ITEX

In-Tube Extraction Sample Preparation for Gas Chromatography

...CTC ANALYTICS

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In-Tube Extraction

ITEX, a new Sample Preparation Technique for Gas Chromatography

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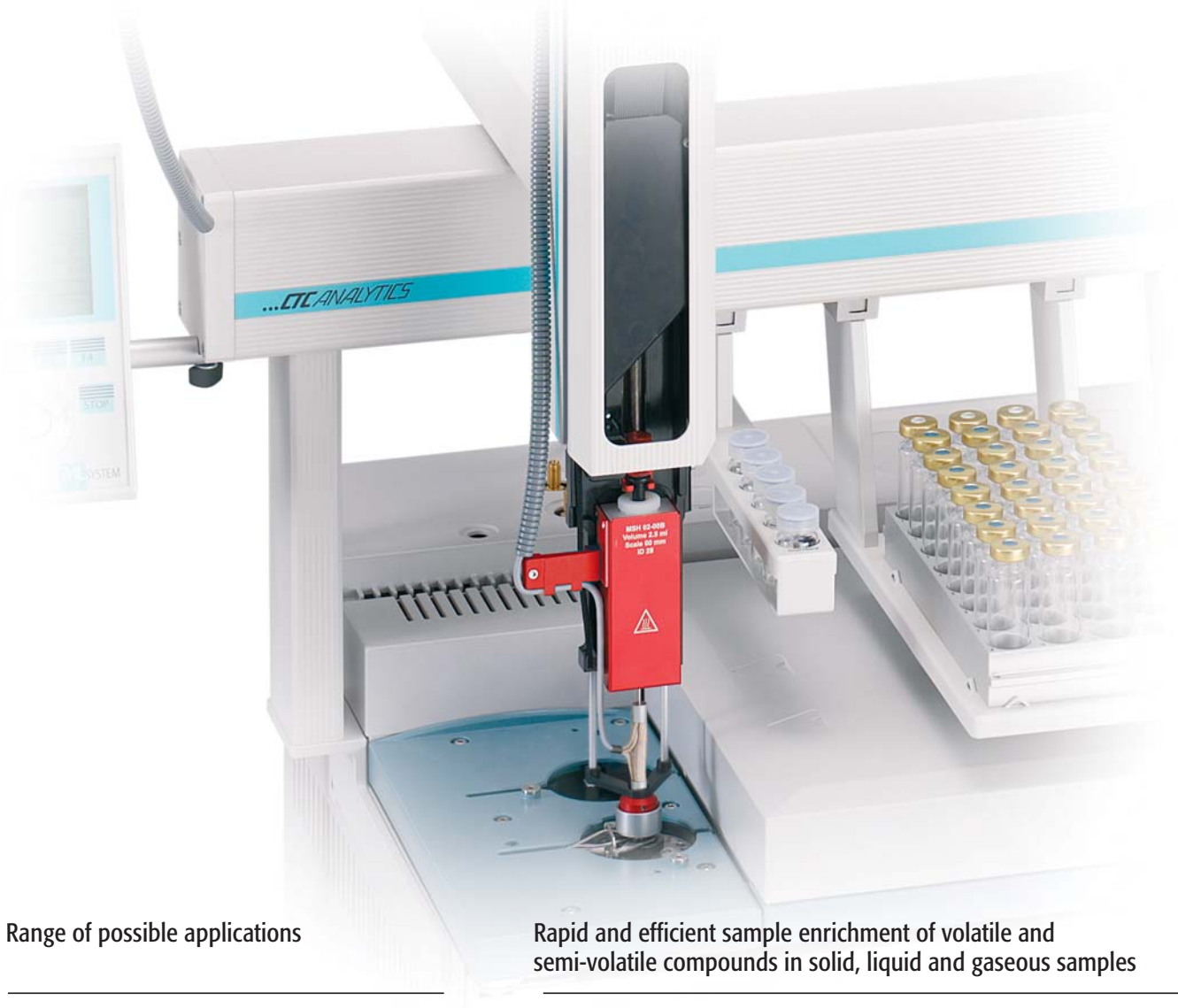


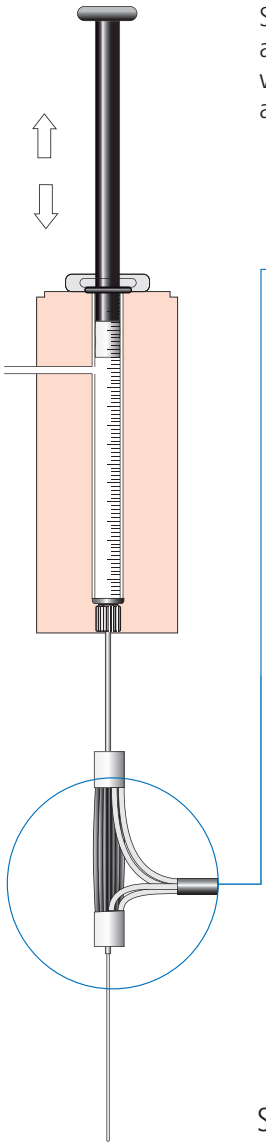
Range of possible applications

- Environmental
- Drinking Water
- Foods / Flavours
- Consumer Products
- Forensics / Toxicology
- Petrochemicals
- Polymers
- Pharmaceuticals
- Residual Solvents

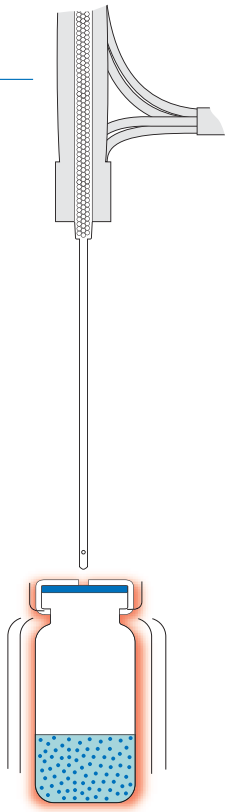
Rapid and efficient sample enrichment of volatile and semi-volatile compounds in solid, liquid and gaseous samples

- In-tube extraction and direct thermal desorption using proven industry standard adsorbents
- Syringe only concept for transparent sample handling, no sample loops, transfer lines, or switching valves
- No GC injector modifications, no cryo-focussing necessary Top mounted on GC's, saves valuable bench space
- Interfaces with any CombiPAL System controlled by all major GC/GC-MS Systems

ITEX Sample Extraction Procedure

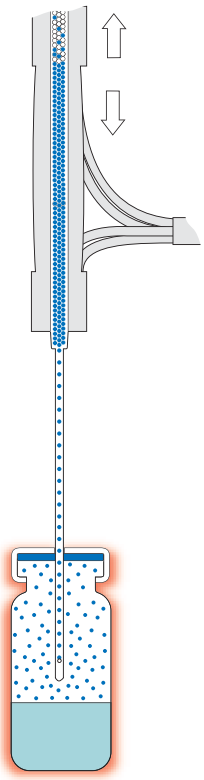


Sample is heated and / or agitated in a sealed sample vial until equilibrium is achieved



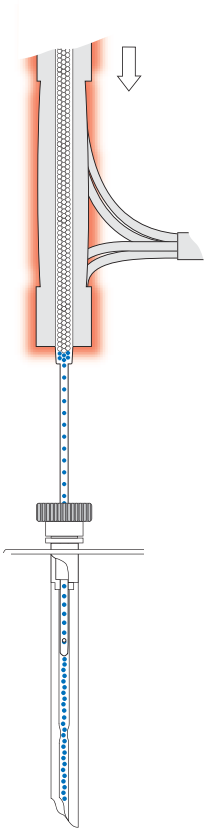
Sample conditioning ⇨

The ITEX Trap needle pierces the sealed sample vial and the heated gas tight syringe pumps the headspace through the ITEX Trap



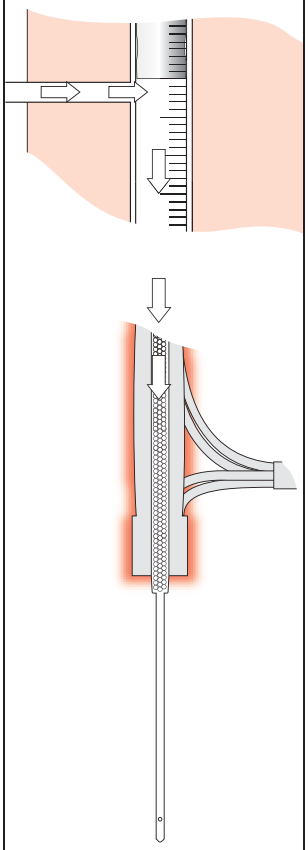
Adsorption ⇨

The loaded ITEX Trap is flash heated up to 350°C and desorbed into the hot GC Injector



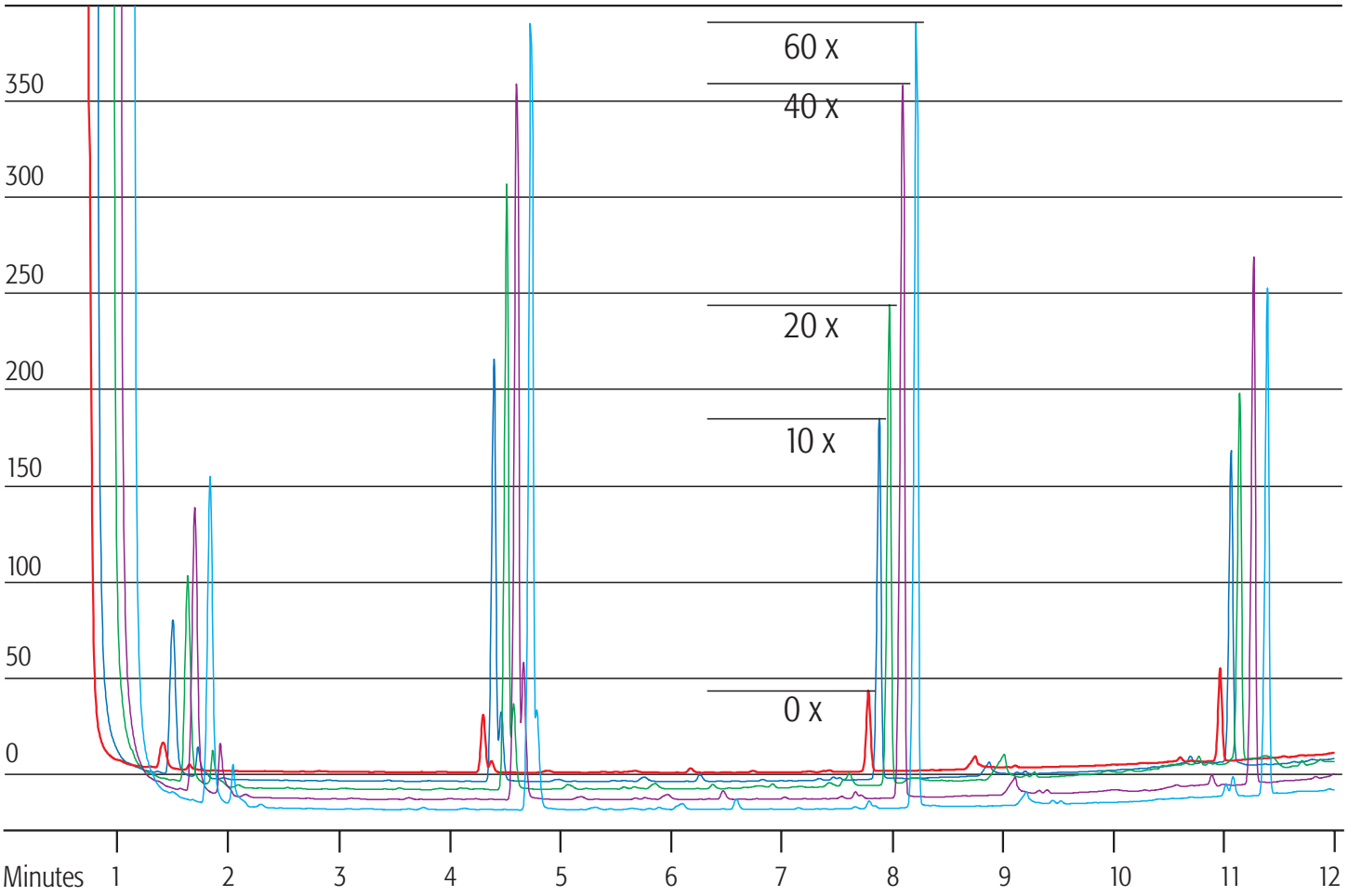
Desorption ⇨

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



Trap cleaning

Enrichment | Relationship between Sample Volume & Sensitivity



Enrichment of Methylesters on a Tenax TA ITEX trap. 1µl of a mixture of C_4, C_6, C_8, C_{10} Methylesters in Methanol (100ng/µl) was injected into a 20ml HS-vial.
After conditioning at 40°C for 10min. 1ml of the headspace using an 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.

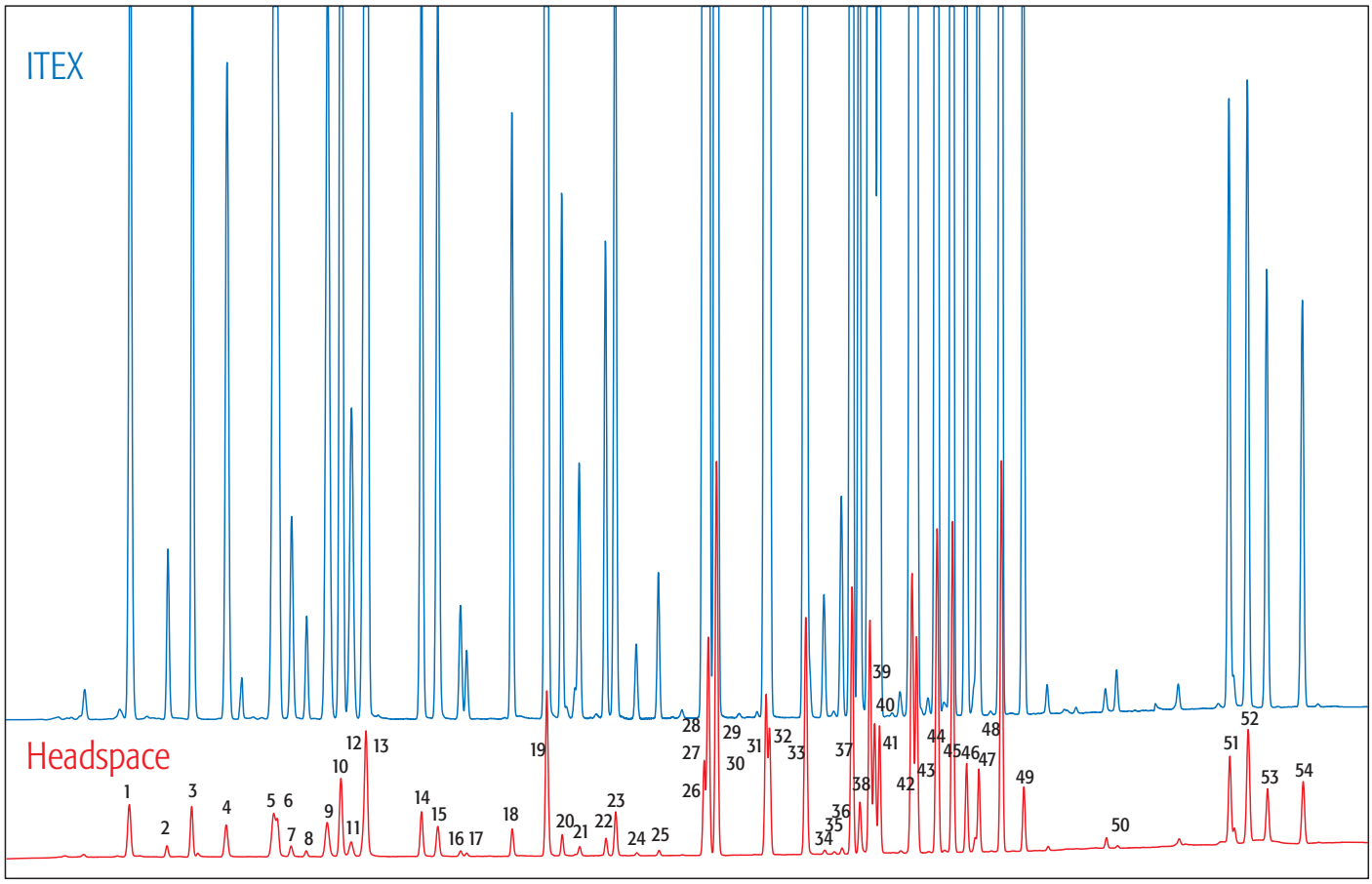
ITEX Parameter:

Extraction Speed: 100µl/sec.
Extraction 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 - 1min. - 10°C/min to 200°C
Detection: FID 250°C

Environmental Application | EPA Method 502.2



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

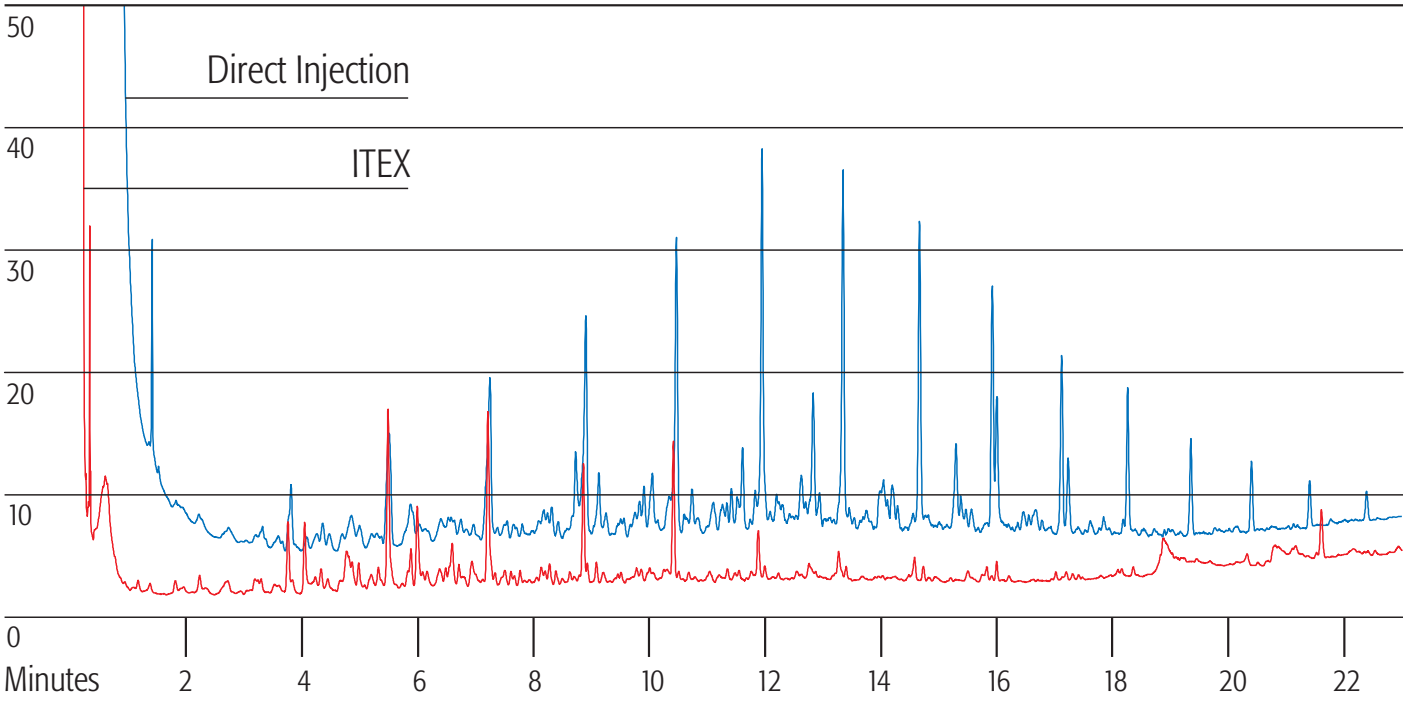
Column: Rtx-502.2 60m x 0.32mm ID, 1.8µm film

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

Detection: FID 250°C

1 1,1-Dichloroethylene
2 Methylene chloride (dichloromethane)
3 trans 1,2-Dichloroethylene
4 1,1-Dichloroethane
5 2,2-Dichloropropane
6 cis-1,2-Dichloroethylene
7 Chloroform
8 Bromochloromethane
9 1,1,1-Trichloroethane
10 1,1-Dichloropropene
11 Carbon tetrachloride
12 1,2-Dichloroethane
13 Benzene
14 Trichloroethylene
15 1,2-Dichloropropane
16 Bromodichloromethane
17 Dibromomethane
18 cis-1,3-Dichloropropylene
19 Toluene
20 trans-1,3-Dichloropropylene
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
35 1,1,2,2-Tetrachloroethane
36 1,2,3-Trichloropropane
37 n-Propylbenzene
38 Bromobenzene
39 1,3,5-Trimethylbenzene
40 2-Chlorotoluene
41 4-Chlorotoluene
42 tert-Butylbenzene
43 1,2,4-Trimethylbenzene
44 sec-Butylbenzene
45 4-Isopropyloluene (p-Cymene)
46 1,3-Dichlorobenzene
47 1,4-Dichlorobenzene
48 n-Butylbenzene
49 1,2-Dichlorobenzene
50 1,2-Dibromo-3-chloropropane
51 1,2,3-Trichlorobenzene
52 Hexachloro-1,3-butadiene (Hexachlorobutadiene)
53 Naphthalene
54 1,2,3-Trichlorobenzene

Comparison Direct Injection vs ITEX | Diesel Oil



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.

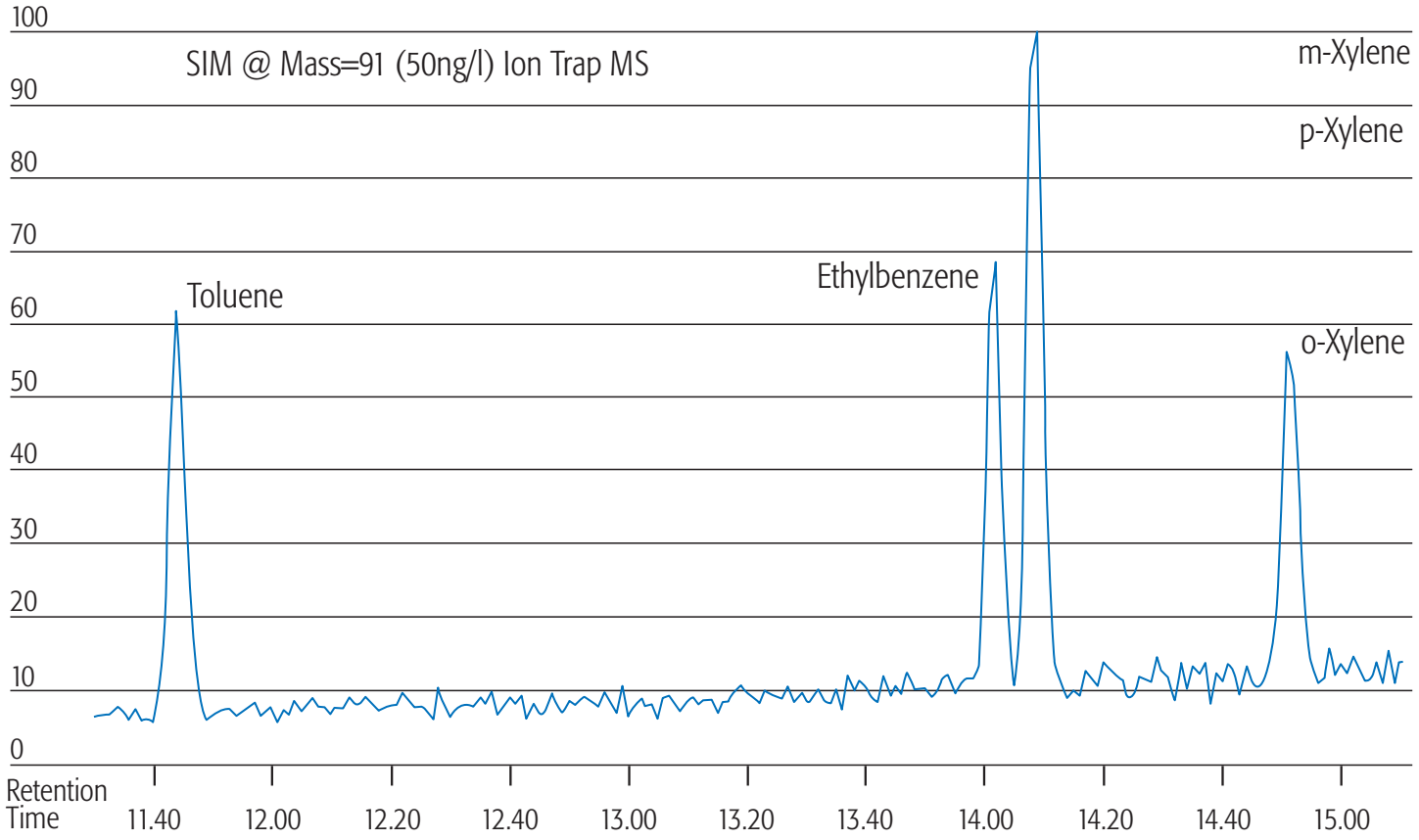
ITEX Parameter:

Extraction Speed: 120µl/sec.
Extraction 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 - 1min. - 10°C /min to 200°C
Detection: FID 250°C

Environmental Application | VOC, BTEX in Water ppt Level



Chromatogram shows BTEX Compounds at a concentration of 50ng/l using 20 Extraction strokes

Monitoring of BTEX and VOC Compounds in Water (EPA Method 502.2)

Key Words: VOC, BTEX, EPA Method 502.2, ITEX

BTEX and VOC Compounds according to EPA Method 502.2 are analysed using ITEX sample preparation technique. Total sample preparation time of less than 15 minutes allows a high sample throughput.

Sample Preparation:
10ml water is filled into 20ml Headspace sample vials. 3g Sodium chloride and 1µl of the internal standard VOC (50ppb Fluorobenzene in Ethanol) is added. After sample conditioning at 60°C during 10 minutes 20 strokes of the headspace are pumped through the ITEXtrap with a velocity of 100µl/sec. The resulting sensitivity is sufficient to obtain the requested detection limit for drinking water of 0.05µg/l.

ITEX Conditions:

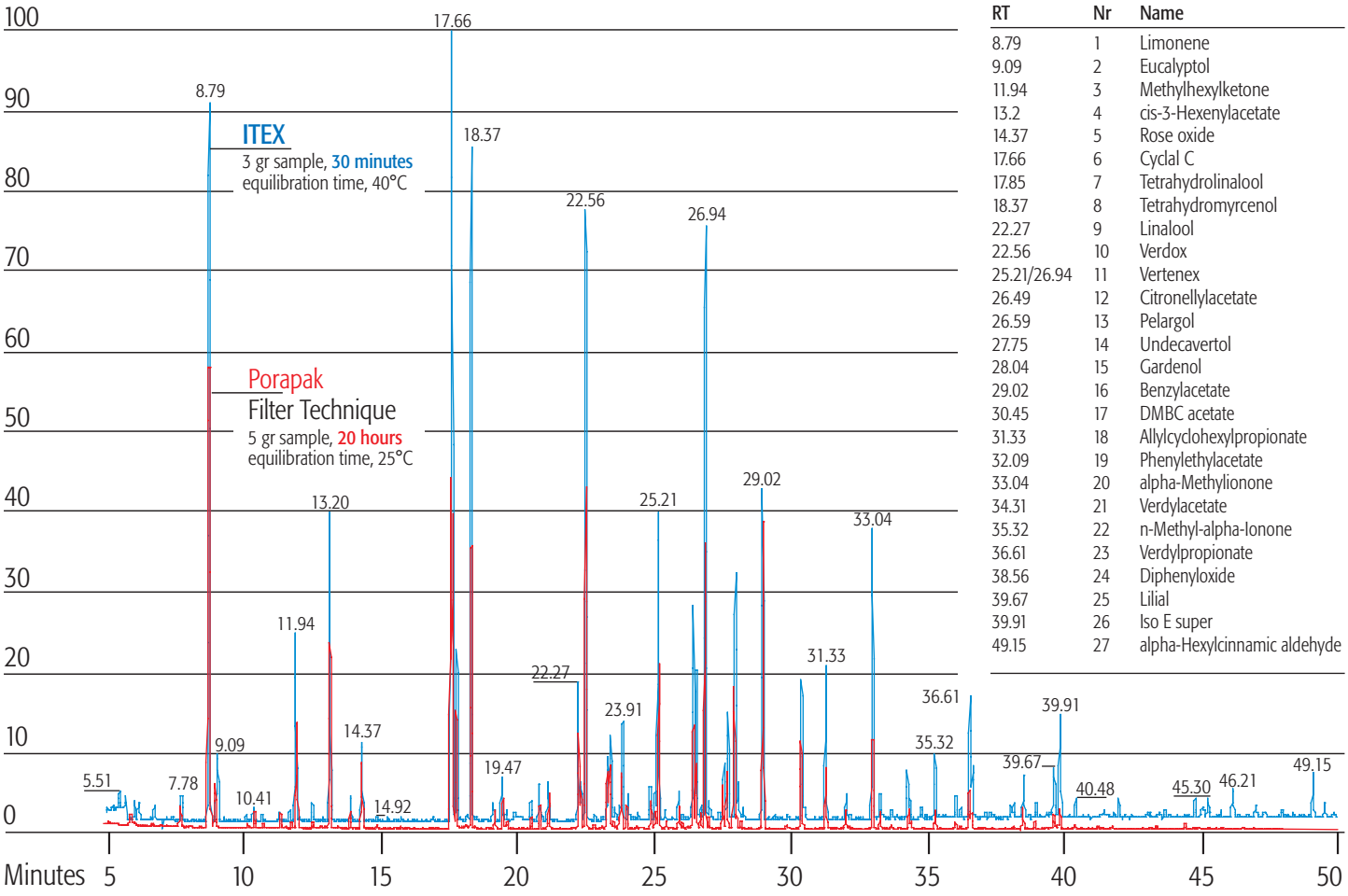
Sample Conditioning @ 60°C, 10 min.
Extraction Strokes: 20 x 1ml
Desorption @ 230°C with 1.3ml Headspace 20µl/sec.

Chromatography:

Column: Rtx-502.2, 60m x 0.32mm, 1.8µm film
Carrier Gas: Helium 20psi
Temperature Program: 40°C - 2 min.- to 240°C - 2 min. at 10°C / min.
Precolumn: 1m x 0.32mm deactivated with DPTMDS
Injector: Gerstel KAS3 with septa @ 150°C isothermal
GC: Varian 3300
Detector: Varian Saturn 4D GC/MS/MS

Courtesy of: Labor Veritas,
Engimattstrasse11, CH-8027 Zürich, Natascha Meggiolaro

Perfume Application | Softener-Comparison Porapak Filter Technique vs. ITEX



RT	Nr	Name
8.79	1	Limonene
9.09	2	Eucalyptol
11.94	3	Methylhexylketone
13.2	4	cis-3-Hexenylacetate
14.37	5	Rose oxide
17.66	6	Cyclal C
17.85	7	Tetrahydrolinalool
18.37	8	Tetrahydromyrcenol
22.27	9	Linalool
22.56	10	Verdox
25.21/26.94	11	Vertenex
26.49	12	Citronellylacetate
26.59	13	Pelargol
27.75	14	Undecavertol
28.04	15	Gardenol
29.02	16	Benzylacetate
30.45	17	DMBC acetate
31.33	18	Allylcyclohexylpropionate
32.09	19	Phenylethylacetate
33.04	20	alpha-Methylionone
34.31	21	Verdylacetate
35.32	22	n-Methyl-alpha-Ionone
36.61	23	Verdylpropionate
38.56	24	Diphenyloxide
39.67	25	Lilial
39.91	26	Iso E super
49.15	27	alpha-Hexylcinnamic aldehyde

Chromatography (Thermo TraceGC):

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

ITEX conditions:

Incubation temp	40°C
Incubation time	30 min
Syringe temp	45°C
Extraction volume	1000 ml per stroke
Extraction strokes	10
Extraction speed	100 ml/sec
Desorption temp	200°C
Desorption speed	100 ml/sec
Flush time	5 min

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

Specifications

ITEX adsorption step out of a sample vial

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 Time:
0 - 300 seconds

Pumping Syringe and Trap Cleaning:
Inert gas purging, 30sec. - 600sec.

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

Conclusion

This new approach, ITEX or In-Tube Extraction, an automated sample adsorption and thermal desorption technique shows a great potential for volatile compounds in various matrices.

The adsorption at the trap does continuously reduce the concentration of analytes in the headspace and disturbs the equilibrium in the vial. The result is a pumping effect of the analytes from the sample matrix to the headspace. This effect is similar as used by the SPME technique but the active control over the pumping strokes allows the user to utilize rather a dynamic headspace as known by the Purge&Trap technique.

The sensitivity level to be reached can be as far down as the ppt level.

Using the flush gas and a user selectable trap heating temperature does allow to clean the trap very efficiently. Blank runs, even after several hundred injections, do not show a carry over effect. We are aware that this statement can not be generalized. It depends on the sample and matrices. The technique is still too young to be able to give a general statement