

ITEX Dynamic Headspace Application Notes



ENVIRONMENTAL



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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.

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Keywords: **VOC, BTEX, EPA Method 502.2, ITEX**

Sample Preparation:

10ml water are filled in 20ml Headspace sample vials. 3g Sodium chloride and 1µl of the internal standard IS 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 ITEX-trap 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.

Trap material: Tenax TA 80/100mesh

Chromatography:

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

Carrier Gas: Helium 20psi

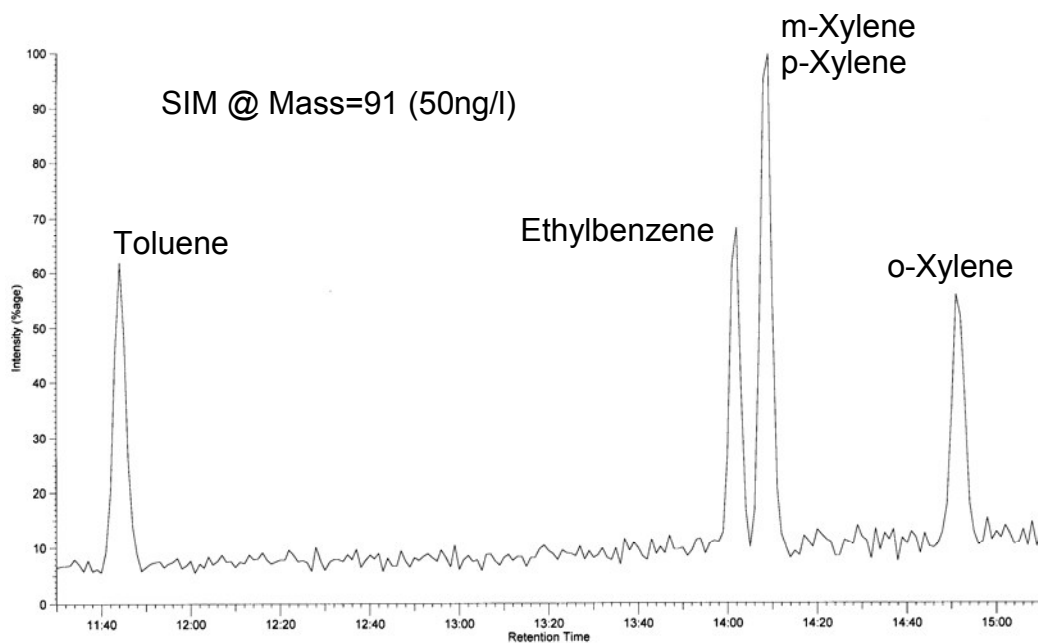
Temperature Program: 40°C (2 min.) to 240°C (2 min.) at 10°C/min.

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



Chromatogram1 shows BTEX Compounds at a concentration of 50ng/l using 20 Extraction strokes

Analysis of Suspected Flavor and Fragrance Allergens in Lotion Samples. A comparison between Static Headspace, SPME, HSSE and ITEX Headspace Sampling

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Keywords: **Allergens, cosmetics, headspace sampling, ITEX**

Abstract

Suspected flavor and fragrance allergens were determined in an alcohol/water based lotion using ITEX headspace sampling. The dynamic headspace sampling was compared to static headspace, SPME and headspace sorptive extraction.

Introduction

According to recent EU regulation [1], 27 suspected allergen compounds should be monitored in cosmetic products. Depending on the sample matrix and solute concentrations, different sample preparation methods are developed and applied [2]. For the determination of suspected allergens in cosmetic products, one of the major problems is related to the presence of detergents that contaminate the analytical system if the samples are introduced without selective sample preparation. Selective extraction or selective sample introduction is however not easy since the target compounds cover a broad volatility range (from limonene to benzyl benzoate) and polarity range (from relatively polar benzyl alcohol, $K_{ow}=1.1$, to apolar benzyl benzoate, $K_{ow}=4.0$). Liquid sample introduction with selective retention of non-volatiles in a PTV liner [3] or sorptive extraction using a PDMS coated stir bar [2] have been used for this application. Sampling from the headspace, using static headspace, dynamic headspace, SPME or headspace sorptive extraction (HSSE) can also be considered as these techniques avoid contamination of the analytical system by high molecular weight material such as detergents. The method of choice should however give ppm sensitivity, on one hand, and avoid discrimination of the target solutes based on relative volatility or polarity, on the other hand. In this application note, the use of dynamic headspace extraction using ITEX is demonstrated. The technique is compared to classical static headspace, solid phase micro-extraction (SPME) and to headspace sorptive extraction, using a polydimethyl siloxane (PDMS) coated stir bar in the headspace of the sample. The latter two techniques are similar in concept, only the total amount of sorptive PDMS phase is different [2,4].

Sample Preparation

As typical sample an alcohol/water based lotion was analysed. The lotion is used in wet wipes and contains besides different detergents also a fragrance. For each method, 100 mg sample was placed in a 20 mL headspace vial. Two internal standards (1,4-dibromobenzene and 4,4'-dibromobiphenyl) were added at 10 ppm level, according to the reference method for the determination of allergens in perfumes described by Chaintreau et al [5].

Static HS conditions

Sample Conditioning @ 80°C, 15 min
HS needle: 2.5 mL, 90°C
Injection: 1 mL; 350 µL/s; 1/10 split ratio

ITEX conditions

Sample Conditioning @ 80°C, 15 min
Extraction Strokes: 10 x 1 mL; 50 µL/s
Desorption @ 250°C with 1 mL headspace; 50 µL/s
Trap Material Tenax TA 80/100mesh

SPME conditions

Fiber: 100 µm PDMS
Sample Conditioning @ 80°C, 15 min

Desorption @ 250°C, 2 min

Headspace Sorptive Stirbar Extraction conditions

Sample Conditioning @ 80°C, 15 min

HSSE sampling in headspace: 10 mm x 0.5 mm df Twister™

Desorption @ 250°C during 10 min in splitless mode

Cryo-focussing @

Injection: -100°C @ 600°C/min to 250°C, 1/10 split ratio

GC conditions

All analyses were performed on an Agilent 6890 GC – 5975 MSD combination.

Column: 30 m x 0.25 mm i.d. x 0.25 µm df HP-5MS (Agilent)

Carrier gas: helium, 168 kPa constant pressure at inlet (column outlet pressure: 28 kPa using AUX EPC and QuickSwap connector) (*)

Inlet: split, 250°C, 1/10 split ratio

Oven temperature program: 50°C, 1 min, 8°C/min to 270°C.

MSD transfer line: 250°C (17 cm x 110 µm i.d. restrictor, 28 kPa)

Detection: MS in scan mode , scan range: 40-350 amu

(*)under these conditions, alpha isomethyl ionone elutes at 15.5 min. These settings were used to performe the analyses under retention time locked conditions [2].

Results

In Figure 1, the total ion chromatogram obtained for the lotion sample using classical static headspace sampling is given. The internal standards, added at the same concentration level, are detected at 10.3 min and 23.1 min respectively. The response for the first internal standard is higher in comparison to the second internal standard, corresponding to their relative volatility.

In this sample, some allergens could be detected. Linalool (peak 1) and hexyl cinnamaldehyde (peak 6) are easily detected. Other allergens are only detected as traces and confirmation of their presence by mass spectral comparison with a library spectrum is difficult.

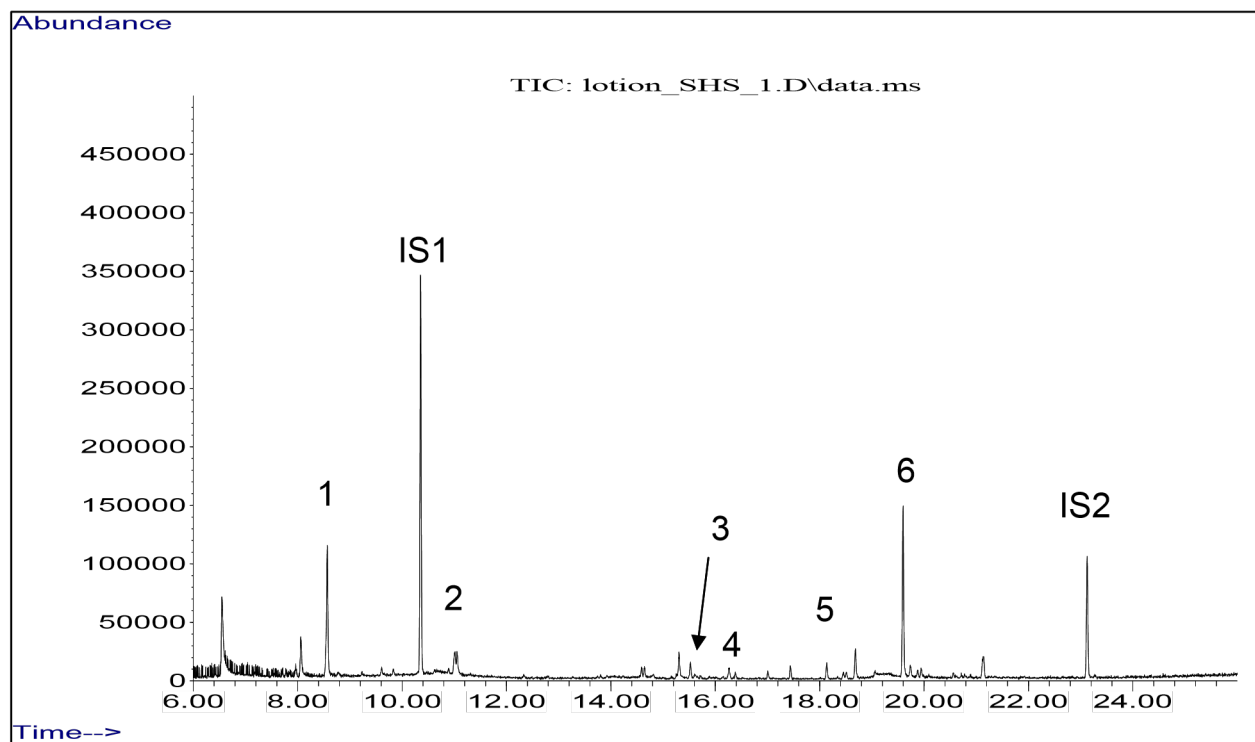


Figure 1: static headspace

The chromatogram obtained by ITEX headspace sampling is shown in Figure 2. A much higher sensitivity is obtained in comparison to static headspace and several flavor and fragrance solutes could be detected. It is very interesting to observe that the response for the two internal standards is nearly equal, corresponding to their equal concentration in the sample. In this analysis, 6 allergens are detected and their presence could easily be confirmed by the mass spectra. Following allergens

are present: 1. linalool, 2. citronellol, 3. alpha isomethyl ionone, 4. linal, 5. amyl cinnamaldehyde and 6. hexyl cinnamaldehyde.

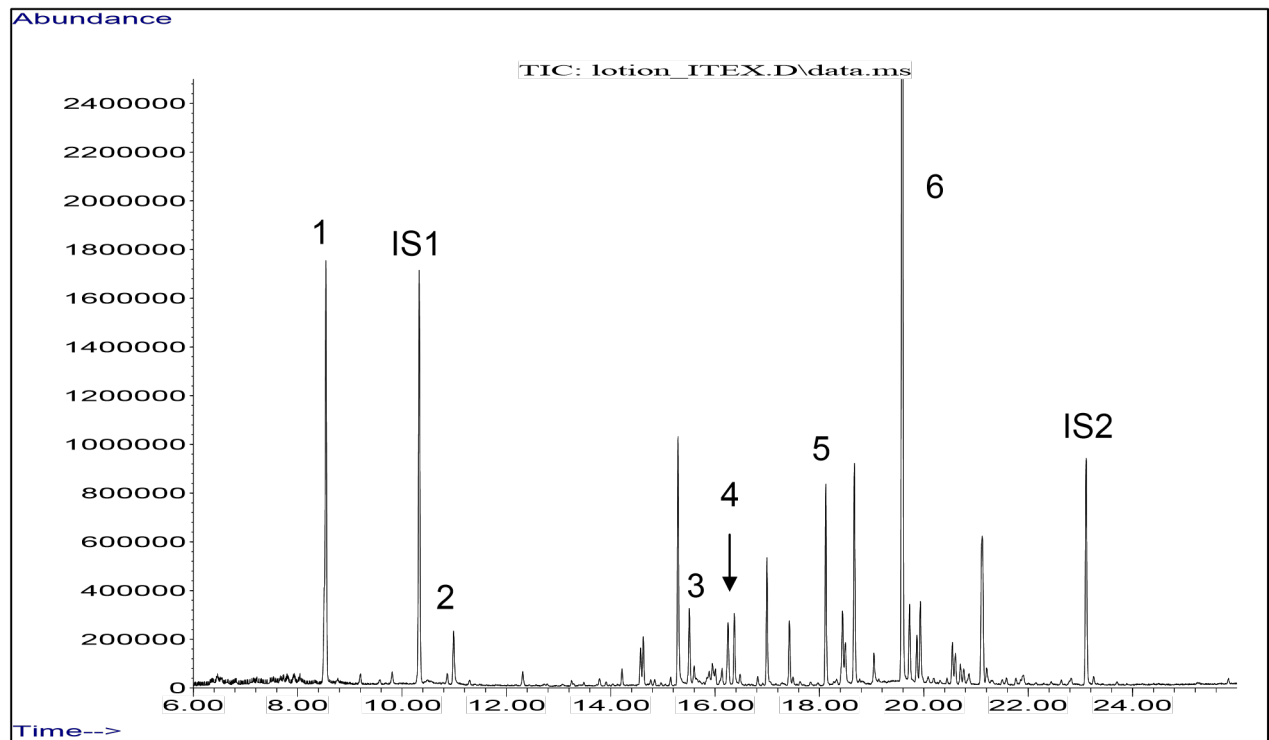


Figure 2: ITEX

The chromatogram obtained by SPME headspace sampling is shown in Figure 3. Excellent enrichment is obtained and all 6 allergens could easily be detected. However, it is interesting to observe that the response of the second internal standard is much higher than for the first eluting internal standard. This difference can be explained due to the higher partitioning coefficient between PDMS and air for the higher molecular weight, later eluting compound. From the whole chromatogram it is clear that the less volatile compounds, having higher $K_{PDMS/air}$ coefficients, are more enriched in comparison to more volatile solutes. The responses of the target solutes largely vary in function of the $K_{PDMS/air}$ coefficients. This corresponds well with theoretical predictions [6].

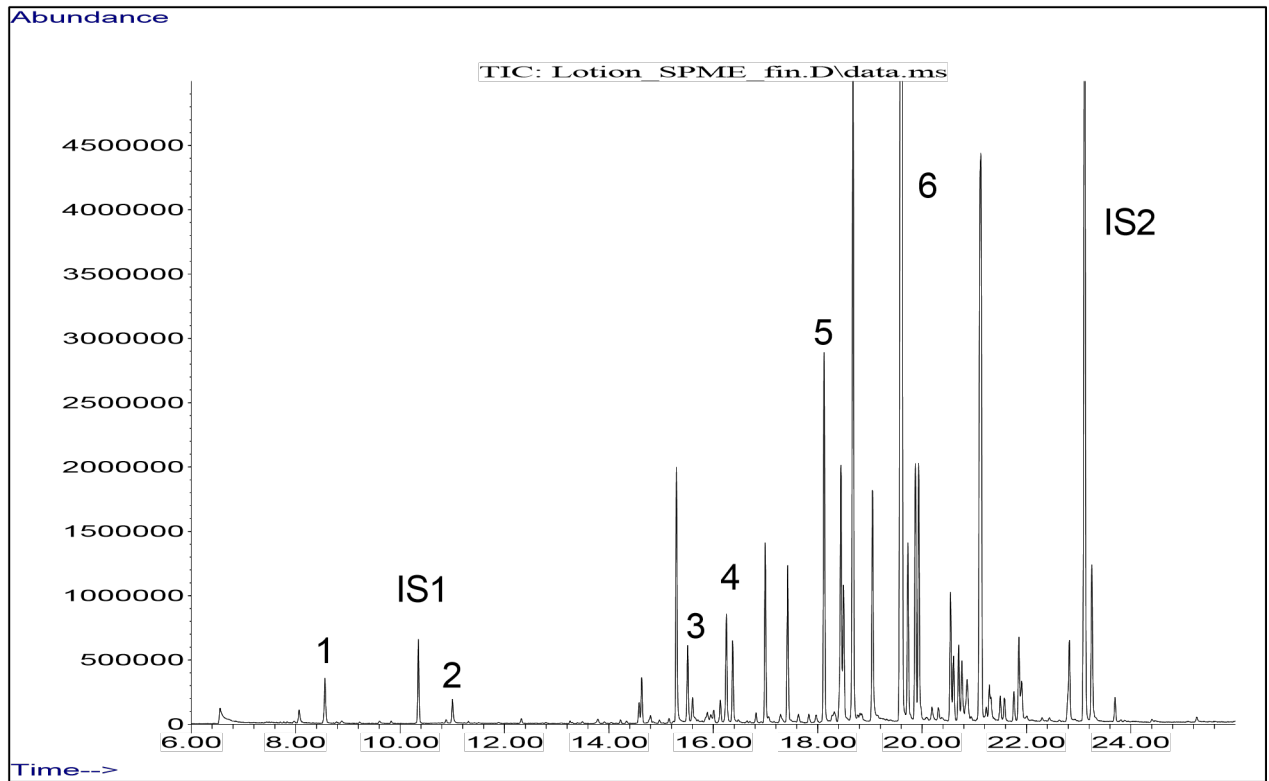


Figure 3: SPME

The chromatogram obtained by headspace sorptive extraction sampling on a 1 cm stir bar coated with 0.5 mm PDMS is shown in Figure 4. As in SPME, excellent enrichment is obtained, but now the response of two internal standards is nearly equal, corresponding to their equal concentration in the sample. Since more PDMS material is available, quantitative recovery is obtained at lower $K_{PDMS/air}$ coefficients and the profile is very similar to the profile obtained by ITEX sampling. In this analysis, 6 allergens are also detected and their presence could easily be confirmed by the mass spectra.

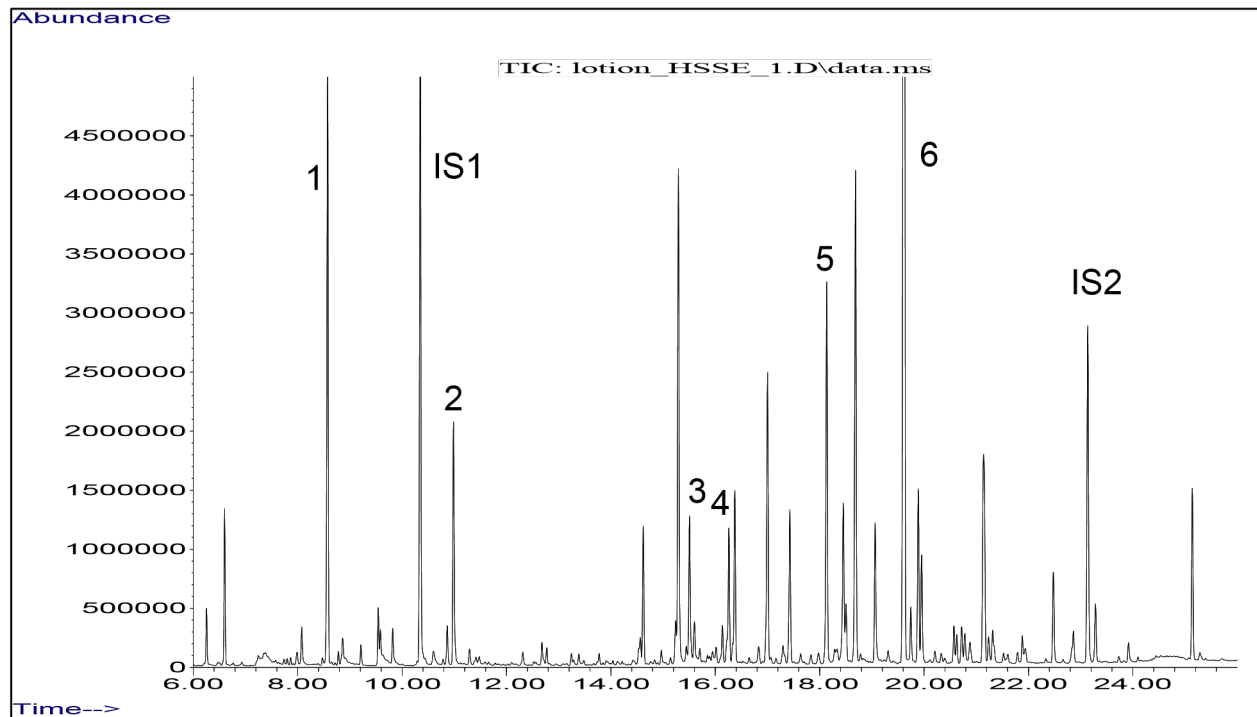


Figure 4: HSSE (Twister™)

Conclusion

For the determination of flavour and fragrance allergens in cosmetics, ITEX headspace sampling results in much higher sensitivity than static headspace. The obtained profile is similar to the profile obtained by headspace sorptive sampling (using a Twister™ stir bar in headspace). In comparison to SPME, the relative response of the solutes is less dependent on the individual $K_{\text{PDMS/air}}$ coefficients of the target solutes. The sensitivity of the ITEX determination can be increased if the number of extraction strokes would be increased.

References

1. Directive 2003/15/EC, Official Journal of the European Union, L 66/26, 11.3.2003
2. F. David, C. Devos and P. Sandra, LC.GC Europe, 19, 602-616, November 2005
3. F. David, C. Devos, D. Joulain, A. Chaintreau and P. Sandra, J. Sep. Science, 29, 1587-1594 (2006)
4. E. Baltussen, P. Sandra, F. David, C.A. Cramers, J. Microcol. Sep. 11, 737-747 (1999)
5. A. Chaintreau, D. Joulain, C. Marin, C.-O. Schmidt and M. Vey, J. Agric. Food Chem, 51, 6398-6404 (2003)
6. B. Tienpont, F. David, C. Bicchi, P. Sandra, J. Microcolumn Separations 12, 577-584 (2000).

Arson Detection using ITEX Headspace Sampling

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Keywords: **Arson Detection, ITEX Headspace Sampling**

Abstract

The analysis of residues of fire accelerants in fire debris samples can be used for arson detection. The analysis can be performed by ITEX sampling, followed by GC-MS analysis. In comparison to static headspace, the sensitivity is increased by a factor of 10 using ITEX enrichment.

Introduction

The detection of residues of fuels (gasoline, naphtha, kerosene) or organic solvents such as paint thinner in fire debris samples is an important application in forensic analysis. The target compounds include C5-C12 hydrocarbons, aromatic hydrocarbons, ethers and alcohols (methanol).

Different methods are used for this analysis including static and dynamic headspace, solid phase microextraction, etc. Using static headspace, the sensitivity of the method is often not high enough to detect traces of solvent residues. Higher sensitivity can be obtained using dynamic headspace with enrichment of the volatile organic compounds that are characteristic for fire accelerators. These solutes can be enriched on a Tenax trap. Consequently the solutes are desorbed from the trap and analyzed by GC-MS. Enrichment of VOCs from the headspace of solid or liquid samples can be done fully automated using the in-tube extraction (ITEX) option on the CTC Combipal sampler. Enrichment is done in a Tenax packed modified syringe.

Sample Preparation

Typically 1-5 g material is placed in a 20 mL headspace vial and the samples are analyzed as such.

Materials introduced are not homogeneous and therefore often multiple samples are analyzed.

For the example showed below, two fire debris samples were taken from a burned wooden floor. Sample A was taken at the place where the fire started and sample B was taken at an area away from the original fire location. From both samples similar amounts were introduced in a 20 mL headspace vial and the vial was sealed.

ITEX conditions

Sample Conditioning @ 80°C, 10 min

Extraction Strokes: 20 x 1 mL; 50 µL/s

Desorption @ 250°C with 1 mL headspace; 50 µL/s

Trap Material Tenax TA 80/100 mesh

GC conditions

The analysis was performed on an Agilent 6890 GC – 5975 MSD combination.

Column: 20 m x 0.18 mm i.d. x 1 µm df DB-VRX (Agilent)

Carrier gas: helium, 170 kPa constant pressure at inlet (column outlet pressure: 28 kPa using AUX EPC and QuickSwap connector)

Inlet: split, 1/10 split ratio

Oven temperature program: 35°C, 2 min, 8°C/min to 190°C, 20°C/min to 250°C, 2 min. Detection: MS in scan mode (33-300 amu)

Results

The total ion chromatogram obtained for sample A (suspected sample) is given in Figure 1. The most abundant peaks are identified as alpha-pinene (12.5 min), camphene (12.9 min), and limonene (14.8 min). These compounds are typical pyrolysis products from wood and are no indicators for fire accelerants.

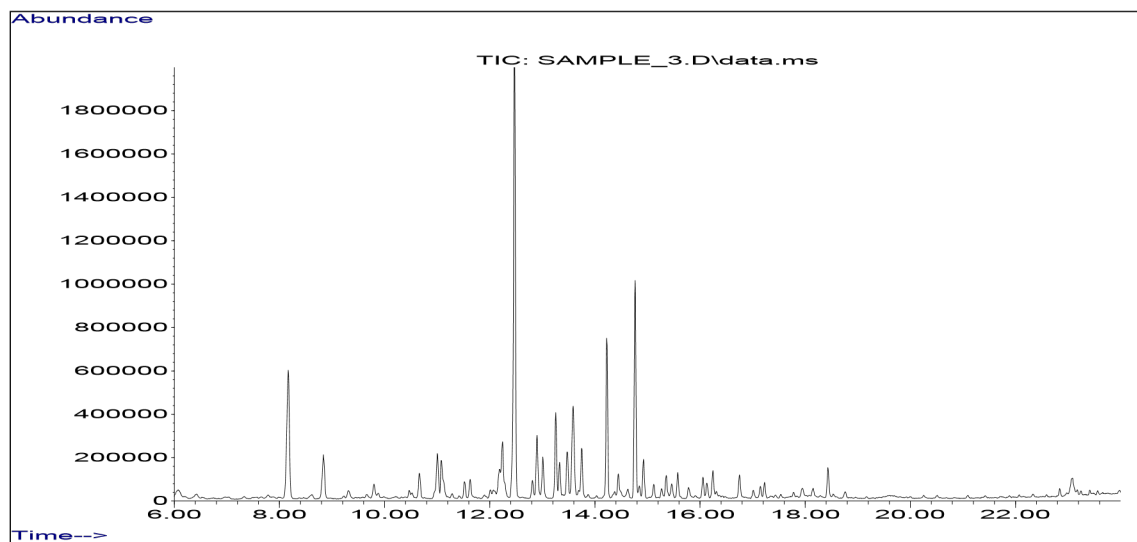


Figure 1

The total ion chromatogram obtained for sample B (believed to be blank) is given in Figure 2. The chromatogram is similar and the major peaks correspond also to the peaks detected in sample A (pyrolysis products of wood).

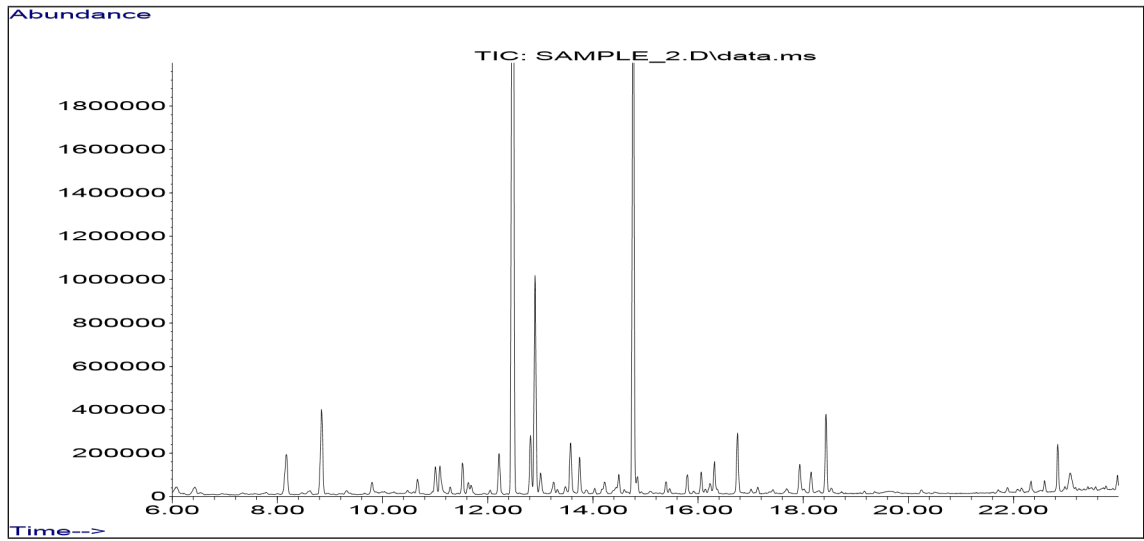


Figure 2

Using extracted ion chromatograms, it is however possible to differentiate both samples. The chromatograms below show the extracted ion chromatograms for m/e 120 (C3-aromatics) and m/e 134 (C4-aromatics) for respectively sample A (Figure 3) and sample B (Figure 4).

It is clear that in sample A, a typical profile of aromatic hydrocarbons is observed, while in sample B only one main peak is detected. This peak was identified as p.cymene and is also a pyrolysis product of wood. The profile of the aromatic hydrocarbons detected in sample A, on the other hand, corresponds to gasoline.

This could be confirmed by analyzing a blank sample spiked with a small amount of gasoline. In Figure 5, the profiles of the C3-aromatics in the spiked sample and in sample A are compared. It is clear that a good correspondence is obtained and that sample A contains traces of a fire accelerant, in this case gasoline.

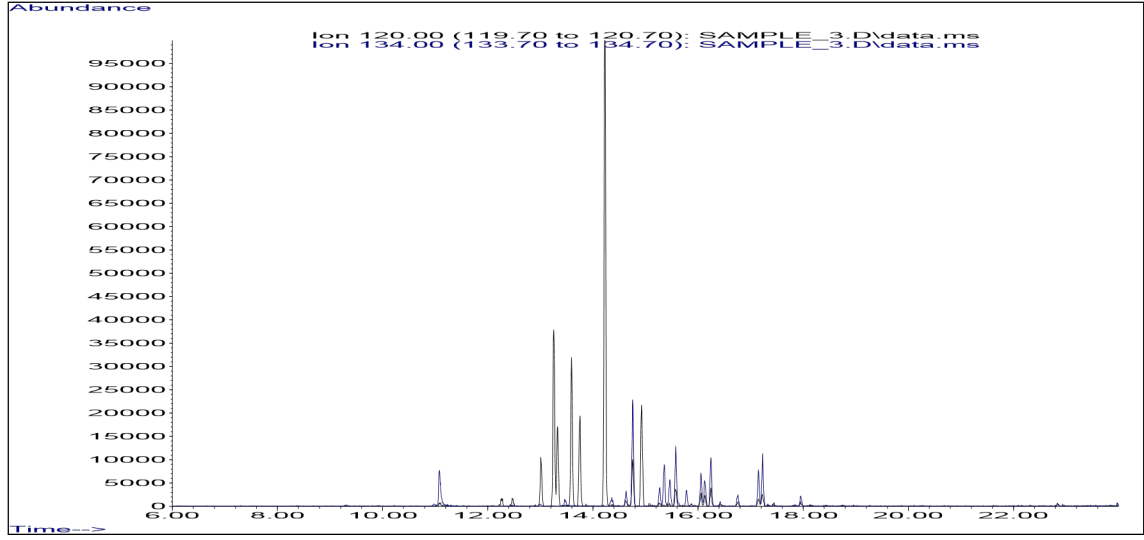


Figure 3

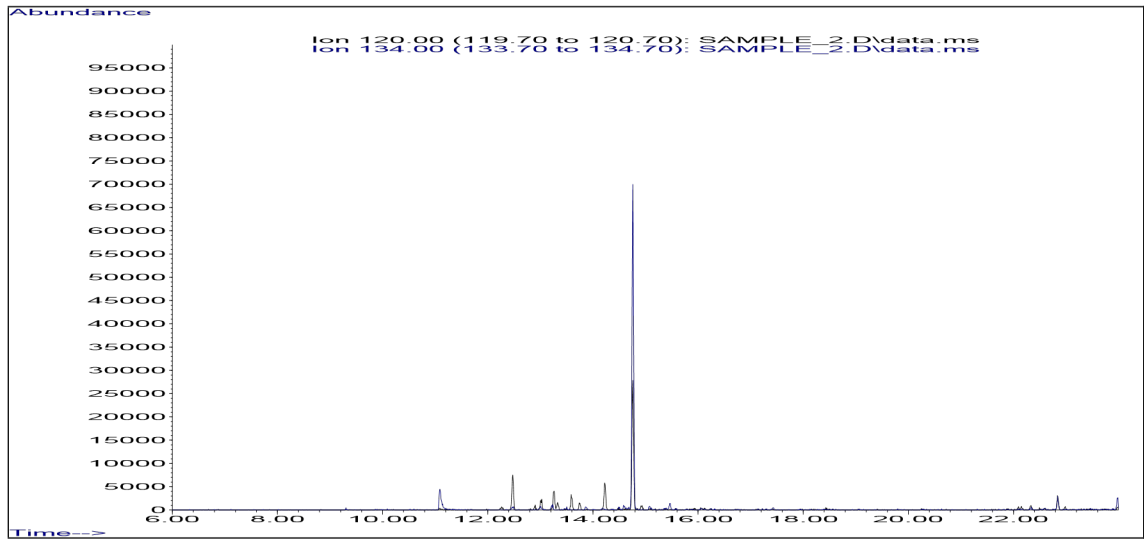


Figure 4

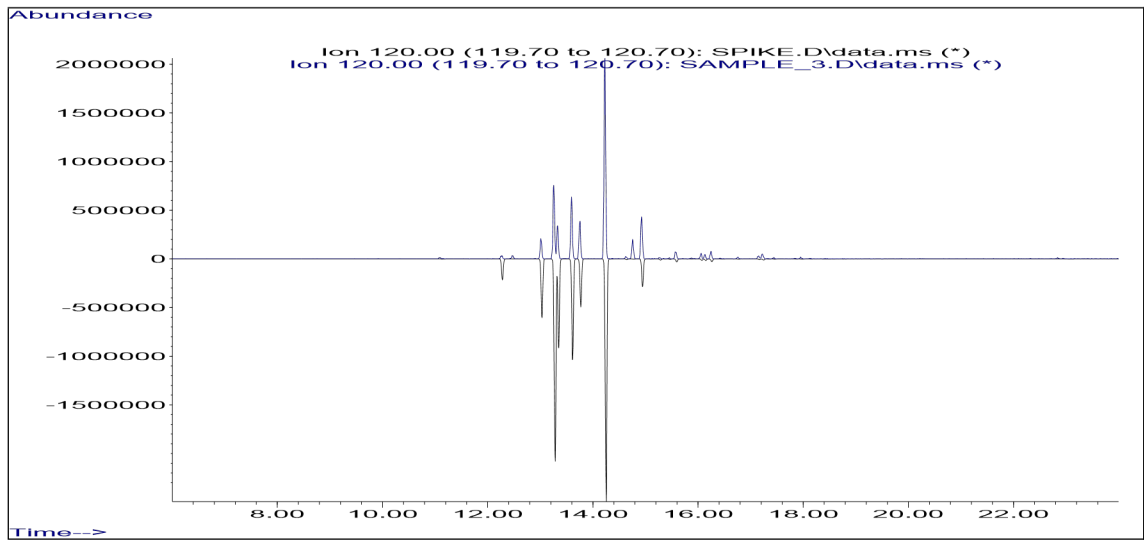


Figure 5: top: sample A (suspected sample); bottom: blank sample spiked with gasoline (reference)

Conclusion

Headspace sampling with enrichment of VOCs using the ITEX option was used for the detection of fire accelerants in fire debris samples. Excellent sensitivities are obtained, allowing detailed profiling of samples. In comparison to static headspace, the sensitivity of the ITEX-GC-MS was increased by a factor of 10, while no discrimination was observed in function of the boiling point of the solutes in the range from C5 to C15.

Analysis of Volatile Organic Compounds in Beer using ITEX Headspace Sampling

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Keywords: **Volatile Organic Compounds, VOC, alcohols, dimethyl sulphide, diacetyl, beer, headspace, ITEX**

Abstract

Using ITEX in combination with GC-MS, different classes of volatile compounds could be measured in the headspace of beer samples using one single run. Alcohols, esters and dimethyl sulfide are monitored at ppm level using ITEX in combination with GC-MS operated in scan mode. Trace levels of diketones, such as diacetyl, are monitored simultaneously at ppb level using MS in SIM mode.

Introduction

In quality control of beer samples, several volatile organic compounds are monitored. These compounds include C3-C5 alcohols, C2-C5 esters, dimethyl sulfide and 1,2-diketones (diacetyl). These compounds are present at different concentration levels ranging from tens of ppm (alcohols) to ppb level (diacetyl, 2,3-pentanedione).

Beer samples are normally analyzed by static headspace in combination with GC. In order to cover all solutes and concentration levels, often several runs are needed per sample. Alternatively, the analysis is performed using effluent splitting to three detectors: FID for alcohols and esters, selective sulfur detection (FPD, PFPD) for dimethyl sulfide and ECD for diketones. The three detectors allow sufficient sensitivity and selectivity, but this set-up is rather complicated and problems with splitters and robustness are often encountered. Using mass spectroscopic detection, all solutes can be detected, either using scan or SIM mode. Recently, simultaneous scan and SIM acquisition have been made possible on benchtop GC-MS systems. However, for some solutes, the sensitivity of mass spectroscopic detection is at the limit, especially in combination with static headspace. Dynamic headspace and even purge and trap sampling have been used to obtain higher sensitivities. Also solid phase micro-extraction can be used, but in general SPME fibers will show different affinities for the solutes and calibration is more difficult.

Enrichment of VOCs from the headspace of the beer can be done fully automated using the in-tube extraction (ITEX) option on the CTC Combipal sampler. Enrichment is done in a Tenax packed modified syringe. After enrichment, thermal desorption is performed by flash heating the syringe needle and injection in a hot GC inlet, followed by GC-MS analysis in simultaneous scan/SIM mode.

Sample Preparation

Typically 10 mL beer sample is placed in a 20 mL headspace vial and the samples are analyzed as such. For the example below, a Belgian pils beer was selected. A comparison was made between classical static headspace and ITEX sampling.

SHS conditions:

Sample Conditioning @ 80°C, 15 min
HS needle: 2.5 mL, 90°C
Injection: 1 mL; 500 µL/s

ITEX conditions:

Sample Conditioning @ 80°C, 15 min
Extraction Strokes: 10 x 1 mL; 50 µL/s
Desorption @ 250°C with 1 mL headspace; 50 µL/s
Trap Material Tenax TA 80/100mesh

GC/MS conditions:

The analysis was performed on an Agilent 6890 GC – 5975 MSD combination.
Column: 20 m x 0.18 mm i.d. x 1 µm df DB-VRX (Agilent)
Carrier gas: helium, 200 kPa constant pressure at inlet (column outlet pressure: 28 kPa using AUX EPC and QuickSwap connector)
Inlet: split, 1/25 split ratio
Oven temperature program: 40°C, 5 min, 10°C/min to 250°C, 10 min.
MSD transfer line: 250°C (17 cm x 110 µm i.d. restrictor, 28 kPa)
Detection: MS in scan/SIM mode
Scan: 29-400 amu
SIM: ions monitored: 43, 57, 86, 100 (50 ms dwell times)

Results

In Figure 1, the total ion chromatograms obtained for a beer sample using classical static headspace sampling (upper trace) and ITEX sampling (lower trace) are compared. Both chromatograms represent the datafiles obtained in scan acquisition mode.

Ethanol, the most abundant peak, elutes at 6 min. The peak at 4 min corresponds to the air peak (MS scan from m/e 29).

It is clear that more peaks are detected using the ITEX sampling. Following peaks could be identified using the mass spectra:
1. 1-propanol; 2. ethyl acetate; 3. 2-methyl-1-propanol; 4. ethyl propanoate; 5. 3-methyl-1butanol; 6. 2-methyl-1-butanol; 7. 2-methyl propyl acetate; 8. ethyl butyrate; 9. 3-methyl butyl acetate; 10. 2methyl butyl acetate.

Also dimethyl sulphide (DMS) could be detected at 7.7 min. Using an extracted ion chromatogram, the peak can be quantified without problem in the beer sample. The signal-to-noise, measured on ion m/e 62 was 70. The concentration of DMS in this sample was in the order of 10 ppb.

In the chromatogram obtained by static headspace, dimethyl sulfide was difficult to detect. Only in an extracted ion trace, a small peak with signal to noise of 5 could be detected, but no library search confirmation was obtained. The sensitivity was thus increased by a factor of more than 10 for this compound using ITEX sampling.

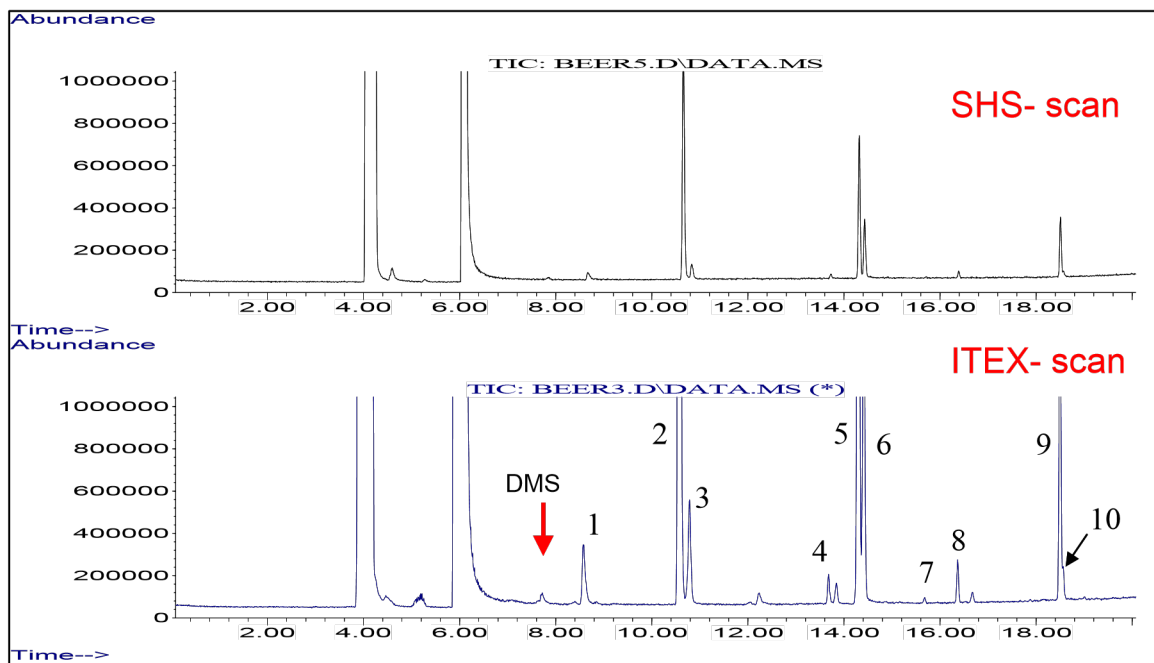


Figure 1

The extracted ion chromatograms for ion m/e 86 obtained by GC-MS in SIM mode are compared in Figure 2. Diacetyl elutes at 13.1 min. The peak can be detected in the chromatogram obtained by static headspace only as a trace ($S/N = 8$). Using ITEX, the peak can be detected more easily and confirmation of the identity through the relative ratios of target and qualifier ions is possible. The S/N value obtained by ITEX was 44 or 6 times higher than with static headspace. The concentration of diacetyl in this beer sample was also in the order of 10 ppb.

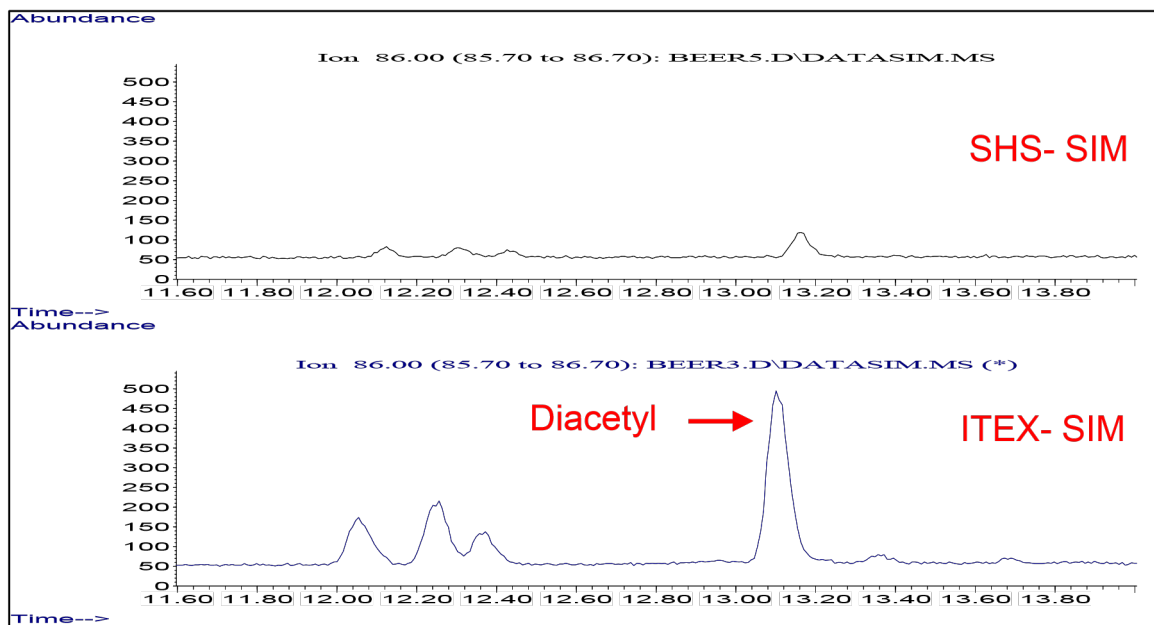


Figure 2

Conclusion

Using ITEX headspace sampling in combination with GC-MS operated in simultaneous scan/SIM mode, different important volatile organic compounds present in beer can be monitored in a single run. Volatile alcohols and esters and dimethyl sulfide can be measured using scan mode. Diacetyl is monitored in SIM. In comparison to classical static headspace, ITEX offers a gain in sensitivity of a factor 5 to 10 using 10 pumping strokes only to shorten the sample preparation time. If a lower detection limit is required can the number of pump strokes be increased?

Determination of Volatile Organic Compounds in Drinking and Surface Water with In-Tube-Extraction comprehensive GC/time-of-flight mass spectrometry

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Keywords: **VOC, Drinking Water, GC-TOF, Comprehensive GC, ITEX**

Abstract

A series of volatile organic compounds (VOCs) in drinking water are determined using In-Tube Extraction comprehensive GC/time-of-flight mass spectrometry (ITEX GCxGC/TOF-MS). A method for determining these compounds has been established and detection limits for a selected number of VOCs are depicted. Detection limits were below 20 ng/L for all compounds. A comparison with 'classical' Purge & Trap GC/MS is made. Repeatability experiments revealed an RSD of less than 20% for all the compounds, well within the demands of the Flemish decree on drinking water.

Introduction and Discussion

Antwerpse Waterwerken (AWW) is one of the largest drinking water companies in the Benelux, producing yearly 160 million m³ of drinking water. Therefore, the daily control of both drinking water and surface water (out of which the drinking water is produced) is an important task. Several parameters are determined under the international standard ISO 17025 in order to keep the quality as high as possible. One of these parameters are the compounds, mentioned in EPA 524.2, i.e. a series of volatile organic compounds.

These compounds are giving a serious threat to human safety. Hence, measuring of a series of VOCs (benzene, 1,2-dichloroethane, tetrachloroethene, trichloroethene, bromodichloromethane and the total concentrations of trihalomethanes and trichlorobenzenes) is demanded by the Flemish government in a decree of 2003. In this decree detection limits are mentioned which have to be reached by the controlling laboratory and are typically 10% of the norm value. Therefore, demanded detection limits are usually above 1 µg/L and do not pose any problem to most analysis methods. However, for 1,2-dichloroethane, being one of the more "difficult" VOCs to analyze, the detection limit should be below 0.3 µg/L in drinking water.

Presently, VOCs in drinking water are measured using two methods: Purge & Trap GC/MS and Headspace GC/MS. The former method is sensitive, but has a number of large drawbacks (e.g. memory effects), while the latter is at least ten times less sensitive. By CTC Analytics a new technique has been introduced, namely In-Tube Extraction Sample Preparation (ITEX®). The principle of this technique is showed in Figure 1. This method gives both easiness to use and sensitivity.

Another problem, which is often encountered, is the occurrence of interferences. Hence, wrong assignments can occur which gives a serious reduction in the quality of the measurement. High chromatographic resolution, as obtained by comprehensive GC/time-of-flight mass spectrometry does give a solution to this problem. Moreover, lower detection limits can be obtained. Using these conditions extremely low detection limits were obtained (see Table 1). A comparison is made with Purge & Trap GC/ion trap mass spectrometry. Repeatability experiments revealed RSDs of less than 20%, as can be seen in Table 1. A typical chromatogram of a 0.2 µg/L is depicted in Figure 2.

Experimental

ITEX (in combination with CTC CombiPAL)

- Trap Material Tenax TA 80/100mesh
- Extraction speed: 100 μ L/s
- Total pumping strokes: 50 x 1 mL
- Temperature Pumping Syringe/sample incubation: 60°C (10 min)
- Desorption: 200°C (15 s; splitless)

Comprehensive GC (Agilent 6890 / LECO modulator)

- 1st column: HP-5ms SV (30 m; 250 μ m id; 0.5 μ m)
- 2nd column: VF-17ms (2m; 100 μ m id; 0.2 μ m)
- Carrier gas: Helium
- Injector temperature: 250°C
- 1st oven temperature program: 40°C (2min) to 200°C at 4°C/min
- 2nd oven temperature is 5°C offset from 1st oven temperature
- Transferline temperature: 250°C
- Modulation time: 5.5 s

Mass Spectrometry (LECO Pegasus 4D TOF)

- Mass range: 45-300 amu
- Acquisition rate: 100 spectra/s
- Detector voltage: 1750 V
- Ion source temperature: 200°C

Compound	Repeatability (%)	Recovery (%)	LOD ITEX (ng/L)	LOD P&T (ng/L)
Chloroform	9.1	103	2.6	40
1,2-Dichloroethane	11.6	109	3.0	46
1,3-Dichloro- <i>trans</i> -propene	8.7	91	2.8	54
Chlorobenzene	4.9	116	1.0	42
1,2,3-Trichloropropane	4.5	96	2.0	33
Butylbenzene	10.1	106	2.5	58
1,2-Dibromo-3-chloropropane	9.8	91	9.7	80
Naphtalene	12.1	89	1.4	57

Table 1: Quality parameters for a selected number of VOCs

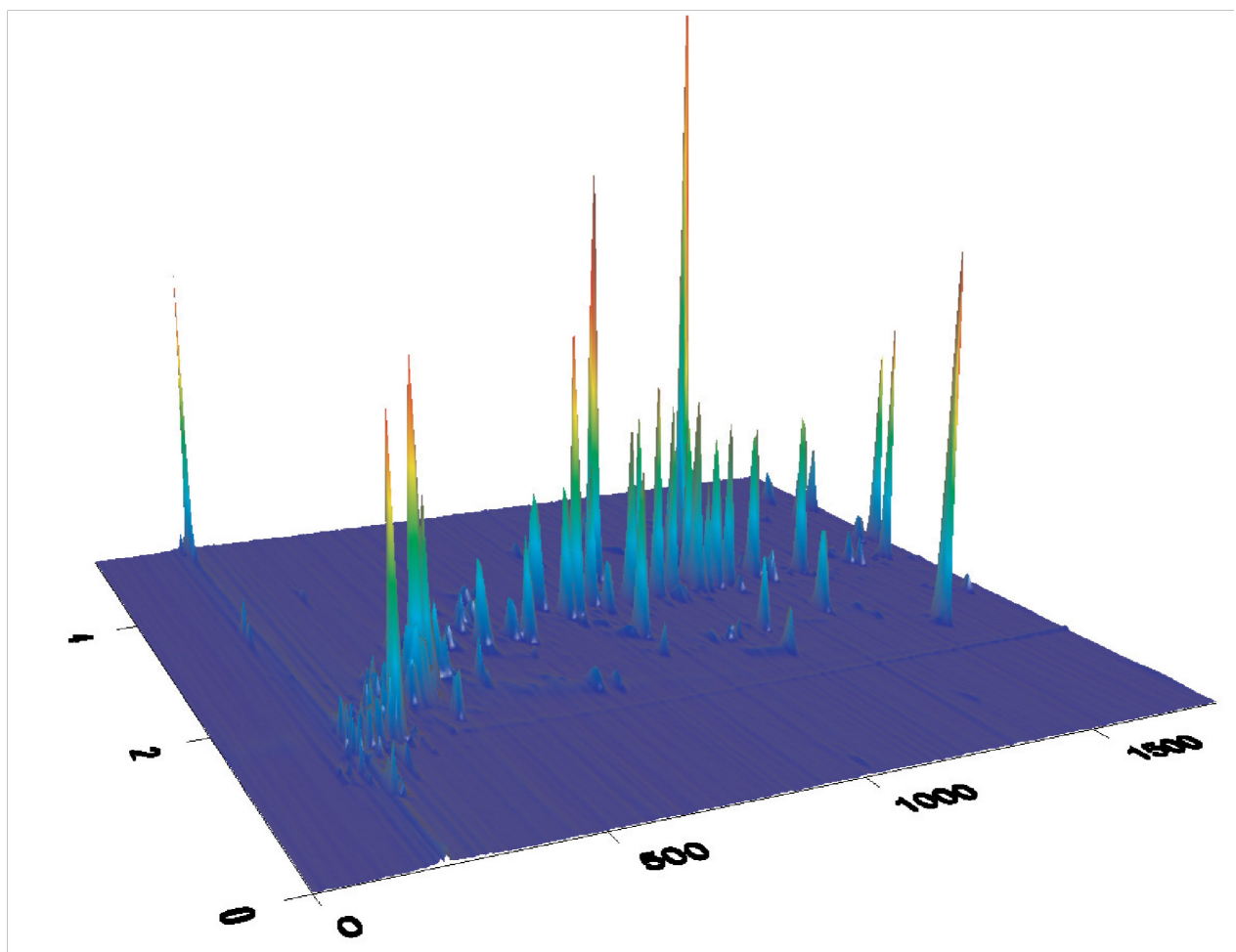


Figure 2: 3D-plot of a mixture of VOCs (concentration: 0.2 µg/L)

In-tube Extraction (ITEX) for Extraction of Volatile Organic Hydrocarbons from Groundwater

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Keywords: In-tube extraction, ITEX, VOCs, groundwater analysis, GC, mass spectrometry

Summary

A full automated in-tube extraction (ITEX) method was evaluated and optimized for the determination of twenty common groundwater contaminants such as halogenated volatiles and monoaromatic compounds. ITEX applies a 2.5 mL headspace syringe with a filled needle body (here Tenax TA). The analytes were extracted from sample headspace by dynamic extraction. The needle body is surrounded by a headable desorber, which is heated for analyte desorption into the injection port of a GC/MS. Method-related parameters such as extraction temperature, number of extraction cycles, extraction and desorption volume as well as extraction and desorption flow rates were determined in detail. The linear dynamic range of the ITEX method was over six orders of magnitude between 0.028 – 1218 µg/L with linear correlation coefficients between 0.990 and 0.998 for the investigated compounds. Method detection limits for monoaromatic compounds were between 28 ng/L (ethylbenzene) and 68 ng/L (1,2,4-trimethylbenzene). For halogenated volatile organic compounds MDLs between 48 ng/L (chloroform) and 799 ng/L (dichloromethane) were obtained. The precision of the method without internal standard was between 3.1 % (chloroform ethylbenzene) and 7.4 % (1,2,3-TMB).

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Optimization strategies of in-tube extraction (ITEX) methods

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Keywords: In-tube extraction, ITEX DHS, method development, parameter optimization

Abstract

Microextraction techniques, especially dynamic techniques like in-tube extraction (ITEX), can require an extensive method optimization procedure. This work summarizes the experiences from several methods and gives recommendations for the setting of proper extraction conditions to minimize experimental effort. Therefore, the governing parameters of the extraction and injection stages are discussed. This includes the relative extraction efficiencies of 11 kinds of sorbent tubes, either commercially available or custom made, regarding 53 analytes from different classes of compounds. They cover aromatics, heterocyclic aromatics, halogenated hydrocarbons, fuel oxygenates, alcohols, esters, and aldehydes. The number of extraction strokes and the corresponding extraction flow, also in dependence of the expected analyte concentrations, are discussed as well as the interactions between sample and extraction phase temperature. The injection parameters cover two different injection methods. The first is intended for the analysis of highly volatile analytes and the second either for the analysis of lower volatile analytes or when the analytes can be re-focused by a cold trap. The desorption volume, the desorption temperature, and the desorption flow are compared, together with the suitability of both methods for analytes of varying volatilities. The results are summarized in a flow chart, which can be used to select favorable starting conditions for further method optimization.

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