PAL SYSTEM
Ingenious sample handling

PAL SPME Fibers
Optimized for Automation

PAL SPME Fiber

Since its introduction by Pawliszyn et al. (ref. 1) Solid Phase Micro Extraction (SPME) has seen a tremendous development. SPME is a very effective way of automated sample preparation. It is used for extracting organics from a matrix (solid, liquid or gaseous) into a stationary phase immobilized on a fiber. The analytes are thermally desorbed directly in the injector of a gas chromatograph. Originally mostly used for extracting solvents with excellent sensitivities from aqueous matrices the range of applications today spans from chemical and environmental to medical applications.

PAL SPME Fibers have been developed and optimized for the most successful SPME sampler, the PAL System Autosampler. The fibers are offered with different coatings and film thicknesses. Their excellent extraction properties have been proven for many important applications.

Reference [1]
Detection of substituted benzenes in water at the pg/ml level using solid-phase microextraction and gas chromatography-ion trap mass spectrometry.
Potter DW, Pawliszyn J., J Chromatogr. 1992 Nov 20;625(2):247-55.

PAL SPME Fiber Order Information

The PAL SPME Fibers are available in order quantities of one, three or five fibers per box. For method development, a set of each fiber type (set of five) is available.

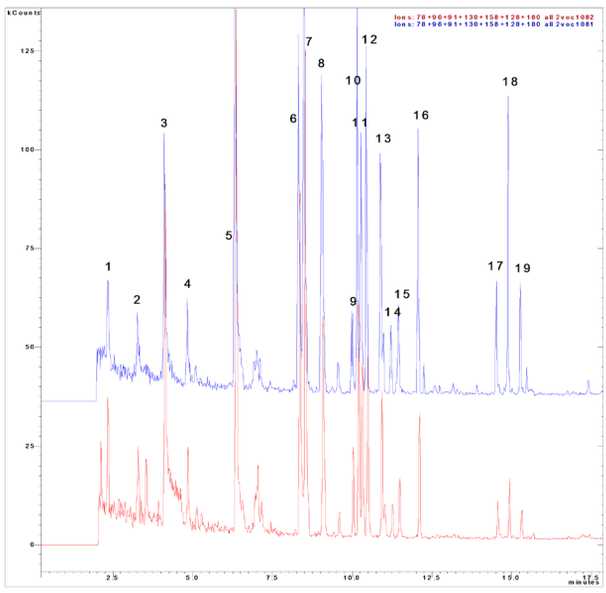
Phase	Color Code	Set of 1 Fiber Description PNo.	Set of 3 Fibers Description PNo.	Set of 5 Fibers Description PNo.	Supelco Color Code
PDMS Fiber (Polydimethylsiloxane) - nonpolar					
7 µm	Green	FIB-P-7/10-P1	FIB-P-7/10-P3	FIB-P-7/10-P5	Green Plain
30 µm	Golden	FIB-P-30/10-P1	FIB-P-30/10-P3	FIB-P-30/10-P5	Yellow Plain
100 µm	Red	FIB-P-100/10-P1	FIB-P-100/10-P3	FIB-P-100/10-P5	Red Plain
Polyacrylate Fiber (PA) - polar					
85 µm	Grey	FIB-A-85/10-P1	FIB-A-85/10-P3	FIB-A-85/10-P5	White Plain
Carbon WR Fiber / PDMS (Carbon Wide Range / PDMS) - bi-polar					
95 µm	Dark Blue	FIB-C-WR-95/10-P1	FIB-C-WR-95/10-P3	FIB-C-WR-95/10-P5	Black Plain
Fiber Collection – Development Kit (1 Fiber of each Fiber Type | Set of 5)					
various				Fiber Collection FIB-SEL5-S1 (one of each type)	

Table 1: PAL SPME Fiber Order Information.

All PAL SPME Fibers have a standard length of 10 mm, gauge 23 and the core material is fused silica. They can be used for a wide range of GC injector models. The assortment of the PAL SPME Fibers and the range of applications will be constantly expanded and developed. For more information register at www.palsystem.com.

Comparison of PAL SPME Fibers with established Fibers

The new PAL SPME Fibers (PDMS fibers 7 µm, 30 µm, and 100 µm and the polyacrylate fiber) yield identical results when compared with the corresponding commercial fibers. For medium and high boiling compounds the PAL SPME Carbon WR Fiber in certain cases shows an even better performance than the established fibers.



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

Fig. 1: Comparison of fibers for the analysis of VOCs: PAL SPME Carbon WR Fiber 95 µm (blue) and Brand X Carboxen® fiber (red).

Choose the right Fiber for your Analytes

Typical applications for the SPME technique are:

- Trace Analysis in foodstuffs
- Drugs and pharmaceuticals
- Herbicides / pesticides
- Medical diagnostics
- Water analysis (organics in water)
- Trace impurities in polymers and solid samples
- Solvent residues in raw materials

The type of the fiber corresponds to the polarity and the molecular weight of the analytes:

- For nonpolar samples a PDMS coated fiber should be chosen.
- For low molecular weights or volatile compounds a 100 µm PDMS-coated fiber is usually the best choice.
- Larger molecular weights or semi-volatile compounds are more effectively extracted using a 30 µm, or 7 µm PDMS-coated fiber.
- For an effective extraction of analytes with a very high polarity from polar samples, the 85 µm polyacrylate-coated fiber is the best alternative.
- For trace-level volatiles analysis, use the 95 µm Carbon WR (Carbon Wide Range / PDMS) coated fiber.

Note: The 100 µm and 30 µm PDMS-coated fibers cannot be used with hexane.

A SPME Tool together with a corresponding holder for the SPME Fibers is available for the PAL RSI and PAL RTC as well as for the PAL and PAL-***xt*** System models dedicated for SPME technique such as Combi PAL or PAL COMBI-***xt***. The main features of the SPME Tools are:

- Easy fiber exchange by hand
- Maximum fiber protection
- Compatible with a variety of different SPME fibers 10 mm or 20 mm fiber length supported

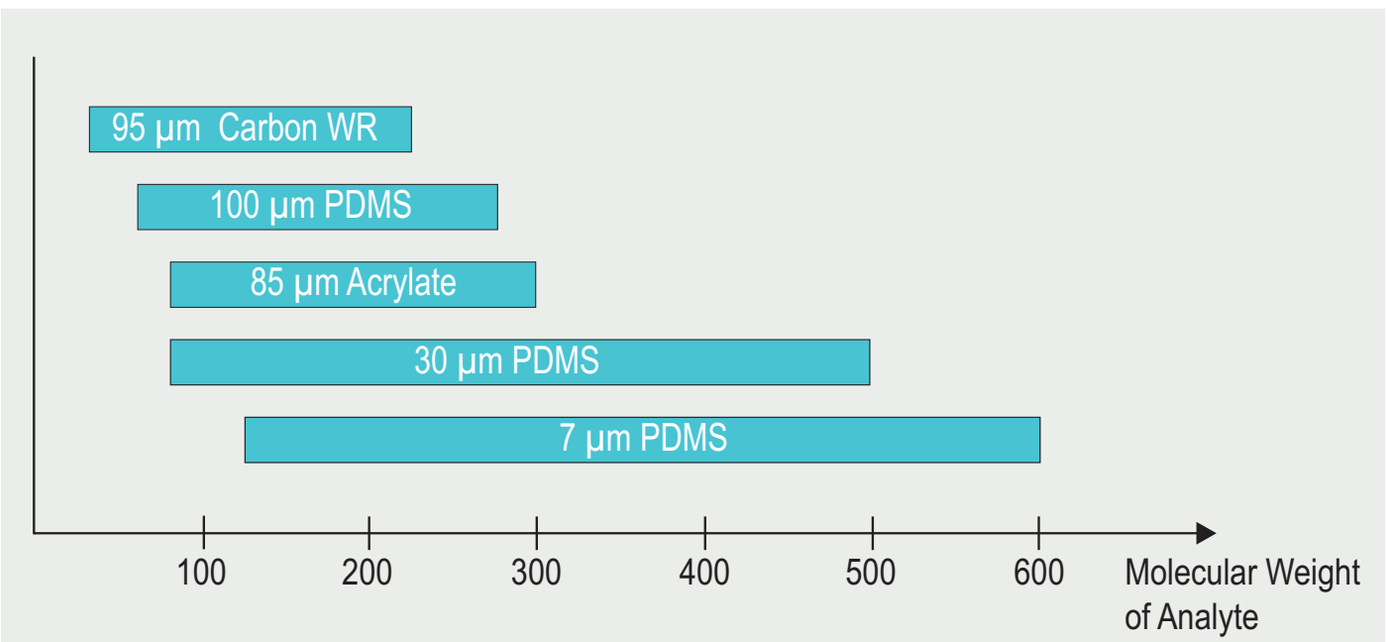


Fig. 2: Correlation between molecular weight of the analyte and the fiber type

Type of Analyte	Molecular Weight	Recommended Fiber
Non-polar high molecular weight compounds	125 - 600	7 µm PDMS (Polydimethylsiloxane)
Non-polar semi-volatiles	80 - 500	30 µm PDMS (Polydimethylsiloxane)
Polar semi-volatiles	80 -300	85 µm Polyacrylate (PA)
Volatiles	60 - 275	100 µm PDMS (Polydimethylsiloxane)
Gases and low molecular weight compounds	30 - 225	95 µm Carbon WR (Carbon Wide Range / PDMS)

Table 2: Which fiber for which type of analyte?

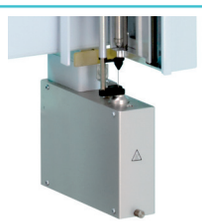
PAL SPME Accessories

To use the SPME technique with a PAL System a dedicated kit is required. Detailed information about the various kits can be obtained from CTC Analytics or directly from the web page www.palsystem.com.

An Agitator is highly recommended for temperature controlled extractions. Furthermore the agitation speeds up the equilibration process.
A second optional module is the SPME Fiber Conditioning Station (PAL and PAL-xt Systems) or the SPME Fiber Conditioning Module for the PAL3 System. The conditioning station has two functions. The first function is the cleaning (bake-out) of the inserted fiber after the analytical process to prepare for the next analysis. The second function is to condition a new fiber in an inert gas atmosphere. This module is strongly recommended since it will help to protect the GC injection port from contamination and free up the port after thermal desorption.

PAL RTC / PAL RSI

The robotic tool change exclusively available with the PAL RTC allows for the automatic exchange of fibers, e.g. for the automated development of SPME methods.

SPME Kit PNo.: PAL3-System-SPME-Kit		Kit containing – SPME Tool with SPME Fiber Holder – SPME Fiber Collection (one of each type) – SPME Performance Evaluation Mix
SPME Fiber Conditioning Module PNo.: PAL3-SPME-Cond		– For the conditioning of SPME fibers prior to sample collection – Temperature range up to 350°C – Purge gas connection for more efficiency – Additional port for a replacement fiber
Agitator Module PNo.: PAL3-Agitator		The Agitator Module provides 6 positions for 20 mL vials for incubation and agitation of samples. – Temperature range 40 – 200 °C – Agitation speed 250 – 750 rpm – Optional adapters for 2 mL or 10 mL vials

PAL / PAL-*xt*

PAL SPME Kit	PNo.: PAL SPME-Kit
SPME Fiber Conditioning Module	PNo.: PAL FibCond
Agitator	PNo.: PAL Incub6x20

Note: For details about the technical specifications of the modules/kit for the PAL-***xt*** please contact your CTC Analytics representative or visit our web page www.palsystem.com.

PAL SPME Fiber Conditioning and Cleaning

Caution:

Without gas protection the fiber surface will be damaged if exposed to elevated temperatures.

Fiber Preconditioning

Prior to analytical use, it is mandatory to precondition each fiber at a specified temperature in an inert gas phase environment. The life span of the fiber can be extended if the fiber is not unnecessarily preconditioned at maximum temperature.

Generally, it is recommended to precondition the fiber 20°C above the planned operating temperature, while respecting the maximum temperature threshold. Recommended temperatures and conditioning times are given in Table 2.

Fiber Conditioning

It is part of the analytical process to condition the fiber after thermal desorption of the analytes has been completed. This conditioning is a preparatory step for the next analytical run. It is necessary to eliminate all possible contaminants from the fiber which have not been desorbed and transferred to the GC column.

To avoid contamination of the GC inlet system and/or the GC column, it is recommended to remove the fiber after the thermal desorption step from the GC injector and move the SPME Tool to the SPME Conditioning Module for the conditioning step.

The large surface of the fiber can trap impurities from the ambient atmosphere if a fiber has been left in the open. Considering this, it is good recommended practice to run a blank prior to running a series of analytical samples. Evaluating the baseline level of the GC detector helps to ensure that the entire system, such as the fiber, the GC inlet, the GC column, and detector, is free from any contaminants.

Rinsing of Fibers

It is possible to clean the fiber using an organic solvent, should the fiber be subject to inappropriate storage, e.g keeping the fiber in the open at ambient environment without protection for a prolonged period, or if obvious dust particles are sticking to the fiber. The recommended types of solvents are listed in Table 2. Do not use any other solvents than those mentioned here. Other solvents can cause a swelling of the fiber which would lead to significant damage. It is important that a fiber is not cleaned mechanically by any means; do not touch the fiber with fingers, not even when wearing gloves.

The cleaning process can be done manually by dipping the fiber into a container filled with the appropriate solvent or in an automated manner by defining a vial for cleaning. To avoid a potential misunderstanding, do not use a wash or waste solvent of the Wash Module from the PAL System. This solvent can be contaminated or the solvent in use may not be suitable for the particular fiber type.

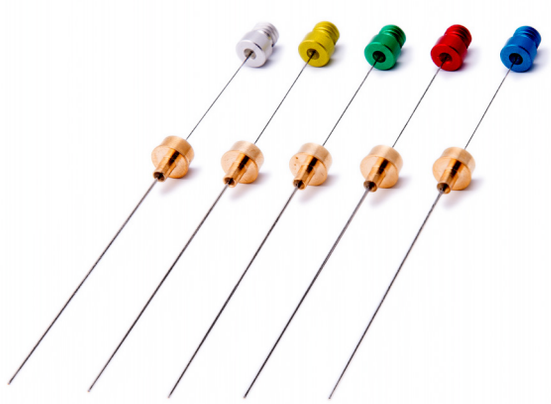
General Remarks for Fiber Conditioning and Cleaning

The table on the right summarizes the various parameters for conditioning and cleaning. The values provided are empirical values which are suitable for a number of applications and give reliable results. The life span of a fiber depends to a great degree on the field and type of application. Using the SPME technique, by inserting the fiber into a liquid with a high degree of matrix, the number of analyses can vary from a few to approximately 100 analyses. If the fiber is positioned in the headspace of a vial and avoids any contact with liquid and matrix, it is typically possible to run several hundred extractions.

It is not possible to visually judge the fiber quality if there are no obvious signs of major mechanical damage, such as a fiber fracture.

Any sign of staining, caused by a starting vitrification of the surface in case of a PDMS fiber, or signs of a yellowish discoloration in the case of a Polyacrylate fiber, does not give any indication on the remaining life span of the particular fiber.

As a rule of thumb, the life span of a fiber can be extended if its exposure to high temperatures is minimized. Do not exceed the maximum temperature for each fiber type as shown in Table 3.



Stationary Phase Fiber Type	Maximum Temp. (°C)	Recommended Operating Temp. (°C)	Preconditioning Temp. (°C) Min. | Max.	Preconditioning Time (min.) Min. | Max. | Recom.	Conditioning Temp. (°C) Min. | Max.	Conditioning Time (min.) Min. | Max. | Recom.	Fiber Rinsing Solvent	Fiber Rinsing Time (min.) Min. | Max. | Recom.
PDMS Fiber (Polydimethylsiloxane)								
7 µm	340	200-340	200 | 340	15 | 120 | 30	200 | 340	1 | 60 | 5	MeOH | EtOH iProp	0.5 | 10 | 2
30 µm	280	200-280	180 | 280	15 | 120 | 30	180 | 280	1 | 60 | 5	MeOH | EtOH iProp	0.5 | 10 | 2
100 µm	280	200-280	180 | 280	15 | 120 | 30	180 | 280	1 | 60 | 5	MeOH | EtOH iProp	0.5 | 10 | 2
Acrylate Fiber (Polyacrylate) 300								
85 µm	300	200-280	180 | 280	15 | 120 | 30	180 | 280	1 | 60 | 5	MeOH aliphatic HC	0.5 | 10 | 2
Carbon WR Fiber / PDMS (Carbon Wide Range / PDMS)								
95 µm	300	220-300	200 | 300	15 | 120 | 60	200 | 300	1 | 60 | 10	MeOH | EtOH iProp	0.5 | 10 | 2

Table 3: Parameters for PAL SPME Fibers. MeOH = Methanol EtOH = Ethanol
iProp = Iso-Propanol (2-Propanol) aliphatic HC = aliphatic hydrocarbons (example n-Hexane)

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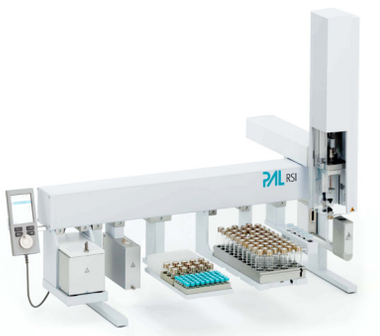

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www.palsystem.com

Part No. PAL Broch-SPME-1