
Addendum to PAL User Manual

PAL SPME Fiber Conditioning Station

Installation and Operation

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Original Instructions

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A. Safety Information

Declaration of Conformity

See Declaration of Conformity sheet enclosed with the instrument



General Considerations

Changes or modifications to this unit not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment. The user should be aware that if the equipment is used in a manner not specified by the manufacturer, the protective and safety features of the equipment may be impaired.

Electrical Hazards

Every analytical instrument has specific hazards, so be sure to read and comply with the precautions as described in the corresponding PAL User Manual. Use only fuses of the type and current rating specified. Do not use repaired fuses and do not short-circuit the fuse holder.



Other Hazards

To avoid injury during PAL System operation, keep hands away from the syringe.

For detailed Safety Information, see the additional warnings in the corresponding PAL or PAL-xt User Manual or in the booklet 'Safety Information and Warnings for Users of the PAL System'.

Commonly Used Symbols

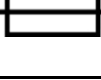
Symbol	Description
	Caution, or refer to User Manual
	Caution, Risk of Needle-Stick Puncture
	Caution, Hot Surface or High Temperature
	Danger of crushing to fingers and hands
	Laser Warning, Barcode Reader
	Biological Hazard
	Direct Current
	Alternating Current
	Protective Conductor Terminal, Ground
	Fuse
	Electrical Power ON Used with Main PAL Power Supply
	Electrical Power OFF Used with Main PAL Power Supply
	Caution, Risk of Electrical shock (high voltage)
	Disposal: Do not dispose in municipal waste. Follow local waste regulations to reduce electrical and electronic waste (WEEE).

Table 1. Commonly Used Safety Symbols

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D. How to Use this Manual

note

This Addendum covers the PAL SPME Fiber Conditioning Station which can be used in combination with PAL Systems (e.g. HTS PAL, or COMBI PAL) and PAL-xt Systems (HTS-xt or PAL COMBI-xt). Issues specific to the PAL-xt System have been emphasized accordingly.

The major sections of this Addendum are:

- Safety Information
- PAL SPME Fiber Conditioning Station Installation
- PAL SPME Fiber Conditioning Station Operation
- Appendices

This Addendum is intended for frequent or new PAL System users who are experienced at using automated systems to run existing analytical methods.

The Appendices provide information on PAL SPME Fiber Conditioning Station and Spare Parts.

note

The COMBI PAL must be installed and set up properly before the SPME Fiber Conditioning Station Operating instructions can be used.

E. Installation of PAL SPME Fiber Conditioning Station

1. General System Overview



Figure 1. SPME Fiber Conditioning Station

The SPME Fiber Conditioning Station is an optional device used for the SPME technique (Solid Phase Micro Extraction).

The purpose is to clean or condition a fiber between chromatographic runs. A flow of inert gas protects the fiber from degradation when it is exposed to elevated temperatures. The temperature for conditioning or cleaning is selectable.

A spare fiber can be conditioned in the front well to ensure that a spare fiber is always ready for use.

1.1. Specifications

Article Number:	PAL FibCond
Description:	PAL SPME Fiber Conditioning Station
Temperature Control:	30 °C to 350 °C in steps of 1 °C
Dimensions and Weight:	L: 50 mm D: 175 mm H: 170 mm Std. bracket holder H: 144 mm Short bracket holder Weight: 820 g

1.2 PAL Hardware Requirements

The PAL SPME Fiber Conditioning Station can be used with any PAL System (e.g. HTS PAL or COMBI PAL) or PAL-**xt** system (e.g. PAL HTS-**xt** or PAL COMBI-**xt**).

A gas supply has to be available. Consequently, a PAL System equipped for headspace or SPME technique would be a logical choice to use with the Fiber Conditioning Station. The electrical heating is provided via an 'Aux' interface, typically 'AUX2'.

1.3 Software Requirements

The heating for the PAL SPME Fiber Conditioning Station is controlled directly by the PAL System. Temperature control via software, such as Cycle Composer or certain data handling systems that control the PAL using Cycle Editor for PAL ICC interpretation, is not possible.

The PAL SPME Fiber conditioning station is compatible with PAL Firmware version 2.0.X or higher. However the minimum firmware version for the SPME technique should be 2.2.7. or higher and for a PAL-**xt** System, PAL Firmware version 4.1.X (or higher) is required.

2. Installation

2.1. Unpacking the Components

The SPME Fiber Conditioning Station is shipped in one box. Check for the following items:

- SPME Fiber Conditioning Station
- PAL FibCondTube, consisting of:
 - Gas tube, PTFE, black
 - Swagelok Union 1/8 inch (includes flow reduction)
 - Reducing Unit M5
- PAL Zub-SPME:
 - Short Bracket (14 mm) with 2 screws and 2 serrated washers.
- Disk: PAL Object Manager Software:
 - Software to add new Objects to the COMBI PAL System

2.2. Assembling the SPME Fiber Conditioning Station

Before beginning the assembly process, determine approximately where the SPME Fiber Conditioning Station will be located. Consider the height of the station, and ensure there is sufficient space to be able to move a 20 mL vial from the Tray to the Agitator and vice versa.

If there is not enough space between the X-axis of the COMBI PAL and the GC top cover, consider installing the short bracket, which is part of the installation kit.

If the Fiber Conditioning Station is to be installed with an existing COMBI PAL System, another hardware module may need to be shifted. Remember to re-position the modules again according the COMBI PAL manual, Chapter 7.

1. Loosen the Torx screw on the mounting clamps located on the top side of the Conditioning Station.
2. Be sure that the clamps fit entirely into the grooves. Tighten the Torx screw until the mounting clamps are firmly in place.
3. Double check whether the Fiber Conditioning Station clamp is correctly attached to the X-axis (see Fig. 5 in the COMBI PAL Manual).

note

The short bracket that is provided is a spacer 14 mm high which could be installed instead of the standard bracket in cases where the total height of the fiber conditioning station does not physically fit the PAL System.

2.3. Electrical Connections

Installing the SPME Fiber Conditioning Station

note

It is important to turn the power off to the COMBI PAL System before the Fiber Conditioning Station is connected electrically.

1. Connect the open end of the cable from the Fiber Conditioning Station to the 'AUX2' connector on the COMBI PAL. See Figure 2.

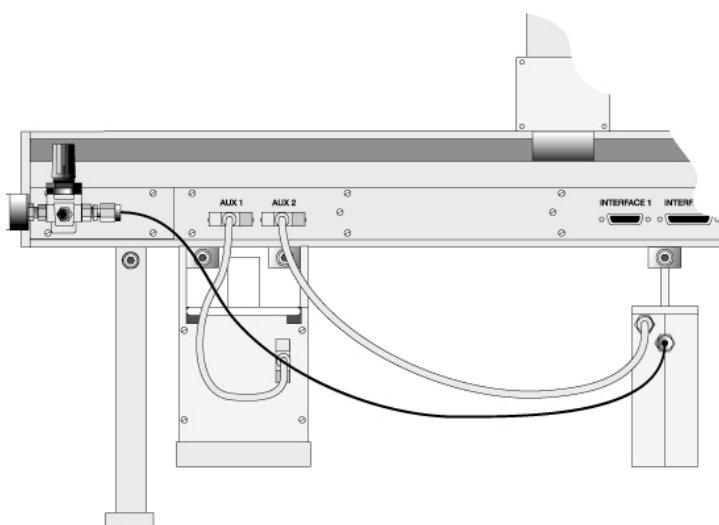


Figure 2. SPME Fiber Conditioning Station, Electrical and Gas Connections

2. Power up the PAL System again. With this warm up, the system again recognizes and references the module connected to 'AUX-Interface'.

note

The electrical connection for the PAL-xt System is identical. Use the 'AUX2' connector as well. Usually, an Agitator is connected by default to the 'AUX1' interface.

2.4. Gas-Line Connections

One end of the supplied black gas tube is connected to the flush gas regulator supplied with the COMBI PAL; see Chapter 6 'Installation' in the COMBI PAL Manual. Remove the blind-plug at the gas regulator and connect the corresponding reducing union supplied with the installation kit. The other side of the gas tube is connected with a 1/8 inch Swagelok nut to the Fiber Conditioning Station. The installation of this second gas line is displayed in Figure 3.

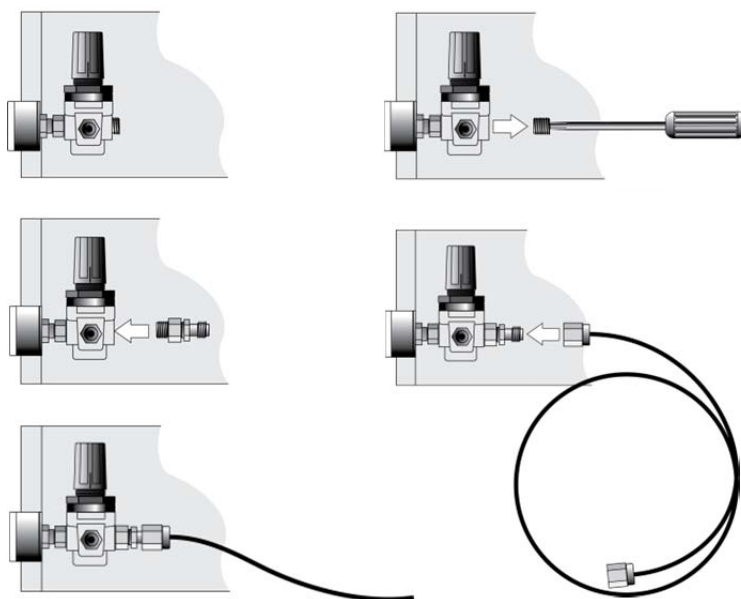


Figure 3. Gas Tube Connection from Pressure Regulator to SPME Fiber Conditioning Station

3. PAL Firmware and PAL Object Installation for PAL SPME Fiber Conditioning Station Option

3.1. PAL Firmware Installation for PAL SPME Conditioning Station

The PAL SPME Fiber Conditioning Station option requires in combination with a PAL System the PAL Firmware version 2.0.X or higher. If a lower firmware version is installed on the unit in use, an upgrade of the firmware to the current version is recommended.

Nevertheless, the minimum firmware version for the SPME technique should be 2.2.7. or higher. It is recommended to upgrade the PAL Firmware version to the current level.

In the case of a combination with a PAL-**xt** System, PAL Firmware version 4.1.X or higher and the control board (PCB) 'APR Control-**xt**' are required.

3.2. PAL Firmware Object List Installation for the PAL SPME Fiber Conditioning Station

A CD-ROM is provided together with the PAL SPME Fiber Conditioning Station, which contains the required PAL Firmware Objects to enable the use of the technique.

Required PAL Object Manager Object Lists:

- PAL System: PAL Object List Revision K or higher.
- PAL-**xt** system : PAL-**xt** Object List Revision C or higher

PAL Systems:

If the revision version of the PAL Object Manager List installed on the computer is lower than 'K', copy the provided folder 'SPME Fiber Conditioning Station' from the CD-ROM to the Object Lists folder which has been installed with the Object Manager. This software is usually installed in the following path:

C:\Program Files\PAL\Object Manager\Object Lists

Using a firmware Object List revision K or higher, this 'SPME Fiber Conditioning Station' folder will become a part of the list.

PAL-xt** Systems:**

If the SPME Fiber Conditioning Station is configured to a PAL-**xt** System, use the provided PAL-**xt** Object List Rev. C or higher.
DO NOT use an Object List from the PAL System (example Rev. K), because

the structure of the Object has changed and is not compatible with the PAL-*xt* Firmware.

Copy the PAL-*xt* Object List from the CD-ROM to the PAL Object Manager software which is usually installed in the directory path:

C:\Program Files\PAL\Object Manager\Object Lists

If the PAL Object Manager software is not installed yet, use the provided 'PAL System CD' or 'PAL-*xt* System CD'. Instructions are provided with the CD.

The following Object Lists are available:

PAL FibCond_Aux1	SPME Cond. Station connected to 'AUX1' interface
PAL FibCond_Aux2	SPME Cond. Station connected to 'AUX2' interface

note

*The Object List names are identical for both 'PAL' or 'PAL-*xt*' Systems, but the actual PAL Object Lists differ between the 'PAL' and 'PAL-*xt*' Systems. The example shown below is the PAL System Object List Rev.J.*

Start Object Manager. The 'PAL SPME Fiber Conditioning Station' folder should be visible in the structure shown in the window 'Choose Object List Folder' on the left side of the Object Manager program window.

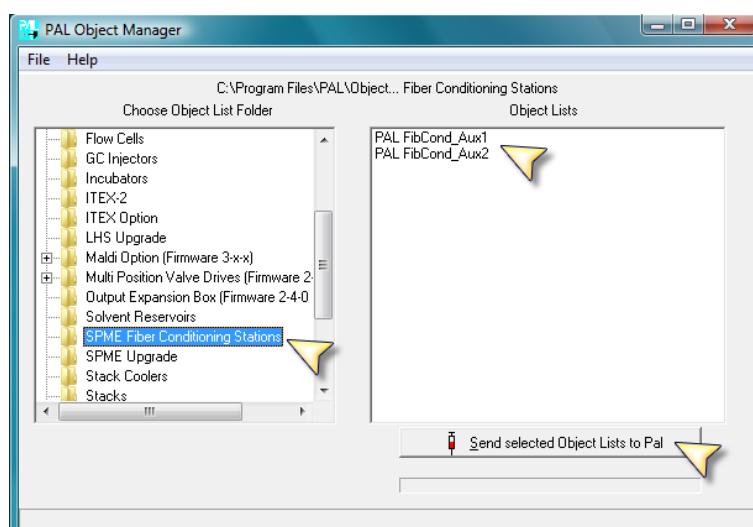


Figure 4. PAL Object Manager 'Choose Object List Folder'

Select the folder 'SPME Fiber Conditioning Station' and choose the 'SPME Fiber Conditioning Station Option' according to the available 'AUX' Interface(s).

Send the selected PAL Firmware Object List to PAL.

note

It is assumed that the PAL Firmware has also been prepared for the SPME technique. If this point has been missed, complete the following steps:

Select the class 'SPME Upgrade' in the Object List Folder, then select 'PAL SPMEOpt' from the Object List.

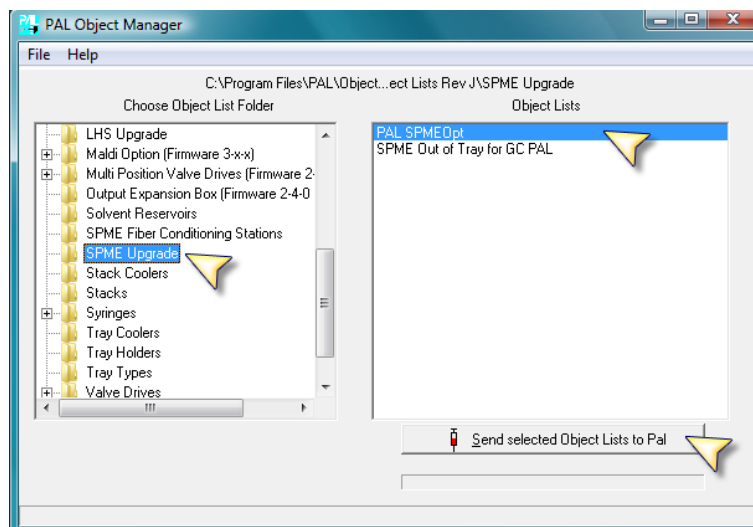


Figure 5. PAL Object Manager, Send Object 'PAL SPMEOpt' to PAL

It is important to understand the meaning of the two Object Lists:

PAL SPMEOpt:	This PAL Firmware Object List loads the Objects 'SPME Fiber' and Position 'FiberExp'. The two Objects are required to enable the automated SPME technique to function, either in standalone mode (from local terminal) or from Cycle Composer or an integration using ICC cycles. The Agitator will be used for this SPME cycle.
SPME Out of Tray for GC PAL:	This PAL Firmware Object List loads the same Objects but will turn OFF the use of the Agitator. 'Out of Tray' means that the fiber goes directly to the selected vial in the Tray, not using the temperature stability function from the Agitator.

note

Load only the required Object List, do not load both types. In case the Object List 'SPME Out of Tray for GC PAL' has been added by mistake, add the PAL Firmware Object List for the Incubator (Agitator) to the PAL System. With this step, the Agitator is again activated.

For details see also the dedicated User Manual for SPME Technique, which is part of the 'SPME Option Kit'.

The PAL SPME Fiber Conditioning Station has the PAL Firmware Object name 'NdlHeater' and is located in the class 'Injectors.' The Object 'NdlHeater' must be positioned in the COMBI PAL System. Follow the instructions described in the COMBI PAL Operating Manual, Chapter 7, Object Positioning.

Path:

Menu | Setup | Objects | Injector | NdlHeatr

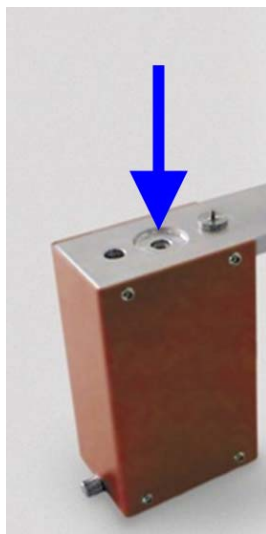


Figure 6. Teaching Position for SPME Conditioning Station.

Use the rear well with the larger diameter as the teaching point.

The lower needle guide, preferably with a red magnetic ring installed, must be positioned in the rear well of the SPME Fiber Conditioning station. The metallic plate of the injection unit needle guide (bridge) will be positioned on the knob of the gas valve. From this point, turn the Z-axis 2.0 mm farther down.

note

The motor current for the injection unit used for teaching is not high enough to press down the spring-loaded valve. Position the lower needle guide (bridge) exactly on the knob of the valve. Changing the Z-axis value manually by 2.0 mm further down then defines the correct position for automated routine. The 'Needle Penetr' value has been optimized by CTC Analytics, it is not necessary to change this value.

3.3. Cycle Composer Control

The SPME Fiber Conditioning Station Option can be used in standalone mode or software controlled, see also the SPME User Manual.

The SPME Fiber Conditioning Station Option can be operated together with the PAL control software Cycle Composer or the Cycle Editor for ICC interpretation in another data handling software. Make sure the hard- and software requirements meet those of your installation before operating the SPME Fiber Conditioning Station.

(See section E 'Installation' point 1.2 Hardware Requirements, and 1.3 Software Requirements.)

3.4. Installation of Macros for Cycle Composer

In order to use the PAL SPME Fiber Conditioning Station in an automated manner for the SPME technique, no dedicated Cycle Composer macro is required. The conditioning station is only a tool to clean the fiber. The SPME method, either as operated from the local terminal (standalone) or software controlled, such as by Cycle Composer, makes use of the conditioning station if required by the application. The activation of its use is a SPME method parameter, selectable by the user.

If the SPME technique is to be used via software control, Cycle Composer or certain data handling systems that control the PAL using Cycle Editor for PAL interpretation, then it will be necessary to load the corresponding macros for the SPME technique – not for the conditioning station as such.

In case the SPME macro(s) has (have) not been added yet, install the software as described in the 'PAL Cycle Composer Software Manual' and add the SPME macros, located in the COMBI PAL folder. The provided macros for SPME are installed with this option. To verify this step, navigate in Explorer to the Cycle Composer folder. This is usually installed in the following path:

C:\Program Files\PAL\Cycle Composer

F. PAL SPME Fiber Conditioning Station Operation

1. Operation and Temperature Setting

1.1. Temperature Setting

The SPME Fiber Conditioning Station is controlled directly by the COMBI PAL firmware. Use the 'Utilities' section to set the desired temperature for fiber conditioning, 'Standby Temp'.

Path:

Menu | Utilities | Injector | NdlHeatr

1.2. Gas Flow Setting

The gas flow is regulated by the Flush Gas Pressure Regulator. The setting of the secondary regulator is the same as for Headspace Analysis: approx. 0.5 bar.

The Fiber Conditioning Station has an internal restriction built-in. The typical flow rate yields approx. 6 mL/min if only the rear position is used. If both positions are used (front and rear), the flow is divided approximately in half.

The gas flow for the two positions is pneumatically separated. Both channels can be used independently of each other.

note

The restriction is a frit installed in the SwageLok union at the conditioning station housing (connection to the gas tube). In case of blockage, clean the frit with an intense flow of gas from the reverse side or wash in an ultrasonic bath with an appropriate solvent. As a good starting point, an aqueous mixture of an alcohol is recommended for washing.

1.3. SPME Fiber Conditioning Station Operation

After completing the installation, it is necessary to restart the PAL system. With this warm up, the COMBI PAL software recognizes and references the SPME Fiber Conditioning Station.

The local SPME Cycle (operated standalone) contains the parameter 'Fiber Bakeout'. If this value is selected (time > 0), the COMBI PAL System Injection Unit will go to the conditioning station after the timer for 'Desorb Time' has reached zero. The fiber will be exposed for the length of time remaining for the 'GC-Runtime'.

A time setting of zero for 'Fiber Bakeout' tells the system to automatically ignore the use of the SPME Fiber Conditioning Station.

Software controlled operation behaves the same way. Depending on the macro used, the same or similar method variable names are used.

note

The automated cycle will go to the conditioning station after the first injection. The conditioning station is ignored by the software before the first injection.

The injection unit needle guide will 'sit' on the spring loaded gas valve. At this point gas flow is activated. Leaving this position will close the gas valve. Gas consumption is reduced to a minimum.

note**Safety Warning:**

Do not use hydrogen or any other explosive gases or gas mixtures for the SPME Fiber Conditioning Station. Recommended gases are helium or nitrogen.

1.4. Using the Front Position to Condition a Spare Fiber

The front position is designed to condition a spare SPME Fiber. The fiber is manually placed into the hole and the fiber is brought to the exposed position. If this second position is used, open the needle valve for gas flow in the second channel (front). The needle valve is the metallic knob in front of the conditioning station.

note

After the spare fiber has been removed from the front position, it is necessary to close the needle valve in front of the conditioning station (metallic knob). Letting gas flow through this open, second channel would reduce the gas flow in the channel for the automated conditioning to a minimum. This could result in damage to the fiber, as it is not protected by inert gas.

G. Appendices

1. Definitions

Job Queue

A Job Queue is a list of sample processing Jobs. Jobs are executed in the order displayed on the JOB QUEUE menu screens. New Jobs may be added to the queue while samples are being processed.

Job

A Job contains the information needed by the PAL to process multiple samples by the same processing steps. The elements of a Job are a Method and a Tray that define the location of the samples to be processed. For identification, Jobs are automatically numbered from 01 to 99 and then restart with 01 when they are added to the Job Queue.

Cycle

A Cycle consists of the specific operations necessary to process one sample. The Cycle operations are repeated for each sample within a Job. Cycles are designed for specific applications.

Method

A Method defines how the samples are processed. The elements of a Method are a Cycle, a Syringe and a Parameter List. Methods have names with up to eight characters and can be edited, copied, and deleted.

Method Parameters

Method Parameters are associated with the Cycle operations. User-assigned Parameter values define how a processing operation is performed. A zero Parameter value will disable a Cycle operation. Cycle Parameters are application-specific.

Tray Holder

A Tray Holder can hold one or more trays. Each Tray Holder has a reference position (X-, Y-, Z-coordinates) that defines its location.

Tray

A Tray holds multiple samples. Trays are defined by designating the Tray Type (see below) and the Tray Holder. Tray names are used to identify the sample source within a PAL Job.

Tray Type

A Tray Type defines the pattern and sampling sequence of sample locations within a Tray.

Stack

A Stack is a particular type of Tray Holder that is designed to hold micro-plates. A six-drawer Stack holds 12 standard micro-plates, two in each drawer. A three-drawer Stack holds six deep-well micro-plates, two in each drawer.

PAL Object Manager

Software to load a PAL Object List to an instrument if a Module (hardware module) has been added to the PAL System. In a special mode Object Manager can also be used to create and maintain Object Lists.

PAL Object List

If a PAL Module (hardware) is added to an instrument, several Objects have to be loaded into the firmware. These Objects are collected in an Object List and stored in a file with the extension '*.pol'.

Object Lists are delivered together with Object Manager Software and are grouped into folders for the different kinds of Modules (e.g. Syringes, Tray Holders, Valve Drives). The name of an Object List starts with the Module part number with variants added (e.g. first or second Stack). The name of the root folder includes the revision which is dependent on the firmware version (e.g. 'Object Lists Rev. K' for firmware 2.X and 3.X).

Objects

Objects are data structures describing the properties of physical modules. Certain modules (e.g. a Stack) require several objects.

Object Class

Each Object belongs to an Object Class (e.g. Syringes, Trays, Injectors). The Object Class defines the Items of an Object.

Object Item

An Object contains several Items which can be numerical values with a physical unit (e.g. X-, Y-, Z-Position, Penetration, Syringe Scale Length, Syringe Volume) or references to other objects. Note that the term 'Parameter' is reserved for 'ATOM Parameter' (PAL Firmware commands to be used for a PAL Cycle or Macro).

Module

PAL hardware module, either part of a standard PAL configuration (e.g. COMBI PAL, HTS PAL) or an optional addition (e.g. Cooled Stack, MALDI Tool, Dilutor).

The term 'Module' is intentionally used to differentiate from 'Object', which is reserved for the PAL Firmware Object.

2. Naming Convention

This section recommends the standard naming convention. Following this convention will allow the Setup to be pre-configured for certain applications, will simplify software backups and application development, and will improve technical support and training.

Tray Type	Tray Description
VT200	Vial Tray, 200 positions (10 x 20) For 7 mm micro-vials 1 mL
VT98	Vial Trays, 98 positions (7 x 14) For 12 mm vials 2 mL
VT78	Vial Tray, 78 positions (6 x 13) For 7 mm micro-vials, 1 mL (opposite side of 98 positions Tray)
VT54	Vial Tray, 54 positions (6 x 9) For 12 mm vials, 2 mL
VT21	Vial Tray, 21 Positions (7 x 14) For 12 mm vials, 2 mL
VT32-10	Vial Tray, 32 positions (4 x 8) For 23 mm headspace vials, 10 mL
VT32-20	Vial Tray, 32 positions (4 x 8) For 23 mm headspace vials, 20 mL

Table 2. Naming Convention

3. Spare Parts

Part No.	Description
PAL Zub-SPME	SPME FibCond short Bracket, 14 mm incl. Screws and serrated washers.
PAL FibCondTube	Gas tube FibCond to Pressure Regulator incl. SwageLok fittings and flow restrictor.

note

Fibers for the SPME technique are solely sold by Supelco which is today part of Sigma-Aldrich. For details see following web page:

[www. sigmaaldrich.com](http://www.sigmaaldrich.com)

Application notes for the SPME technique are also provided by Supelco. Check the web page: www.sigmaaldrich.com/spme to request the 'SPME Application CD'.