

MFX (Multi Fiber eXchange) PAL RSI and PAL RTC systems



MFX (Multi Fiber eXchange)

AUTOMATED SPME USING THE NEW PAL AUTOSAMPLER

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.

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.

In SPME, you can adsorb analytes from a liquid sample, by immersion or headspace extraction, or a solid sample, by headspace extraction, using a polymer-coated fused silica fiber. Analytes are desorbed from the fiber by exposing the fiber in the injection port of a GC or in the desorption chamber of an SPME/ HPLC interface.

Following the concentration step, the SPME fiber is inserted into the injector for thermal desorption and transfer of analytes to the GC/ MS system. The use of a septumless system, eliminating the common problems with septum coring seen with blunt-tipped SPME needles and standard GC inlets.

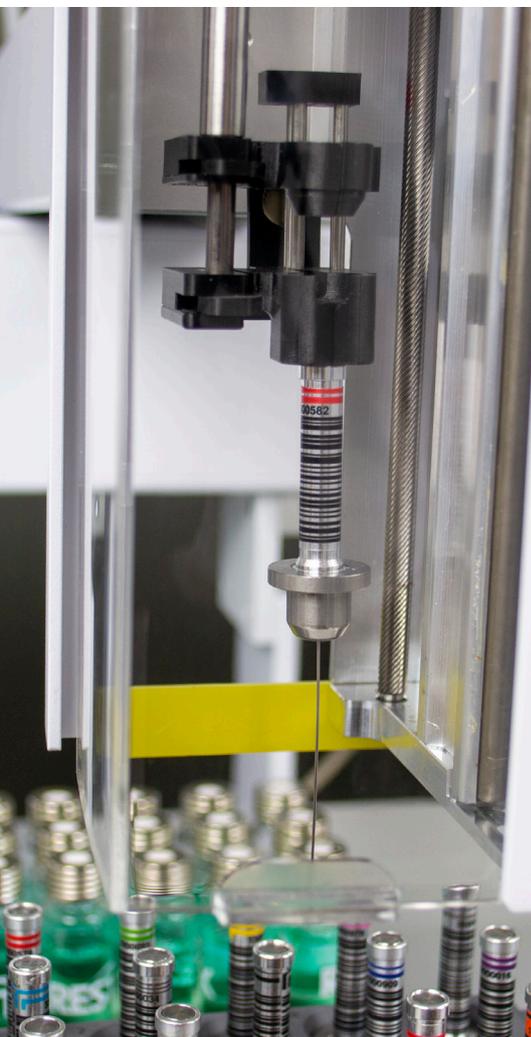
Derivatization can be automatically performed directly on the fiber, either prior to or following the SPME extraction step, enabling the use of water sensitive derivatization reagents even for aqueous samples.

CTC Sample Control software enables highly flexible and efficient SPME analysis. The user can set up the method and the sequence table and clicks run.

CTC PAL3 systems are highly flexible and based on a modular concept.

Standard SPME systems can be upgraded with automated SPME Multi Fiber Exchange (MFX) and with a long list of other options that can cover your every need.

The MFX system for CTC PAL systems is distributed exclusively by SRA INSTRUMENTS FRANCE.



AUTOMATED SPME

- The Multi Fiber eXchange System (MFX) performs automated exchange of SPME fibers (Standard and Arrow Fibers). Creating a new method will be quick and easy, you can also create multi-method sequences that use fibers with different phases (without stopping the autosampler and the GC)
- With Tools exchange solution of PAL RTC, it's possible to manage all syringes operations like standard addition or derivatization with SPME routine steps
- Exchange of fibers SPME manually can damage SPME fibers. The use of MFX system reduces this problem
- The complete automated management of the analysis process improves the reproducibility of the methods



PAL RTC (Robotic Tool Change) with Park Station.



Multi Fiber eXchange (MFX)

SPME is easily automated, but until now, replacing a fiber had to be performed manually, limiting the scope of automated method development and making it difficult to run large series of analyses due to limited fiber life span.

The Multi Fiber eXchange (MFX) accessory for the PAL System enables fully automated exchange of SPME fibers within a sequence.

MFX allows both analysis conditions and SPME fibers to be easily and automatically changed at any time within a method or a sequence. MFX simplifies method development and enhances the productivity of routine SPME analysis by allowing samples to be run without interruption - 24/7 while delivering accurate results.

To use MFX option on PAL system, the PAL Sample Control software is the user-friendly tool for the daily routine jobs. With a few clicks sample lists are generated or imported. Now PAL Sample Control starts the operation and the data acquisition. Since PAL Sample Control Interfaces seamlessly with most of the major chromatographic or MS data systems only one sample list has to be handled. Different user levels ensure process safety.



MFX BENEFITS

- The development of new methods is faster. By setting a sequence that uses more fibers on the same sample, it is easy to understand which fiber highlights the best results for analytes of interest.
- Automatic replacement of fibers can be done at any time inside the sample list. Long analysis series can also be performed with different fibers and the same fibers can be cleaned automatically. You can also replace fibers used after a defined number of samples, so they always use fibers at maximum efficiency.
- In a tray can stay all the available phases of the SPME fibers (as in the picture)
- Automated analysis of SPME fibers that have been used as passive air samplers
- MFX tray for 15 fibers available (up to 45 fibers for each trayholder)
- Automated analysis of samples using multiple fibers, covering a wide range of analytes
- Multi-method sequences using different SPME phases for more selectivity
- Longer fiber life expectancy, mechanical impact is minimized during fiber change and operation.
- SPME-FFA and SPME-FFA Arrow available from Chrom-line Srl

SPME: Innovative, low-cost and versatile. The technique is solvent-free and reduces sample preparation times and manual manipulation of the samples.

A BIT OF HISTORY

The SPME® technique was born thanks to the intuitions of Janusz Pawliszyn, a professor at the University of Waterloo in Canada, who managed to immobilize an appropriate stationary phase on a thin fus of fused silica. The technique is patented in 1990 by the University of Waterloo (Canada) and the first automation devices are developed by Varian (AS 8200) in the following years.

WHAT IS FIBER

SPME, also known as “SPEE MEE”, is a solvent-free technique that is based on adsorption / desorption mechanisms. The device consists of coated fibers that are used to isolate and concentrate the analytes within a wide range of coating materials.

After extraction, the fibers are transferred to an analytical tool for the separation and quantification of target analytes.

This step is carried out both manually or through the use of self-amissions, in both cases devices are used (called holders) that allow the exposure of the polymer phase within the instrument. Some holders are made so as to protect the fiber and analytes during the sample conservation phase.

HOW FIBER WORKS

The fiber, contained inside the needle of a syringe, is thermally desorbed in the injector of a gas chromatograph equipped with an internal diameter liner of 0.75 mm.

The stationary phase deposited, constitutes the fiber coating and has a volume of $690 \times 10^{-12} \text{ m}^3$ for the thickness of 100 μm and $140 \times 10^{-12} \text{ m}^3$ for thicknesses of 30 μm .

ITALIAN PATENT

Chromline, in 2006 has registered the patent that reinvents the use of this technique and add some new features capable of improving the robustness of SPME fibers and their use in the various analysis phases.

The fibers are housed in a metal body that protects the most fragile parts and also allows the introduction of a unique barcode that identifies each fiber.

BARCODE AND TRACEABILITY

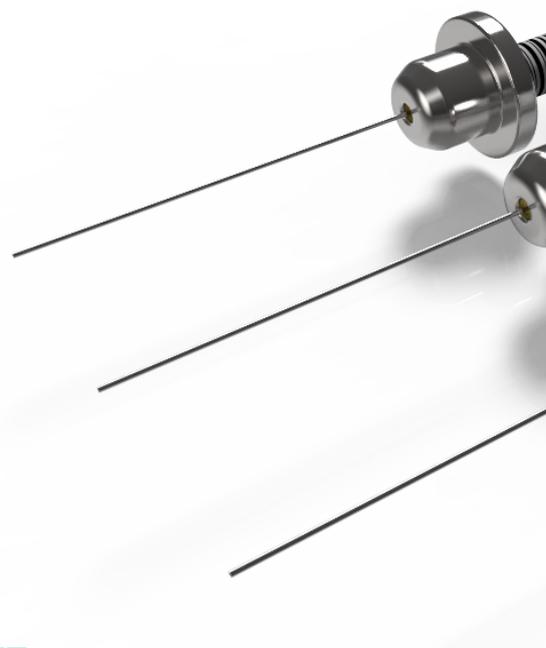
A complete traceability of the sample is possible with new FFA devices made by Chromline. The Barcode provides the user and information relating to the SPME-FFA fibers that are using. In particular, 3 different information can be identified on the fiber that generate a characteristic unique code of each fiber:

A. The color indicates the phase type. The color is the same as the colored hub present on traditional SPME fibers. The SPME-FFA Arrow fibers have a color-based encoding that considers the diameter of the needle and the phase diameter (based on the type of fiber used there will only be the color of the phase, the color with a line or the color with two lines);

B. The first two numbers (three on Arrow fibers) identify the type of fiber and represents the phase and the size of the needle;

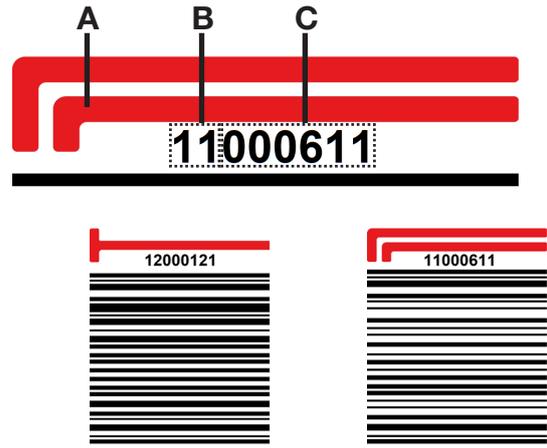
C. The remaining numbers indicate the unique code of each fiber to ensure a perfect traceability of the samples.

The new Solid phase MicroExtraction (SPME) Arrows are available

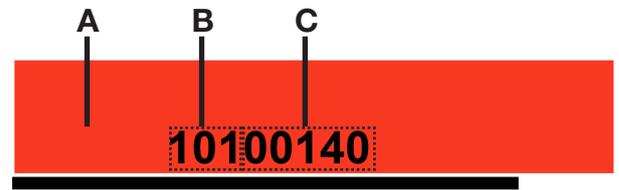




Standard fibers coding



Arrow fibers coding

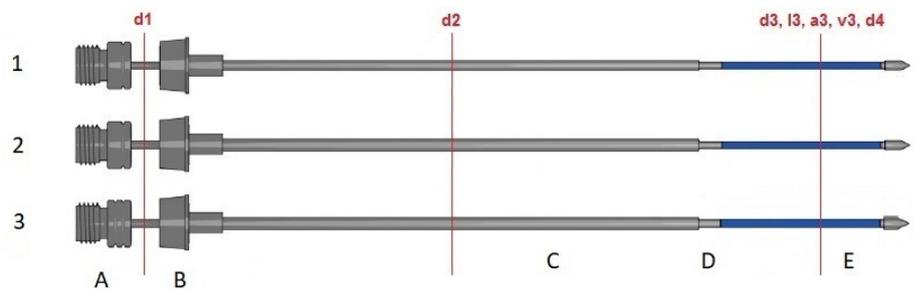
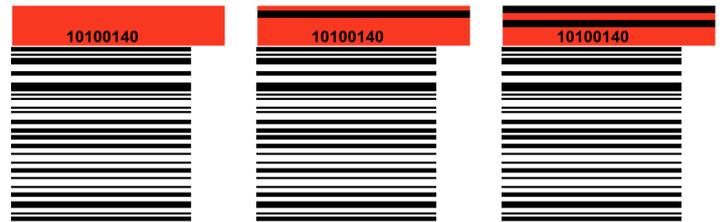


able in 3 different configurations:

- (1) 1.1 mm SPME Arrow;
- (2) 1.5 mm Wide Sleeve SPME Arrow
- (3) 1.5 mm SPME Arrow.

All SPME Arrows composed of the following parts:

- **A:** a color coded screw hub;
- **B:** a sealing septum;
- **C:** a septum piercing needle;
- **D:** a fiber attachment needle;
- **E:** a coated metal fiber;
- **d1:** Support tubing;
- **d2:** septum piercing needle;
- **d3:** phase diameter;
- **d4:** phase support tubing diameter;
- **l3:** phase length;
- **a3:** phase area;
- **v3:** phase volume.



Label	Description	Units	1.1 mm SPME Arrow	1.5 mm Wide Sleeve SPME Arrow	1.5 mm SPME Arrow
d1	Support tubing	mm	0.804	1.01	1.01
d2	Septum piercing needle	mm	1.1	1.50	1.50
d3	Phase diameter	mm	0.721	0.721	0.912
d4	Phase support tubing diameter	mm	0.647	0.647	0.498
l3	Phase length	mm	20.0	20.0	20.0
a3	Phase area	mm ²	44.0	44.0	62.8
v3	Phase volume	μl	3.80	3.80	11.8

CASE STUDY

NEEDLE GAUGE

A PROPER CHOICE CAN AFFECT THE FINAL RESULT

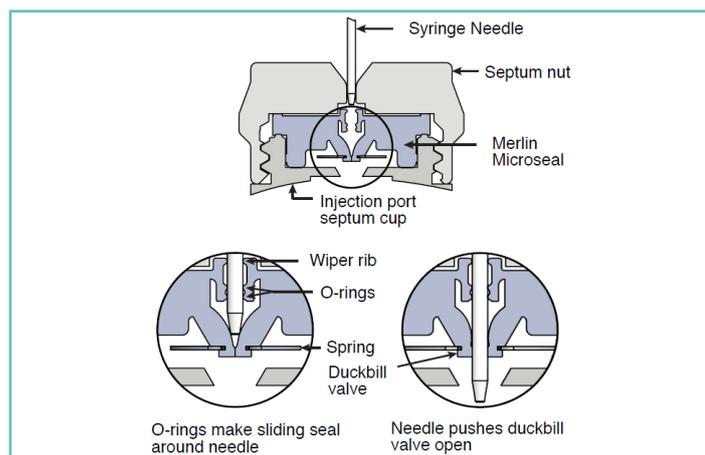
DIFFERENCES

Like the standard fibers produced by Supelco, even the new SPME-FFA fibers are available with two-dimensional needles, 23Ga and 24GA. The fibers with 24GA needles are used with standard injectors and by performing injections manually and using self-ampers. The small size of the needle 24ga minimize shavings formation problems inside the injector and promote better penetration through the septum seen the flat shape of SPME needles.

The fibers with 23ga needles are instead made for injectors with Merlin Microseal™ system.

Due to the relatively large diameter of Restek PAL SPME Arrows, you must modify the GC inlet using an instrument-specific conversion kit prior to use. Converted inlets are compatible with all standard injection techniques (SPME Arrow, liquid syringe, headspace, etc.); no need to switch inlets after installation.

Restek PAL SPME Arrow GC-Specific Conversion Kits are easy to install. For the weldment, install according to your instrument's owner's manual. An installation instruction sheet for Agilent 7890 Split/Splitless injectors is provided below for reference.



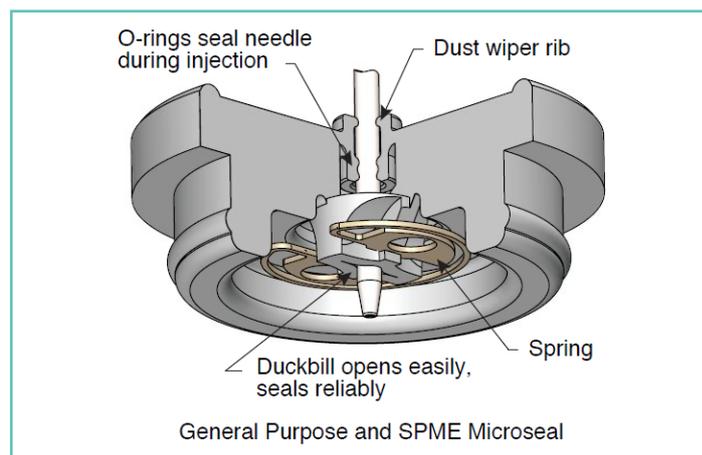
Injection sequence with Merlin Microseal™ system.

MERLIN MICROSEAL™

This Septum-Less system guarantees a longer life of the injector block, reducing the stop machine for septum replacement. Since there is no pierce septum, the septum shavings will not be formed and therefore there will be no accumulation inside the injector.

The Merlin system contains a series of gaskets that allow the entrance of a needle, maintaining a leak-free seal. The septum can only be used with a syringe equipped with a flat-pointed needle or 23GA SPME needles. The larger needle sizes are necessary to guarantee the necessary contact with septum seals.

Now the conversion kits for Merlin systems dedicated to the new SPME Arrow fibers are also available, to obtain a complete compatibility on all GC systems.



Internal view of the Merlin Microseal™ system.

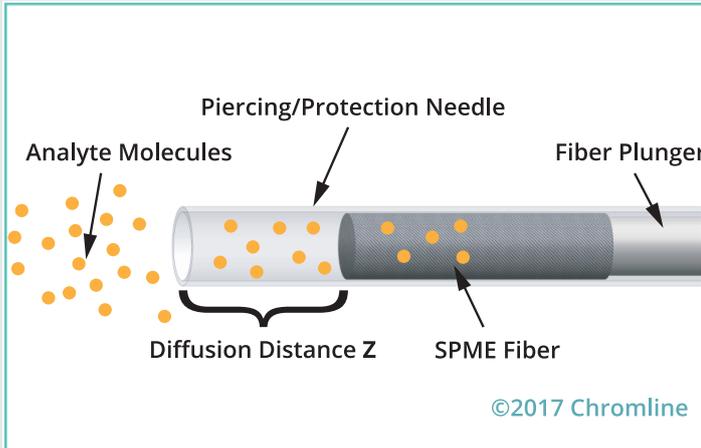
Ago	Diametro esterno nominale			Diametro interno nominale			Spessore parete nominale		
	inches	mm	toll. inches (mm)	inches	mm	toll. inches (mm)	inches	mm	toll. inches (mm)
23	0.02525	0.6414	±0.00025 (±0.0064)	0.01325	0.337	±0.00075 (±0.019)	0.006	0.1524	±0.00025 (±0.0064)
24	0.02225	0.5652	±0.00025 (±0.0064)	0.01225	0.311	±0.00075 (±0.019)	0.005	0.1270	±0.00025 (±0.0064)

Table of needle size for standard SPME fibers.

VERSATILITY

SOME EXAMPLES OF SPME APPLICATIONS

Some examples are reported to explain the different applications in which SPME technique can be used, but these represent only a small part of opportunity to use SPME and solve analytical problems. Food, packaging, pharmaceutical, organic, toxicological, forensic and environmental matrices can be analyzed and applications continue to increase thanks to new publications that are made every year.



Passive sampling mechanism with SPME fibers.

PASSIVE SAMPLER

BENEFITS:

- ✓ Long sampling times (up to 8 hours)
- ✓ TLW Cealing
- ✓ No air collection equipment
- ✓ Low costs

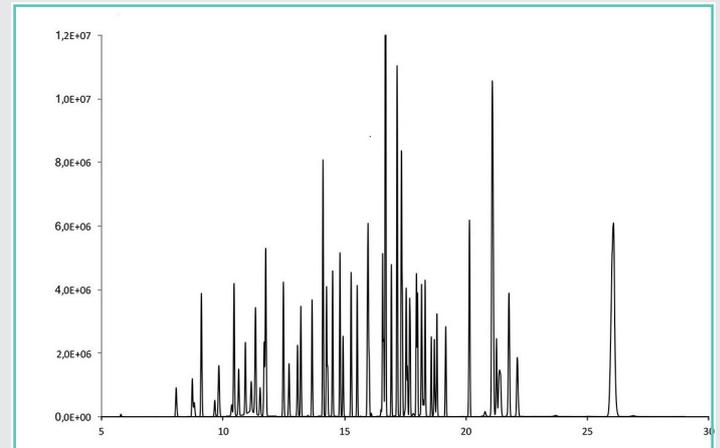
SKILL LEVEL: HIGH

DRINKING WATER

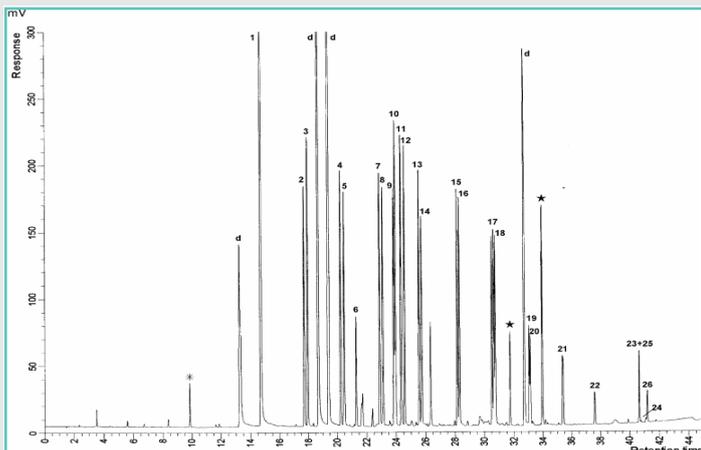
BENEFITS:

- ✓ Small quantities of sample
- ✓ Polar and apolar analytes
- ✓ Short extraction time
- ✓ Low costs

SKILL LEVEL: LOW



The water sample were analyzed according to the drafted ISO Standard 17943 with HS-SPME.



SPME with on-fiber PFBHA derivatization has been applied to environmental monitoring of aldehydes and ketones.

DERIVATIZATION

BENEFITS:

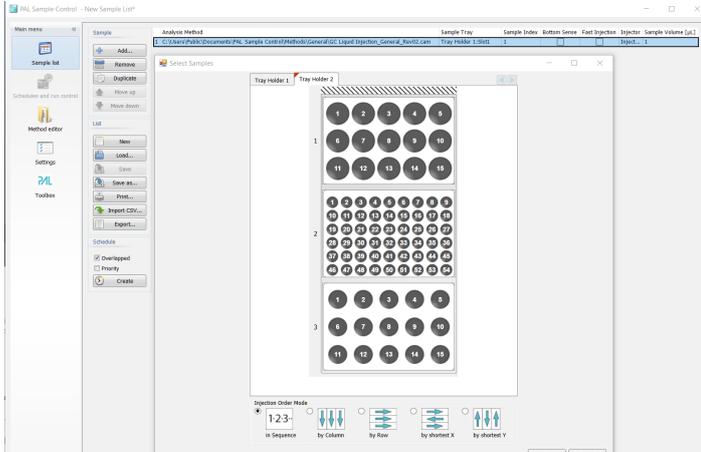
- ✓ Greater specificity
- ✓ Lod increase
- ✓ Better separation
- ✓ Makes some analytes detectable

SKILL LEVEL: MEDIUM

SOFTWARE

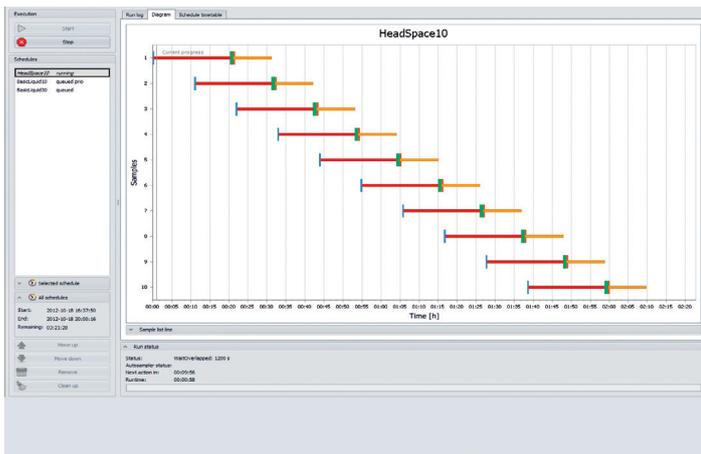
PAL SAMPLE CONTROL

PAL SYSTEM SOFTWARE



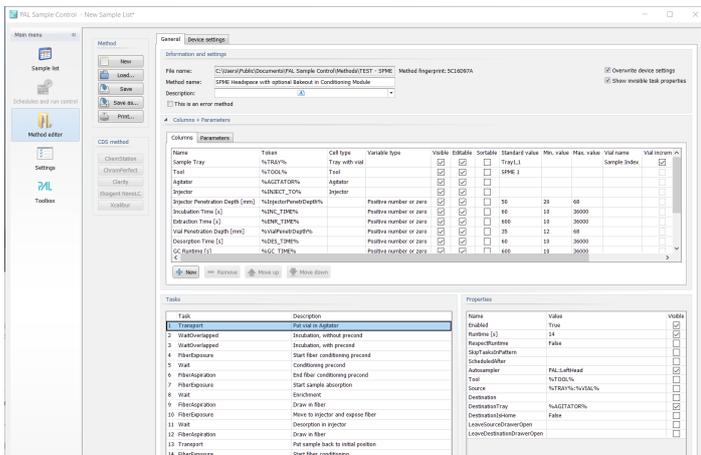
SIMPLE TO USE

PAL Sample Control software is the user-friendly tool for the daily routine jobs. With a few clicks sample lists are generated or imported. Now PAL Sample Control starts the operation and the data acquisition. Since PAL Sample Control Interfaces seamlessly with most of the major chromatographic or MS data systems only one sample list has to be handled. Different user levels ensure process safety.



INCREASE PRODUCTIVITY

New PAL software, PAL Sample Control, allows overlapping of time consuming steps. It optimizes automatically the timing of various steps in a sample preparation process and generates a schedule that minimizes the runtimes of sequences. This increases sample throughput greatly and boosts productivity.



METHOD EDITOR AND CUSTOM PLUGIN

The PAL Sample Control allows both standard operations and the construction of fully customized methods. The basic methods provided, can be modified, updated or used as a reference to build more complex methods. The many tasks available, allow the construction of very complex workflow and new methods. Chromline has developed tasks to use MFX on PAL systems and is able to customize these instructions according to the needs of its customers.