

Sample Preparation for Gas Chromatography



GC Sample Preparation

Environmental
Drinking Water

Foods/Flavours/
Consumer Products

Forensics/Toxicology

Petrochemicals/
Polymers

Pharmaceuticals
Residual Solvents

PAL Rapid and efficient sample enrichment of volatile and semi-volatile compounds in solid, liquid and gaseous samples

PAL In-tube extraction and direct thermal desorption using proven industry standard adsorbents

PAL Syringe only concept for transparent sample handling, no sample loops, no transfer lines, no switching valves

PAL No GC injector modifications, no cryo-focussing necessary
Top mounted on GC's, saves valuable bench space

PAL Interfaces with any CombiPAL System
controlled by all major GC/GC-MS Systems



CombiPAL equipped with ITEX Option



ITEX traps containing proven industry standard adsorbents



ITEX adsorption step out of a sample vial

CTC Analytics' aim is to supply instruments to customers which make the operation of sample processing simple and transparent. In-line with today's lab requirements for productivity, CTC expanded the application range of its GC Injector System CombiPAL introducing the ITEX Option. The ITEX Option consists of an add-on module which can be used with any existing or new CombiPAL System. It performs enrichment of volatile or semi-volatile compounds during headspace analysis. A microtrap filled with adsorbent material, such as Tenax or activated charcoal is placed between the heated CombiPAL Headspace syringe and the syringe needle. Using the HS syringe as a pump, a part of the gaseous phase of the pre-conditioned sample vial is pumped repeatedly through the microtrap. This system setup allows rapid, simple and efficient extraction of volatile and semi-volatile sample compounds. To gain sensitivity simply the number of pumping strokes can be increased or several different vials containing the same sample can be extracted. During thermal desorption into the GC Injector the microtrap is rapidly flash heated and the analytes reach the GC column as a narrow band. No cryofocusing is needed to obtain sharp peaks. To prepare the next extraction, the hot trap is conditioned outside the injector with clean purge gas.

ITEX trap material examples

Carbotrap/Carbopack
 Non-porous graphitized carbon blacks (GCBs)
 Hydrophobic properties minimized sample displacement by water
 Carbosieve/Carboxen
 For very volatile compounds, e.g. Vinylchloride, Freon compounds
 Tenax TA
 Volatile and semivolatile compounds, temperature limit of 350°C

Specifications ITEX Option

Pumping Syringe Size:

2.5ml with 1/4" 28 UNF fitting

Extraction Speed:

Selectable from 10µl / sec. up to 1000µl / sec.

Extraction Strokes:

Selectable from 1 - 999

Extraction Volume:

Selectable from 250µl - 2500µl / 1 stroke

Desorption Temperature:

+5°C above ambient - 350°C selectable in 1°C increments

Desorption Speed:

1µl/sec. - 500 µl/sec.

Pumping Syringe and Trap Cleaning:

Inert gas purging, 30 sec. - 3600 min.

Heated Pumping Syringe:

+5°C above ambient - 150°C selectable in 1°C increments

Incubator Oven:

6 heated vial positions for 2ml / 10ml / 20ml vials
 +5°C above ambient - 200°C selectable in 1°C increments

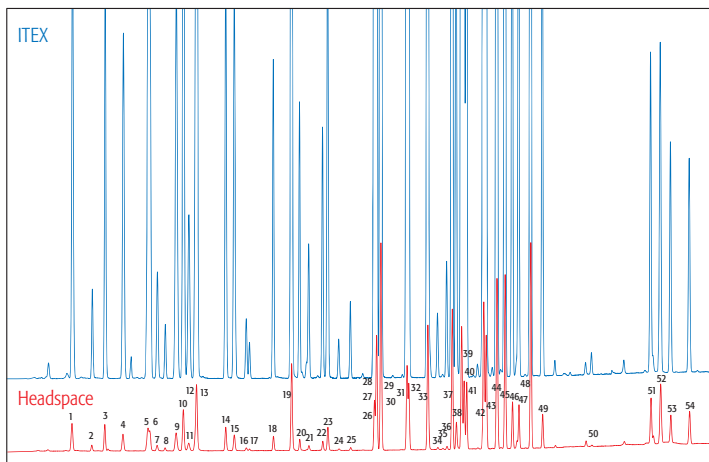
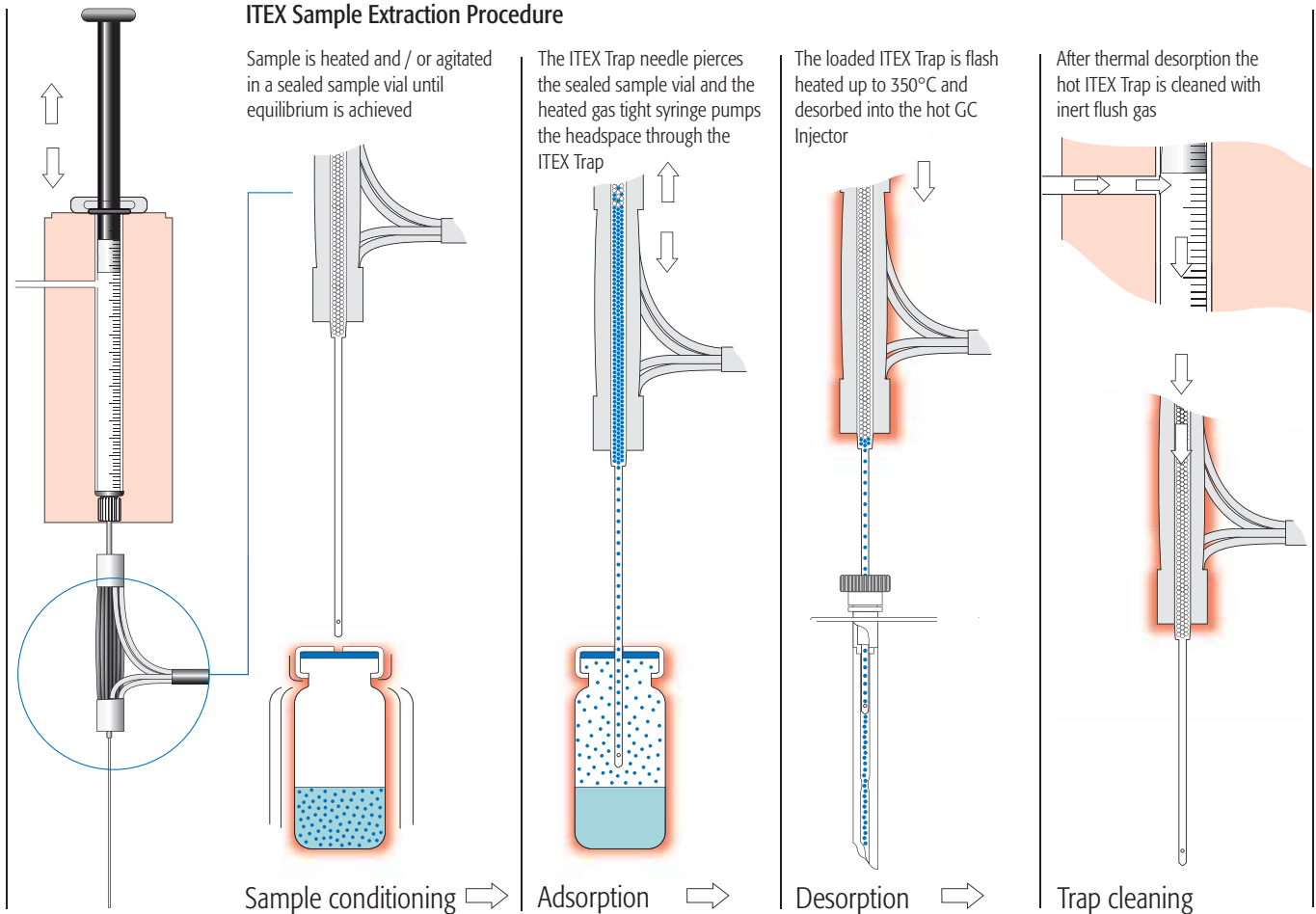
Agitation:

Interval shaking 250rpm - 750rpm, selectable in 1rpm increments

Incubation Time:

Up to 999 minutes selectable in 1 second increments

ITEX Sample Extraction Procedure



Comparison of ITEX analysis versus Static Headspace
Sample: Purge and Trap calibration mix (Restek Cat.No. 30431 502.2 CAL2000 Mega-Mix)

Static Headspace Parameter

60°C / 10min / 1ml sample volume

ITEX Parameter

Extraction Speed: 100µl / sec.

Total Pumping Strokes: 50

Temperature Pumping Syringe / Sample Incubation: 60°C / 10min.

Desorption at 200°C, 15sec. splitless

Chromatography:

Injection: Splitless 15sec. at 250°C / Carrier gas: 0.2bar hydrogen

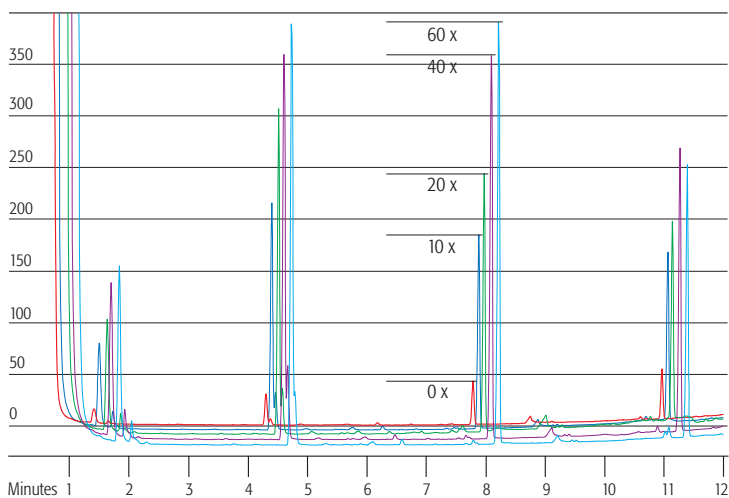
Column: Rtx-502.2 60m x 0.32mm ID, 1.8µm film

Temperature Program: 40°C - 1min. - 10°C / min to 220°C

Detection: FID 250°C

- | | | | |
|----|--------------------------------------|----|--|
| 1 | 1,1-Dichloroethylene | 35 | 1,1,2,2-Tetrachloroethane |
| 2 | Methylene chloride (dichloromethane) | 36 | 1,2,3-Trichloropropane |
| 3 | trans-1,2-Dichloroethylene | 37 | n-Propylbenzene |
| 4 | 1,1-Dichloroethane | 38 | Bromobenzene |
| 5 | 2,2-Dichloropropane | 39 | 1,3,5-Trimethylbenzene |
| 6 | cis-1,2-Dichloroethylene | 40 | 2-Chlorotoluene |
| 7 | Chloroform | 41 | 4-Chlorotoluene |
| 8 | Bromochloromethane | 42 | tert-Butylbenzene |
| 9 | 1,1,1-Trichloroethane | 43 | 1,2,4-Trimethylbenzene |
| 10 | 1,1-Dichloropropene | 44 | sec-Butylbenzene |
| 11 | Carbon tetrachloride | 45 | 4-Isopropyltoluene (p-Cymene) |
| 12 | 1,2-Dichloroethane | 46 | 1,3-Dichlorobenzene |
| 13 | Benzene | 47 | 1,4-Dichlorobenzene |
| 14 | Trichloroethylene | 48 | n-Butylbenzene |
| 15 | 1,2-Dichloropropane | 49 | 1,2-Dichlorobenzene |
| 16 | Bromodichloromethane | 50 | 1,2-Dibromo-3-chloropropane |
| 17 | Dibromomethane | 51 | 1,2,3-Trichlorobenzene |
| 18 | cis-1,3-Dichloropropylene | 52 | Hexachloro-1,3-butadiene (Hexachlorobutadiene) |
| 19 | Toluene | 53 | Naphthalene |
| 20 | trans-1,3-Dichloropropylene | 54 | 1,2,3-Trichlorobenzene |
| 21 | 1,1,2-Trichloroethane | | |
| 22 | 1,3-Dichloropropane | | |
| 23 | Tetrachloroethylene | | |
| 24 | Dibromochloromethane | | |
| 25 | 1,2-Dibromoethane (EDB) | | |
| 26 | Chlorobenzene | | |
| 27 | 1,1,1,2-Tetrachloroethane | | |
| 28 | Ethylbenzene | | |
| 29 | m-Xylene | | |
| 30 | p-Xylene | | |
| 31 | o-Xylene | | |
| 32 | Styrene | | |
| 33 | Isopropylbenzene | | |
| 34 | Bromoform | | |

Enrichment of Methylsters on a Tenax TA ITEX trap. 1µl of a mixture of C₄, C₆, C₈, C₁₀ Methylsters in Methanol (100ng / µl) was injected into a 20ml HS-vial. After conditioning at 40°C for 10min. 1ml of the headspace using a ITEX trap without packing material was injected to determine a "static headspace value" (0x). Afterwards the needle was replaced by the TENAX TA ITEX trap. The enrichment of the solutes on the trap was studied using various numbers of pumping strokes.



ITEX Parameter

Extraction Speed: 100µl / sec.

Total Pumping Strokes: 0 / 10 / 20 / 40 / 60

Temperature Pumping Syringe / Sample Incubation: 40°C / 10min.

Desorption at 250°C, 15sec. splitless

Chromatography

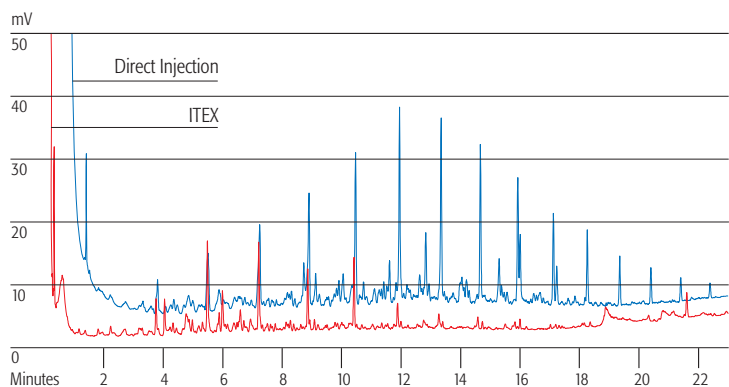
Injection: Splitless 15sec. at 250°C, Carrier gas: 0.2bar Hydrogen

Column: BGB-1 15m x 0.32mm ID, 1.0µm film

Temperature Program: 40°C - 1 min. - 10°C / min to 200°C

Detection: FID 250°C

1µl of a Diesel dissolved in Methanol (500ng / µl) was injected (splitless 15sec.) into the injector to determine a "100% value". 1µl of the same solution was added to 12ml water in a 20ml Headspace vial and then analysed with ITEX.



ITEX Parameter:

Extraction Speed: 120µl / sec.

Total Pumping Strokes: 120

Temperature Pumping Syringe / Sample Incubation: 50°C / 10min.

Desorption at 250°C, 15sec. splitless

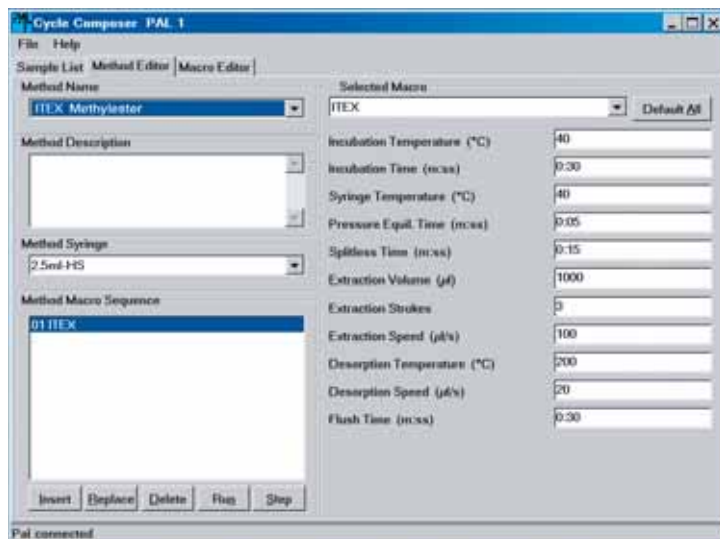
Chromatography:

Injection: Splitless 15sec. at 250°C / Carrier gas: 0.2bar Hydrogen

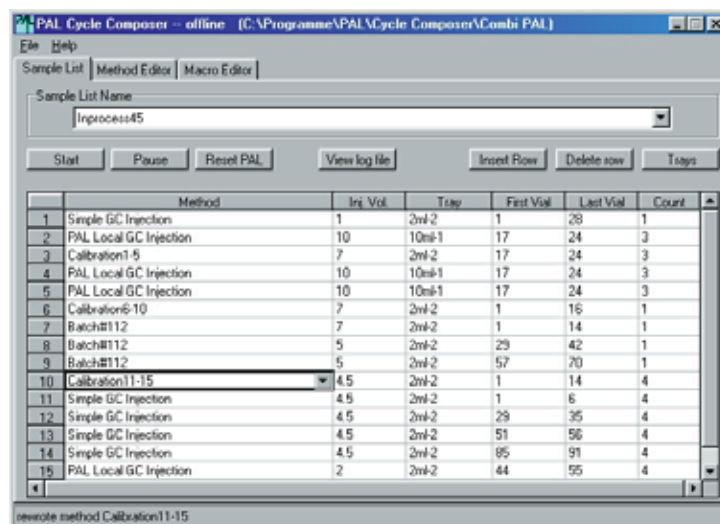
Column: BGB-1 15m x 0.32mm ID, 1.0µm film

Temperature Program: 40°C - 1 min. - 10°C / min to 200°C

Detection: FID 250°C



ITEX parameter control by Cycle Composer



Cycle Composer sample list

Flexible Software Control

Choose between two options to control your CombiPAL ITEX Option. For individual application requirements the standalone PC based Windows 2000/XP software Cycle Composer is available.

For single keyboard operation of a whole GC/GC-MS system, the following third party CombiPAL drivers are available*.

Vendor	Software
Agilent	ChemStation
Agilent	EZChrom Elite
DataApex	Clarity
Dionex	Chromleon
Justice Software	Chromperfect
Leco	ChromaTOF
Shimadzu	GCMSsolution
Thermo Scientific	Xcalibur
Varian	Star
Varian	Galaxie
Waters	Masslynx
Waters	Empower

* certain drivers may not support the ITEX cycle



CombiPAL General Specifications

System Type

XYZ robot with syringe only concept, no tubing in sample path

Local User Interface

Control panel with 4 function keys, graphical LCD display, unique scroll knob for teach functions

Remote Control

Cycle Composer control software Windows 2000 / XP
Third party instrument drivers for all major GC/GC-MS Systems

Maintenance

Accessibility to all maintenance parts from front
Preventative maintenance kits available

Electrical Control

2x RS232
3x TTL Input
2x Opto Coupler Input
2x Relay Output

Power Requirements

100-240V, 120W, 50/60Hz

Environment

4°C - 40°C constant temperature, < 80% humidity (non condensing)

Weight

~ 10kg (without accessories)

Dimension

Length 828mm Depth 385mm Height 575mm

Electrical Safety Standards

CAN/CSA C22.2 No. 61010-1 / ANSI/UL 61010-1 / EN 61010-1

Sample Capacity*

up to 600	1ml micro vials (78 1ml vials standard)
294	2ml vials (98 2ml vials standard)
96	10ml or 20ml vials
4	deepwell microplates (96/384 wells)
8	standard microplates (96/384 wells) (* depends on GC model)

GC Mounting Kits

Agilent Technologies 5890 / 6850 / 6890
Thermo Scientific Trace 2000 / GC 8000top / Focus
Varian GC 3400 / 3600 / 3800 / 3900
Shimadzu GC 14 / 17A / 2010 / 2014
Perkin Elmer Autosystem XL / Clarus 500
GL Sciences GC 353 / 393 / 4000

Order details for ITEX Option (part no. PAL ITEXOpt)

Description

1pc	2.5ml Syringe with 1/4" 28 UNF fitting
1pc	Replacement plunger 2.5ml
2pc	ITEX trap TENAX TA 60/80 mesh
1pc	Trap heater incl. electrical connections
1pc	Endplate left side
1pc	Syringe heater side bracket
1pc	ITEX Injection Unit
1pc	CD-ROM including ITEX Cycle (requires Cycle Composer)

Consumables

ITEXTrapTXTA	1pc ITEXTrap Tenax TA
ITEXTrapTXTA3	Set of 3pcs. ITEXTrap Tenax TA
ITEXTrapCstm3	Set of 3pcs. ITEXTrap custom filling (specify adsorbent)
SYRC ITEX2.5	1pc replacement ITEX Syringe 2.5ml
PLG G2500	Replacement plunger for 2.5ml syringe

Specifications are subject to change without notice

PAL GC Sample Injection Systems

Static Headspace - Liquid Injection - SPME - ITEX Extraction
combined in one single instrument



Distributed by:

CTC Analytics has dedicated the last 15 years to the continued development and high reliability of advanced sample injection technology. To learn more about the unique PAL Series of GC/GC-MS sample handling systems or any of our LC/LC-MS sample injection systems contact your CTC Analytics distributor.

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Where design meets performance

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