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Evaluation of a small prototype passive sampler for airborne volatile organic compounds¹

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Abstract

A small prototype passive sampler assembled from relatively inexpensive and readily available materials was improved and evaluated for the determination of airborne volatile organic compounds (VOCs). Automated injection of extract aliquots directly from samplers into a gas chromatography system after in situ solvent extraction of VOCs with CS₂ is an attractive feature of the method. The performance of the method was compared with that based on the use of the Organic Vapour Monitors 3500 (OVM3500) from 3M Co. at different concentrations (ca. 0.01 to 30 mg/m³) of 25 VOCs in controlled atmospheres for ca. 8 h sampling periods. For most compounds, concentration values obtained with the prototype sampler were within $\pm 30\%$ of those obtained with the OVM3500 at concentrations below ca. 5 mg/m³. This method is useful primarily for exposure/workplace monitoring but does not have sufficient sensitivity for general ambient air measurements. © 1998 Elsevier Science B.V.

Keywords: Instrumentation; Sample preparation; Air analysis; Environmental analysis; Passive sampler; Volatile organic compounds

1. Introduction

Passive samplers were originally developed for the measurement of time-weighted-average exposure to airborne pollutants at relatively high concentrations in the occupational workplace, and thus have been widely used in this area. Although they are very simple and convenient to use compared with the active sampling methods, passive sampling methods are only used occasionally for the measurement of volatile organic compounds (VOCs) at ppb (v/v) (ppbv) or sub-ppbv levels in outdoor and indoor air,

e.g., Refs. [1-3]. One of the reasons for its less popularity in the ambient air VOC monitoring programs is that each passive sampler type has to be fully validated for each chemical to be monitored according to the validation protocols, which is very time consuming and expensive to carry out. Thus, very limited validation work has been done so far [4-9]. The other reason is due to the fact that current commercially available passive samplers are still relatively expensive, and consideration of their cost may detrimentally influence the design and scope of monitoring studies.

To explore the possibility of reducing costs of future investigation, a small, inexpensive passive sampler was designed, constructed, and briefly evaluated previously [10]. The analytical method based on

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the use of this prototype sampler allows in situ extraction of VOCs from the adsorbent and automated analyses without transfer of the extract from the sampler. In the preliminary evaluation [10], the potential usefulness of the device and method was demonstrated, but a need for improvement and further evaluation of the method also was indicated. The relatively poor detection limits (ca. 50 μ g/m³ for selected hydrocarbons, 8 h sampler exposure) were partly due to interfering substances in the extraction solvent (carbon disulphide, CS_2) and the carbonaceous adsorbent. A decrease in method blank readings would be beneficial. Also the range of target compounds and concentrations was quite limited in the preliminary tests. An assessment of the method for use with a wider range of compounds and concentrations typically found in indoor air was required to better determine the reliability of the method for use in VOC monitoring studies.

2. Experimental

2.1. Chemicals

The prototype sampler was evaluated for 25 VOCs which previously were monitored in indoor air [2]. The purities of these chemicals used for preparation of standard solutions were all better than 97%. CS₂ hydrocarbon, 99%) from Caledon (low in (Georgetown, Canada) and Aldrich (Milwaukee, WI, USA) was used for the preparation of standard solutions and for the extraction of VOCs in samplers. Since the CS₂ contained interfering substances at significant levels, which varied from batch to batch, the solvent was cleaned by the following procedure, based on that prescribed by OSHA [11].

About 70 g molecular sieve (type 13X, $\frac{1}{8}$ in. pellets; BDH, Toronto, Canada), which previously had been heated at 290°C for 24 h, was packed into a clean 50 cm×1.7 cm I.D. Pyrex glass column (1 in.=2.54 cm). About 40 ml of CS₂ was added to the top of the column and collected at a flow-rate of ca. 2 ml/min in a glass vial. The CS₂ was cleaned, in the same manner, a second time by use of the same molecular sieve column. Each 70 g batch of heat treated molecular sieve could be used to obtain ca. 120 ml of cleaned CS₂.

Stock standard solutions of VOCs were prepared by transferring a measured amount of each standard into a 40 ml amber glass vial containing 20 ml cleaned CS_2 . A series of working standard solutions containing each VOC at a nominal concentration of 0.05, 0.25, 1, 2.5 and 15 ng/µl were prepared by diluting the stock standard solution.

2.2. Preparation of prototype passive samplers

The construction of the prototype passive sampler has been described previously [10]. For this study, the sampler consisted of a 2 ml capacity, amber glass vial containing a 200 μ l capacity glass insert, which in turn contained a carbonaceous adsorbent disk at bottom of insert. Fig. 1 shows the schematic diagram of this prototype sampler.

The procedure for sampler preparation was improved to reduce blank levels. The adsorbent disks $(6.67\pm0.15 \text{ mg}, 3.5\pm0.1 \text{ mm}$ in diameter, $0.7\pm0.05 \text{ mm}$ in thickness) were cut from the activated charcoal cloth (W.L. Gore and Associates, Elkton, MD, USA). Contaminants on these disks were extracted, first with 1 ml CS₂ for 30 min, and then with two portions of 1 ml CS₂. The solvent cleaned disks were transferred to the flat-bottom glass inserts supported in 2 ml capacity amber glass vials, and heated in a clean oven at 290°C for 5 h. Laboratory air was



Fig. 1. Schematic diagram of prototype passive sampler.

introduced through the purge port into the oven at about 6 l/min by means of a metal bellows pump. After 5 h, ultra-high purity grade nitrogen gas was introduced into the oven at a flow-rate of ca. 4 l/min to cool the oven. The vials were removed from the oven and sealed with screw caps with septa for storage. For sampling of airborne VOCs, the screw caps and silicone disks on the prototype passive samplers were replaced by screw caps fitted with draft shields cut from a glass fibre filter (type A, binderless, 1 μ m porosity).

2.3. Extraction of VOCs

VOCs in exposed and blank prototype passive samplers were extracted in situ overnight (ca. 12–18 h) with 30 μ l CS₂. For the Organic Vapour Monitors 3500 (OVM3500) samplers (3M Co., St. Paul, USA), VOCs were extracted with 1.5 ml CS₂ for 30 min with frequent shaking of the sampler. Then the extract was transferred to a 2 ml capacity amber vial for analysis.

To simulate the storage of extracts, 30 μ l aliquots of working standard solutions (0.25 and 15 ng/ μ l) were added to prototype samplers, which were then sealed with septum screw caps and stored at room temperature (ca. 24°C) and normal indoor light conditions for either 48 h or 1 week before analysis of the contents. The effect of storage on recoveries of VOCs (Table 2, below) was calculated by comparison of the analytical results for the samplers with those of standard solutions in inserts/vials. Tests with group A and group B compounds were done separately to reduce interferences during gas chromatography–mass spectrometry (GC–MS) analyses.

2.4. Exposure chambers and tests

A Plexiglas exposure chamber (38 cm \times 38 cm \times 29 cm) was used in the sampler exposure tests. The organic vapour generating system which has been described previously [12] was used to prepare dynamic test atmospheres in the chamber. Depending on the target concentrations to be generated in the chamber, a mixture of group A or B standards in a gas tight syringe of size 0.5 ml to 5 ml was injected at a rate ranging from 0.2–0.7 ml/h into a sealed heated metal block where nitrogen gas swept the

organic vapours at a rate ranging from 0.8-3 l/min into a 2 l glass mixing chamber. Here the vapours were mixed with laboratory air at a rate ranging from 0.8-4 l/min and then led into the Plexiglas chamber before they were directed into a fume hood. An electric fan was used to ensure adequate air velocity (1.5-2.5 m) within the chamber. The air temperature and relative humidity inside the chamber were $24\pm1^{\circ}$ C and $35\pm2\%$. Before exposure of the samplers, the electric fan was started, and the organic vapour mixture was then introduced for about 2 h.

Tests with group A and group B compounds were done separately. For the sampler comparison tests (Table 1), three samplers of each type were exposed (8–12 h) at each of four concentrations, and unexposed samplers (blanks) were also extracted and analysed. For each of the prototype sampler storage tests (0.02–0.03 and 10–20 mg/m³), six samplers were exposed for 8 h. Three samplers were extracted immediately and analyzed, while the other three samplers were stored at room temperature for 1 week before extraction and analysis.

2.5. GC-MS analysis

A Hewlett–Packard (HP) 5890 Series II gas chromatograph equipped with a HP 5972A massselective detector operated in the selected ion monitoring (SIM) mode was used for all analyses. The GC system was equipped with a HP 7673A automatic sampler, and a DB-624 capillary column (60 $m \times 0.32$ mm I.D., 1.8 µm film thickness; Chromatographic Specialties, Brockville, Canada). The column head pressure was 6 p.s.i., and the oven temperature was held at 40°C for 4 min, raised to 220°C (0 min) at 10°C/min, then to 250°C (6 min) at 20°C/min (1 p.s.i. = 6894.76 Pa). The injector and the transfer line temperatures were 250°C.

Aliquots (3 μ l) of extracts and standard solutions in the 2 ml vials were injected automatically in the splitless mode, and the injector purge gas flow (helium, 30 ml/min) was started 0.5 min after injection. Analytical data acquisition and editing were done by means of a custom designed, SIM method based on one quantification ion and two qualifier ions per target compound. An external standard method based on peak areas was used for quantification.

Compound	12 h Exposure				9 h Exposure			8 h Exposure			8 h Exposure					
	OVM3500		Prototype		OVM3500		Prototype		OVM3500		Prototype		OVM3500		Prototype	
	(mg/m ³)	(%)	(mg/m ³)	(%)	(mg/m ³)	(%)	(mg/m ³)	(%)	(mg/m ³)	(%)						
Group A																
n-Hexane	3.8	1.9	6.8	5.8	0.72	4.5	1.1	7.6	0.17	11	0.17	8.3	0.03	9.2	0.03	9.4
Chloroform	8.0	0.7	20	4.9	2.0	4.3	4.1	3.9	0.10	9.9	0.15	9.4	0.02	7.8	0.03	5.7
Benzene	8.9	2.1	16	5.1	1.8	2.4	2.7	5.4	0.23	9.5	0.22	10	0.02	16	0.03	3.3
Trichloroethylene	11	2.5	23	2.7	2.4	2.6	3.9	5.0	0.22	10	0.27	7.8	0.03	12	0.03	7.4
Toluene	7.9	4.6	14	4.3	1.1	4.7	1.1	8.2	0.22	13	0.19	12	0.01	27	0.01	25
Tetrachloroethylene	13	4.4	26	1.9	2.6	3.1	3.8	7.1	0.25	10	0.28	8.2	0.05	9.2	0.05	23
Ethylbenzene	6.9	4.9	11	6.2	1.2	2.2	1.3	10	0.18	11	0.18	7.8	0.00	3.3	NDb	
(m+p)-Xylene	18	4.5	27	6.3	3.2	2.5	3.0	11	0.49	11	0.45	7.6	0.02	2.3	0.01	22
o-Xylene	10	5.2	13	6.4	1.7	1.3	1.5	11	0.25	11	0.23	8.5	0.01	13	0.00	31
n-Dichlorobenzene	23	5.0	23	10	3.9	3.4	3.1	9.2	0.25	14	0.24	11	0.04	13	0.03	28
p-Dichlorobenzene	15	5.3	16	11	2.4	3.7	2.1	7.3	0.26	13	0.31	5.8	0.03	16	0.03	21
Naphthalene	6.7	2.8	3.3	14	0.69	4.3	0.29	3.1	0.10	15	0.12	14	0.02	5.1	0.02	39
Group B																
1,2-Dichloroethane	5.8	0.3	10	6.6	3.5	6.3	5.0	9.2	0.10	2.0	0.15	9.8	0.03	4.8	0.04	14
Styrene	10	6.4	7.3	13	3.1	8.2	1.3	17	0.15	0.70	0.02	43	ND^{b}		ND^{b}	
e-Pinene	9.2	5.0	13	7.9	5.5	8.1	6.1	12	0.06	13	0.05	12	0.03	3.7	0.03	20
,1,2,2-Tetrachloroethane	13	4.0	19	8.4	9.3	7.7	11	9.8	0.05	6.6	0.06	17	0.03	2.4	0.03	27
a-Decane	13	7.4	13	5.9	6.9	9.4	4.8	4.1	0.01	28	0.01	43	0.02	20	0.01	30
,3,5-Trimethylbenzene	15	5.9	18	6.0	8.1	8.4	8.2	12	0.03	13	0.03	25	0.02	17	0.02	25
,2,4-Trimethylbenzene	16	4.7	19	5.8	9.4	8.8	7.5	14	0.02	19	0.01	21	0.02	8.8	0.02	35
Pentachloroethane	14	3.7	24	6.4	11	8.2	14	12	0.03	7.7	0.04	10	0.03	2.2	0.04	21
l-Limonene	20	5.5	24	5.3	11	7.8	9.6	15	0.01	9.4	0.01	49	0.03	3.5	0.02	43
-Cymene	26	5.2	30	5.1	13	7.8	13	13	0.03	10	0.03	25	0.06	1.6	0.06	18
Hexachloroethane	14	4.7	22	5.4	11	7.1	13	13	0.06	2.6	0.08	12	0.10	6.0	0.14	17
1,2,4-Trichlorobenzene	29	6.4	16	18	18	5.9	10	14	0.02	12	0.01	25	0.04	2.3	0.04	28

Table 1 Exposure chamber VOCs concentration (mg/m^3) and relative standard deviation $(\%)^a$ as determined by OVM3500 and prototype samplers

^aThree samplers, calculation based on more significant figures than shown.

^bNot detected.

3. Results and discussion

3.1. Blank levels and detection limits

The relatively poor detection limits were of some concern after the preliminary examination of the prototype sampler method [10]. Treatment of the CS₂ with molecular sieve significantly improved the solvent blank readings for some compounds. Blank readings for the adsorbent disks were also reduced by use of the revised heat treatment procedure, but still contributed to the prototype sampler blank readings for some compounds. In addition to nhexane detected at levels about 0.14 ng/µl, chloroform, benzene, trichloroethylene, toluene, tetrachloroethylene, p-dichlorobenzene and naphthalene were also detected at levels of about 0.01-0.03 $ng/\mu l$ in the extracts of cleaned prototype samplers. When prototype samplers were stored at room temperature for 3 weeks, there was no significant increase in blank readings compared to readings for samplers stored for shorter periods.

Although the instrument detection limit (ca. 5 pg injected or 0.002 ng/µl) was quite good, the sampler method blank readings limited the sensitivity of the method. To estimate the method detection limits for airborne VOCs, an average sampling rate of 0.1 cm³/min was used, 100% extraction recoveries were assumed, and the estimated minimum detectable amounts of VOC were considered. For an 8 h sampler exposure, the method detection limits for *n*-hexane, benzene, toluene, *p*-dichlorobenzene, styrene, and 1,2,4-trichlorobenzene, respectively, were 37, 22, 10, 15, 63, and 19 µg/m³. Values ranged from 3 to 7 µg/m³ for the other compounds.

3.2. Comparison of prototype and OVM3500 samplers

It was considered prudent to compare the performance of the prototype sampler method with the commonly used OVM3500 based method to aid in the assessment of the former method. The results for side-by-side exposures of the two types of samplers at four different concentrations of the target VOCs are reported in Table 1.

It can be seen from Table 1 that, for exposures to relatively low concentrations (below ca. 0.5 mg/m^3),

the results obtained by means of the prototype samplers were generally similar to those obtained with the OVM3500, with differences less than 20% for most of the compounds. Notable exceptions were naphthalene, 1,2-dichloroethane, and hexachloroethane, for which the values obtained with prototype samplers were about 30–60% higher than those obtained with the OVM3500. For styrene and 1,2,4trichlorobenzene, respectively, the concentrations obtained with the prototype sampler were 87 and 50% lower than those obtained with the OVM3500.

At concentrations of ca. $1-10 \text{ mg/m}^3$, the results obtained with the prototype samplers were generally similar to those obtained with the OVM3500, with differences less than 20% for most of the compounds. Exceptions were n-hexane, chloroform, benzene, trichloroethylene and hexachloroethane for which the values obtained with the prototype samplers were ca. 30-50% higher than those for the OVM3500. Naphthalene, styrene, and 1,2,4-trichlorobenzene showed lower values for the prototype sampler than for the OVM3500. However, at higher exposure concentrations (ca. $10-30 \text{ mg/m}^3$) and the longer, 12 h exposures, the concentrations for the prototype samplers were consistently higher (ca. 40-90%) than those for the OVM3500, for most of the compounds. Exceptions were *m*-dichlorobenzene, *p*dichlorobenzene, n-decane, 1,2,4-trimethylbenzene, for which the prototype sampler values were quite similar to those obtained with the OVM3500. For naphthalene, styrene, and 1,2,4-trichlorobenzene, prototype values were lower than OVM3500 values.

It is difficult to explain why readings obtained by means of the OVM3500 sampler were considerably lower than those found with the prototype sampler at the highest exposure concentration (Table 1). Saturation of the adsorbent could be an explanation for low readings. According to available information [13], saturation of the OVM3500 adsorbent occurs when about 25 mg of VOCs have been collected. If we use the information in Table 1, it can be calculated that only about 10 mg of VOCs were collected over 12 h at the highest VOCs concentration.

The precision values for replicate determinations at low concentrations were generally considerably higher than those at the higher exposure concentrations. Overall, slightly better precision was obtained with the OVM3500 than with the prototype sampler. R.S.D. values <10% generally were observed for the highest exposure concentration, and some group A compounds at the lower concentrations.

If determination of the accuracy of the prototype sampler method is judged by the above results, and a arbitrary criterion somewhat of **OVM3500** value ±30% is used as an acceptable range for prototype method values, some conclusions can be drawn. At the highest exposure concentration only a few prototype sampler values were within this range, but at concentrations below ca. $1-5 \text{ mg/m}^3 \text{ most of}$ the compounds had prototype values within this range. Compounds which consistently had values outside the ±30% range were chloroform, 1,2-dichloroethane, styrene, hexachloroethane and 1,2,4trichlorobenzene. No explanation for the outliers could be determined. However, for chloroform, use of the theoretical instead of the experimental OVM3500 sampling rate (SR) for calculation of concentration resulted in OVM3500 values which were quite similar (within $\pm 35\%$) to the prototype values.

In conclusion, if prototype values within $\pm 30\%$ of corresponding OVM3500 values are acceptable, the prototype method could be judged accurate for monitoring for most of the target VOCs, particularly at concentrations below 5 mg/m³.

3.3. Storage effects

As undesirable changes may occur in stored exposed samplers and extracts, two types of tests were done to determine the potential for storage effects. In the first series of tests, 30 µl aliquots of standard solutions were stored in prototype samplers at room temperature (ca. 24°C) and normal indoor light conditions for either 48 h or 1 week and then analyzed. The results in Table 2 show that the recoveries of most target VOCs were similar to those measured for samplers stored for 12 h. However, the extraction recoveries for styrene, α -pinene, and dlimonene decreased significantly during storage, especially at low VOCs loading levels. The recovery for styrene at the loading level of 15 ng/µl did not change significantly after 48 h storage, but dropped by more than 50% after 1 week. Due to low recovery, this compound could not be detected at the 0.25 ng/ μ l loading. The recoveries for α -pinene and *d*-limonene only changed slightly over 48 h, but decreased significantly during 1 week of storage, except for *d*-limonene recoveries which only changed slightly at the high concentration. The R.S.D. for replicate determinations was usually below 10%, and always below 15%, except for styrene with 21% R.S.D. at 15 ng/ μ l.

Based on the above results, it may be concluded that, if reasonable analytical accuracy is desired for these three compounds, the prototype sampler extracts should not be stored for more than 2 days before analysis. The results also support the earlier notion that the two terpenes may degrade in the presence of a carbonaceous adsorbent and CS_2 .

The effect of storage on VOCs collected in the prototype samplers was also examined in tests in which samplers were exposed to airborne VOCs. For one half of the samplers, extraction and analyses were done immediately as for earlier tests. The other samplers were stored at room temperature for 1 week before extraction and analyses. The results in Table 2 show that there was no significant storage effect for any of the target compounds, including styrene, α pinene and d-limonene, at the two test concentrations. Based on the results in Table 2, it can be concluded that exposed samplers should not be extracted immediately if there will be a delay before analyses of the extracts, and that the samplers can be stored for 1 week without a significant effect of the storage on results.

4. Summary

The results of these investigations show that the prototype sampler method is reliable for determination of most of the 25 target VOCs at concentrations between ca. 0.01 and 5 mg/m³. Use of the method for determination of some compounds, such as styrene, α -pinene, and *d*-limonene, is not recommended due to inadequate detection limits, extraction recoveries, precision of results, stability and other reasons. The method precision is not quite as good as that based on the OVM3500. Disadvantages of the prototype sampler method are the relatively high detection limits, typically 10–20 µg/m³ for 8 h exposures.

Compound	Extractio	n recovery	(%)	Ratio of conc. before and after						
	0.25 ng/	սլ		15 ng/u	1		storage ; exposure concentration			
		h			-		$0.02 - 0.03 \text{ mg/m}^3$	$10-20 \text{ mg/m}^3$		
	Week	48 h	12 h	Week	48 h	12 h	-	-		
Group A										
<i>n</i> -Hexane	100	96	103	104	108	100	0.97	0.99		
Chloroform	98	90	100	95	97	92	0.98	1.03		
Benzene	104	93	99	95	98	94	0.96	1.02		
Trichloroethylene	100	94	101	97	100	95	0.97	1.02		
Toluene	96	88	99	93	100	94	0.96	0.99		
Tetrachloroethylene	98	101	99	96	105	94	1.08	0.97		
Ethylbenzene	95	87	99	94	105	97	0.96	1.01		
(p+m)-Xylene	87	93	97	91	100	94	0.98	1.01		
o-Xylene	91	86	89	86	95	88	0.96	1.04		
<i>m</i> -Dichlorobenzene	87	86	86	70	76	74	0.82	1.11		
<i>p</i> -Dichlorobenzene	b	b	89	83	87	84	1.00	1.12		
Naphthalene	b	b	28	10	12	11	0.85	0.96		
Group B										
1,2-Dichloroethane	107	94	90	89	96	95	1.02	1.09		
Styrene	0	0	0	11	35	34	0.00	0.95		
α-Pinene	1.3	47	59	69	98	89	1.02	1.04		
1,1,2,2-Tetrachloroethane	80	80	79	74	82	82	0.95	1.13		
<i>n</i> -Decane	86	92	99	105	108	99	0.98	1.04		
1,3,5-Trimethylbenzene	87	96	95	96	102	96	0.97	1.00		
1,2,4-Trimethylbenzene	92	99	96	84	96	92	0.97	0.94		
Pentachloroethane	98	95	98	89	99	95	0.95	0.99		
d-Limonene	7.8	65	80	93	101	97	0.92	0.90		
p-Cymene	120	117	105	101	109	101	0.97	1.01		
Hexachloroethane	107	105	96	94	100	97	0.95	1.14		
1,2,4-Trichlorobenzene	66	93	94	58	63	75	1.10	1.10		

Table 2				
Desults of storess	tasts for metatring comm	and containing standard	colutions on armoad	to sinhoma VOC

^aRatio of VOC concentration in extract from sampler extracted and analysed after 1 week storage and in extract from sampler extracted and analysed immediately after exposure.

^bSamplers were contaminated.

One advantage of the prototype method is its lower cost compared to other commercially available passive samplers. In addition, the amount of potentially hazardous CS_2 used for the method is considerably less than that required for the OVM3500 method, and sample preparation is limited to the addition of CS_2 to the sampler.

Use of the prototype sampler would be most practical and cost-effective for large air quality surveys, especially for exposure/workplace monitoring. Although detection limits can be improved by increasing the sampler exposure time, the potential for adsorbent saturation should be considered if VOC concentrations are too high. Depending on the expected application, additional evaluation of the method may be required, and improvement in the sampler design should be considered.

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