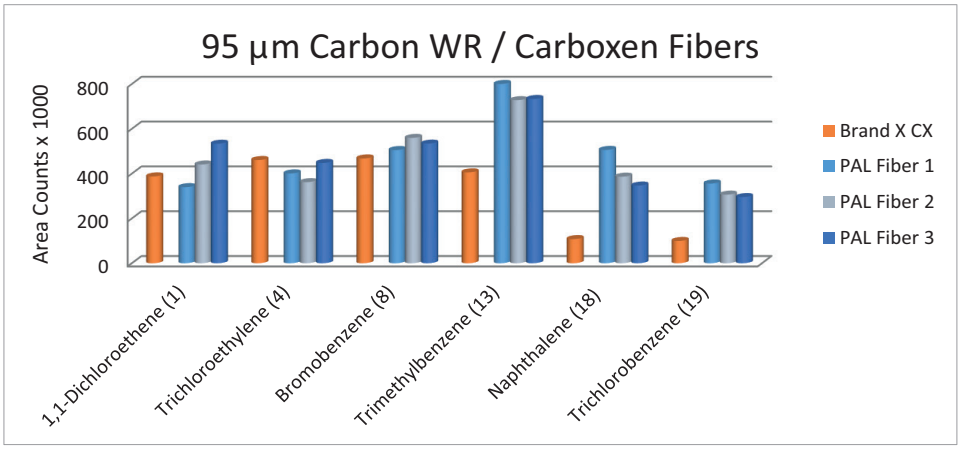


Figure 7: Quantitative Comparison of PAL 95 µm Carbon WR Fiber and Brand X Fiber for Six selected Target Molecules.

Fiber to Fiber Reproducibility

30 µm PDMS Fiber

Test Procedure	
Sample:	7.5 mL Water + Performance Mix (Nitrobenzene,2-Nitrotoluene 10 µg, each in 100 µL Methanol) in 10 mL Vial
Extraction:	10 min Immersion at 25 °C
Thermal Desorption:	1 min 280 °C in SPME Liner 0.8 mm ID
Carrier Gas:	Hydrogen 70 kPa
Column:	ZB-5 15 m x 0.25 mm ID, Film Thickness 0.25 µm
Temp. Progr. GC Oven:	40 °C hold 1 min; Afterwards 5 °C/min up to 80 °C
Autosampler:	Combi PAL
GC-FID:	Thermo Trace 2000, with FID at 250 °C
GC MS:	Varian 3400 with Varian Saturn Ion Trap

Performance Test

In order to ensure consistent quality of the fibers, tests according to the performance test of a well-established brand were performed. The absolute values obtained are of minor importance for the analyst, as quantification of SPME analysis always requires an internal standard method. Nevertheless, those values are interesting for fiber production, as any change in the performance e.g. variations of the film thickness or changes of the physical properties of the phase can be detected. The diagram shows the peak areas for two selected molecules obtained from eight different fibers (30 µm PDMS) that had been subjected to the SPME performance test.

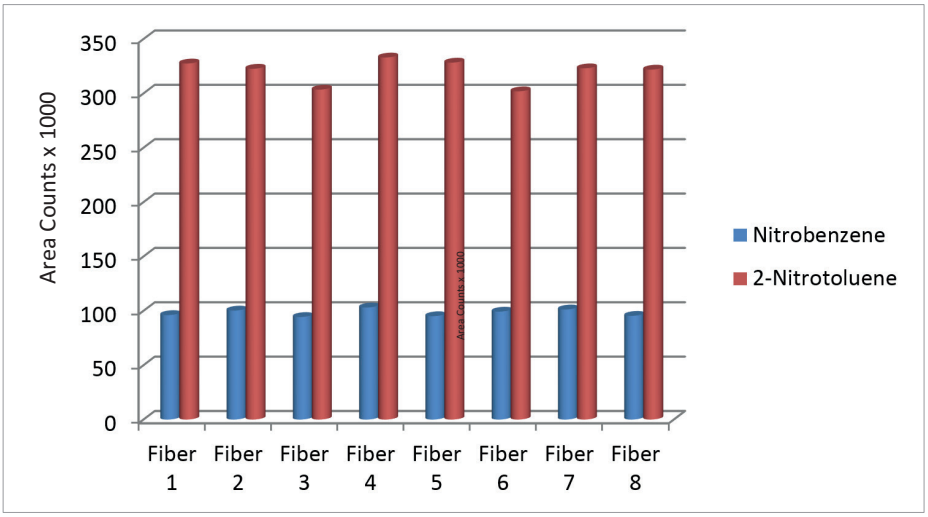


Figure 8: Fiber to Fiber Reproducibility Test.

Parameters of PAL SPME Fibers

Coating	Film Thickness (µm)	Operating Temperature (°C)	Maximum Temperatur (°C)	Hub Color Code	P/N
PDMS	7	200-340	340	Green	FIB-P-7/10
PDMS	30	200-280	280	Gold	FIB-P-30/10
PDMS	100	200-280	280	Red	FIB-P-100/10
Polyacrylate	85	200-280	300	Grey	FIB-A-85/10
Carbon WR	95	220-300	300	blue	FIB-CWR-95/10

Compatibility

PAL SPME Fibers are compatible with all holders currently in use for automated applications.

Conclusion

The new PAL fibers (PDMS fibers 7 µm, 30 µm, and 100 µm and the Acrylate fibers) yield identical results when compared with the corresponding Brand X fibers.
For medium and high boiling compounds the PAL Carbon WR Fibers show a significantly better performance than the respective Brand X fibers.
The reproducibility of the fiber production is very high. Nevertheless the use of an internal standard for quantification is recommended, as it is common practice for quantitative SPME.

Data Sheet

A detailed data sheet is provided with informations on (pre-) conditioning, operating temperatures and cleaning in order to gain full performance and an extended lifespan of PAL SPME Fibers.