Addendum to PAL User Manual
PAL ITEX-2 Option (ITEX: In-Tube-Extraction [1])
Installation and Operation

Printing History

Edition 1 April 2009
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Original Instructions

[1] EPO patent granted. Patent number: EPO 1697736

A. Safety Information

Declaration of Conformity

See the Declaration of Conformity sheet enclosed with the instrument.

General Considerations

Any changes or modifications to this unit not expressly approved by the party responsible for compliance could nullify 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 Hazard

Every analytical instrument features specific hazards. 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.

Safety Warning:
Do not touch the surface of the Trap Heater during operation. The surface temperature can reach a potentially harmful level when operated at a high desorbing or cleaning temperatures.
The Trap surface can reach approximately 270 °C if operated at the maximum trap temperature of 350 °C.

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 Safety Symbols

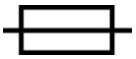
Symbol	Description
	Caution, or refer to User Manual
	Caution, Risk of Needle-Stick Puncture
	Caution, Hot Surface or High Temperature
	Danger of crushing to fingers and hands
	Laser Warning, Barcode Reader
	Biological Hazard
	Direct Current
	Alternating Current
	Protective Conductor Terminal, Ground
	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.

B. Table of Contents

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C. List of Figures and Tables

D. How to Use this Manual

note

*This manual covers the COMBI PAL and the PAL COMBI-**xt** systems. Issues specific to the PAL COMBI-**xt** model have been emphasized accordingly.*

The major sections of this Addendum are:

- Safety Information
- PAL ITEX-2 Option Installation
- PAL ITEX-2 Option Operation
- Explanations and Description of the ITEX Technique
- Appendices

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

The Appendices provide information on PAL ITEX-2 options and spare parts. A performance test to demonstrate the enrichment of the analyte is described.

note

The COMBI PAL must be installed and set up properly before following the PAL ITEX-2 Option operating instructions.

E. PAL ITEX-2 Option Installation

1. General System Overview

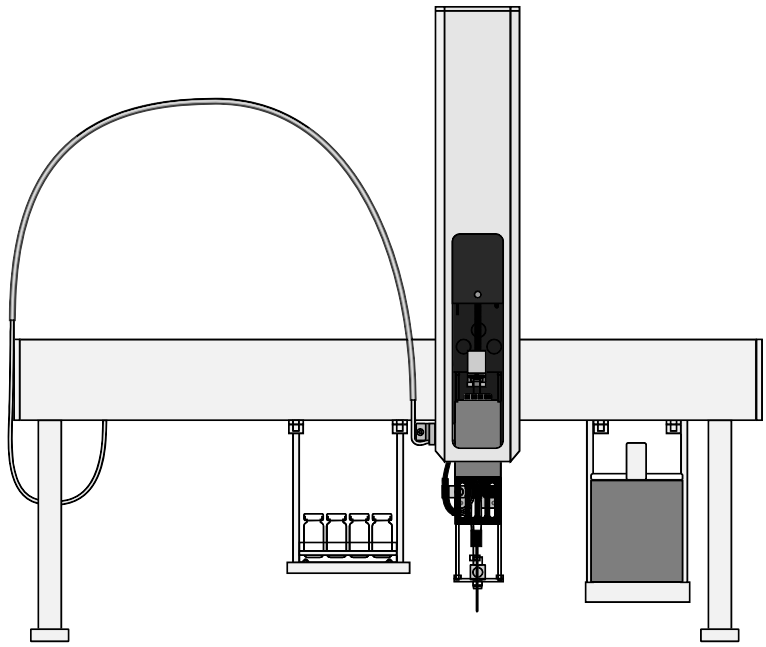

Figure 1. PAL ITEX-2 Option for COMBI PAL System, X-Axis Length 80 cm.

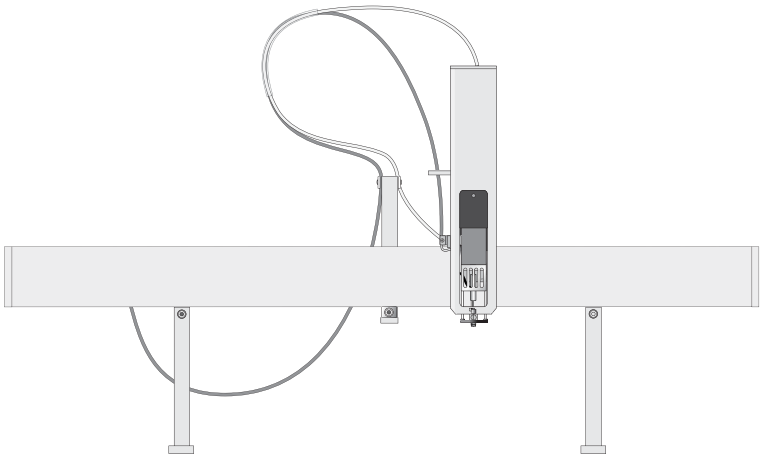

Figure 2. PAL ITEX-2 Option for PAL LHX System, X-Axis Length 120 cm

The PAL ITEX-2 Option for the PALLHX System requires a fixation for the electrical cable and the flush gas tube. See Figure 2.
When used with the 120 cm X-Axis, the swivel-mounted tubing may collapse into a bow form. Without the proper fixation and guiding, there is the possibility that it will catch on a module.
The installation is described in detail, see Section E, Point 2.2 'Installation PAL LHX System with X-Axis Length 120 cm'.

Introduction to the ITEX Technique

The PAL ITEX-2 Option is an add-on module for the PAL and PAL-xt Systems to extend the range of headspace techniques. ITEX can be considered an automated dynamic headspace technique. In classical headspace technique, one sampling is done per vial. In this device, analytes are adsorbed (loaded) on a sorbent trap contained within a side-hole needle attached to a gas-tight syringe. Following this loading procedure, analytes are thermally desorbed and led directly into the GC injection port, then analyzed by the required technique, mainly using GC-MS detection.

Loading takes place by continuously moving the headspace syringe plunger up and down, passing the headspace gas phase that exists above a sample through the sorbent trap inside the needle numerous times.

Dynamic headspace extraction increases the concentration of analyte in order to extend the range of detection as compared to conventional static headspace techniques. Required levels of detection can be reached by applying more pumping (up and down) cycles. To reach the detection limit in the range of a ppm to ppb concentration, three to twenty strokes are typically applied and, for the ppt-level of concentration, 40 to 60 pumping strokes may be necessary.

The PAL ITEX-2 Option consists of a dedicated gas-tight syringe with an adapter for a replaceable side-hole needle and a heated syringe cartridge. The side-hole needle includes an integrated trap (standard trap material is Tenax TA) and a trap/desorber heater.

1.1. Specifications

Part number:	PAL ITEX-2Option
Description:	PAL ITEX-2 Option for COMBI PAL (80 cm X-Axis)
	PAL ITEX-2Opt-LHX PAL ITEX-2 Option for LHX System (120 cm X-Axis)
Headspace Syringe:	PNo.: SyrC ITEX-2-1.3, Volume 1.3 mL*
Syringe Heater:	PNo.: MSH 05-01; Syringe ID#: 25 Temperature range: 30-150 °C
Trap Heater:	PNo.: ITEX-2 HT Description: ITEX-2 Trap Heating Unit (X-Axis 80 cm) PNo.: ITEX-2 HT-LHX Description: ITEX-2 Trap Heating Unit (X-Axis 120 cm) Temperature range: 30 – 350 °C (requires AUX interface connection, standard is AUX2)

note

**Nominal volume: 1.3 mL.*
Syringe Maximum Scale Length: 27 mm = 1.125 mL actual syringe volume.

ITEX-2 Trap

- Material: Stainless steel, deactivated by SilTek®
- Needle: Injection needle gauge 23 (O.D. 0.63 mm)
Point style 5 (side-hole)
Injection needle length: 41 mm
- Trap: Upper part of needle 3.20 mm O.D.
Standard trap material: Tenax TA
80/100 mesh, fill volume 44 mg
Upper temperature limit: 350 °C
(Other trap material is available; see section 'Appendix, Spare Parts'.)
- Gas Flow: 4 mL/min +/- 1 mL/min
Restrictor for pressure regulator outlet is provided.

Net Weight:	Approx. 210 g (total weight including trap heating cable)
Dimension:	Height: 160 mm Width: 78 mm (total width including side adapter of cartridge)

PAL Firmware:	PAL System: Version 2.5.2 or higher PAL-*xt* System: Version 4.1.X or higher
Software Control:	Using Cycle Composer Software Version 1.6.0 (current version 1.6.0; any lower version is suitable as long as serial communication is selected), or PAL ICC (Cycle Editor).

1.2. Hardware Requirements

The PAL ITEX-2 option can be used with any PAL System equipped for static headspace technique, e.g. the COMBI PAL or PAL COMBI-*xt* Systems. The revision level of the PAL Injection Unit must be Rev. E or higher, a slot at the left side is required. Example: PNo.: MZ 02-00 Revision E.

1.3. Software Requirements

The PAL ITEX-2 Option requires PAL Firmware 2.5.2 (or higher) for the PAL System and firmware version 4.1.X (or higher) for the PAL-*xt* System. It can be controlled using PAL control software, the Cycle Composer, or any CDS (chromatography data system) software that controls the PAL System, including those using the Cycle Editor for PAL ICC interpretation (e.g. Analyst, ChemStation, Empower, EZChrom, MassLynx, Xcalibur).

For details, see Point 3: PAL Firmware and Firmware Object Installation for PAL ITEX-2 Option.

1.4 PAL ITEX Option compared to PAL ITEX-2 OPTION

The PAL ITEX Option, as used until now, is the name of the first generation module designated for the ITEX technique. It was based on a 2.5 mL headspace syringe with a standard syringe cartridge and a trap/injection needle combination. This earlier version was longer (or higher) in dimension than the current models. The resulting total length required a special PAL Injection Unit, PNo. MZ 010-00E. This earlier setup had certain limitations. For example, a change-over to other injection techniques, such as liquid injection or SPME, was not possible without changing the PAL Injection unit back to the standard device.
With the new ITEX-2 Option, it is possible to use standard PAL equipment for the ITEX application. A special PAL Injection Unit is no longer required. Any PAL System installed in the field, fulfilling the hardware and software requirements, is compatible with the new ITEX-2 Option. The hardware and software requirements are described above, in Points 1.2 and 1.3.

The first-generation ITEX Option parts are not compatible with those of the new ITEX-2 Option. All product number details for spare parts are provided in the Appendix.

2. Installation

2.1 PAL System with Axis Length 80cm

2.1.1 Unpacking the Components

The PAL ITEX-2 Option is shipped in one box. Check for the following items (some parts are preassembled):

1. 1 pc Headspace Syringe Heater, ID # 25, for syringe with 1.3 mL volume;
2. 1 pc Plunger Holder;
3. 1 pc Fixation Clamp for Syringe Heater;
4. 2 pcs ITEX-2 Trap, standard packing Tenax TA, 80/100 mesh;
5. 1 pc ITEX-2 Syringe 1.3 mL, complete, including Plunger;
6. 1 pc Replacement Plunger for ITEX-2 Syringe;
7. 1 pc ITEX-2 Trap Heater;
8. 2 pcs End Cover, left side; one each for PAL or PAL-***xt*** System
9. 1 pc Flow Restrictor for Pressure Regulator;
10. 1 pc CD-ROM containing:
    - Object Manager 'ITEX-2' Lists
    - Cycle Composer macros and ICC cycles.

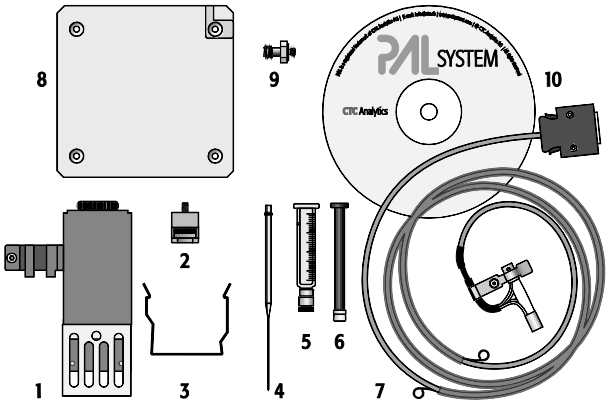

Figure 2. PAL ITEX-2 Option Components.

2.1.2. Installing the PAL ITEX-2 Option (PAL X-Axis Length 80 cm)

note *Turn off the power to the PAL System before beginning the installation process.*

1. Replace the X-axis end cover on the left side of the unit with the cover provided, which permits fixing the guide wire of the electrical cable. Connect the guide wire as shown in detail in Figure 4 below.

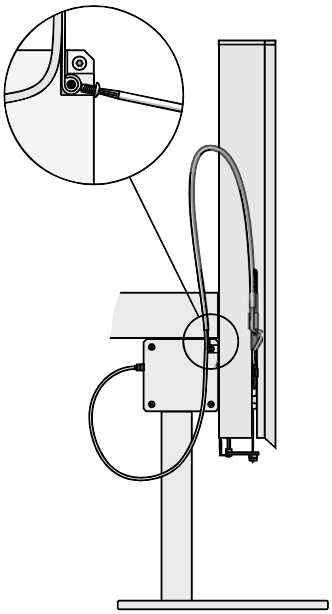

Figure 4. Connecting the Guide Wire to the X-axis End Cover.

2.2 PAL LHX System with X-Axis Length 120 cm

2.2.1. Unpacking the Components

The 'PAL ITEX-2 Option for PAL LHX System' is shipped in one box. Check for the following items (some parts are preassembled):
The ITEX-2 Standard parts as shown in Figure 5 are described above (point 2.1)

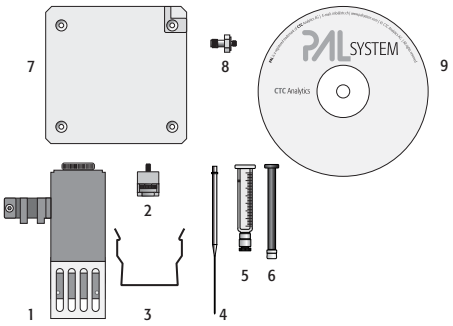

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Figure 5. ITEX-2 Option Standard Components

The dedicated parts for the ITEX-2 PAL LHX Option are shown in Figure 6.

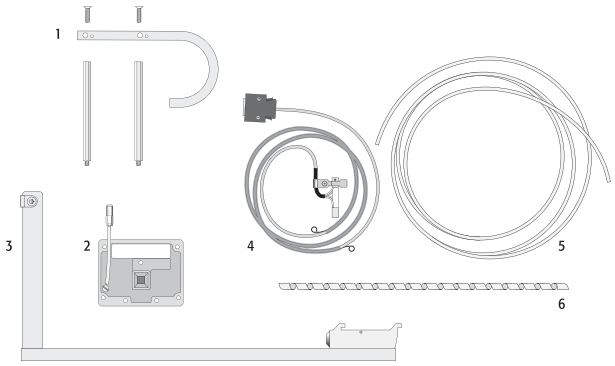

Figure 6. ITEX-2 Option for PAL LHX

Following additional parts are required for the ITEX-2 Option in combination with the PAL LHX System (X-Axis 120 cm):

1. 1 pc Hoop Guard with 2 pcs. Distance Holders;
2. 1 pc PAL Y-Axis Front Cover (Hoop Guard and Front Cover is assembled);
3. 1 pc Holder for Fixation of electrical cable and flush gas tube;
4. 1 pc ITEX-2 Trap Heater for PALLHX System;
5. 1 pc Flush Gas Tube;
6. 1 pc Cable Strap

2.2.2. Installing the PAL ITEX-2 Option for PALLHX System

note — *Turn off the power to the PAL System before beginning the installation process.*

1. If the Injection Unit is already installed, remove it from the Y-Axis.

2. Remove the original Y-Axis Front Cover and replace it with the supplied Front Cover from this kit.

3. Mount the Hoop Guard (guide arm) on top of the Front Cover as shown in Figure 7.

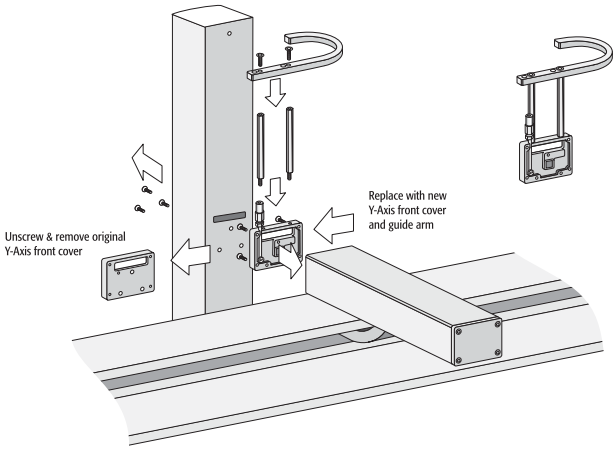

Figure 7. Replacing original Y-Axis Front Cover

4. Reinstall the Z-Axis.

5. Install the Holder for Fixation of the electrical cable and the flush gas tube.

   Important: It is mandatory to install the holder in the middle of the X-Axis to ensure that the cable and tube form a controlled arc. See Figure 9.

6. Connect the Flush Gas Tube to the gas adapter at the Y-Axis Front Cover. Guide the tube to the top of the injection unit; use the tube adapter for fixation as shown in Figure 8.
   Continue guiding the tube to the top of the Holder, use the provided tube adapter for fixation and finally connect the tube to the elbow adapter at the far left hand side of the X-Axis (looking from behind the unit).

note — *Use the natural bow of the tube to avoid any restraint when the Z-Axis is moved.*

7. Connect the electrical cable to the ITEX cartridge as described in the Addendum ITEX-2 Option. If the original cable is already connected, replace it with the provided one. The lengths of the cables are different. Guide the cable through the Hoop Guard and connect it to the top part of the Holder as shown in Figure 8.

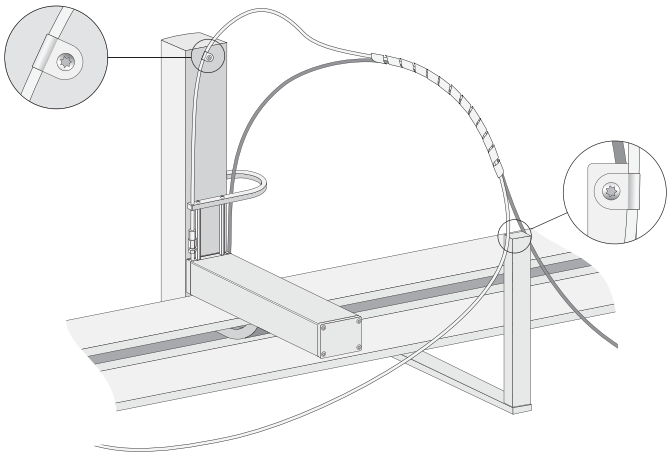

Figure 8. Connection of the Electrical Cable and Flush Gas Tube

8. Use the provided Cable Strap to hold the Flush Gas Tube and electrical Cable together as shown in Figure 8.

note

Cover the Flush Gas Tube and Cable with the cable strap only to the extent shown in Figure 6. Do not completely cover the Tube and Cable.
A certain freedom for movement is required.

9. Complete the connections of the electrical cable to the interface 'AUX2'.
   See Figures 9 and 10 illustrating the final connections.

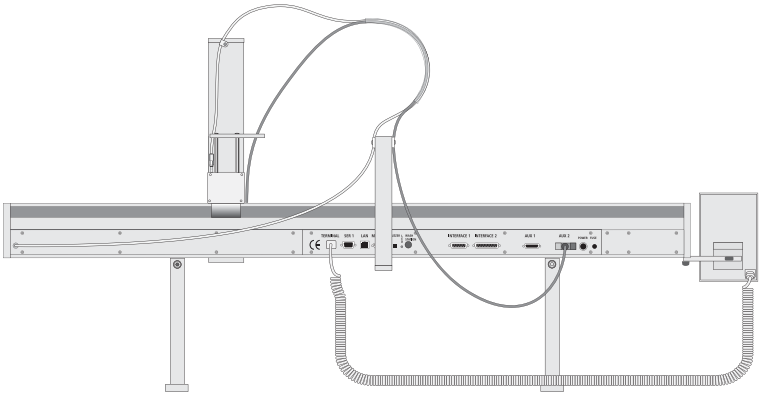

Figure 9. Final Connections of the Flush Gas Tube and Electrical Cable

Figure 8 shows the setup and connections in side view.

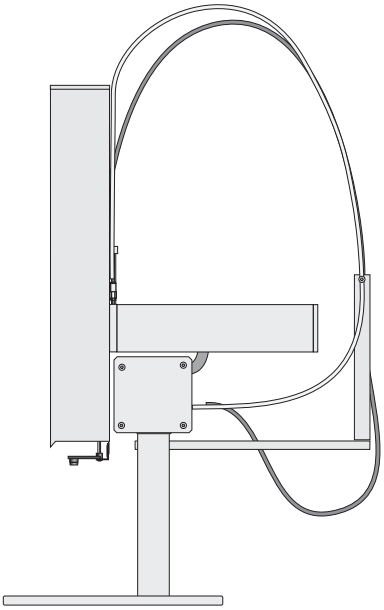

Figure 10. Side View of Setup and Connections

10. Check the form and stability of the bow formed during movement of the Injection Unit.
    Move the arm manually back and forth and check that the tube or electrical cable does not collide or catch with any part of the PAL System. See Figure 11 for illustration.

    This figure shows clearly why it is important to install the Holder in the middle of the X-Axis.

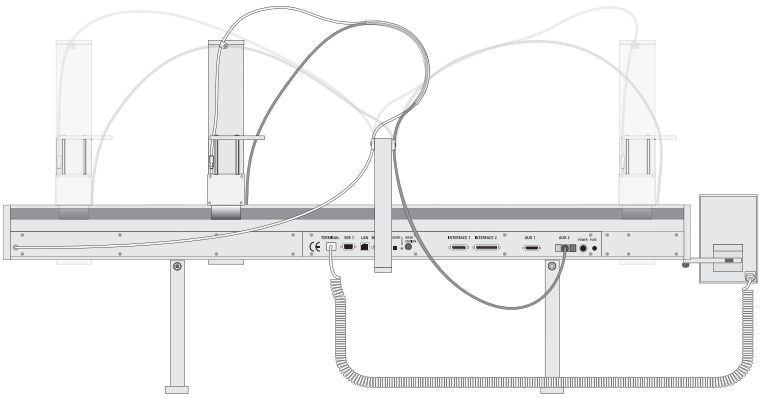

Figure 11. Checking Movement of Injection Unit

11. This completes the installation and setup of the ITEX-2 Option for the PAL LHX System. All other steps for installation and operation of the ITEX-2 Option are described in the Addendum for 'ITEX-2 Option'.

2.3 Installing the ITEX-2 Heated Cartridge

note

Installing the ITEX-2 Heated Cartridge is common for both version of ITEX-2 Option for X-axis length 80 and 120 cm.

1. The heated cartridge is prepared such that the electrical cable for the Trap Heater is attached to the side adapter of the heated cartridge. Check the assembly by comparing the details as shown in Figure 12.

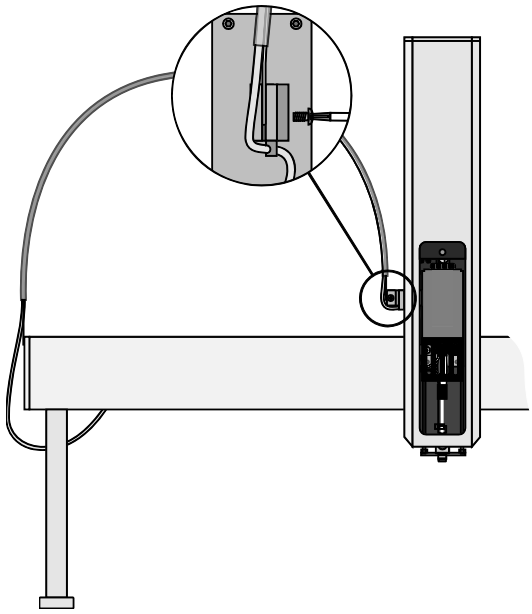

Figure 12. Guide Wire connected to the Heated Cartridge.

2. Assemble the headspace syringe and the trap/needle, as shown below.

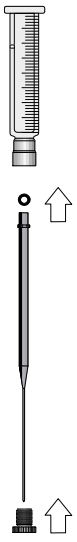

Figure 13. ITEX-2 Headspace Syringe Assembly with Trap/Needle.

3. Insert the assembled ITEX-2 syringe into the Syringe heater as shown below. For enhanced access, remove the grid from the syringe heater, as shown in Figure 14.

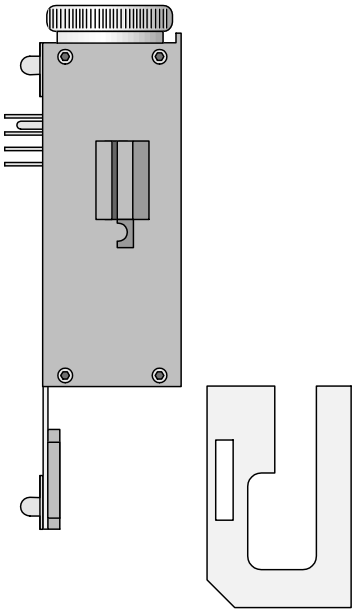

Figure 14. Grid removed from the Syringe Heater Unit.

The heated cartridge is prepared such that the electrical cable for the Trap Heater is fixed by a U-bolt to the Trap itself. See Figure 15.

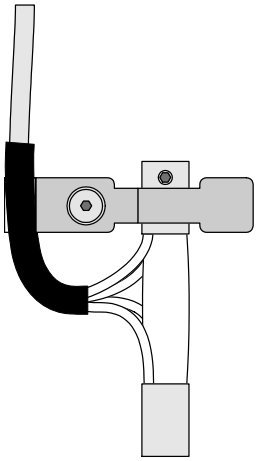

Figure 15. Prepared Cartridge with Trap Heater.

Thread the needle into the trap heater as shown in Figures 16 and 17.
Reinstall the grid onto the syringe heater to protect from high temperatures.

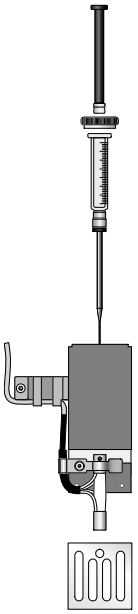

Figure 16. Assembled ITEX-2 Syringe inserted into Syringe Heater.

note

Install an O-Ring in the syringe glass barrel to protect the flange (not shown in Figure 8).

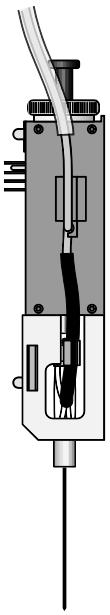

Figure 17. ITEX-2 Syringe and Heater Assembly.

In its final position, the trap needle fits into the conical shape of the trap heater, as shown in Figure 18. Tighten the set screw to fix the heater onto the trap.

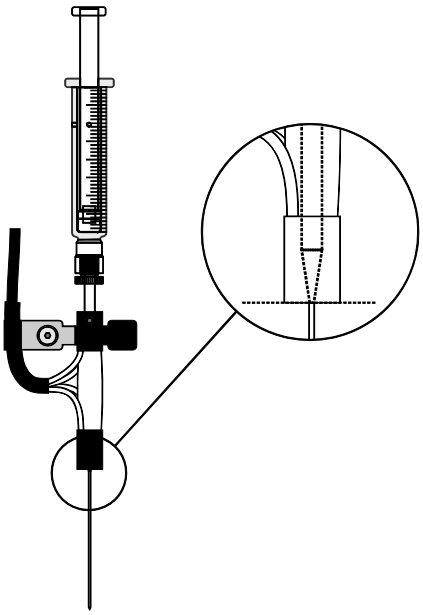

Figure 18. Positioning the Trap Heater onto the Trap/Needle.

Please note that the U-bolt fixation has to be installed with an angle as shown in Figure 91 (viewed from the bottom of the cartridge).

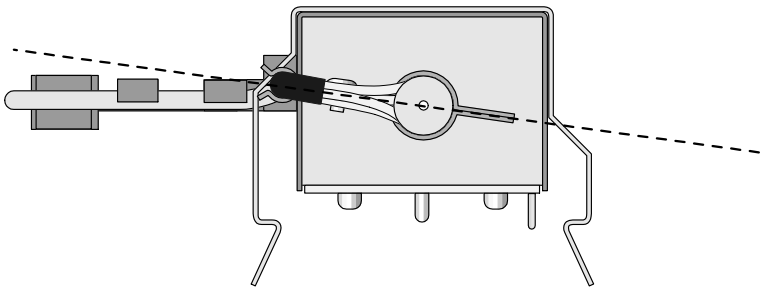

Figure 19. Bottom View of Cartridge with angled Trap Fixation.

The explanation for this specific angled position is illustrated in Figure 20. The adapter size for the long tension cord at the Z-axis syringe slider prevents a square fixation of the U-bolt on the Trap Heater.

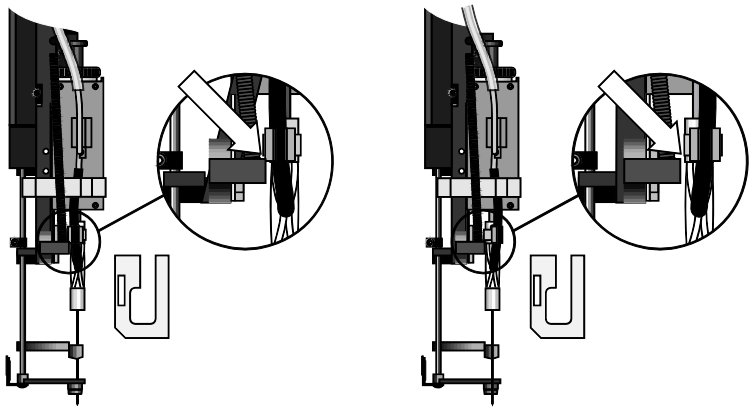

Figure 20. Illustrating the Space required in-between the Adapter and the U-bolt fixation.

5. Attach the plunger holder to the syringe plunger. The final assembly of the ITEX-2 headspace syringe, inserted in the syringe heater and trap heater, is shown in Figure 21.

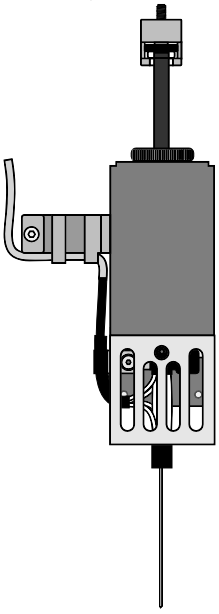

Figure 21. Final Assembly of ITEX-2 Syringe, Heater and Trap.

If a syringe or a Trap Needle has to be replaced and is inside the grid, use the hole provided to access the set screw with a screw driver from the front without removing the grid.
Be aware that the grid itself can be hot at the surface. Be careful when touching any parts around the ITEX2 Headspace Syringe Heater.

note

Safety Warning:
Do not touch the surface of the Trap Heater during operation. The surface temperature can reach a potentially harmful level when operated at high desorbing or cleaning temperatures.
The Trap surface can reach approximately 270 °C if operated at the maximum trap temperature of 350 °C.

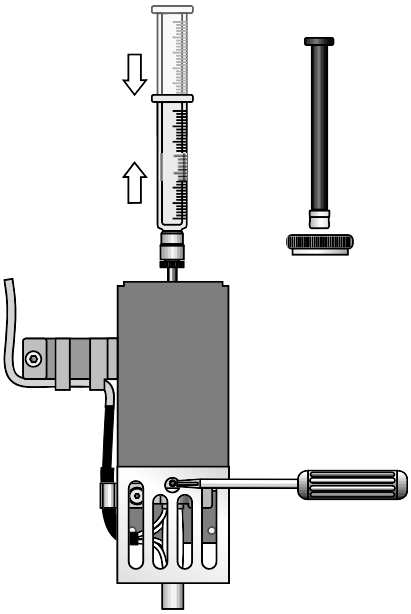

Figure 22. Accessing the Set Screw to replace a Syringe or Trap Needle.

2.4. Inserting the PAL ITEX-2 Syringe Assembly into the Injection Unit

1. Move the PAL Injection Unit manually to the side to allow free movement of the syringe slider. Lower the syringe slider as shown in Figure 11 in order to gain access to install the syringe/heater assembly.
   Lift the lower needle guide, sliding it into the upper needle guide. Insert the syringe needle tip and match the magnetic pins of the syringe holder with their corresponding positions on the syringe slider.
   Let the lower needle guide return slowly and make sure the needle tip does not catch on the guide. See Figures 23 and 24.

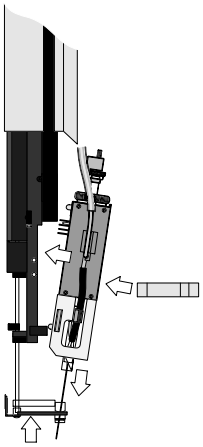

Figure 23.

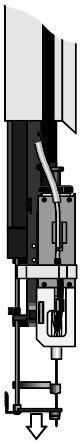

Figure 24.

2. Press the heated cartridge firmly against the Z-axis slider to ensure that the Syringe heater Unit engages. Move the plunger up (plunger holder) until the thread of the screw catches the thread of the plunger bushing. Tighten the screw to fix the plunger holder and attach the holding clamp around the cartridge. See Figures 25 and 26.

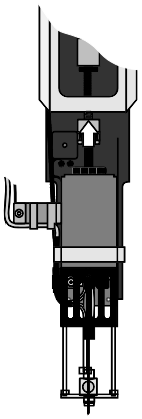

Figure 25.

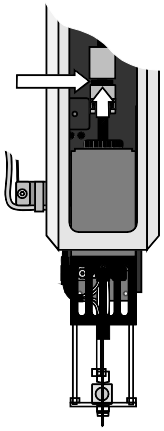

Figure 26.

Figures 15 to 18. Inserting the ITEX-2 Cartridge into the Z-axis.

3. Connect the electrical cable from the heated trap to the interface 'AUX2' at the back side of the unit, as shown in Figure 27.

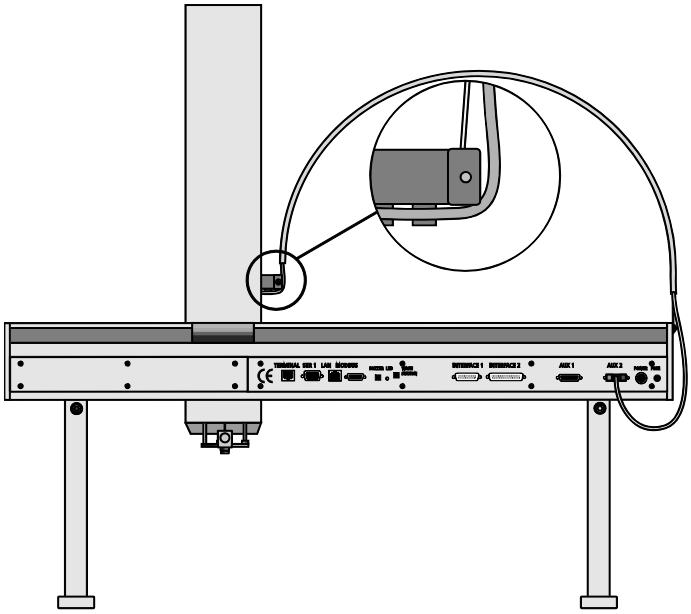

Figure 27. Heated Trap connected to the AUX2 Interface.

note

*Figure 19 shows the electrical connection to the new electronic board for the PAL-**xt** version, APR Control-**xt**.*
The 'AUX2' interface is also used for the PAL System.

2.5. Flush Gas Considerations

A gas flow level greater than 6 mL/min can destroy the bed of the trap, which will result in non-reproducible trapping efficiency.

2.5.1. Flush Gas Pressure Settings

A gas flow restrictor union for the pressure regulator is provided with the ITEX-2 Option kit, item #9 in Figure 2. Remove the original adapter from the pressure regulator body and replace it with the ITEX-2 adapter with the built-in flow restriction frit. This restriction allows a flow of approximately 7 mL/min helium or approximately 5 mL/min nitrogen at a pressure setting of 0.5 bar (at room temperature). See Figure 28.

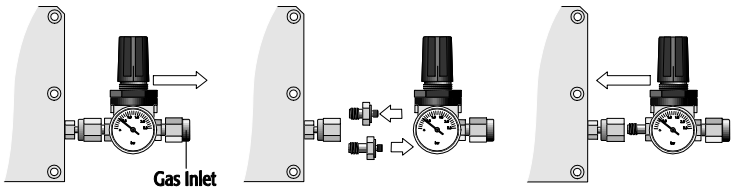

Figure 28. Replacing the Original Adapter with the Flow-restricted ITEX-2 Adapter at the Pressure Regulator.

note

The original adapter removed from the pressure regulator must be stored for future use. If the PAL system is to be used for static headspace technique, the flow setting of approx. 4 mL/min may be too low to flush the standard headspace syringe. A maximum flush volume of two or three fold per minute is the lower limit. For MHE (Multiple Headspace Extraction) technique, the required pressure of 1.0 to 1.5 bars cannot be reached with the ITEX-2 flow–restricted adapter installed. For static headspace and MHE techniques, it is recommended that the original adapter without any flow restriction be re-installed.

2.5.2. Gas Flow Adjustment

If your system was previously used for static headspace techniques, you must adjust the gas flow to ITEX technique requirements. If the original adapter for Headspace (without flow restriction) is in place, it is likely that the pressure setting of 0.5 bar needs to be lowered.

Adjust the gas flow to approximately 4 mL/min +/- 1 mL/min.

This flow of gas will not damage the bed of the trap, but the entire system is flushed about three times per minute. Standard flush time is two minutes. This means that the ITEX-2 device is flushed six times with gas for cleaning purposes.

note *The Cycle Composer macro 'ITEX2_GasFlowCheck' can be used to measure the actual gas flow through the trap/needle. See the section 'Operation'.*

Gas flow adjustment without an installed restrictor can be done as described below. This procedure is necessary in case the ITEX Option is installed, if the restriction frit was not provided or if it has been lost or is clogged.

1. Release the pressure entirely from the PAL pressure regulator. Use a liquid (e.g. n-hexane) to check whether any bubbles form.

2. Turn the pressure on very slowly and check for bubble formation in the liquid again. If a good stream of bubbles is visible, measure the gas flow.

2.5.3. Flush Gas Type

For gas chromatographic technique, three different carrier gases are typically used: nitrogen, helium, and hydrogen.
Nitrogen and helium are inert gases and therefore can be safely used as flush gases for the ITEX technique.

Because of a slightly lower solubility in water, the preferred gas for headspace analysis is helium. Whenever possible, helium should be used for the ITEX technique.
If helium is used as a carrier gas, the carrier gas line can be divided and teed-up for a carrier and a flush gas. Activating the flush gas valve should not result in irregularities in the GC detector baseline.

note ***Safety Warning:*** *Hydrogen, widely used as a carrier gas for GC capillary techniques, is a flammable, explosive gas. For safety reasons do not use hydrogen as a flush gas for the ITEX technique.*

2.5.4. Flush Gas Quality

The minimum quality level is grade 55 (99.9995 % purity). Grade 59 (99.9999 % purity) or Grade 60 (99.99990 % purity) are preferable.

Good laboratory practice is to install a charcoal and a moisture filter to ensure that a high quality of gas is supplied to the GC system.

note *For further details on flush gas and contamination, see section G, 'Principles'.*

note *If the installation procedure has been carried out in the order described, it is necessary to power up the PAL System at this point. Powering up executes a 'warm-start'. During this procedure the software references the interfaces and the ITEX-2 trap heater is identified at 'AUX2'.*

3. PAL Firmware and Firmware Object Installation for PAL ITEX-2 Option

3.1. PAL Firmware Installation for PAL ITEX-2 Option

The PAL ITEX-2 Option requires PAL Firmware version 2.5.2 or higher for the PAL System and version 4.1.X or higher for the PAL-***xt*** System. Firmware version 3.0.X is not compatible for the ITEX-2 technique.

The ITEX-2 Option can be controlled either by a Cycle Composer macro or by any CDS (chromatography data system) software that controls the PAL or PAL-***xt*** System using for example Cycle Composer macros or PAL ICC cycles (e.g. Analyst, ChemStation, Empower, EZChrom, MassLynx, Xcalibur).

note

*The PAL-**xt** System requires PAL Firmware 4.1.X. (or higher), the PAL-**xt** Object List Revision B or higher the control board, APR Control-**xt**.*

3.2. PAL Firmware Object List for PAL ITEX-2 Option

A CD-ROM is provided together with the PAL ITEX-2 Option, 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 J or higher.
- PAL-***xt*** system : PAL-***xt*** Object List Revision B or higher

note

In case an upgrade is made from ITEX to ITEX-2, it is advisable to first delete the PAL Firmware Objects of the previous version 'ITEX Option' from the firmware. This applies mainly to the object in the class Injector 'Desorber'. The objects in the class Out-Signal, 'CarGas' and 'SpltPurg' can be deleted as well; see below.

If an earlier version of the Object Manager Lists are used, copy the 'ITEX-2 Option' folder to the Object Lists folder which has been installed with the PAL Object Manager. This software is usually installed in the following path:

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

Start PAL Object Manager software. The 'ITEX-2 Option' 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.
Select 'ITEX-2 Option' on the left side. Select 'PAL ITEX2Opt' on the right side and click 'Send selected Object Lists to PAL'.
The example shown in Figure 29 is the PAL System Object Lists.

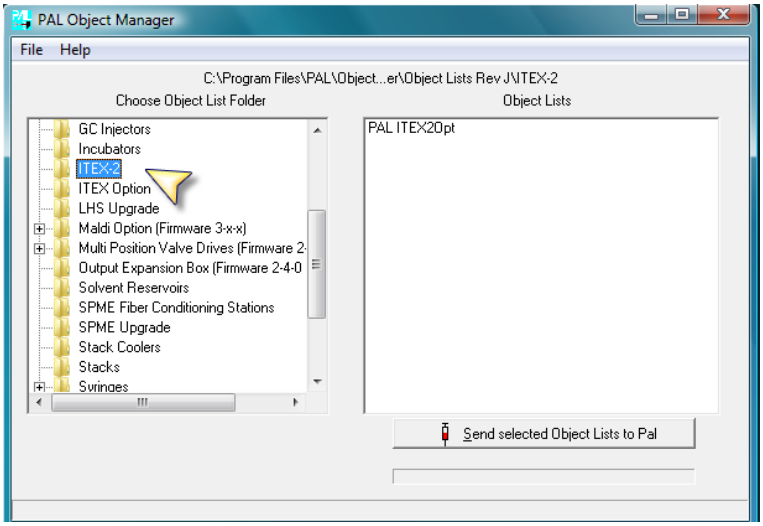

Figure 29. PAL Object Manager, loading ITEX-2 Firmware Objects.

The following PAL Firmware Objects are loaded:

- Class Syringe: - 1.3ml-IT
  (1.3 mL ITEX-2 Syringe)
- Class Injectors: - Desorb2
  (Heater 'H2Desorb' assigned)
- Class Heaters: - H2Desorb
  (temperature range 30 to 350 °C)
  - HSyr)
  (Heater for ITEX 1.3 mL Syringe,
  temperature range 30 to 150 °C)
- Class Motors_Drives: - MPlgHS
  (Plunger Motor drive for Headspace syringe)
- Class Vial: - Fiber Exp
  (position for 'Blocking Needle Guide')

note

PAL Firmware Objects of the classes 'Out Signals' ('CarGas' and 'SpltPurg') have been included with the first generation, the PAL ITEX Option. During the developmental phase of the ITEX technique, it became evident that these options were no longer necessary for the technique.

3.3. Installation of Macros for Cycle Composer or Cycles for PAL ICC interpretation

Every PAL ITEX-2 Option is shipped with a CD-ROM containing the various macros or cycles as described below.
Copy these files to the corresponding folder as described below.

3.3.1. Installation of Macros for Cycle Composer

Navigate in Explorer to the Cycle Composer folder. This is usually installed in the following path:

C:\Program Files\PAL\Cycle Composer

If you wish to create a separate Method Folder for ITEX-2, copy the folder 'ITEX-2' from the CD to the Cycle Composer folder.
In the Cycle Composer use menu 'File/Options/Choose Method Folder...' select the 'ITEX-2' folder as the Method Folder.
If you wish to add the ITEX-2 macros to an existing method folder, just copy the macro (*.pma) and method (*.pme) files from the 'ITEX-2' folder on the CD to the appropriate method folder.

Use the provided test methods to run a short test to verify the operation of the instrument.

3.3.2. Installation of ICC-Cycles

Navigate in Explorer to the Cycle Editor folder and/or the corresponding folder of the application within an integrated system.
If you wish to add the ITEX-2 cycles to an existing method folder, just copy the cycles (*.cyx) files from the 'ITEX-2' folder on the CD to the application folder.

4. Adjusting Object Reference Positions

The PAL ITEX-2 Option does not require a special reference position.
The needle tip should be at exactly the same position as if a liquid or headspace syringe were being used.
However, it is good practice to check the individual positions by using the PAL 'Utilities' functions as described in the COMBI PAL User Manual.

F. PAL ITEX-2 Option Operation

The PAL ITEX-2 Option can only be used together with the PAL Cycle Composer control software or with the Cycle Editor, assuming the intent is to achieve ICC interpretation using another data handling software system. Verify that your system meets the basic hardware and software requirements before operating the ITEX-2 Option.
(See points 1.2 Hardware Requirements and 1.3 Software Requirements.)

The PAL ITEX-2 Option is shipped with a CD-ROM containing various cycles (macros) for the ITEX application.
Copy these macros to your Cycle Composer method folder or corresponding folder for application within an integrated system, as described under Section E, point 3.3 'Installation of Macros for Cycle Composer or Cycles for ICC Interpretation'.

note *To operate the PAL ITEX-2 Option using Cycle Composer software, it is assumed that the user is familiar with the PAL Cycle Composer control software.*

note *Safety Warning:*
Do not touch the surface of the Trap Heater during operation. The surface temperature can reach potentially harmful levels when operated at high desorbing or cleaning temperatures.
The Trap surface can reach approximately 270 °C if operated at the maximum trap temperature of 350 °C.

note *If a new Trap is installed the material has to be conditioned first. Use the macro or cycle 'ITEX2_CleanTrap' for the cleaning step and verify if a blank baseline can be reached with the specific GC-configuration to be used for the analysis.*

1. PAL ITEX-2 Option Cycle Composer Macros or ICC-Cycles

1.1. Macro or Cycle 'ITEX2'

Standard Macro/Cycle for ITEX-2 application for medium and low volatility compounds.
At the moment of thermal desorption the plunger is in the uppermost position. After reaching the Trap desorption temperature, injection into the GC inlet system follows. For medium or low volatile analytes, the injection speed can be set low, typically 30 to 250 µL/s.
This macro should not be used for the analysis of highly volatile compounds. At the moment of thermal desorption, there could be partial loss of compounds. See the macro description below.

Macro Description	Macro Variable
The syringe Agitator and ITEX-2 Trap are heated to the required temperature.	Syringe Temperature Trap Starting Temperature Incubation Temperature Desorption Temperature
The vial is transported to the Agitator and equilibrium is established.	Incubation Time Agitator Speed
The ITEX-2 Trap/Syringe needle is inserted into the vial 18 mm. With pumping cycles as defined in the counter 'Extraction Strokes' the plunger is moved up and down to load the analyte (sample) into the trap.	Extraction Strokes Extraction Volume Extraction Speed Pullup Delay
The plunger is moved up by the volume as defined by the injection volume in the Sample List.	SL.Volume Pullup Delay *Remark to 'SL.Volume': This is not a Macro Variable.*
The 'Ready-Signal' from the GC system for the PAL system is awaited at this stage. After receipt of the signal, the next step is executed.	
The needle is removed from the vial and the ITEX-2 device is moved to the GC-Injector, the inlet is selectable. The needle is inserted. Fixed needle penetration 36 mm into GC injector.	Inject to; INJECTOR
The ITEX-2 Trap is heated to the selected desorption temperature.	Desorption Temperature
The sample volume is injected into the GC system. A start signal 'Injected' is sent out to start the GC system. The ITEX-2 device is moved back to 'Home' Position.	Desorption Speed (= injection speed) *Remark:* *Post Injection delay is fixed to 20 seconds.*
The Trap temperature is lowered to 70 +/- 2 °C. The treated sample vial is brought back to its original position in the Tray Holder.	*Remark: This step is required to avoid possible contamination of the trap/syringe system in case a substance was only partially desorbed. Pulling up the plunger at the desorption temperature for gas flush cleaning could bring the substance into the syringe part of the device.*
The Trap cleaning step is executed with the 'Flush' command. At the same time the temperature for the Trap is raised to the selected cleaning temperature.	Trap Cleaning Temperature Trap Cleaning Time (= Flush Time) *Remark: 'Cleaning Temperature' can be higher than 'Desorption Temperature'. Observe the maximum operating temperature for the trap material.*

(*Table 2 continues*)

After stopping the gas flow, the Trap temperature is lowered to the starting conditions for the next run.	Trap Starting Temperature
With the variable 'GC Ready Time' the time is calculated for the next sample to be placed into the Agitator, and to establish equilibrium. This optimization of cycle time is useful in case of a longer sample preparation time compared to the GC run time.	GC Ready Time *Remark: Time from GC start to GC ready.*

Table 2. Description of the Macro 'ITEX2'.

1.2. Macro or Cycle 'ITEX2_Volatile'

Macro/Cycle used for very volatile compounds.
Examples: Vinyl chloride, dichloroethane, etc.
The difference between this and the standard macro is that at the moment of thermal desorption, the plunger moves up slowly to keep volatile compounds in the system, not permitting them to diffuse from the trap to the GC inlet port.

Macro Description	Macro Variable
The syringe Agitator and ITEX-2 Trap are heated to the required temperature.	Syringe Temperature Trap Starting Temperature Incubation Temperature Desorption Temperature
The vial is transported to the Agitator and equilibrium is established.	Incubation Time Agitator Speed
The ITEX-2 Trap/Syringe needle is inserted 18 mm into the vial. With pumping cycles as defined in the counter 'Extraction Strokes', the plunger is moved up and down to load the analyte (sample) into the trap.	Extraction Strokes Extraction Volume Extraction Speed Pullup Delay
The plunger is moved up by **50 % of the volume** defined as the injection volume in the Sample List.	SL.Volume * 0.5 Pullup Delay *Remark to 'SL.Volume': This is not a macro variable.*
The 'Ready-Signal' from the GC system for the PAL system is awaited at this stage. After receipt, the next step is executed.	
The needle is removed from the vial and the ITEX-2 device is moved to the GC-Injector, the inlet is selectable. The needle is inserted. Fixed Needle penetration of 36 mm into GC injector.	Inject to; INJECTOR
The trap heater is started to achieve the selected 'Desorption Temperature'. At the same time the plunger is moved up slowly to reach the total volume (injection volume) as defined in the Sample List.	Desorption Temperature SL.Volume * 0.5
The sample volume is injected into the GC system. A start signal 'Injected' is sent out to start the GC system. The ITEX-2 device is moved back to 'Home' Position.	Desorption Speed (= injection speed) *Remark:* *Post Injection delay is fixed to 20 seconds.*

(*Table 3 continues*)

The Trap temperature is lowered to 70 +/- 2 °C. The treated sample vial is brought back to its original position in the Tray Holder.	*Remark: This step is required to avoid possible contamination of the trap/syringe system in case a substance is only partially desorbed. Pulling up the plunger at the desorption temperature for gas flush cleaning, could bring the substance into the syringe part of the device.*
The Trap cleaning step is executed with the 'Flush' command. At the same time the temperature for the Trap is raised to the selected cleaning temperature.	Trap Cleaning Temperature Trap Cleaning Time (= Flush Time) *Remark: 'Cleaning Temperature' can be higher than 'Desorption Temperature'. Observe the maximum operating temperature for the trap material.*
After stopping the gas flow, the Trap temperature is lowered to the starting conditions for the next run.	Trap Starting Temperature
With the variable 'GC Ready Time' the time for the next sample to be placed in the Agitator, and to establish equilibrium is calculated. This optimization of cycle time is useful in case of a longer sample preparation time compared to the GC run time.	GC Ready Time *Remark: Time from GC start to GC ready.*

Table 3. Description of the Macro 'ITEX2_Volatile'.

1.3. Macro or Cycle 'ITEX2_TestHS'

Macro/Cycle used to run ITEX-2 test procedure.
One plunger stroke to simulate static headspace without a concentration step.

Macro Description	Macro Variable
This macro is identical with the macro 'ITEX2'. The exception is the fixed counter step 'Extraction Strokes', see below.	Remark: Unused variables for this simplified macro are eliminated. The remaining variables are identical as used for 'ITEX2'.
The counter 'Extraction Strokes' is replaced by a fixed one-fill stroke to simulate static headspace technique.	*Remark: Extraction Strokes = 1. Counter is not available.*
Waiting for a timer tolerance is not necessary at the end of the cycle for a single test.	Variable 'GC Ready Time' is eliminated.

Table 4. Description of the Macro 'ITEX2_TestHS'.

1.4. Macro or Cycle 'ITEX2_TestITEX'

Macro/Cycle used to run ITEX-2 test procedure.
Ten plunger cycles (up and down plunger movements) to demonstrate the concentration factor for the trap.

Macro Description	Macro Variable
This macro is identical with the macro 'ITEX2'. The exception is the fixed counter step 'Extraction Strokes', see below.	Macro variables are identical with variables from Macro 'ITEX2'.
The counter 'Extraction Strokes' is replaced by a fixed number of ten pumping cycles.	Extraction Strokes = 10. Variable eliminated. Fixed number.
Waiting for a timer tolerance is not necessary at the end of the cycle for a single test.	Variable 'GC Ready Time' is eliminated.

Table 5. Description of the Macro 'ITEX2_TestITEX'.

1.5. Macro or Cycle 'ITEX2_CheckGasFlow'

Macro/Cycle used to check and adjust gas flow through the Trap.

This Macro/Cycle provides the option of blocking the needle guide for the sake of convenience at the moment of gas flow measurement. If this option is used, the position 'FiberExp' (PAL Firmware class 'Vials') must be defined first. The lower needle guide of the Z-axis has to be positioned on the lid of the Agitator such that one screw of the lower needle guide sits at the edge, the red magnetic ring does not touch the Agitator, and the needle can move freely (test by manually lifting up the lower needle guide). Adjust the X-, Y-, and Z-positions accordingly. The same concept of blocking the needle guide is used for the SPME technique as well.

For details on 'Flush Gas' see the 'Installation' section, point 2.3.

At the moment when gas is flowing through the ITEX syringe/trap assembly, a flow meter can be connected to measure the gas flow.
A simple test to check if gas is flowing is to fill a vial with solvent and insert the needle into the liquid. A steady stream of gas bubbles should be observed. The selected solvent should be an organic solvent with a lower boiling point, such as n-hexane. Gas bubbles do not form readily in water.

note *Insert the needle/trap tip into the vial only after the gas flush has been started. Avoid sucking liquid into the trap device.*

Macro Description	Macro Variable
This macro provides the option to block the needle guide of the Z-axis for better and more convenient access to the trap needle at the moment of flow measurement. With a counter, a decision is made to enable needle guide blocking or to leave this step out.	NdlGuide Blocking YES_1 NO_0
If '1' (Yes) is selected, the Z-axis moves to the position 'FiberExp'. A needle penetration of 25 mm is executed. The needle guide is blocked, and the needle is freely exposed for 25 mm.	
If '0' (No) is selected, the next step is executed. The lower needle guide has to be lifted up manually at the moment the needle is inserted into the vial containing the liquid.	
The Injection Unit is moved to position 'Home'.	
The gas flush is executed, the plunger is moved up to allow a gas flow through the Syringe/Trap assembly. A timer is started to allow enough time to insert the needle into a vial containing liquid to check the flow of gas or to apply a flow meter to measure the gas flow. We recommended a flow rate of 4+/- 1 mL/min.	Flush Time
After stopping the flush gas, a timer is started to allow removing equipment from the unit before the needle guide is released.	Time to Finish Test
After the timer has run back to zero, the gas flush mode is stopped and the needle guide is released again.	

Table 6. Description of the Macro 'ITEX2_CheckGasFlow'.

1.6. Macro or Cycle 'ITEX2_CleanTrap'

Macro/Cycle used to clean or condition the Trap.

The Trap can be conditioned or cleaned at a selectable temperature and time. For the cool down process, the choice is open to use a continuous gas flush to protect the trap material or, if the Trap should be cooled in a passive way, not to have gas protection.

Macro Description	Macro Variable
From the 'Home' position, the gas flush mode is activated.	
Temperature and Time for Trap cleaning or conditioning are selectable.	Trap Cleaning Temperature Trap Cleaning Time
After reaching the time limit for cleaning, the Desorber heater is set to 'Cool_Off Temperature'. This temperature can be a higher temperature than ambient, set such that the trap material can be cooled-off to a level where gas protection can be turned off.	Cool_Off Temperature *Note: Temperature 'Accuracy' = 0. Temperature is not checked within a range, next step is executed immediately.*
The user has the choice to cool-off the trap in an active or passive manner by selecting or not selecting the flush mode again. We recommend activating the flush mode again to speed up the cooling process and protect the trap material with an inert gas stream.	Cool_Off with Flush YES_1 NO_0
If '1' (YES) has been selected, the flush mode is activated again. Gas flow is provided until 'Cool_Off Temperature' is reached within +/- 5 °C.	Cool_Off Temperature
If '2' (NO) is selected, the Trap material will cool-off passively. The variable 'Cool_Off Time' is obsolete.	
A second temperature setting allows cool-down to ambient (or near to ambient) without a gas stream. Possible condensation can be eliminated.	Standby Temperature (Accuracy +/- 2 °C.)

Table 7. Description of the Macro 'ITEX2_CleanTrap'.

2. PAL ITEX-2 Cycle Step-by-Step

Step1.
The sample vial is placed into the Agitator and the equilibrium is established (time and temperature controlled).

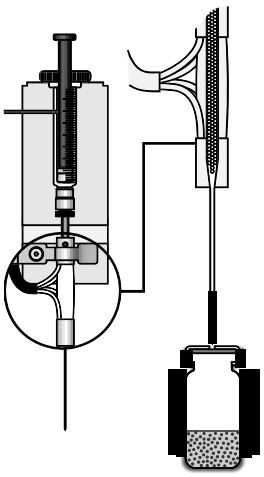

Figure 30.

Step 2.
The sample is loaded on the trap by continuously pumping the syringe plunger up and down (dynamic headspace).

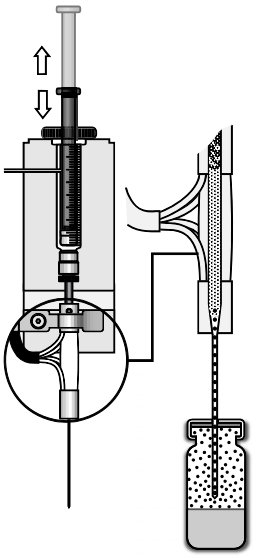

Figure 31.

Step 3.
Thermal desorption of analyte directly into the GC inlet system.

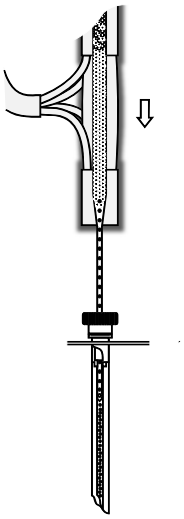

Figure 32.

Step 4.
The Trap material is cleaned with inert gas, Trap Cleaning Time/Flush Time.
The Trap temperature can be raised for the cleaning step.

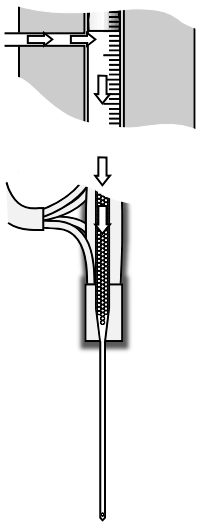

Figure 33.

Figures 30 to 33. Step-by-Step Illustration of an ITEX-2 Cycle.

3. Quantification of ITEX Technique

The quantification of results obtained from the ITEX technique is based on the same principles as for static headspace or any other sample enrichment technique such as SPME, for example.

In the case where analyte(s) is (are) combined with matrix, it is important to be able to have the calibration standard in the same matrix environment. Since the ITEX technique could be considered a dynamic headspace technique, the lack of identical or missing matrix could be compensated for by performing an exhaustive extraction. Be aware that such a compromise must be validated very carefully.

4. Available Trap Material

The standard trap material shipped with a PAL ITEX-2 Option is:

- Trap material: Tenax TA
- Mesh size: 80/100
- Fill volume: 44 mg
- Bed length: Approx. 36 mm
- Positioning inserts: Silanized glass wool
- Upper temperature limit: 350 °C
- Trap/Needle material: stainless steel, inside wall neutral deactivated for analysis of, e.g. organo-sulfur compounds

See also Appendices; Section 3, 'PAL ITEX Option Spare Parts', point 3.3. 'Ordering Information for Custom Traps'.

5. Cleaning the Trap

note

Safety Warning:
Do not touch the surface of the Trap Heater during operation. The surface temperature can reach potentially harmful levels when operated at high desorbing or cleaning temperatures.
The Trap surface can reach approx. 270 °C if operated at the maximum trap temperature of 350 °C.

It is advisable to clean a trap initially to eliminate all volatile contaminants. Use the provided Cycle Composer macro 'ITEX2_CleanTrap'.
A practical check is to run a blank injection, monitor the detector baseline to check for sufficient cleanness of the trap.

note

Observe the maximum operating temperature for the trap material.

6. Trap Heating-up Rate and Cooling-off Time

In the second generation of the ITEX apparatus, improvements have been made to the heating-up rate. The cool-off time, however, became slightly longer.

Parameter	ITEX-2 Option	ITEX Option
Heating-up rate: 30 to 250 °C	25 to 30 s	approx. 30 s
Cooling-off time (passive): 250 to 70 °C 250 to 35 °C	 approx. 180 s approx. 360 s	 approx. 120 s approx. 300 s

Table 8. Trap Heating-up and Cooling-off Times.

7. Performing ITEX-2 Test

This section describes a short test procedure to demonstrate the enrichment of the analyte in the trap as compared to that obtained using static headspace technique.

7.1. ITEX-2 Test Sample

The ITEX-2 Test sample is composed of:

- 1,2-Dichlorobenzene CAS# 95-50-1
- 1,3-Dichlorobenzene CAS# 541-73-1
- 1,4-Dichlorobenzene CAS# 10646-7

The concentration is 100 µg/mL each, dissolved in Methanol (solvent P&T quality).

The test sample is available from Restek – Chromatography Products, order number: RT-564309. For details, see Appendix 4.

These compounds were selected because they exhibit good response with many GC detectors, including the flame ionization detector (FID), the electron capture detector (ECD) and the mass selective detector (MSD). Furthermore, separation of the three isomers is possible with all common GC column phases in the range of non-polar to polar phases.
An equal response with all three isomers is expected.

The test consists of two experiments:

- first, running one sample simulating static headspace (one sampling stroke only);
- second, using the enrichment technique by applying 10 pumping cycles (plunger up and down movements).

7.2. Preparation of Test Standard Solution

The test or calibration standard solution as received (concentration 100 µg/mL of each compound) is diluted 1:10 with methanol.
This secondary standard solution is used for the tests as described below.

note — *Ensure that the methanol used for the dilution step is of high purity.*

7.2.1. Selective GC Detector, e.g. MSD and ECD

Add 1 µL of the secondary standard solution directly into a 20 mL Headspace vial and seal the vial immediately. Use a microcap device or a GC syringe to dose the aliquot of the standard solution.

- Secondary standard solution: Concentration 10 µg/mL
- 1 µL aliquot = 10 ng/ µL

7.2.2. GC Detector FID, TCD or any other Non-selective Device

Add 10 µL of the secondary standard solution directly into a 20 mL Headspace vial and seal the vial immediately. Use a microcap device or a GC syringe to dose the aliquot of the standard solution.

- Secondary standard solution: Concentration 10 µg/mL
- 10 µL aliquot = 100 ng/ µL

If the calibration standard is used only for the less sensitive GC Detectors, such as FID or TCD, then the primary standard with a concentration of 100 µg/mL can be used directly. Add a 1 µL aliquot of the primary standard solution into a 20 mL headspace vial.

7.3. Instrument Preparation for the ITEX Test

7.3.1. Cycle Composer Macros or ICC- Cycles

The Cycle Composer macros or ICC-cycles used for the ITEX-2 tests are:

- Static Headspace: ITEX2_TestHS
- ITEX enrichment: ITEX2_TestITEX

7.3.2. GC Method Parameters

- GC column: nearly any capillary column can be used to separate the compounds in the test sample
- GC Injector temperature: 200 to 250°C, split less for 1 min.
- Detector: settings depend on the detector used
- GC oven temperature: 40 ^{0}C, hold 1 minute, 10 ^{0}C/min up to 150 ^{0}C, hold 2 minutes

7.3.3. PAL Injection Parameters

Parameter	Value	Parameter	Value
Syringe Temperature	80 °C	Pullup Delay	5 s
Trap Starting Temperature	35 °C	Desorption Temperature	250 °C
Incubation Temperature	80 °C	Inject to	GC Inj
Incubation Time	5:00 min	Desorption Speed	100 µL/sec
Agitator Speed	500 rpm	Trap Cleaning Temperature	250 °C
Extraction Volume	1000 µL	Trap Cleaning Time	2:00 min
Extraction Strokes	10	GC Ready Time	10:00 min
Extraction Speed	100 µL/sec	Injection Volume (1)	500 µL

(1) Injection Volume is specified in Sample List

Table 9. PAL Injection Parameters used for the Performance Test.

An example of the test is given in Appendix 4 'ITEX-2 Performance Test'.

note

For the syringe temperature setting, a compromise has to be made. One has to avoid condensation in the syringe, but if the temperature is too high the 'hot' gas stream could have a desorption effect when pressed through the bed of the trap during the plunger down movement.

As higher the Incubation Temperature is selected as higher is the pressure in the vial if a solvent (e.g. water) is part of the matrix.

The ITEX-2 performance test can be done with settings such as Syringe Temperature 65 °C and Incubation Temperature 60 °C. These lower temperature settings are always a good starting point.

G. Principles of ITEX-2 Enrichment

1. Introduction

As described above, ITEX is a powerful sample enrichment technique to be used in conjunction with gas chromatographic separation and detection. As designed, the ITEX sample preparation device lends itself only to sampling from the headspace phase.
Sampling from a water phase will coat or possibly even saturate the trap material with a liquid film. By the process of thermal desorption this liquid would be brought onto the GC column and would influence the chromatographic separation, perhaps even to the point of losing the separation. Therefore, the removal of water (humidity) should be a first step – outside of the GC inlet system. The danger here is loss of volatile compounds at the dry-off stage. This stepwise desorption is not provided with the available ITEX-2 device.

For this reason, the only recommended application for the ITEX sample enrichment technique is for the gas phase, the headspace in the vial.

The ITEX technique can be used with various sample concentration levels. The only parameter that varies is the counter for the plunger pumping cycles. For example, a few pumping cycles can easily reach the ppb level, which is often good enough for detection and quantification of the target compounds. The most common application in the ppt range is the determination of organic impurities in drinking water. To date, the recommended technique for drinking water analysis has been the 'Purge & Trap' technique. It has been proven experimentally by various groups that the same analysis can be done with the ITEX sample enrichment technique. The ITEX technique makes it possible to reach the ppt level, even with very volatile compounds such as vinyl chloride. The ease of handling of this technique and the rapid replacement of a trap or a syringe, when needed, are the main advantages of this enrichment technique.
The compatibility of ITEX with the GC capillary technique is another important point to consider. The resulting gas flow from the thermal desorption step can be controlled by the plunger movement. The gas flow into the GC inlet port must be within the range required by the capillary column and detection system.

2. Instrumentation for the ITEX Technique

In the early stage of experimental work with the ITEX technique, it was thought that all gas sources (carrier gas, septum purge) should be shut off and the split outlet closed during the thermal desorption process. There was concern that too much gas would pass into the GC column. However, it was experimentally proven that these precautions are not necessary.

2.1. GC-Injector Type or Mode

The recommended GC instrumentation is a split injector with a split liner to provide enough volume to receive the gas phase from the ITEX thermal desorption step. Whether the split is closed or open depends greatly on the particular application and must be defined case by case.

2.2. GC-Injector Septum

The standard injector septum can be used; however, ITEX needle gauge 23 is appropriate for the Merlin adapter.
If a Merlin adapter is used, be aware that the seal can leak if used with a cold injector (cold injector to start with, ramping the temperature up at the start of injection; see text below).

2.3. GC-Injector Temperature

As a standard starting condition, injection into a hot injector is recommended, typically 200 °C isothermal. Check the peak shape and reproducibility and vary the parameters if necessary to improve the results.

For extremely volatile compounds, such as vinyl chloride or 1,2-dichlorethane, different experiments have been done, such as starting with a cool injector and ramping the temperature up quickly. Another approach has been to cryofocus the sample before the actual transfer to the GC column.
This provides some ideas on how to adapt if problems arise with a specific application. The final method selected depends mainly on the available GC equipment and application.

2.4. GC-Injector EFC/EPC

One important point to be aware of is the combination of high column backpressure and electronic flow or pressure carrier gas control. If column backpressure is relatively high, for example 2 bars (or approx. 25 to 30 psi) and a gas plug from the ITEX-2 device is brought into the injector, the injector pressure will rise and will possibly reach the EFC/EPC limit to control the flow/pressure efficiently and accurately. Reaching a critical pressure limit for the electronic control causes the GC control to open the split until the pressure returns to a normal level. This opening of the split is not very obvious to the user; the peak shape is still good but reproducibility can be very bad, relative standard deviations of 20 to 40 % have been observed.
For the user it is easy to monitor the split flow. Connect a flow meter to the split-outlet line and monitor the split flow during the injection process.
Such high backpressures may be due, for example, to a capillary column of 0.25 mm I.D. and length of 60 to 80 meters.

If a split opening is observed, either reduce the injection speed from the ITEX-2 device, which should be done but without sacrificing the peak shape, especially as seen in early eluters, or reduce the column backpressure. Some GC manufacturers provide special EFC/EPC devices with different plumbing from the EFC/EPC to the injector, which adapts to such special cases and stabilizes the baseline as well. For further information contact your GC manufacturer representative.

2.5. GC Column Oven

No recommendation can be given for column oven temperature. This parameter is highly dependent on the application. For very highly volatile compounds, select a starting temperature of 30- to 35 °C. This low temperature permits focusing the sample at the column inlet. Cryo-cooling the oven to this low starting temperature is advised.

2.6. GC Column

The type of GC column also depends greatly on the application and detection device for the GC system. If a low starting temperature is used, check also the lower temperature limit of the GC column. Some column phases cannot be used at such low temperatures.

For highly volatile compounds, focusing at the column entrance is important. Use a greater film thickness to permit better trapping. A greater film thickness normally causes greater bleeding, which will affect the baseline and detector stability. Compromise on film thickness with the temperature range used for the column and the detection type (MSD is critical to bleeding).

2.7. Injection Speed

The injection speed depends to the volatility of the analyte. Very highly volatile compounds can be injected (thermally desorbed) at a speed of approximately 100 µL/s. Less volatile compounds can be injected at a speed of 20 to 100 µL/s. Finding the right injection speed for a specific application is based mainly on judging the peak shape. Is any distortion or even peak splitting visible?

3. Contamination of the Analytical Device

It is important to realize that the ITEX Technique is a very efficient sample enrichment technique. As described, detection levels in the ppt range can be achieved. However, each step in the handling and device setup must be considered with care with respect to any possible contamination. Contamination of the analytical device originates from several sources, as described below.

3.1. Gas Quality

If the gas quality is rather poor, impurities can be enriched in the trap and can cause interference, or so-called 'ghost peaks' in the chromatogram.

3.2. Gas Supply, Lines, etc.

Extreme precautions must be taken with respect to the gas supply, the gas lines, and the entire handling thereof. It is good practice and, for the ITEX technique, highly recommended to install an appropriate gas filter or filter set to adsorb any impurities from the gas supply system. See also the section 'Installation', point 2.3.
If in doubt, install a dedicated gas cylinder of known gas quality and check the system thoroughly. Be aware that possible contaminants may have already migrated into the GC plumbing and pressure control system.
The material of the carrier gas tube should be heat-cleaned cooper, stainless steel or a selected polymer tube, or a polymer tube which does not release softener and is non-permeable to ambient gases.
Good laboratory practice must be applied when handling the material. Ultra clean material only should be used; greasing or the like is strictly forbidden.

3.3. Gas Supply System Inside the COMBI PAL

The gas supply system inside the COMBI PAL system is outlined for static headspace technique. In general, no contamination was observed in routine work. A known exception for the static headspace technique is possible contamination from the glue of the headspace syringe. This can be observed if the syringe is operated at the highest temperature (150 °C), sample preparation time is longer than 60 minutes, and a very sensitive detection device is used, such as GC-MSD. All three listed parameters are necessary to be able to detect a possible contaminant.
The design of the ITEX-2 syringe and needle fixation are different from the standard headspace syringe, therefore the points described above do not apply to the ITEX-syringe. See also point 3.4 below.

With the high enrichment factor achieved by using the ITEX technique, there is a higher risk that contamination can occur. Experimentally, all components of the PAL internal gas supply have been tested, namely the pressure regulator, gas flush valve, gas flush tube, and all connections including the gas distribution block mounted at the end cover of the X-axis.

In case a contaminant is detected in the chromatographic and/or mass-range detection, it is advisable to install a filter between the gas inlet and the injection unit and after the built-in gas flush valve.

3.4. ITEX-2 Syringe and ITEX-2 Trap

Contamination of the ITEX syringe has not yet been observed. The needle connection is different from that used with the static headspace syringe. Contamination due to glue out-gassing is eliminated by design.

Contamination of the trap can occur during the application or due to insufficient cleaning. However, if certain rules are observed this risk can be minimized.

- Sample from the gas phase only (not from the liquid layer)
- Extend the cleaning time (flush)
- Increase the trap temperature after thermal desorption to a higher cleaning temperature. Observe the maximum temperature for the trap material.

In case the trap material has been contaminated and cannot be cleaned by applying reasonable effort, replace the trap. This only delays routine work by a few minutes.

As described above in section F, point 5 'Cleaning the Trap', is it necessary to clean or condition a newly-installed trap first.

3.5. Sample Vial – Septum – Cap

Vial, septum and the cap can be contaminated by the laboratory environment. When working in trace analysis, it is advisable to bake-out contaminants and cool down in a protected atmosphere, such as by using a desiccator.

3.6. Contamination Summary

This list of possible sources of contamination is not complete. However, it should help in identifying the main sources of contamination and gives ideas of other areas to explore when faced with this problem.

H. Appendices

1. Definition of Terms

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 restarting 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.

Objects

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

Tray Holder

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

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.

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.

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 in 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 kind 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. L' for firmware 2.X and 3.X or 'PAL-*xt* Object Lists Rev.B').

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 distinguish from 'Object', which is reserved for the PAL Firmware Object.

2. Naming Convention

This section recommends standard naming conventions for PAL ITEX-2 targets. Following these conventions will allow the PAL ITEX-2 setup to be pre-configured for certain applications. This will simplify software backups and application development, and will improve technical support and training.

Tray Type	Tray Description
VT32-20	Vial Tray, 32 positions (4 x 8) for 23 mm headspace vials, 20 mL
VT32-10	Vial Tray, 32 positions (4 x 8) for 23 mm headspace vials, 10 mL
VT21	Vial Tray, 21 positions (7 x 14) for 12 mm vials, 2 mL
VT54	Vial Tray, 54 positions (6 x 9) 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)
VT98	Vial Tray, 98 positions (7 x 14) for 12 mm vials, 2 mL
VT200	Vial Tray, 200 positions (10 x 20) for 7 mm micro-vials, 1 mL

Table 10. Naming Convention

3. PAL ITEX Option Spare Parts

3.1. PAL ITEX-2 Option Spare Parts

Part No.	Description
SyrC ITEX-2-1.3	ITEX-2 Syringe 1.3 mL with Adapter M7x0.5
ITEX-2 PLG1.3	Replacement Plunger for ITEX-2 Syringe
MSH 05-01	ITEX-2 Syringe Heater Unit
ITEX-2 HT	ITEX-2 Trap Heating Unit (COMBI PAL, X-Axis Length 80 cm))
ITEX-2 HT-LHX	ITEX-2 Trap Heating Unit for LHX PAL (X-Axis length 120 cm)
ITEX-2 TrapTXTA	ITEX-2 Trap Tenax TA 80/100 mesh
ITEX-2 TrapTXTA-3	ITEX-2 Trap Tenax TA 80/100 mesh, set of 3 pcs.

Table 11. ITEX-2 Option Spare Parts.

3.2 PAL ITEX Option Spare Parts

Parts for the first generation ITEX are NOT compatible with the ITEX-2 Option. The Trap needle for the ITEX Option has an outer diameter of 2.0 mm at the top and the ITEX-2 trap has an outer diameter of 3.2. Therefore, the Trap Heater Unit is not interchangeable.
The Syringe Heater Unit for ITEX-2 is shorter because the syringe has a volume of 1.3 mL compared to 2.5 mL from the older ITEX Option.
Use the order information as shown below for the first generation ITEX Option spare parts.

Part No.	Description
SyrC ITEX2.5	ITEX Syringe 2.5 mL with Adapter ¼"-28 UNF Fitting
PLG G2500	Replacement Plunger for ITEX Syringe 2.5 mL
MSH 02-00	Syringe Heater Unit for 2.5 mL HS Syringe
ITEX HT	ITEX Trap Heating Unit
ITEX Trap TXTA	ITEX Trap Tenax TA 80/100 mesh
ITEX TrapTXTA-3	ITEX Trap Tenax TA 80/100 mesh, set of 3 pcs.

Table 12. ITEX Option Spare Parts.

3.3. Ordering Information for Custom Traps

Tenax TA is the standard material for the ITEX-2 Trap. Other materials can be custom filled into the ITEX-2 trap (or ITEX Trap).

An exact description of the material, including mesh size and expected fill volume, must be provided. If a two-layer bed is required for the trap, the order of the layers must be specified.

After receiving the ordering information, BGB-Analytik AG will verify feasibility and, if in doubt, will contact the customer.

Minimum order quantity is three traps of the same kind.
Custom traps are handled and provided solely through:

BGB Analytik AG
Rohrmattstrasse 4
4461 Boeckten / Switzerland

Phone No.: +41 (0) 61 991 0046
Fax No.: +41 (0) 61 991 0025
E-mail: sales@bgb-analytik.com
Web: www.bgb-analytik.com

4. ITEX-2 Performance Test

4.1. Supplier and Order Information for ITEX-2 Test Sample

The test or calibration standard for ITEX-2, as described in section F, point 7 'Performing ITEX-2 Test', can be ordered directly at:

3.4.1. Supplier

- Restek – Chromatography Products: www.restek.com or

- BGB Analytik AG (address see above) or

- Any other Restek distributor.

3.4.2. Order Information:

- Article No.: RT-564309
- Description: ITEX Calibration Standard,
  o-, m-, p-Trichlobenezene 100 µg/mL each, solved in P&T Methanol.

4.2. ITEX-2 Performance Test

The purpose of the test is to verify the enrichment of the analyte(s) on the trap and to understand the technique as such. The enrichment factor can be expressed as the ratio of the simulation of the static headspace sampling compared to the ITEX trapping. A quantitative conclusion is not the aim of this test.

The chromatogram below shows an overlay of the performance test 'Static Headspace simulation' and the enrichment of the analytes using the ITEX-2 technique.

Note that the ITEX-2 enrichment step was performed 50 times, 50 'Extraction Strokes'. The test as described calls for 10 plunger strokes only. This number of Extraction Strokes is sufficient to demonstrate the enrichment technique within a reasonable time frame.

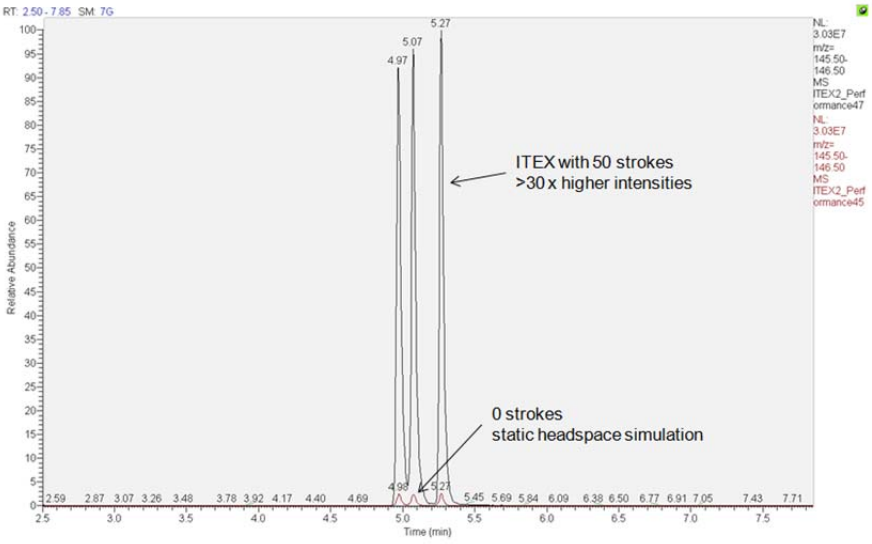

Figure 34. Test Chromatogram for Static Headspace and ITEX-2 Enrichment.

4.2.1. GC and PAL Conditions

- GC and PAL conditions as described in section F, point 7 'Performing ITEX-2 Test';

- GC Column: ZB-5MS; 30 m x 0.25 mm; Film thickness 0.25 µm;
- Flow Rate: 1.5 mL/min; Constant Flow; Helium;
- GC-MS Detection: Mass Range 50-250 m/z; Full Scan; Scan Time 0.200 s.

Acknowledgment: Data provided by:
Brechbühler AG, 8952 Schlieren ZH, Switzerland; Operator UH/TF.

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