ELSEVIER

Journal of Chromatography A, 826 (1998) 191–200

JOURNAL OF CHROMATOGRAPHY A

Determination of benzene, toluene, ethylbenzene and xylenes in indoor air at environmental levels using diffusive samplers in combination with headspace solid-phase microextraction and high-resolution gas chromatography–flame ionization detection

K. Elke, E. Jermann, J. Begerow, L. Dunemann*

Medizinisches Institut für Umwelthygiene, Department of Analytical Chemistry, Auf'm Hennekamp 50, D-40225 Düsseldorf, Germany

Received 7 July 1998; received in revised form 7 September 1998; accepted 8 September 1998

Abstract

An improved analytical method for passive air sampling is presented based on a combination of commercially available diffusive samplers with headspace solid-phase microextraction and high-resolution gas chromatography with flame ionization detection (HRGC–FID). This procedure is targeted for short-term BTEX (benzene, toluene, ethylbenzene and *o*-, *m*- and *p*-xylenes) determinations at environmental concentrations and can be applied for sampling intervals between 30 min and 24 h. The analytes are adsorbed onto the charcoal pad of a passive sampler and then extracted with carbon disulphide–methanol. After removal of the carbon disulphide by xanthation, the BTEXs are enriched on a Carboxen SPME fiber, thermally desorbed and analysed by HRGC–FID. Detection limits for a sampling interval of 2 h are between 0.4 and 2 $\mu g/m^3$, within-series precision ranges between 6.6 and 12.8%, day-to-day precision is between 11.1 and 15.2%. The results obtained with this procedure are validated by comparison with active sampling. Detection limits and a further reduction of the sampling time are limited by blanks of the chemicals and the diffusive samplers. Procedures to eliminate these blanks are described in detail. Applications such as the determination of BTEXs in indoor air inside buildings, inside a train and a car are presented, indicating the usefulness of the described procedure for short-term measurements of environmental BTEX concentrations. An advantage of passive samplers is the storage stability for at least six months, which is essential for its use in large epidemiological studies.

Keywords: Air analysis; Environmental analysis; Sample handling; Headspace solid-phase microextraction; Benzene; Toluene; Ethylbenzene; Xylenes; Volatile organic compounds

1. Introduction

In the past several years volatile organic compounds (VOCs) such as benzene, toluene, ethylbenzene, and *o*-, *m*- and *p*-xylenes (BTEXs) have become of particular interest in the field of indoor air quality. Since people spend on average about 90% of the day indoors, attention is mainly paid to indoor air instead of outdoor air pollution. Indoor air pollution due to BTEXs results from smoking, building or furnishing materials, paints, adhesives, other consumer products and burning processes [1,2]. The main outdoor sources, which also contribute to

*Corresponding author. Tel.: +49 211 3389375, Fax: +49 211 3389331.

0021-9673/98/$ – see front matter
PII: S0021-9673(98)00736-5

indoor air pollution via ventilation, are automobile exhaust and industrial emissions.

Exposure to low-level VOC concentrations in indoor air are suspected to contribute to a variety of non-specific symptoms such as headache, eye, nose and skin irritation, which are known under the term ''sick building syndrome''. Benzene, moreover, is known to be carcinogenic to humans.

Sampling of BTEX compounds can be performed by active or passive sampling techniques. Passive samplers are well established for indoor and outdoor air measurements at environmental concentrations [1–5], because they are easy to handle and require cheap and user-friendly equipment. As passive samplers were originally developed for the assessment of occupational exposures, they are, at environmental concentrations, only suitable for long-term sampling periods of approximately 1–4 weeks [2–6]. For short-term sampling periods, active sampling requires expensive equipment and a skilled staff.

An innovative technique for air sampling is solid-phase microexctraction (SPME) developed by Pawliszyn and co-workers [7–10]. Its principle is based on a partition equilibrium of the analytes between the sample itself or the headspace above the sample and a fused-silica fiber coated with a stationary phase. The amount of analytes extracted by the fiber depends on the type of fiber and is proportional to the initial analyte concentration in the matrix, e.g., water or air. After sampling, the fiber is thermally desorbed into the gas chromatography (GC) injector. SPME which combines sampling, enrichment, and sample introduction in one step [7,8], has already found widespread use in environmental analysis. It has, for example, been used for the determination of VOCs [11,12], phenols [13], pesticides [14], polycyclic aromatic hydrocarbons [15] and polychlorinated biphenyls [15,16] in different environmental matrices such as drinking water or wastewater. Martos and Pawliszyn [17] used the SPME fibers as air sampling device for VOCs in ambient and industrial air and found excellent agreement with traditional air sampling methods. Other authors [7,8,18,19] used grab sampling with stainless steel canisters or glass bulbs in combination with SPME. One of the problems, the dependence of the adsorption rate on humidity and temperature of the air can be overcome by the use of correction factors [17]. But the main drawback of these procedures is the poor storage stability of the exposed SPME fiber, because uncontrolled losses of analytes can occur by evaporation from the fiber [at least with the poly(dimethylsiloxane) (PDMS) phase]. Preliminary studies of time-averaged sampling were performed by retracting the fiber into the needle and diffusion of the analytes through the needle opening to the fiber [7,17]. The first results are promising, but this method is not routinely applicable until now.

This paper presents an improved procedure which is routinely applicable for integrated short-term passive air sampling in indoor air at environmental concentrations. It is based on a combination of conventional passive samplers with headspace solid-phase microextraction (HS-SPME) and high-resolution gas chromatography with flame ionization detection (HRGC–FID).

2. Experimental

2.1. Reagents

Before use, all materials and chemicals coming into contact with the samples or standards were routinely checked for contamination. Glass vials were heated at 150°C for 24 h and stored under a clean bench equipped with activated charcoal filters (Bleymehl, Jülich, Germany).

All reagents and standards were of analytical-reagent grade, except sodium methanolate, which could only be obtained in synthesis quality. Methanol ''purge-and-trap quality'' was purchased from Lab-Scan (Dublin, Ireland), ''low benzene'' carbon disulphide was obtained from Promochem (Wesel, Germany), sodium methanolate ''for synthesis'' and acetone ''for organic trace analysis'' were purchased from Merck (Darmstadt, Germany). A solution of sodium methanolate (5 mol/l) was prepared in methanol. Due to contaminations, methanol and the sodium methanolate solution were purified by shaking with 1 g/l Carboxen (Supelco, Deisenhofen, Germany) for 10 to 12 h. The other chemicals were used as purchased.

2.2. Sampling

2.2.1. Passive sampling

Passive sampling was performed with diffusive

samplers (3500 OVM, 3M, Neuss, Germany). For 24-h sampling periods the monitors were used as purchased, for 2-h periods the samplers were preconditioned by agitating them first with 2 ml carbon disulphide (60 min) and then with 2 ml acetone (30 min). Residues of the solvents were removed by treating the samplers at 80°C under vacuum. To prevent preconditioned samplers from contamination, different storage conditions were tested: (i) the sampler was put into its original package and covered with an activated charcoal sheet (Alkol, Norit, Düsseldorf, Germany), that had been trimmed to size of the package. (ii) The sampler was wrapped into tin foil, put into its original package, and covered with an activated charcoal sheet. (iii) The sampler was wrapped into tin foil and put into its original package. (iv) The sample was shrink-wrapped into a polyethylene foil and put into its original package.

Determination of monitor blanks were performed immediately, after five days, and after six weeks.

2.2.2. Active sampling

For active sampling charcoal tubes (GK 26-16, SKC, Eighty Four, PA, USA) containing 800 mg of charcoal in the sample section and 200 mg in the back-up section were used in combination with a sampling pump Model GS 312 (Desaga, Heidelberg, Germany), which was operated at a flow of 2 l/min for the 24-h sampling period and 12 l/min for the 2-h sampling period.

2.3. Sample preparation

2.3.1. Passive sampling

The charcoal pad of the monitor was extracted with 1.5 ml solvent (carbon disulphide and methanol in various ratios). After addition of the solvent, the monitor was closed again and mechanically shaken for 30 min. Subsequently a xanthation reaction [20–22] was performed by mixing 500 μl of this extract with 2 ml sodium methanolate solution, according to the following equation:

$$Na^+OCH_3^- + CS_2 \rightarrow S{=}C{-}OCH_3 \quad (1)$$
$$\qquad\qquad\qquad\quad |$$
$$\qquad\qquad\qquad\quad S^-Na^+$$

After a reaction time of 15 min, 5 ml ultrapure water was added and a strong basic solution (pH 14) resulted. HS-SPME was applied using a 75-μm Carboxen–PDMS fiber (Supelco, Deisenhofen, Germany). The Carboxen–PDMS fiber was chosen, because it has a higher selectivity and extraction efficiency for VOCs than other types of fiber such as PDMS fibers [23]. To avoid any damage of the fiber, which does not tolerate pH 14, 40-ml vials containing 32.5 ml headspace volume were used for all experiments. Extractions were performed by exposing the fiber to the headspace over the sample under rapid magnetic stirring. The extraction temperature was varied between −10°C and +60°C and the extraction time between 1 and 60 min. After extraction the fiber was retracted, directly transferred to the GC injector and thermally desorbed at 300°C for 30 s.

2.3.2. Active sampling

The sample and back-up section of the active samplers were extracted with 3 ml and 1.5 ml carbon disulphide (containing 1% methanol), respectively, and mechanically shaken for 60 min. The extract was centrifuged (5 min, 4000 rpm), then decanted into glass vials and analysed by GC applying liquid sample introduction via a split/splitless injector.

2.4. Capillary gas chromatography

The analysis of the passive sampler extracts were performed with a Hewlett-Packard 5880A gas chromatograph equipped with a flame ionization detector. The split/splitless injector was operated at 300°C with the purge flow closed for 30 s. Helium (ultrapure, 99.999%, flow: 2 ml/min) served as carrier gas. For separations, a 60 m×0.32 mm I.D. DB-1701 column (J&W Scientific, Köln, Germany, 1.0 μm film thickness) was used. The following temperature program was used: 35°C for 10 min, ramp at 5°C/min to 150°C, held for 0.1 min, 20°C/min to 200°C, held for 10 min. The FID temperature was 220°C.

Analyses of active sampler extracts were carried out on a HRGC 5300 (Carlo Erba, Hofheim/Taunus, Germany) gas chromatograph equipped with FID. The split/splitless temperature programmable multi-injector MFA 515 (Carlo Erba) was operated at 50°C for 1 s and then ballistically heated to 250°C.

Injection (2 μl) was performed with the split being opened at a flow of 10 ml/min. Helium (ultrapure, 99.999%, flow: 2 ml/min) served as carrier gas. For separations a dual-column system was applied which was described in detail in a former paper of our group [4]: the carrier gas flow was split via a Y-connector and led onto two columns of different polarity, a 60 m×0.32 mm I.D. DB-1701 (J&W Scientific, 1.0 μm film thickness) and a 60 m×0.32 mm I.D. DB-5 (J&W Scientific, 1.0 μm film). The following temperature program was used: 35°C for 5 min, ramp at 4°C/min to 160°C, which was held for 30 min. The FID temperature was 330°C.

2.5. Calibration and calculation

Calibration for the SPME procedure was obtained with a blank solution and a set of nine standards containing 0.1 μg/l to 50 μg/l BTEX. Standards were made from high-purity reagents (Riedel-de Haën, Seelze, Germany) by diluting them first with methanol and then with carbon disulphide. For matrix adaptation the standards were also treated with sodium methanolate and water prior to SPME.

Calibration for liquid injection of the active sampler extracts was obtained using a blank and a set of four standards containing 8 mg/l to 35 mg/l BTEX. Standards were also made from high-purity reagents (Riedel-de Haën) by diluting them first with methanol and then with carbon disulphide resulting in a final solution containing 1% of methanol.

For quantification the blanks of the analytical procedure were subtracted.

2.6. Figures of merit

The detection limits were calculated as the threefold standard deviation of the monitor blanks ($n=10$). Recoveries of the compounds using passive samplers were determined by spiking the charcoal pads of the samplers with a known concentration of BTEXs. After a waiting period of 24 h the passive samplers were extracted with carbon disulphide–methanol and analysed as described. Sampling rates were adopted from those given in [24]. Precision was checked by spiking the samplers, extracting them and measuring them within-series and on different days, respectively. To check the accuracy of the passive sampling procedure, active and passive sampling were applied simultaneously and compared with each other. Indoor air was sampled for 2 h in a smoker room and for 24 h sampling in a non-smoker room.

2.7. Applications

We tested the described method under real-life conditions. Samplers without preconditioning were employed to check the efficiency of clean benches. In that study, one sampler was placed in the clean bench and another was exposed directly to the air of the laboratory. More validation of the method was carried out to determine the BTEX concentrations in a smoker day room (exposure period: 60 min), a smoker compartment of a commuter train (30 min) and inside a car (30 min, motorway). Two samplers were exposed in parallel in each case.

3. Results and discussion

3.1. Measures to shorten the exposure period

Using the described procedure, passive samplers without preconditioning (cleaning) are suitable for sampling periods of at least 24 h. If the samplers have to be exposed for shorter sampling periods, the samplers must be cleaned prior to use. Preconditioning results in a drastic reduction of the blanks up to a factor of about 80 in case of benzene and toluene. The blanks before and after cleaning are summarized in Table 1 and illustrated in Fig. 1 given as ng/sampler and converted to $\mu g/m^3$ for sample intervals of 2 h and 24 h.

Other types of passive samplers are less suitable for the proposed method because it is more difficult to precondition them. Tube-type samplers, additionally, have smaller cross-sectional areas and lower sampling rates resulting in a lower sensitivity.

To prevent preconditioned samplers from an accidental contamination during storage, it is absolutely necessary to shield them with an additional layer of prepressed activated charcoal (Fig. 2). Ubiquitous BTEX compounds are then effectively adsorbed onto the shielding activated charcoal. Other packages, like

Table 1
Blank values of samplers without and with preconditioning

	Sampler without preconditioning		Sampler with preconditioning
	24-h interval ($\mu g/m^3$)	2-h interval ($\mu g/m^3$)	2-h interval ($\mu g/m^3$)
Benzene	2.0	23.5	0.3
Toluene	2.1	24.8	0.3
Ethylbenzene	0.6	7.2	2.2
m-, *p*-Xylene	2.1	24.8	6.9
o-Xylene	1.1	12.7	3.9

tin foil or PE-foil, are not useful to prevent the samplers from contamination.

3.2. Optimization of HS-SPME

Adsorption profiles were obtained by monitoring the peak areas as a function of both time and temperature. The extraction temperature was varied between −10°C and +60°C. An extraction temperature between 0°C and +20°C was found to be optimal. In this range the adsorbed amount of BTEXs did not vary significantly. This finding is in accordance with the results given by Arthur et al. [25] using a PDMS fiber. For further experiments we chose an extraction temperature of 0°C which was provided by an ice bath to obtain constant extraction conditions for SPME. The influence of the extraction time was investigated by varying it between 1 and 60 min. The results are illustrated in Fig. 3. In agreement with Popp and Paschke [23] the highest extraction yields were obtained after an extraction time of 30 min. Since this is also a typical time for a GC run, the next sample can already be extracted during the GC run, so that samples can be prepared and measured without any delay.

3.3. Xanthation

Activated charcoal samplers are typically extracted with carbon disulphide (CS_2). Unfortunately, CS_2 is not compatible for the analysis of BTEX with Carboxen SPME fibers. Due to the similar polarity of CS_2, the analytes, and the SPME fiber, the amount of analytes extracted by the fiber is extremely low and

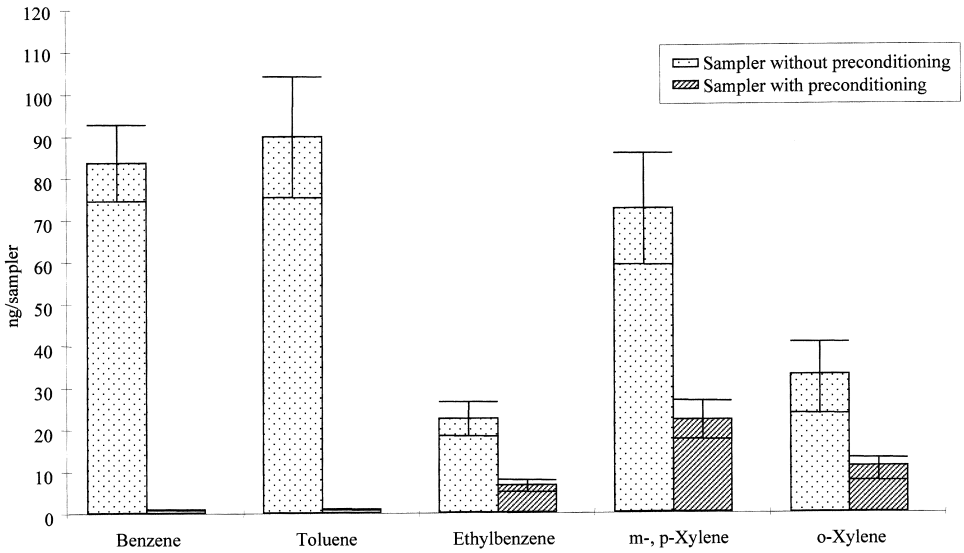

Fig. 1. Effect of preconditioning on the blanks of passive samplers.

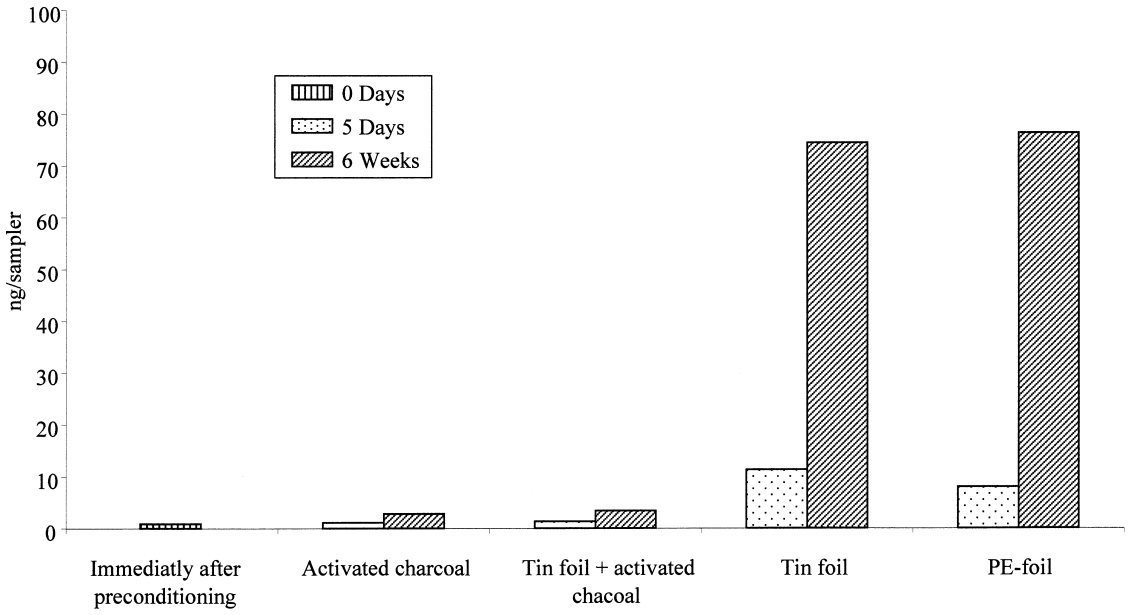

Fig. 2. Blank benzene levels of unexposed samplers in dependence of storage conditions using preconditioned samplers.

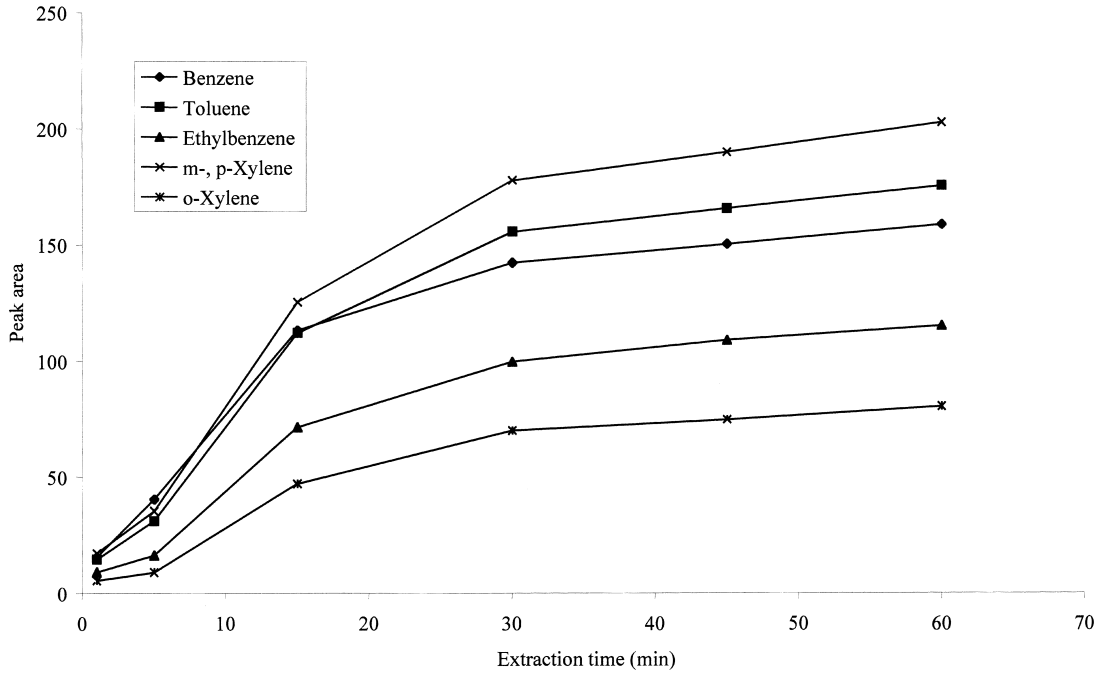

Fig. 3. Optimization of extraction time (extraction temperature 0°C).

the fiber can become overloaded and the analytes may be displaced from the adsorption sites. If a solvent with a higher polarity such as methanol is used, the compounds show a higher tendency towards the fiber and the adsorption efficiency is greater. On the other hand, CS_2 is absolutely necessary for the desorption of BTEX compounds from activated charcoal which does not work with pure methanol (recovery only about 1%). Thus, the CS_2 has to be removed from the extract after desorption of the BTEXs from the charcoal pad. This can be achieved by reaction of CS_2 with sodium methanolate (reaction 1), resulting in a xanthate which is soluble in water and organic polar solvents such as alcohol. In aqueous solution this xanthate exists in its ionic form, has a low vapour pressure, and shows no tendency to adsorb onto the SPME fiber. As a result of this xanthation reaction the adsorbed mass of BTEXs and thus the sensitivity of the described procedure increase by a factor of 200 (Fig. 4). Investigations using different mixtures of CS_2 and methanol in combination with xanthation of CS_2 and SPME showed that a methanol–carbon disulphide ratio of 60:40 is the optimum.

3.4. Figures of merit

The figures of merit of the described method are summarized in Table 2. The absolute detection limits were expressed in terms of $\mu g/m^3$ for a 2-h, 24-h or 4-week exposure interval. Detection limits ranged between 0.4 and 2.0 $\mu g/m^3$ for a 2-h sampling interval and between 0.4 and 1.1 $\mu g/m^3$ for a 24-h interval (Table 2). They were comparable with those detection limits obtained for a 4-week sampling period using direct liquid injection of the CS_2 extract and subsequent GC–FID analysis (no enrichment with HS-SPME) which were between 0.1 and 0.4 $\mu g/m^3$ [4]. It is obvious, that the detection limits for short-term sampling in combination with SPME are comparable to those of conventional long-term sampling procedures. Our procedure is thus sensitive enough for short-term BTEX determinations at environmentally relevant concentrations [3,4].

Within-series precision was between 6.6 and 12.8%, day-to-day precision was between 11.1 and 15.2% (Table 2). These findings are in agreement with those of Popp and Paschke [23], who reported relative standard deviations in the range between 7

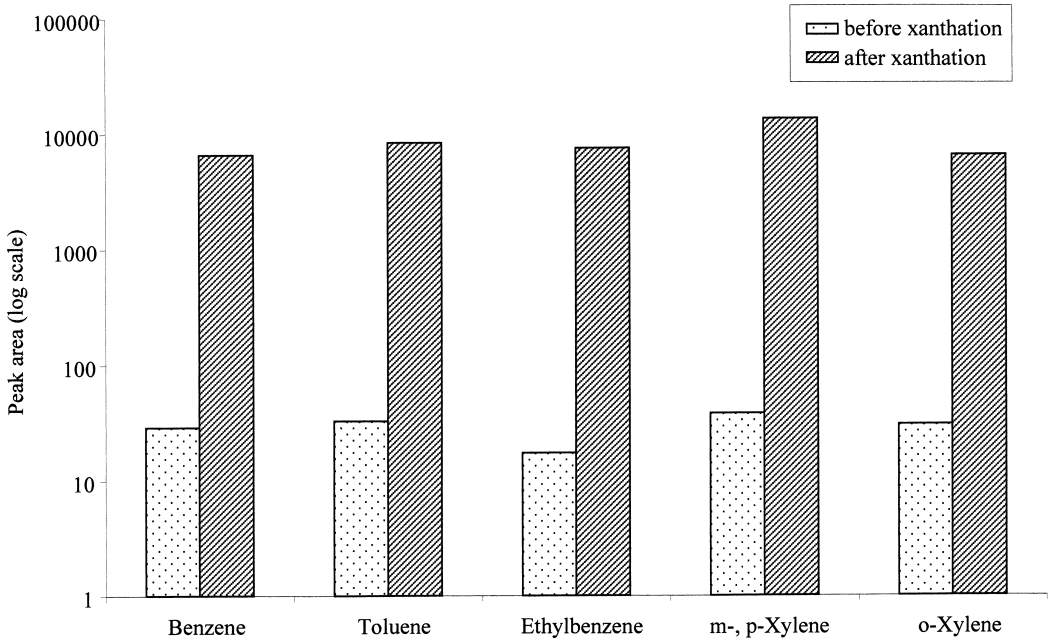

Fig. 4. Effect of xanthation (logarithmic scale).

Table 2
Figures of merit

	Detection limit (μg/m³) (n=10)			Precision[a] (%) (n=10)		Recovery (%) (n=10)	
	2 h, SPME	24 h, SPME	4 weeks, direct	Within-series, SPME	Day-to-day, SPME	2 h[a], SPME	24 h[b], SPME
Benzene	0.36	0.54	0.13	6.6	15.2	94.0	89.2
Toluene	0.77	0.87	0.15	8.1	15.0	88.3	93.1
Ethylbenzene	0.36	0.36	0.21	10.3	11.4	95.1	99.8
m-, *p*-Xylene	2.02	1.11	0.36	9.6	11.1	83.8	94.5
o-Xylene	0.37	0.80	0.14	12.8	12.8	89.0	83.7

[a] $c = 2.6$ nl/l = 15–18 μg/m³.
[b] $c = 28.3$ nl/l = 13–18 μg/m³.

and 10% for direct SPME extraction from water. For the determination of BTEX in indoor air using diffusive sampling and liquid injection, within-series precisions from 9.6 to 17.9% [3] and from 2.8 to 12% [6] were reported. Recoveries ranged between 83.7 and 99.8% for both preconditioned and non-preconditioned samplers (Table 2).

In agreement with other authors [3,26,27] the results obtained by short-term passive and active sampling were, with the exception of *o*-xylene, comparable (−16 to +19%) (Table 3). The higher values for *o*-xylene determined by passive sampling may be the result of an interference caused by α-pinene, which according to Begerow et al. [4] has the same retention time on the used DB-1701 column as *o*-xylene. Samples obtained by active sampling have to be regarded as correct because a dual column system (DB-1701 and DB-5) was applied. In this case the DB-5 column was used for the determination of o-xylene. The BTEX results for the 2-h sampling period were higher because the samples were taken in a smoker room whereas the samples over a 24-h interval were taken in a non-smoker room.

Mangani and Cenciarini [18] compared passive sampling using graphitized carbon black SPME fibers with active sampling and obtained relative deviations between −11 and +20%. Martos and Pawliszyn [17] used PDMS fibers for 30 min integrated industrial air sampling of styrene and compared the results with active charcoal sampling and passive batch sampling. They found 56 μg/l styrene with SPME sampling, 54 μg/l with active sampling and 72 μg/l with passive batch sampling. Their results show a good agreement between direct exposure of the SPME fiber and active sampling with charcoal tubes. The results thus indicate that air sampling with SPME fibers is a promising alternative to active charcoal tube sampling or passive batch sampling, but analysis must be performed within several hours, because losses of the highly volatile compounds occur [7]. Thus, passive sampling has the advantage that storage of the exposed samplers is possible for at least six months [3]. Another approach is the use of canisters in combination with SPME [7,12], which extends storage capacity to two weeks. But for epidemiological studies even a two-week storage may be still not sufficient.

Table 3
Comparison between active and passive sampling

	Sampling 2 h (μg/m³)		Sampling 24 h (μg/m³)	
	Active sampling	Passive sampling	Active sampling	Passive sampling
Benzene	27.7	31.3	3.3	3.3
Toluene	64.9	54.7	5.7	6.8
Ethylbenzene	11.4	11.4	2.5	2.8
m-, *p*-Xylene	29.2	30.7	27.0	31.4
o-Xylene	9.2	22.6	1.6	3.0

Table 4
24-h sampling with non-preconditioned samplers in chemical laboratories

	Laboratory A		Laboratory B	
	Inside the clean bench	Outside the clean bench	Inside the clean bench	Outside the clean bench
Benzene	1.55	1.63	<0.54	1.84
Toluene	4.59	5.36	<0.87	4.53
Ethylbenzene	3.69	4.06	<0.36	2.94
m-, *p*-Xylene	51.6	60.5	<1.11	22.0
o-Xylene	3.42	3.66	<0.80	4.47

3.5. Applications

To demonstrate the suitability of our method, we monitored the air quality at some typical sites such as chemical laboratories, a smoker day room, a smoker compartment of a train and inside a car (Tables 4 and 5).

We investigated the efficiency of two clean benches A and B (laminar flow cabins) equipped with charcoal filters. As can be seen in Table 4, the filters in clean bench B works efficiently whereas those in A have to be renewed. The sampling period was 24 h using samplers without preconditioning.

The results obtained with preconditioned samplers, which were exposed for 30 min in a smoker compartment of a commuter train during the rush hour, show that even such a short exposure time is sufficient for screening purposes (Table 5). At such slightly elevated concentrations like those inside a car during a drive (without smoking), a 30 min sampling period gives reliable results (Table 5). The BTEX concentrations we found inside the car are comparable with those given by Fromme et al. [28]. Compared with those given by Mücke et al. [29], who determined VOCs inside a car in the city centre of Munich, our results were two- to four-times lower. This may be explained by the fact that the study of Mücke et al. was conducted in 1984, at which time fuel consumption and VOC emissions of automobiles were higher than today. In addition, the car type, its age and traffic density have great influence on the air quality inside the car.

For the determination of environmental BTEX concentrations inside buildings, which may result from furnishing, paints, adhesives, or smoking for example, the sampling interval has to be extended to 60 min. The results of BTEXs in a smoker day room (Table 5) are in a good agreement with the findings of other authors [2–4] for environmental concentrations in indoor air.

4. Conclusions

The present work has shown that passive batch samplers in combination with HS-SPME using a Carboxen–PDMS fiber are suitable for short-term measurements of BTEXs in indoor air at environmental concentrations. Xanthation followed by HS-SPME provides an effective tool for preconcentration

Table 5
Applications of short-term sampling (for details see Section 2.7 and 3.5)

	30 min smoker compartment			30 min indoor air of a car			60 min smoker day room		
	Sample 1	Sample 2	Mean	Sample 1	Sample 2	Mean	Sample 1	Sample 2	Mean
Benzene	15.5	11.7	13.6	15.9	23.5	19.7	10.3	10.8	10.6
Toluene	70.0	38.9	54.5	52.1	58.5	55.3	19.8	21.4	20.6
Ethylbenzene	11.8	7.9	9.9	21.5	23.3	22.4	8.7	9.5	9.1
m-, *p*-Xylene	27.8	18.5	23.2	51.7	59.1	55.4	14.7	15.2	15.0
o-Xylene	20.5	15.7	18.1	31.7	32.8	32.2	11.2	11.5	11.4

of BTEXs from the headspace of CS_2 extracts. Compared to traditional passive sampling techniques this results in a drastic gain in sensitivity and enables detection limits for BTEXs below 1 $\mu g/m^3$ for 2-h and 24-h sampling intervals. For sampling periods of 24 h, the passive samplers can be used as supplied by the manufacturer. If shorter sampling periods (30 min to 24 h) are required, it is necessary to precondition the passive samplers prior to use to reduce the background level.

For the first time short-term passive sampling at environmental concentrations is now feasible within the scope of large epidemiological studies and to record day-time fluctuations. The described procedure can be easily extended for the determination of other volatile and semi-volatile organic compounds in air.

References

[1] B. Seifert, D. Ullrich, Atmos. Environ. 21 (1987) 395.
[2] L.C. Holcomb, B.S. Seabrook, Indoor Environ. 4 (1995) 7.
[3] J. Begerow, E. Jermann, T. Keles, U. Ranft, L. Dunemann, Fresenius J. Anal. Chem. 351 (1995) 549.
[4] J. Begerow, E. Jermann, T. Keles, T. Koch, L. Dunemann, J. Chromatogr. A 749 (1996) 181.
[5] H.C. Shields, C.J. Weschler, J. Air Pollut. Control Assoc. 37 (1987) 1039.
[6] B. Seifert, H.-J. Abraham, Int. J. Environ. Anal. Chem. 13 (1983) 237.
[7] M. Chai, J. Pawliszyn, Environ. Sci. Technol. 29 (1995) 693.
[8] C. Grote, J. Pawliszyn, Anal. Chem. 69 (1997) 587.
[9] C.L. Arthur, J. Pawliszyn, Anal. Chem. 62 (1990) 2145.
[10] J. Pawliszyn, Solid Phase Microextraction: Theory and Practice, Wiley–VCH, New York, 1997.
[11] I. Valor, C. Cortada, J.C. Moltó, J. High Resolut. Chromatogr. 19 (1996) 472.
[12] T. Nilsson, F. Pelusion, L. Montanarella, B. Larsen, S. Facchetti, J. Madsen, J. High Resolut. Chromatogr. 18 (1995) 617.
[13] M. Möder, S. Schrader, U. Franck, P. Popp, Fresenius J. Anal. Chem. 357 (1997) 326.
[14] M.T. Sng, F.K. Lee, H.A. Lakso, J. Chromatogr. A 759 (1997) 225.
[15] D.W. Potter, J. Pawliszyn, Environ. Sci. Technol. 28 (1994) 298.
[16] Y. Yang, D.J. Miller, S.B. Hawthorne, J. Chromatogr. A 800 (1998) 257.
[17] P.A. Martos, J. Pawliszyn, Anal. Chem. 69 (1997) 206.
[18] F. Mangani, R. Cenciarini, Chromatographia 41 (1995) 678.
[19] M. Chai, C.L. Arthur, J. Pawliszyn, R.P. Belardi, K.F. Pratt, Analyst 118 (1993) 1501.
[20] M. Ragg, W. Green, Chem.-Z. 11 (1910) 82.
[21] G. Edelmann, in: E. Bartholomé, E. Biekert, H. Hellmann, H. Ley, W.M. Weigert, E. Weise (Eds.), Ullmann's Encyklopädie der Technischen Chemie, VCH, Weinheim, 4th ed., 1983, p. 511.
[22] M. Bögemann, S. Petersen, O.-E. Schultz, H. Söll, in: E. Müller (Ed.), Houben-Weyl: Methoden der Organischen Chemie, Vol. IX, Georg Thieme Verlag, Stuttgart, 4th ed., 1955, p. 810.
[23] P. Popp, A. Paschke, Chromatographia 46 (1997) 419.
[24] Organic Vapor Monitor Sampling and Analysis Guide, 3M, St. Paul, MN, 1996.
[25] C.L. Arthur, L.M. Killam, K.D. Buchholz, J. Pawliszyn, J.R. Berg, Anal. Chem. 64 (1992) 1960.
[26] M.A. Cohen, P.B. Ryan, Y. Yanagisawa, S.K. Hammond, J. Air Waste Manage. 40 (1990) 993.
[27] R. Pristas, Am. Ind. Hyg. Assoc. J. 52 (1991) 297.
[28] H. Fromme, A. Oddoy, T. Lahrz, M. Piloty, U. Gruhlke, Zbl. Hyg. 200 (1998) 505.
[29] W. Mücke, D. Jost, W. Rudolf, Staub-Reinhalt. Luft 44 (1984) 374.