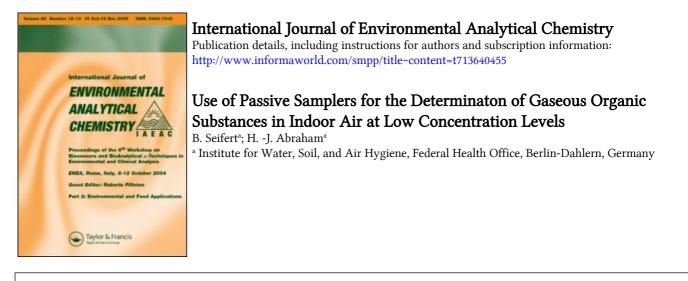
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Use of Passive Samplers for the Determination of Gaseous Organic Substances in Indoor Air at Low Concentration Levels

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The design and calibration of a passive sampler operating according to the diffusion principle and its application to the analysis of indoor air are described. Taking aliphatic and aromatic hydrocarbons as representative pollutants, it is demonstrated that at constant concentration, the amount of substance trapped by the sampler is a linear function of the time of exposure. An equation is given relating this amount of substance to the mean pollutant concentration. The detection limit is of an order of $300 \,\mu g/(m^3.h)$. For test gas atmospheres variation coefficients of between 5 and 10% were determined for a 24-hour exposure in an atmosphere with concentrations of the individual hydrocarbons between 150 and $600 \,\mu g/m^3$.

KEY WORDS: Indoor air pollution, passive sampling, hydrocarbons.

1. INTRODUCTION

Statistical surveys^{1,2} show that most people spend much more time within enclosed spaces than outside buildings. A correct evaluation of the effects of air pollutants, especially in epidemiological studies, can thus only be carried out if indoor air quality is considered. Furthermore, there will be an increasing need for indoor air quality studies in the future as air exchange rates inside buildings are reduced to conserve energy.

In principle, the same methods of analysis that are used in ambient air studies may be used for the analysis of indoor air. In the special case of organic compounds, substances are generally collected by sucking the air sample through a tube filled with a suitable adsorbent.^{e.g.3-7} Sampling by such methods requires skilled staff and elaborate sampling equipment. The

use of passive samplers⁸ which are frequently employed for control purposes in working areas, would permit a considerable reduction of expenditure compared with active methods. With passive samplers the pollutants are absorbed onto a small pad generally made of charcoal. After exposure, the pad is analyzed in the laboratory. The total amount of substance collected can be converted into the mean pollutant concentration during the exposure period using equations based on Fick's diffusion law. So far, the corresponding equations have been validated only for gaseous organic substances at concentration levels to be expected in working areas⁹, i.e. the ppm level. In this paper, information is presented on the use of passive samplers for the measurement of lower pollutant concentrations (ppb level), e.g. in the case of indoor air pollution studies.

2. EXPERIMENTAL

2.1 Test gases

Test gases with known concentrations of a number of hydrocarbons were prepared, with the aid of permeation tubes and using purified N₂ for dilution, as shown in Figure 1. The permeation rates varied between 5 and $30 \mu g/h$. The higher concentrations were produced by closing valves 1 and 2 and opening valve 3 while not operating the pump. The lower pollutant concentrations in the exposure vessel were produced by opening valve 1 (additional dilution of the amount of pollutant released within the permeation vessel) and valve 2, closing valve 3 and operating the pump. In such a way, the gas flow through the exposure vessel could be maintained constant even at high dilutions and the excess could escape through valve 2. Atmospheres containing between 75 and $5,000 \mu g/m^3$ of benzene, toluene, *m*-xylene, pentane, hexane, heptane and 1-hexene were studied. The exposure periods varied between 5 and 65 h.

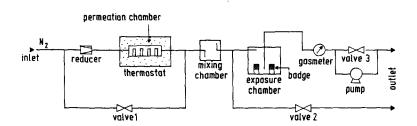


FIGURE 1 Device for generation of test gas atmospheres and exposure experiments.

2.2 Passive samplers

The passive samplers used in this work were GasbadgeTM samplers (now manufactured by National Mine Service Co., Oakdale, PA 15071, USA). The samples consist of a $5.5 \text{ cm} \times 8 \text{ cm} \times 2 \text{ cm}$ plastic casing containing a small glass wool pad coated with charcoal and having an effective surface of 9.54 cm^2 . The charcoal pad is separated from the surrounding air by means of a glass fibre filter which is kept at approximately 1.3 cm distance from the pad by means of a distance piece. When exposing the passive sampler, the organic vapours to be determined diffuse through the filter and the buffer zone to become finally adsorbed onto the charcoal pad. More detailed descriptions are given elsewhere.^{9,10} After exposure, the sampler is wrapped in aluminum foil if the analysis can be performed within one day. If a longer storage is necessary the charcoal pad is taken off the sampler and put in a tightly screwed glass vial. Suitable vials are available from the manufacturer of the sampler.

2.3 Analysis

The substances adsorbed during the exposure period were extracted from the pad in the vial with 2 ml carbon disulphide in the presence of $20 \mu g$ cyclooctane as an internal standard. A short ultrasonic treatment enhanced the extraction process. Subsequent analysis was made by temperature programmed gas chromatography of a $0.5 \mu l$ aliquot using a 50 m OV-101 glass capillary column (12 min at 30°C, then up to 150°C with 2.5°/min).

2.4 Calculation of the result

The analysis gives the amount of pollutant extracted from the charcoal pad. To convert this value into the mean concentration c of the pollutant during the exposure period the following equation is given by the manufacturer of the sampler for the ppm range:

$$c\,(\text{ppm}) = 3,360 \cdot A/(MDt\epsilon) \tag{1}$$

where A —extracted amount of pollutant (in μg)

- M-molecular weight of the pollutant
- D —diffusion coefficient at 25°C and 1 bar (in cm^2/s)
- ε —desorption efficiency of the pollutant
- t —exposure period (in seconds).

B. SEIFERT AND H.-J. ABRAHAM

The factor 3,360 includes the molar volume amd a number of conversion factors and also takes into account the geometry of the GasbadgeTM.

As the concentrations to be expected in the analysis of indoor air will mostly be below the ppm level normally found in working areas, c may also be calculated in ppb:

$$c\,(\text{ppb}) = 3.36 \cdot 10^6 \cdot A/(mDt\varepsilon) \tag{2}$$

An even more simplified equation results if the units "ppb" and "second" are replaced by " $\mu g/m^3$ " and "hour", respectively, and if—in a first approximation— ε is assumed to be unity:

$$c(\mu g/m^3) = 41.6 \cdot A/(Dt)$$
 (3)

On the one hand assuming $\varepsilon = 1$ would greatly reduce the work for calculating the results since no individual correction of the different compounds would be necessary; on the other hand this simplification will lead to a certain error because the desorption efficiency will generally be lower than 1. In Section 3.3 an estimate of the magnitude of this error will be given for the different compounds on the basis of experimental results.

3. RESULTS AND DISCUSSION

3.1 Calibration of passive samplers

In a preliminary series of experiments, the linearity of the relationship between the exposure period and the adsorbed amount of substance was checked. For this purpose, the samplers were placed in the exposure chamber (Figure 1) which was purged with the test gas at about 301/h. The test gas contained a mixture of benzene, toluene and *m*-xylene as representatives of aromatic substances, 1-hexene as a representative of the more reactive group of olefines and pentane, hexane and heptane as aliphatic hydrocarbons. Pentane (b.p. 36.1° C) also served to test the performance of the GasbadgeTM in measuring low boiling compounds. In Table I, the boiling points and diffusion coefficients of the different substances are listed as well as the approximative concentrations in the undiluted gas stream leaving the permeation chamber (Figure 1).

The results of the calibration experiment carried out at 25° C are shown in Figures 2 and 3. Each bar represents the range of three duplicate measurements. From Figure 2 it is clear that for aromatic substances there is a linear relationship between exposure period and adsorbed amount of pollutant. In agreement with what is expected from a consideration of

TABLE I

Substance	Boiling point (°C)	Diffusion coefficient in air ^a (cm ² /sec)	Approximative concentration in the undiluted gas stream (Figure 1) (ppm)
Pentane	36.1	0,084	0.3
Hexane	69.0	0.073	0.2
Heptane	98.4	0.067 ^b	0.1
1-Hexene	63.4	0.07°	0.3
Benzene	80.1	0.093	0.2
Toluene	110.6	0.085	0.1
m-Xylene	139.1	0.067	0.05

Boiling points, diffusion coefficients and concentrations of various hydrocarbons used in the experiments.

At 25°C and 1 bar; according to Ref. 10.

^bThis value has been found by graphical interpolation using the diffusion coefficients for pentane, hexane and octane (D=0.062).

"This value is an estimate based on the diffusion coefficients for alkanes.

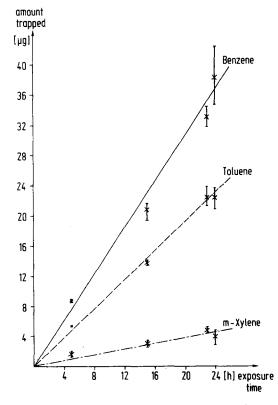


FIGURE 2 Relationship between exposure time and amounts of aromatic hydrocarbons trapped by the GasbadgeTM sampler.

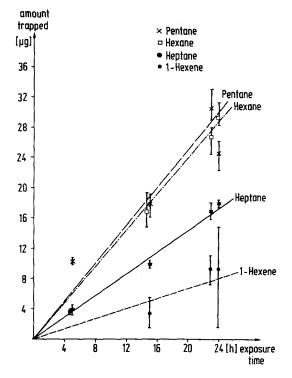


FIGURE 3 Relationship between exposure time and amounts of aliphatic hydrocarbons trapped by the GasbadgeTM sampler.

diffusion coefficients, the amount of pollutant trapped on the charcoal pad decreases as the boiling point of the pollutant increases. The results are similar for hexane and heptane as can be seen from Figure 3. However, in the case of the low-boiling pentane there is a remarkable scattering of the individual values, especially for brief exposure periods. Similarly bad results must be expected for other low-boiling compounds. Even greater difficulties arise with 1-hexene; the reproducibility is very poor and the mean values are not well distributed along a straight line. This can probably be attributed to the double bond which takes part in uncontrolled reactions at the surface of the charcoal thus leading to varying losses in each individual experiment. This assumption is supported by the results of recovery experiments with 1-hexene (cf. Section 3.3).

3.2 Reproducibility of passive sampler measurements

In addition to the mean values, Figures 2 and 3 also include the ranges of

individual results in order to give information on the reproducibility of the experiments. For purposes of statistical evaluation, however, the standard deviation is a more suitable parameter than the range. To determine the standard deviation, an experiment was carried out involving the simultaneous exposure of several passive samplers in a test gas. For technical reasons, the number of samplers had to be limited to four per experiment. For exposure times up to 24 h, the relative standard deviation in the amount of hydrocarbon trapped was of the order of 5 to 10% or less (Table II) except for 1-hexene, where a higher standard deviation was found for reasons mentioned in Section 3.1. Since the values given in Table II have been obtained under optimal conditions, i.e. a constant gas stream during the exposure and an analysis carried out immediately after the exposure, the standard deviation to be expected in practice will probably be slightly higher.

The reproducibility of measurements with passive samplers might be influenced by the fact that the adsorption of pollutants will not occur at a constant rate during practical exposure periods. The amount trapped by a sampler may vary according to whether it has been subject to a particular pollutant concentration during the first 24 hours of a one-week exposure period and a low pollutant atmosphere for the remaining six days of the week or, conversely, it has been placed into a comparatively clean atmosphere for the first six days with higher concentrations occurring only during the last 24 hours. In the first of these two cases, desorption of part of the adsorbed amount of substance could not be excluded. This problem is of considerable practical importance since, as a rule, short-term peak concentration of defined pollutants occur frequently when using certain household products.

To study this problem, five passive samplers were exposed in an

simultaneously (all values in µg/sampler).								
	Hexane	Heptane	1-Hexene	Benzene	Toluene	m-Xylene		
Badge 1	27.5	18.9	13.5	35.3	21.6	4.3		
Badge 2	27.6	18.5	10.2	40.7	23.0	5.5		
Badge 3	26.6	18.2	d 1.4	36.7	22.4	4.7		
Badge 4	26.9	18.5	9.4	35.8	22.8	5.4		
Arithmetic mean Relative standard	27.2	18.5	11.1	37.1	22.3	5		
deviation (%)	1.8	1.6	16	6.6	2.8	12		

 TABLE II

 Reproducibility of Gasbadge[™] measurements: four samplers have been exposed

atmosphere of pure nitrogen with a flow of only 300 ml/h, thus simulating an air exchange rate of approximately 0.3/h. For the type of passive sampler used, the influence of air velocity can be neglected even at low flow rates since there is a draft shield separating the surrounding air by 1.3 cm from the charcoal pad. According to Brown *et al.*,¹¹ an air gap of 15 mm between the diffusion barrier and the adsorptive sink eliminates external flow effects. The good results obtained when comparing the results obtained with GasbadgeTM samplers and adsorption tubes under uncontrolled field conditions¹⁵ support these findings.

The samplers were removed from the N₂ atmosphere according to the time-table given in Table III and placed into the exposure vessel shown in Figure 1. The vessel was purged with nitrogen flowing at 901/h and containing definite pollutant concentrations. The concentrations were about one third of those given in Table I. These concentrations were higher than those used in the other experiments because the effect to be studied could be assumed to be greater in the case of higher loadings. The time-schedule which was followed and the amount of substance trapped by the individual samplers are given in Table III. On the one hand it is clear that a 7-day exposure in an atmosphere of pure nitrogen results in a negligibly small loading of the charcoal pad and on the other hand that, once loaded, the pad will not release the adsorbed compounds even if it remains in an unpolluted atmosphere for a couple of days. By combining the values of the different compounds for samplers 1 to 5 (in the case of sampler no. 5 the values given in brackets were used), the relative standard deviations were calculated for each pollutant. The respective figures are also given in Table III. Although the exposure level was relatively high, the data are a good proof of the quality of results to be obtained with passive samplers and confirm the statistical characteristics given in Table II.

3.3 Calculation of analytical results

In Sections 3.1 and 3.2, results have been given in terms of "amount of substance/pad". These results must be converted into the mass concentration of pollutants in order to be comparable to results of outdoor air measurements. For this purpose Eq. 3 was used. The validity of this equation was checked experimentally by exposing GasbadgeTM samplers to test gas atmospheres with known pollutant concentrations over varying periods, calculating the pollutant concentrations using Eq. 3 and the diffusion coefficients given in Table I, and finally comparing these concentrations to the known concentrations of the test gases. Part I of Table IV gives the results of this experiment in which 1-hexene and

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TABLE III Influence of exposure conditions on the amount trapped by the GasbadgeTM sampler.

Sampler No.	Exposure conditions	Hexane	Heptane	Amount trapped (µg/sampler) 1-Hexene Benzene	d (μg/sampler) Benzene	Toluene	m-Xylene
0	168 h N ^a	<	-	1.1	2.4	1.0	~
2	24 h P ^b 144 h N	130	69	. 115	138	86	21
2	24 h N _b 24 h P 120 h N	128	71	160	145	104	22
£	48 h N 24 h P 96 h N ₆	126	72	149	144	103	22
4	72 h N 24 h P 72 h N	114	71	131	141	104	22
5°	96 h N	372	209	413	419	301	62
	4 u 7/	(124)	(10)	(138)	(140)	(100)	(21)
Relative standard (%) deviation (samplers 1-5)		5.0	9.1 1.6	12.4	2.0	2.7	2.5

GASEOUS ORGANIC SUBSTANCES IN INDOOR AIR

245

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	Mean of differences (%)	-4.3	+ 5.3
Part II	Difference (%)	15.7 24.1 - 25.9 - 6.4 1.2 - 34.6	11.8 37.8 - 11.4 - 8.6 0
	Concentration found using Equation 4 (µg/m ³)	5,090 3,290 1,290 1,460 810 170	3,400 2,550 1,090 320 180
	Mean of differences (%)	- 16.7	-7.9
Part I	Difference (%)	0.7 7.9 - 35.6 - 18.6 - 12.5 - 42.3	-2.6 20.0 -22.8 -10.9 -11.1
	Concentration found using Equation 3 (µg/m ³)	4,430 2,860 1,120 1,270 700 150	2.960 2,220 950 490 160
	Given concentration (µg/m³)	4,400 2,650 1,740 800 260	3,040 1,850 1,230 550 350 180

Substance

Benzene

Toluene

B. SEIFERT AND H.-J. ABRAHAM

Difference between given and measured pollutant concentration.

TAB',E IV

246

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	+15.1					-4.6						+ 5.3				
8.2	15.3	46.1	-9.1	0	1.6	- 14.0	-31.0	-8.7	-13.3	33.3	12.4	21.1	13.5	7.9	4.0	- 27.3
5,160	980	760	300	5,000	3,090	1,720	1,380	840	260	100	3,070	2,010	1,220	1,160	520	120
	0					- 16.2						- 8.9				
- 5.9	0	26.9	-21.2	- 13.0	-11.5	- 25.0	40.0	- 20.6	-23.3	20.0	-2.2	5.4	- 1.4	- 6.0	- 10.0	- 39.4
4,490	850	099	260	4,350	2,690	1,500	1,200	730	230	90	2,670	1,750	1,060	1,010	450	100
4,770	850	520	330	5,000	3,040	2,000	2,000	920	300	75	2,730	1,660	1,075	1,075	500	165
m-Xylene				Hexane							Heptane					

pentane have not been included due to the difficulties mentioned in Section 3.2.

It should be pointed out at this stage that literature values of diffusion coefficients, even if given for the same temperature, may vary considerably in the case of certain substances. Major differences can be found especially for those organic molecules that contain other atoms in addition to carbon and hydrogen. One example is carbon tetrachloride for which values of 0.0828^{12} and $0.091 \text{ cm}^2/\text{s}^{13}$ are given. For the substances mentioned in Table I, however, only the data for *n*-hexane showed a range greater than 5% with 0.073^{12} and $0.0797 \text{ cm}^2/\text{s}^{13}$. For all other compounds listed, data in the literature vary by less than 5%.

Because the diffusion coefficient depends on temperature, the influence of this parameter must be kept in mind when exposing passive samplers under conditions of varying temperature. As an example, the effect of temperature variations leads to errors in the order of 0.7%/°C for the 0 to 20° C range in the case of benzene as can be estimated from the diffusion coefficients given for 0 and 20° C¹³. Lautenberger *et al.*¹⁴ give lower values: Using the relationship $D = f(T^{3/2})$, these authors concluded that for measurements above 77°F (25°C) the results should be reduced by 1% for each 10°F (approximately 5°C), while for measurements below 77°F a corresponding positive correction would be necessary. Similar corrections have been recommended by Palmes *et al.*¹⁵ Since in the case of indoor air studies the deviations from calibration temperature will normally not be greater than 5°C and bearing in mind the variations in diffusion coefficient values found in literature even for one temperature the influence of temperature can be neglected.

From Table IV (Part I) it follows that there is an incomplete recovery of the given pollutant concentration if Eq. 3 is used, i.e. if possible desorption losses during treatment of the activated carbon pad are neglected ($\varepsilon = 1$). The recovery varies for different compounds and may go down to 60% in some cases. On average, the recovery approaches 100% as the boiling point of the compound increases: The mean for *m*-xylene which has the highest boiling point of the compounds investigated (b.p. 139.1°C) does not show any difference between calculated and given concentrations, while in the cases of hexane and benzene (b.p. 69.0 and 80.1°C, respectively), average losses of 16% are found. These results show that the simplification made when establishing Eq. 3, i.e. $\varepsilon = 1$, does not hold for all compounds.

In addition, Table IV shows that there is a tendency in recovery. The parameter having the greatest influence on recovery might be the boiling point since for *m*-xylene (b.p. 139°C) with a boiling point close to that of the internal standard (cyclo octane b.p. 149° C) the experimental results

agree very well with the known concentration. For clarification, experiments were carried out in which a standard solution with known concentration of the individual substances was dropped onto an unloaded charcoal pad which was then treated as usual.

It can be seen from Table V that in fact for substances with lower boiling points (pentane, hexane, 1-hexene) losses were greater than for the high-boiling compounds which showed recoveries only slightly below 100%. Similar results have been obtained by Halliday and Anderson¹⁶ in experiments with halothane (b.p. 50°C) using the GasbadgeTM sampler. In four experiments at various concentrations they observed a mean recovery even slightly above 100%.

In order to avoid the use of different values of ε for each compound, it was decided to use only one mean correction factor, taking into consideration that the error thus made would generally not exceed by much that of the individual measurement. Thus, an approximative value of $\varepsilon = 0.87$ was used for substances in the 70 to 140°C boiling range. Accordingly, Eq. 4 should be used instead of Eq. 3 for calculation of the pollutant concentration:

$$c (\mu g/m^3) = 47.8 \cdot A/(Dt)$$
 (4)

Using this equation, the results of the experimental series involving benzene, toluene, m-xylene, hexane and heptane were again calculated. The concentrations thus obtained are listed in Table IV, Part II. It can be seen that Eq. 4 is far more suited for calculating the pollutant concentration in the air from the amounts of pollutant found on the charcoal pad than is Eq. 3.

Since the factor ε which is meant to account for losses during handling of the exposed charcoal, may also be dependent upon the amount of

Results of recovery experiments (each value is an average of 4 measurements).

Substance	Amount of substance given	(µg/sampler) found	Recovery (%)
entane	9.3	8.4	90
Hexane	7.1	6.6	93
leptane	9.1	8.8	97
Nonane	10.1	10.3	102
-Hexene	4.6	3.3	72
Benzene	9.8	9.3	95
Foluene	12.7	12.6	99
n-Xylene	11.8	11.3	96

TABLE V

pollutant adsorbed and as in the majority of cases low concentrations of pollutants are to be expected in indoor air studies, the concentration range below $1,000 \,\mu g/m^3$ was studied in more detail in another series of experiments. In these experiments, test atmospheres with known concentrations of benzene, toluene, m-xylene, hexane and heptane (cf. Figure 4) were produced and 8 pairs of GasbadgeTM samplers were exposed for periods of 5, 15, 23 and 24h. From the amounts trapped, the concentration of substances in the test gas was calculated using Eq. 4. In Figure 4 which is a plot of measured against known concentrations, the mean values are shown as well as the ranges of the individual values. The conclusion can be drawn that even for concentrations below 1,000 μ g/m³, Eq. 4 is well suited for converting the amount trapped on the pad into the concentration of a pollutant. As a further proof of the validity of Eq. 4 it should be mentioned that in a field study where passive samplers and adsorption tubes according to Grob⁵ had been used in parallel for an 8h sampling period, the results of both methods agreed within 20%. Details of this work are given elsewhere.¹⁰ These findings are comparable to those obtained for benzene at the 8 to 50 mg/m³ level by Lautenberger et al.¹⁴ In a comparative study with charcoal adsorption tubes and passive

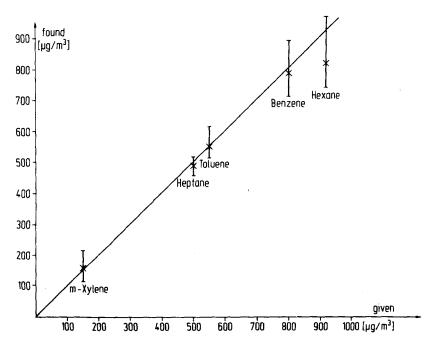


FIGURE 4 Determination of the hydrocarbon content of test gas atmosphere with GasbadgeTM samplers.

samplers having a design similar to those used in the present study, they found the passive sampler results to be lower by 3 to 16%.

3.4 Detection limits

As can be seen from Figures 2 and 3, the amount trapped by the passive sampler, A, at a constant pollutant concentration in the air, c, shows a linear relationship to the exposure period t as predicted by Eq. 4 for c = constant and D = constant. Thus, a passive sampler is suited for analysis of low concentrations in indoor air provided long exposure periods are selected which permit sufficient amount of substance to be trapped on the charcoal pad. Using the GasbadgeTM sampler and the analytical procedure described in Section 2.3, the detection limit is of an order of $0.5 \,\mu\text{g/sampler}$. With this figure and assuming a one hour exposure period and an average diffusion coefficient of $0.08 \,\text{cm}^2/\text{s}$ one can calculate from Eq. 4 that a mean pollutant concentration of about $300 \,\mu\text{g/m}^3$ can be determined. Thus, the detection limit is of an order of $300 \,\mu\text{g/(m}^3 \cdot h)$. It should be pointed out that this figure is only informative since it does not take into account any variation in gas chromatographic response factors or diffusion coefficients.

3.5 Applications

A passive sampler is less suited for measuring pollutant concentrations subject to short-term variations. Rather, its main usefulness will be in the determination of relatively constant exposure levels of extended duration. Thus, interesting applications may be for instance recording pollutant concentrations in long-term decay studies or tracing the sources of indoor air pollution. The following examples are taken from practical measuring programmes carried out by the authors.

During a routine check of the pollutant level within a flat, a toluene concentration exceeding typical values was found in some of the rooms. While normally concentrations between 50 and $100 \,\mu g$ toluene/m³ are encountered¹⁷, levels as high as $2,000-4,000 \,\mu g/m^3$ were measured in the kitchen and corridor of this flat (at the same time, the concentrations of other hydrocarbons were of the order of $20 \,\mu g/m^3$). A search for the source of these abnormal concentrations revealed that the tenant of the flat had been storing considerable amounts of freshly printed newspapers and magazines in the kitchen. A control analysis carried out in a newsstall led to an elevated toluene content of the air of more than $1,000 \,\mu g/m^3$. It can be concluded that the printed matter was the reason for the high toluene concentration in the flat.

B. SEIFERT AND H.-J. ABRAHAM

In a second case, it had to be checked whether the textile floor covering of a room was a source of toluene. For this purpose, three passive samplers were exposed: one approximately in the centre of the room near the lamp, another in a bookshelf made of chipboard and the third one in one of the corners of the room close to the floor covering. It can be seen from Table VI that except for toluene, all compounds were present in approximately the same concentrations irrespective of the sampler's location. Since the toluene concentration at the floor level amounted to only approximately one half of that measured in the bookshelf, the floor covering could be excluded as a source of toluene. Most probably, the toluene present in room air had its origin in the material of the bookshelf.

	Concentration $(\mu g/m^3)$						
Hydrocarbon	In book-shelf	Near lamp	Near carpet				
n-Hexane	5	5	4				
n-Heptane	3	3	4				
n-Octane	4	4	3				
N-Nonane	13	13	12				
n-Decane	24	26	23				
n-Undecane	15	17	14				
n-Dodecane	7	5	6				
Benzene	6	8	6				
Toluene	97	64	55				
m/p-Xylene	17	17	15				
o-Xylene	5	5	5				
Ethyl benzene	6	7	6				
1, 2, 4-Trimethyl benzene	14	13	14				

TABLE VI

Concentration of various hydrocarbons measured at different places in a living room (exposure time: 180 h)

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