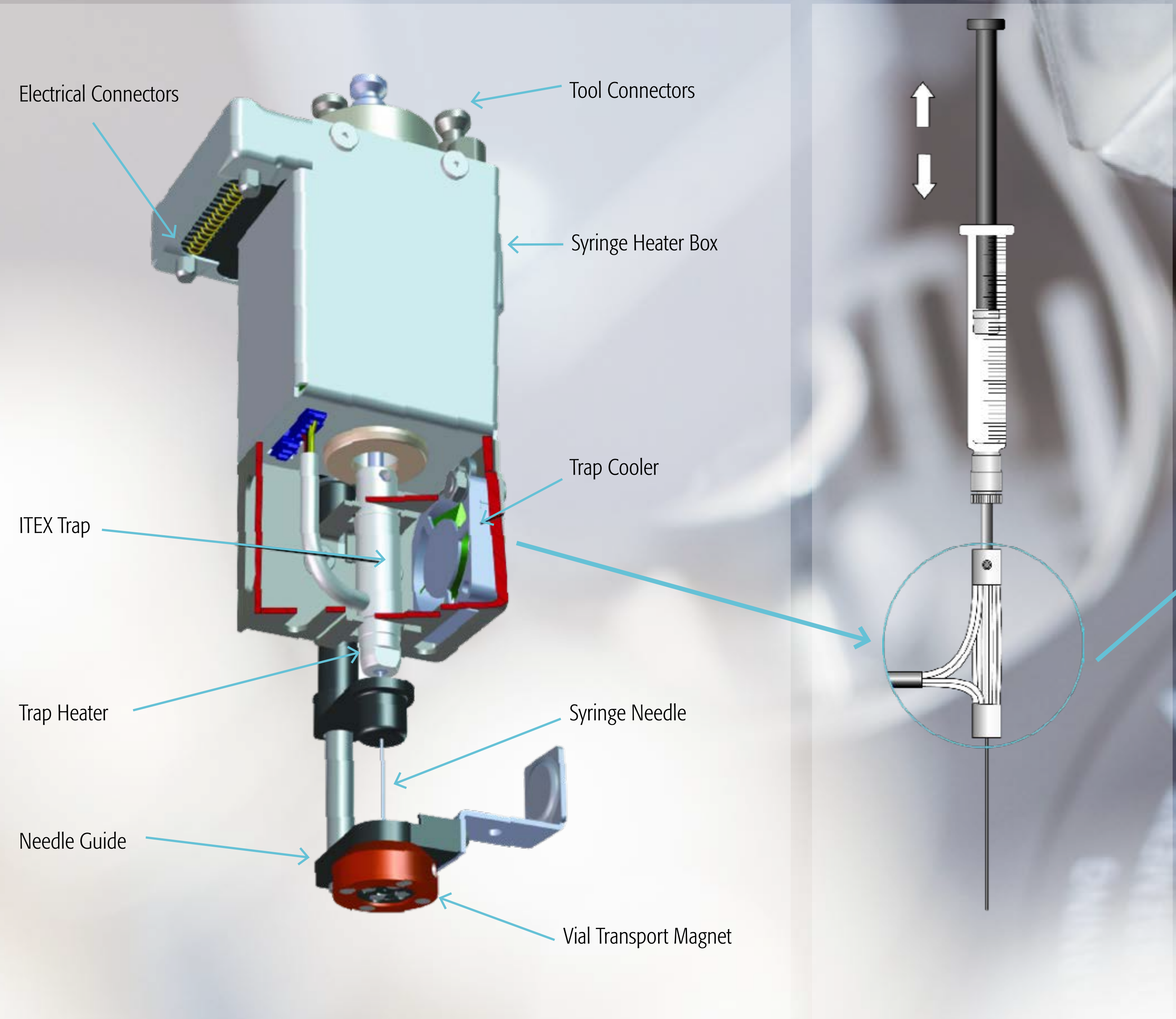
PAL SYSTEM
Ingenious sample handling


# ITEX Dynamic Headspace
# Powerful Sample Enrichment for GC

# ITEX (In-Tube Extraction) Dynamic Headspace Tool

## Rapid and efficient enrichment of volatile and semi-volatile compounds from solid, liquid and gaseous samples




- In-Tube Extraction and thermal desorption using industry standard adsorbents
- Syringe only concept: no sample loops, transfer lines, or switching valves that could be contaminated
- Different ITEX Traps available for optimal performance in your application
- Active cooling allows for rapid sample preparation and short cycle times
- No modifications of the GC injector needed
- PAL Robotic Tool Change enables headspace, SPME and ITEX sampling within the same sequence on one system

## ITEX Dynamic Headspace Extraction Procedure

The sample is heated and / or agitated in a sealed vial.

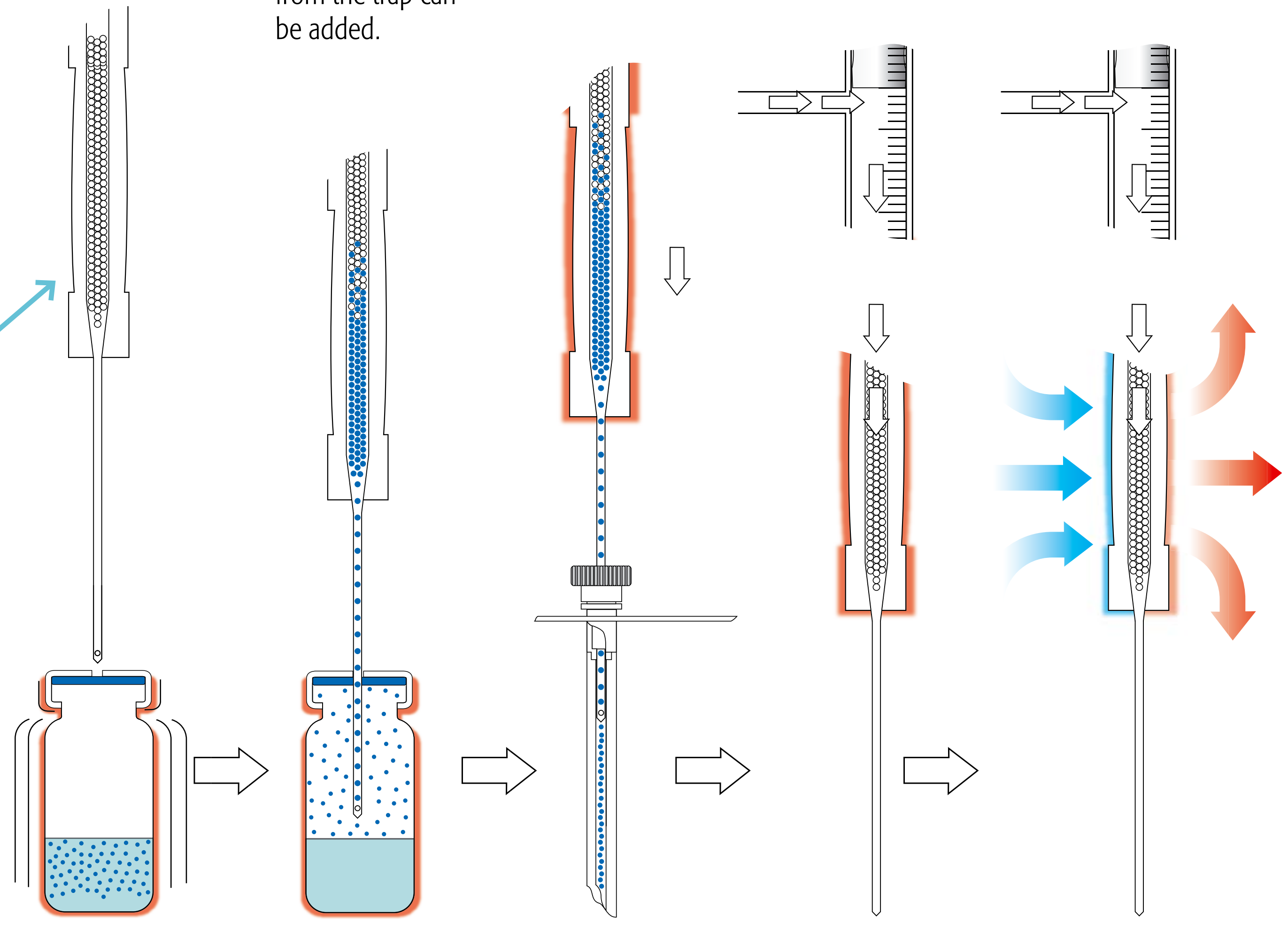
The ITEX needle pierces the sealed vial and the heated syringe pumps the the headspace gas through the cold trap.

An additional step to remove water from the trap can be added.

The loaded ITEX Trap is flash heated up to 350°C and analytes are desorbed into the hot GC injector.

After thermal desorption the hot ITEX Trap is cleaned with inert flush gas.

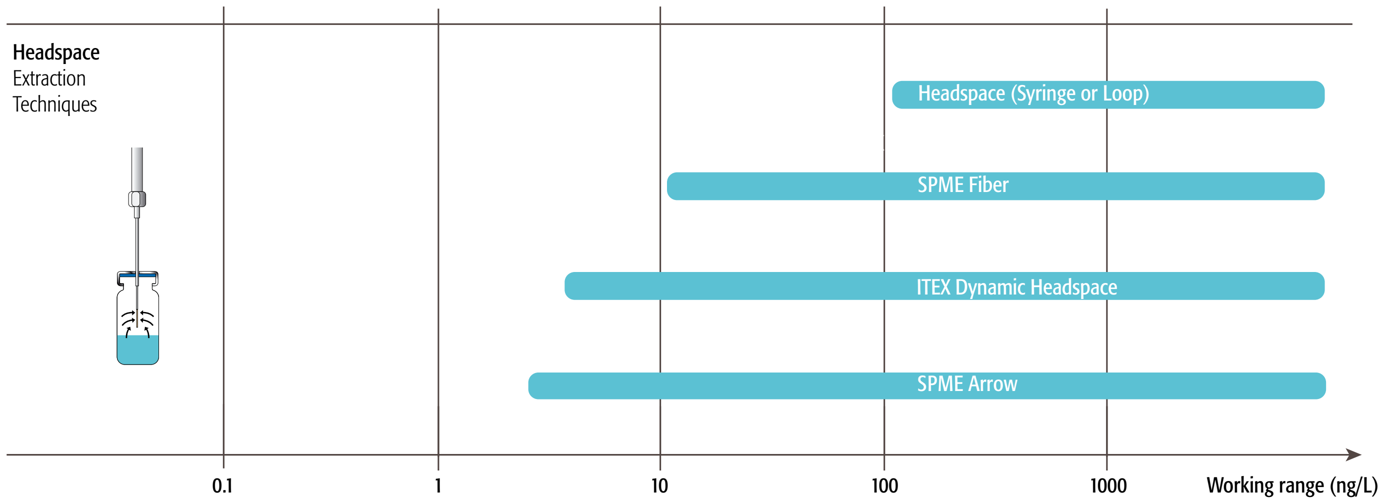
Active cooling allows for short cycle times.



Sample Conditioning | Adsorption | Desorption | Trap Cleaning | Active Cooling

Find more information about [ITEX DHS]

# Sensitivity diagramm




# Sensitivity comparison between ITEX Dynamic Headspace and Purge & Trap

Sensitivity Comparison of ITEX with Purge & Trap

| | ITEX Dynamic Headspace | | | | | Purge & Trap | |
|---|---|---|---|---|---|---|---|
| Compound | MDL [µg L$^{-1}$] | Linear range [µg L$^{-1}$] | $R^2$ | RSD [%] | Recovery [%] | MDL [µg L$^{-1}$] | RSD [%] |
| Vinylchloride | 0.008 | 0.02-2.0 | 0.999 | 5.3 (n=63) | 103 | 0.008 | |
| Dichloromethane | 0.01 | 0.03-2.7 | 0.999 | 5.5 (n=63) | 97 | | |
| MTBE | 0.004 | 0.01-1.5 | 0.999 | 6.1 (n=63) | 88 | 0.001 | 4.7 |
| ETBE | 0.001 | 0.004-1.5 | 0.998 | 6.6 (n=77) | 94 | 0.009 | 5.1 |
| Chloroform | 0.004 | 0.007-2.9 | 0.999 | 5.4 (n=77) | 99 | 0.008 | |
| Benzene | 0.001 | 0.002-1.8 | 0.999 | 5.4 (n=84) | 89 | 0.002 | 5 |
| TAME | 0.001 | 0.004-1.5 | 0.999 | 5.8 (n=77) | 95 | 0.01 | 3 |
| 1,2-dichloroethane | 0.002 | 0.006-2.5 | 0.999 | 5.3 (n=77) | 97 | | |
| Trichloroethylene | 0.001 | 0.007-2.9 | 0.999 | 5.2 (n=77) | 95 | 0.003 | 5.3 |
| Bromodichloromethane | 0.001 | 0.002-4.0 | 0.999 | 6.1 (n=91) | 97 | 0.007 | 5.2 |
| 1,4-Dioxane | 0.07 | 0.1-2.1 | 0.998 | 8.9 (n=49) | 59 | | |
| Toluene | 0.005 | 0.009-1.7 | 0.998 | 7.3 (n=70) | 96 | 0.001 | 4.5 |
| Tetrachloroethylene | 0.001 | 0.003-3.2 | 0.999 | 5.7 (n=84) | 97 | 0.004 | 7.3 |
| Dibromochloromethane | 0.005 | 0.02-4.9 | 0.999 | 5.8 (n=70) | 98 | 0.001 | 4.1 |
| Ethylbenzene | 0.002 | 0.009-1.7 | 0.999 | 8.7 (n=70) | 93 | 0.001 | 5.5 |
| p-Xylene | 0.004 | 0.009-1.7 | 0.999 | 8.9 (n=70) | 117 | 0.001 | 4.9 |
| o-Xylene | 0.005 | 0.02-1.7 | 0.999 | 6.9 (n=63) | 90 | 0.002 | 4.7 |
| Bromoform | 0.002 | 0.006-5.8 | 0.999 | 5.7 (n=84) | 94 | | |
| 2-Methylisoborneol | 0.03 | 0.1-2.0 | 0.999 | 5.5 (n=49) | 94 | 0.001 | 5.6 |
| Geosmin | 0.06 | 0.1-2.0 | 0.999 | 5.1 (n=49) | 88 | 0.002 | 6.1 |

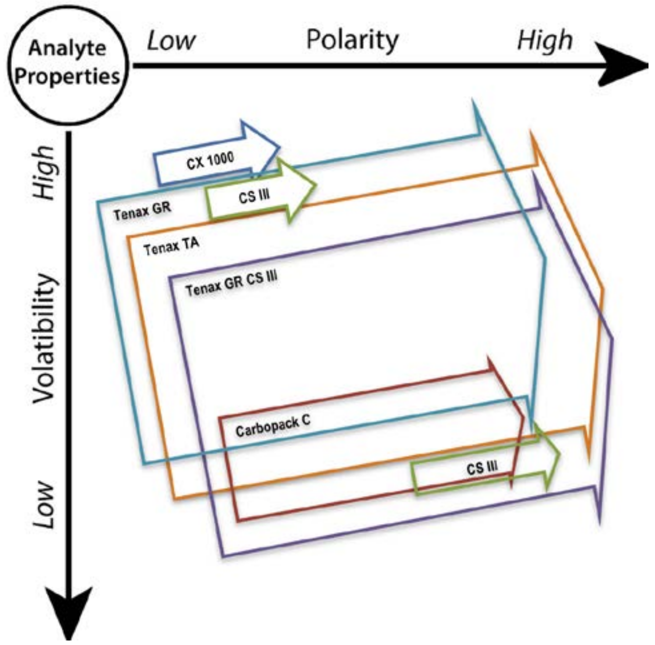
MDL: Method detection limit
Ref: Laaks J, Jochmann MA, Schilling B, Schmidt TC ; Anal. Chem. 2010, 82, 7641-7648



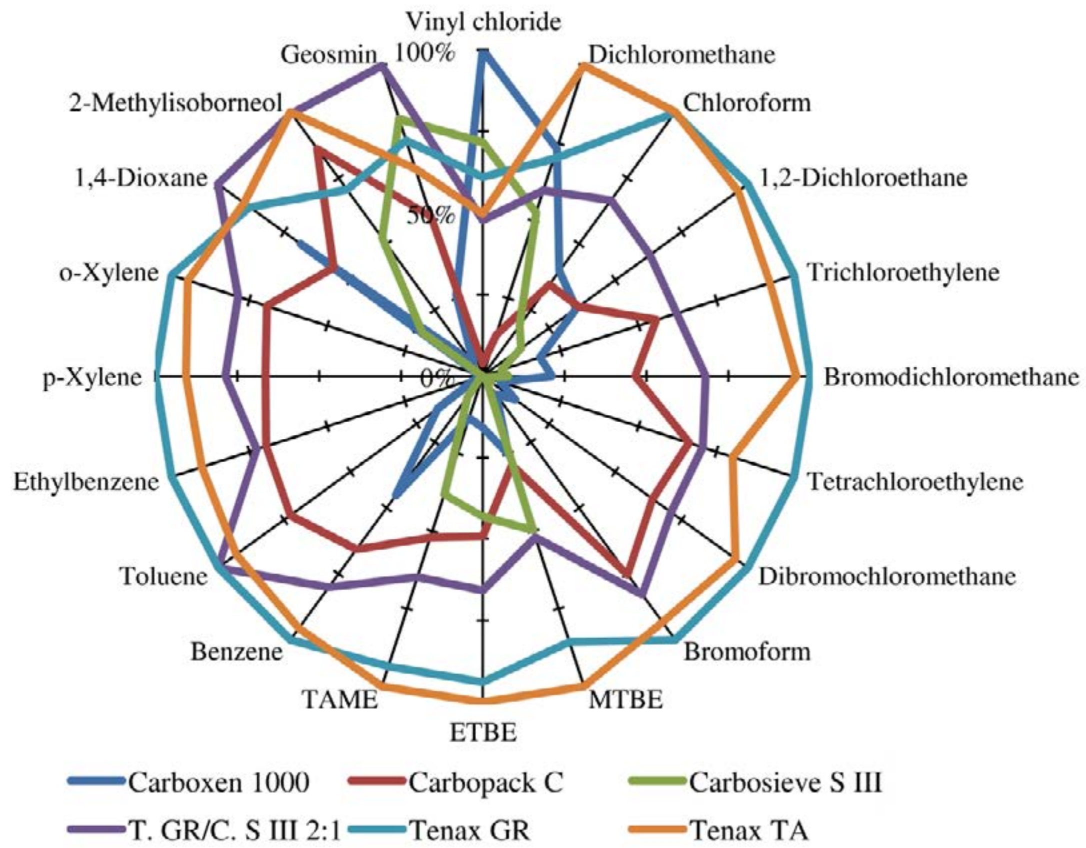
# ITEX Traps

| | |
|---|---|
| PAL3-ITEX-Trap-Carb-S3 | PAL ITEX Trap Carbosieve SIII -60/ 80 mesh |
| PAL3-ITEX-Trap-Carb1000 | PAL ITEX Trap Carboxen 1000 -60 / 80 mesh |
| PAL3-ITEX-Trap-CarbC | PAL ITEX Trap Carbopack - C 80 /100 mesh |
| PAL3-ITEX-Trap-TxGR | PAL ITEX Trap Tenax GR - 80 /100 mesh |
| PAL3-ITEX-Trap-TXTA100 | PAL ITEX Trap Tenax TA 80 /100 mesh |
| PAL3-ITEX-Trap-Mix-1 | PAL ITEX Trap Mix1 Tenax TA : Carboxen1000 (Bottom: Top - 1: 1) |
| PAL3-ITEX-Trap-Mix-2 | PAL ITEX Trap Mix2 Carbopack C : Carbosieve SIII (Bottom: Top - 2: 1) |
| PAL3-ITEX-Trap-Mix-3 | PAL ITEX Trap Mix3 Tenax GR : Carbosieve SIII (Bottom: Top - 1: 1) |

# Analyte affinity of ITEX Traps

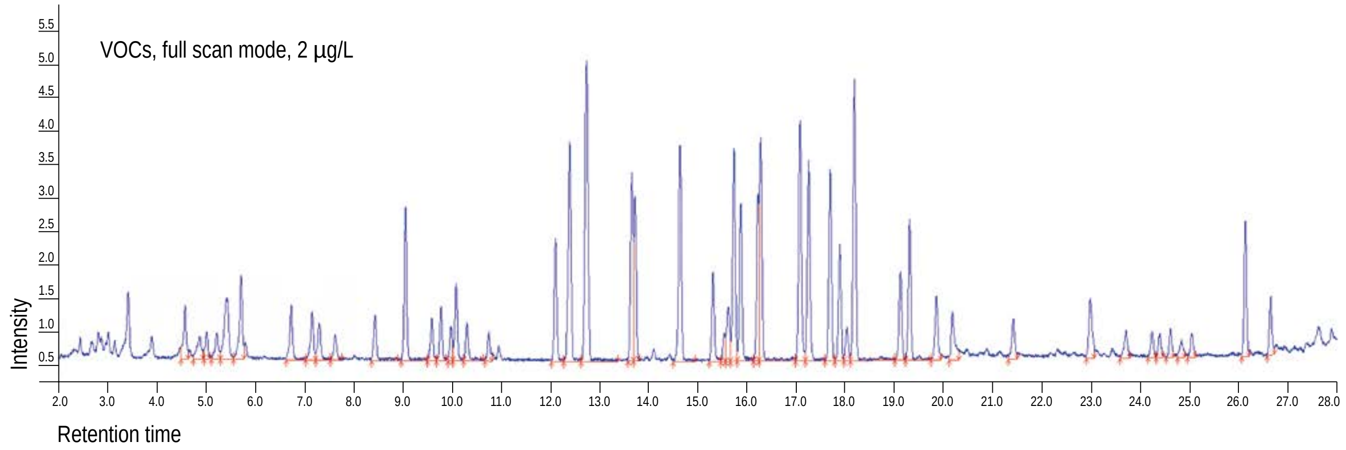


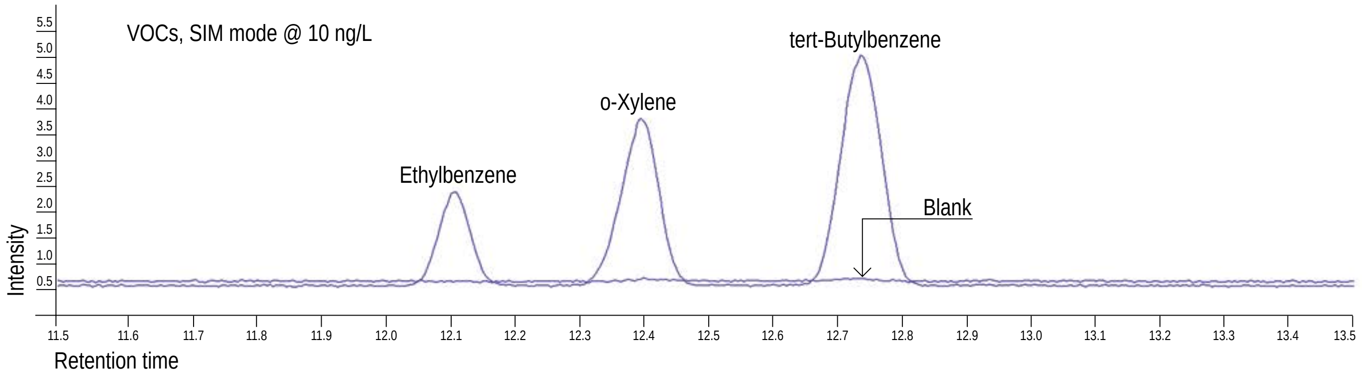

# ITEX Comparison of Volatile Compounds




J. Laaks, M. Jochmann, B. Schilling, T. Schmidt; Optimization strategies of ITEX methods - Analytical and bioanalytical chemistry 407/2015

# Environmental application
# VOCs, BTEX in water at ppt levels, EPA 502.2




## Sample preparation:

Full scan data: 2 µg/L MegaMix® Standard, 524.2 73 components), Restek PN 30601

SIM scan data: 10 ng/L MegaMix® Standard, 502.2 (54 components), Restek PN 30432

10mL water + standard, filled into 20 mL headspace vial, + 3g NaCl

## Chromatography (Shimadzu GC-2010 Plus):

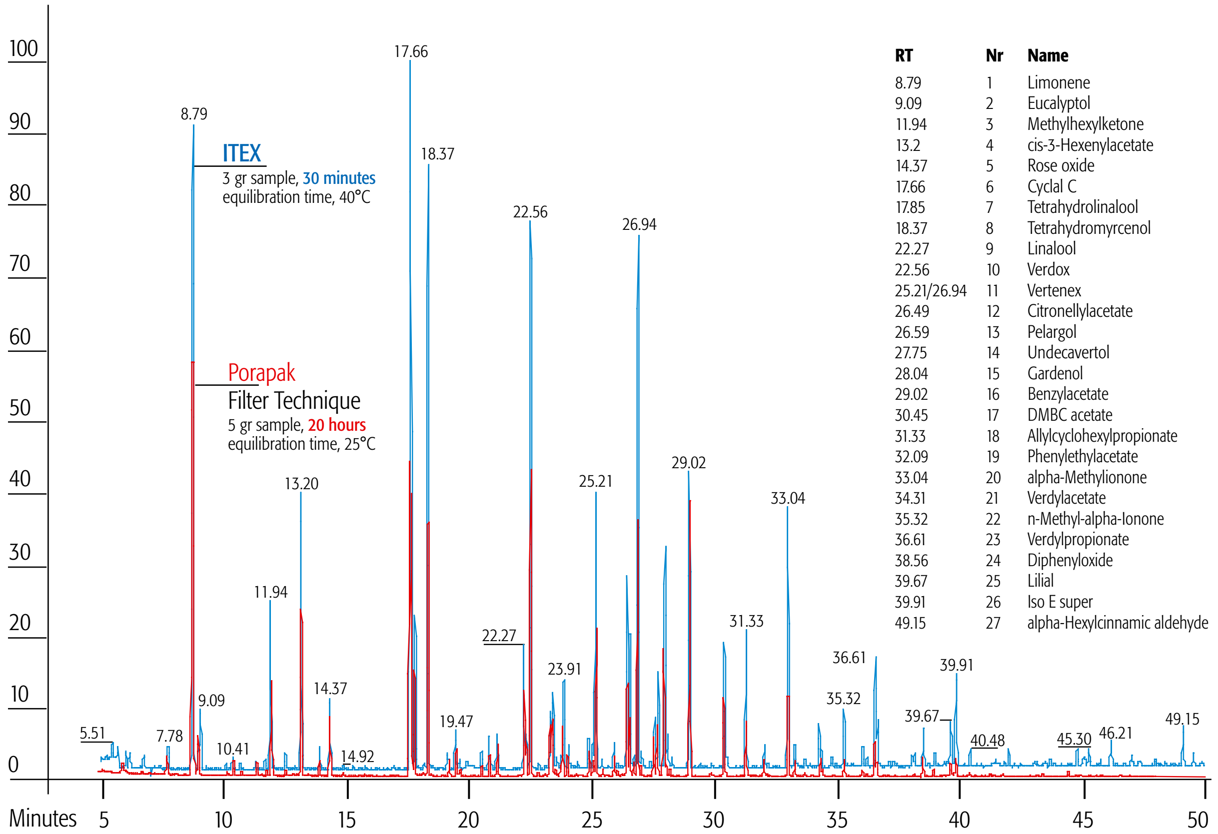
| | |
|---|---|
| Column | Rxi® 624 Sil MS, 30 m x 0.32mm, 1.8 µm df |
| Carrier gas | helium, 93.2 kPA |
| Temperature program | 40°C for 1 min, 5°C/min to 250°C, 2 min hold |
| Injector | split/split less at 225°C |
| Liner | Restek catalog # 23321.1, 3.5 mm Splittless Single Tpr Gsnk |
| GC | Shimadzu GC-2010 Plus |
| Detector | Shimadzu GCMS-W2010E |

## ITEX Dynamic Headspace conditions

| | |
|---|---|
| Incubation temperature | 40°C |
| Incubation time | 3 min |
| Syringe temperature | 50°C |
| Extraction strokes | 3 x 1 ml at 40°C (12 min) |
| Extraction speed | 100 µl/s |
| Desorption temperature | 200°C with 1 ml headspace |
| ITEX trap material | Tenax TA |



# Food application
# Comparison of flavors/softeners
# ITEX Dynamic Headspace vs. Poropak Filter




## Chromatography (Thermo TraceGC):

| | |
|---|---|
| Column | Stabilwax 30 m x 0.25 mm, ID x 0.25 mm film thickness |
| Oven | 35°C - 0.5 min 15°C/min 50°C - 0 min 5°C/min 220°C/1 min |
| SSL | splitless with surge, surge pressure 20 kPa/0.4 min (0.5 ml), split flow 100 ml/0.3 min |
| Carrier | He, 1 ml/min constant flow with vacuum compensation |

## MS conditions (Thermo Trace MS system)

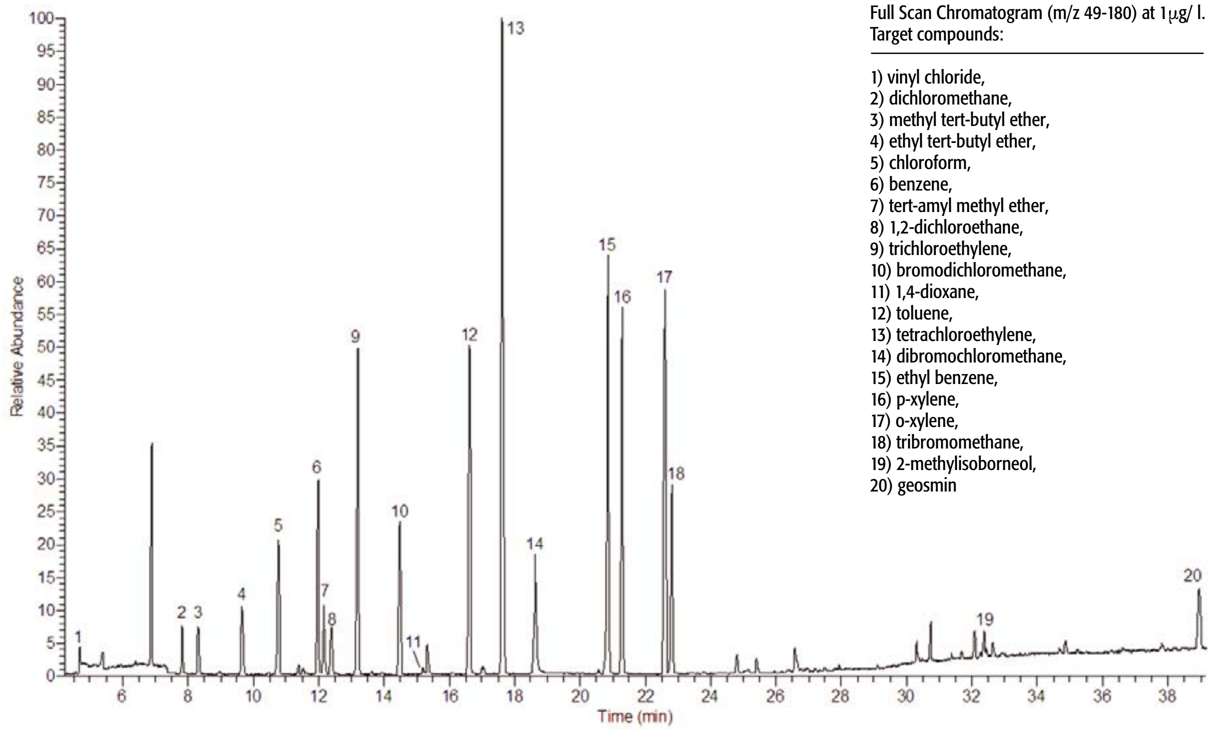
| | |
|---|---|
| Ionisation mode | EI+ |
| Source temperature | 230°C |
| Interface | 220°C |
| Mass | 20 - 350 amu |

Courtesy of: Givaudan Research Company, CH-8600 Dübendorf, Zürich, Switzerland, H. Koch

## ITEX Dynamic Headspace conditions

| | |
|---|---|
| Incubation temperature | 40°C |
| Incubation time | 30 min |
| Syringe temperature | 45°C |
| Extraction volume | 1000 µl/stroke |
| Extraction strokes | 10 |
| Extraction speed | 100 µl/s |
| Desorption temperature | 200°C |
| Desorption speed | 100 µl/s |
| Flush time | 5 min |

# Environmental application
# Purgable organic compounds (VOCs) in water by GC/MS, EPA 524.2




ITEX ppt sensitivity for volatile or semi-volatile compounds

## Instrumentation / Conditions

| Instrumentation | | Conditions |
|---|---|---|
| GC | Thermo Trace GC Ultra | 40°C for 1 min, 4°C/min to 130°C, 10°C/min to 200°C for 10 min |
| MS | Thermo DSQII | EI 70evm Scan mode m/z 49-180 |
| Injector | AtasGL Optic 3 with cryofocusing unit | 280°C Crytrap - 165°C for 30 s, 30°C/s to 250°C |
| GC Column | Restek VMS 60 m x 0.32 mm i.d., 1.8 µm film | 1.5 ml/min constant flow |
| Autosampler | PAL RTC with ITEX Dynamic Headspace, Tenax GC/ Carbosieve S3 trap | 10 ml sample in 20 ml headspace vial |

## ITEX Dynamic Headspace conditions

| | | | |
|---|---|---|---|
| Sample storage in tray cooler | 25°C | Extraction volume | 1000 µl |
| Tray temperature | 40°C | Extraction flow rate | 100 µl/s |
| Syringe temperature | 60°C | Desorption volume | 500 µl |
| Incubation time | 5 min | Desorption flow rate | 10 µl/s |
| Agitator speed | 500 rpm | Desorption temperature | 300°C |
| Extraction temperature | 60°C | Trap cleaning temperature | 350°C |
| Extraction stroke | 60 | Trap cleaning time | 5 min |



# More application examples

## Environmental

**In-Tube Extraction of Volatile Organic Compounds from Aqueous Samples: An Economical Alternative to Purge and Trap Enrichment**
Jens Laaks,† Maik A. Jochmann,*,†
Beat Schilling, and Torsten C. Schmidt†
Anal. Chem. 2010, 82, 7641–7648

**Multi-walled carbon nanotubes as sorptive material for solventless in-tube microextraction (ITEX2)—a factorial design study**
Thorsten Hüffer & Xochitli L. Osorio & Maik A. Jochmann & Beat Schilling & Torsten C. Schmidt
Instrumental Analytical Chemistry, University of Duisburg-Essen, Universitätsstrasse 5, 45141 Essen, Germany; BGB Analytik GmbH, Rohrmattstrasse 1, 4461 Boeckten, Switzerland
Anal Bioanal Chem (2013) 405:8387–8395

**Microwave-assisted nonionic surfactant extraction of aliphatic hydrocarbons from petroleum source rock**
A. Akinluaa, , M.A. Jochmannb, J. Laaksb, A. Ewertb, T.C. Schmidtb
a Fossil Fuels and Environmental Geochemistry Group, Department of Chemistry, Obafemi Awolowo University, Ile-Ife, Nigeria
b Instrumental Analytical Chemistry, University Duisburg-Essen, Universitätsstr, 5, 45141 Essen, Germany

**Predominance of Biotic over Abiotic Formation of Halogenated Hydrocarbons in Hypersaline Sediments in Western Australia**
A. Ruecker,† P. Weigold,† S. Behrens,† M. Jochmann,‡ J. Laaks,‡ and A. Kappler*,†
†Geomicrobiology, Center for Applied Geosciences, University of Tübingen, Tübingen 72074, Germany
‡Instrumental Analytical Chemistry, Faculty of Chemistry, University of Duisburg-Essen, Essen 45141, Germany

**Sub-ppt determination of butyltins, methylmercury and inorganic mercury in natural waters by dynamic headspace in-tube extraction and GC-ICPMS detection†**
Jing Hu,ab Enea Pagliano,b Xiandeng Hou, a Chengbin Zheng,a Lu Yang *b and Zoltan Mesterb

## Chemical

**Microwave-assisted nonionic surfactant extraction of aliphatic hydrocarbons from petroleum source rock**
A. Akinlua[a],*, M.A. Jochmann[b], J. Laaks[b], A. Ewert[b],
T.C. Schmidt[b] - Analytica Chimica Acta 691 (2011) 48–55

**Optimization strategies of in-tube extraction (ITEX) methods**
Jens Laaks1 & Maik A. Jochmann1 & Beat Schilling2 & Torsten C. Schmidt1

**Unbiased profiling of volatile organic compounds in the headspace of Allium plants using an in-tube extraction device**
Miyako Kusano1,2*, Makoto Kobayashi2, Yumiko Iizuka2,3, Atsushi Fukushima2 and Kazuki Saito2,4

**Multi-walled carbon nanotubes as sorptive material for solventless in-tube microextraction (ITEX2)—a factorial design study**
Thorsten Hüffer & Xochitli L. Osorio & Maik A. Jochmann & Beat Schilling & Torsten C. Schmidt

## Food

**In-tube Extraction and GC–MS Analysis of Volatile Components from Wild and Cultivated sea buckthorn (Hippophae rhamnoides L. ssp. Carpatica) Berry Varieties and Juice**
Sonia A. Socaci,a Carmen Socaciu,a* Maria Tofan,a Ioan V. Ratib and Adela Pinteaa - Phytochem. Anal. 2013, 24, 319–328

**Automated and quantitative headspace in-tube extraction for the accurate determination of highly volatile compounds from wines and beers**
Julián Zapata, Laura Mateo-Vivaracho, Ricardo Lopez, Vicente Ferreira*
J. Chromatography A, 1230 (2012) 1– 7

**In-Tube Extraction-GC/MS as High Capacity Enrichment Technique for the Analysis of Alcoholic Beverages.**
Laaks J., Jochmann M.A., Schilling B., Molt K., Schmidt T.C.
†Instrumental Analytical Chemistry, University Duisburg-Essen, Universitätsstrasse 5, D-45141 Essen, Germany;
§BGB Analytik AG, Lettenstrasse 97, CH-8134 Adliswil, Switzerland
J. Agric. Food Chem. 2014, [Epub ahead of print]

**Determination of Wine Aroma Compounds by Head Space "In tube extraction" Technique and Gas Chromatography (HS-ITEX-GC/MS)**
Luminita VÂRVA1, Sonia SOCACI1, Anca FARCAS1, Liana SALANTA1, Ana-Viorica POP (CUCEU)1, Elena MUDURA2 and Maria TOFANA1*

## Clinical / Life Science

**Headspace In-Tube Extraction Gas Chromatography–Mass Spectrometry for the Analysis of Hydroxylic Methyl-Derivatized and Volatile Organic Compounds in Blood and Urine**
Ilpo Rasanen, Jenni Viinamäki, Erkki Vuori, and Ilkka Ojanperä*
J Analytical Toxicology, 34, 2010, 113-121

**Metabolic Profiling of Oxidized Lipid-Derived Volatiles in Blood by Gas Chromatography/Mass Spectrometry with In-Tube Extraction (ITEX)**
Shoji Kakuta,1 Yasuhiko Bando,2 Shin Nishiumi,3 Masaru Yoshida,3,4,5 Eiichiro Fukusaki,1 and Takeshi Bamba*,1
1 Department of Biotechnology, Graduate School of Engineering, Osaka University, 2–1 Yamadaoka, Suita, Osaka 565–0871, Japan
2 AMR Inc., 13–18 Nakane-2, Meguro-ku, Tokyo 152–0031, Japan
3 Division of Gastroenterology, Department of Internal Medicine, Kobe University Graduate School of Medicine,
7–5–1 Kusunoki-cho, Chu-o-ku, Kobe, Hyogo 650–0017, Japan
4 e Integrated Center for Mass Spectrometry, Kobe University Graduate School of Medicine, 7–5–1 Kusunoki-cho, Chu-o-ku, Kobe, Hyogo 650–0017, Japan 5 Division of Metabolomics Research, Kobe University Graduate School of Medicine, 7–5–1 Kusunoki-cho, Chu-o-ku, Kobe, Hyogo 650–0017, JapanMedicine, 7–5–1 Kusunoki-cho, Chu-oku, Kobe, Hyogo 650–0017, Japan Mass Spectrometry 2013, 2, A0018

**Metabolomic signature of brain cancer†**
Renu Pandey1, Laura Caflisch2, Alessia Lodi1, Andrew J. Brenner2,3, Stefano Tiziani1,4

# ITEX Dynamic Headspace Specifications

| | |
|---|---|
| Trap temperature range | 40°C - 350°C |
| Syringe temperature range | 40°C - 120°C |
| Extraction flow rates | 10 µl/s - 1000 µl/s |
| Extraction stroke cycles (strokes/sample) | 0 - 1000 |
| Extraction volume | 0 µl - 1300 µl |
| Incubation time | up to 600 min. |
| Standard adsorption material | Tenax TA 8/100 mesh, other material see page 11 |
| Water removal step with adjustable trap temperatures | 40°C - 150°C |
| Ventilated trap for quick sample processing | Fast cooling of trap < 2 minutes |


# PAL System Tools and Modules complementing ITEX Dynamic Headspace

| | | |
|---|---|---|
| ITEX Kit<br><br>PAL3-SITEX-Kit |  | PAL ITEX Tool, including:<br>1 pc Smart ITEX Syringe 1.3 mL (SITEX-1300-T)<br>2 pcs ITEX Trap TENAX TA 80/100 mesh (PAL3-ITEX-Trap-TXTA100)<br>compatible with all PAL RTC/PAL RSI models from firmware version 2.3. or higher<br>requires firmware version 3.n or higher and PAL RTC/PAL RSI Series II for Smart functionality |

# Mandatory Modules

| | | |
|---|---|---|
| Agitator<br><br>PAL3-Agitator |  | For the incubation and agitation of samples<br>• 6 positions for 20 ml vials<br>• Temperature range 40 - 200 °C<br>• Agitation speed 250 - 750 rpm<br>• Optional adapters for 2 ml or 10 ml vials |

# Optional Headspace Tools and Modules

| | | |
|---|---|---|
| Headspace tools<br><br>PAL3-TH-HS1000<br>PAL3-TH-HS2500<br>PAL3-TH-HS5000 |  | Consistent headspace sampling<br>– 1000, 2500 and 5000 µl Smart Syringes available<br>– Syringe flush with inert gas<br>– Magnetic vial transport for 2 ml, 10 ml, 20 ml vials |
| SPME Smart SPME Fiber Kit<br><br>PAL3-SPME-SFib-Kit |  | Smart SPME Kit, consisting of:<br>1 pc SPME Tool<br>1 pc SPME Fiber Assortment Kit (SFIB-SEL5-S2)<br>1 pc SPME Performance Evaluation Mix |
| SPME Smart SPME Arrowr Kit<br><br>PAL3-SPME-SArr-Kit |  | Smart SPME Arrow Kit without GC adaption kit consisting of:<br>1 pc SPME Arrow Tool<br>1 pc Smart SPME Arrow Assortment Kit (SARR1115-SEL5-S2)<br>1 pc SPME Performance Evaluation Mix<br>requires adaptation kit for GC specific injector |
| Heatex Stirrer<br><br>PAL3-HeatexStirrer |  | For powerful mixing and heating in sample prep and SPME Arrow<br>• Temperature range 40 ° - 150 °C<br>• Stirring speed up to 1600 rpm (i.e. 200 cycloidal loops)<br>• Optimized for 20 ml vials (for 10 ml vials special adapters are required) |
| Vortex Mixer<br><br>PAL3-Vortex-Mixer |  | For efficient mixing (dilution / extraction)<br>• Standard vial sizes: 2 ml / 10 ml / 20 ml<br>• 1 additional slot for custom specific vials<br>• Provides efficient mixing with up to 2000 rpm |

PAL SYSTEM
Ingenious sample handling

Contact the experts for sample preparation:

Or find your nearest value added reseller.

For more information on PAL System visit:

www.palsystem.com



PAL Broch-ITEX
Rev 3 | August 2024