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Screening method for the determination of 28 volatile organic compounds in indoor and outdoor air at environmental concentrations using dual-column capillary gas chromatography with tandem electron-capture–flame ionization detection

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Abstract

A gas chromatographic method is presented for the simultaneous determination of 28 volatile organic compounds (VOCs) in indoor and outdoor air at environmental concentrations. Using diffusive (passive) samplers, the VOCs were adsorbed onto charcoal during a four-week sampling period and subsequently desorbed with carbon disulphide. After injection, using a cold split-splitless injector, the mobile phase was split via a Y-connector and led onto two capillary columns of different polarity switched in parallel. This dual-column configuration provides additional information about the VOC components and can be obtained for verification purposes. Detection was in both cases performed by connecting each column with a non-destructive electron-capture detector and a flame ionization detector switched in series. By this procedure sensitivity is increased because no effluent splitting is required. At the same time, sample throughput is enhanced drastically since several items of information are obtained simultaneously. The procedure has been successfully applied in the context of a large field study to measure outdoor air concentrations in three areas with different traffic density. It is applicable to indoor air measurements in like manner.

Keywords: Air analysis; Environmental analysis; Volatile organic compounds; Hydrocarbons; Halocarbons; Passive sampling

1. Introduction

During the last few years, interest has grown in indoor and outdoor air quality. As a result, the concentration of selected volatile organic compounds (VOCs) in ambient air, in particular, has been the subject of many investigations. In this context, aromatic hydrocarbons like benzene, toluene and xylenes or aliphatic halocarbons like tetrachloroethene have mostly been the focus of interest. Less attention has so far been paid to the determination of other air pollutants, such as aliphatic hydrocarbons, ketones, and esters.

VOCs are ubiquitous in the air we breathe and include a multitude of components which can cause a variety of adverse health effects. Automobile exhausts and industrial emissions are the main outdoor VOC sources. In addition to the penetration of outdoor pollutants into the indoor environment, numerous indoor sources exist, like tobacco smoke,

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gas or kerosene heaters, building or furnishing materials, paints, adhesives and other consumer products. Because of this complex, multisource exposure, more VOCs need to be identified and quantified routinely in order to expand our knowledge of the occurrence of VOCs in indoor and outdoor air and potential health effects. The characterization of the VOC pattern can additionally contribute to the identification of the exposure sources and can draw the attention to compounds which had so far been regarded as of no significance as environmental pollutants.

In this context, a simple and reliable screening method is urgently demanded which can also be routinely applied in large field studies. The analytical procedures suitable for these purposes involve the use of passive (diffusive) samplers containing activated charcoal as adsorbent followed by a gas chromatographic separation in conjunction with one or more GC detection methods including universal detection methods as the flame ionization detection (FID), less-specific detection methods, such as electron-capture detection (ECD) and nitrogen-phosphorous detection (NPD) or specific detection methods as mass spectrometry (MS) or ion trap detection (ITD) [1–7]. When exposed for long sampling periods of several weeks, these passive samplers (originally designed for occupational exposures) have been proven to be an excellent tool for the investigation of chronic exposures to various non-polar to semi-polar VOCs at environmental concentrations. Although in the past exclusively chosen for indoor air measurements, the usefulness of passive monitors for outdoor air sampling has been recently demonstrated [1,2,4,7].

While the non-specific detectors are less expensive and in some cases (e.g. ECD in case of halocarbons) more sensitive than specific detectors, they are more prone to interference because the identification of a compound is performed only by its retention time. Misidentification due to overlapping peaks can be drastically reduced by using simultaneous detection by different detectors or by comparing retention times from GC columns of different polarity for validation.

In this paper a procedure is described for the simultaneous determination of 28 VOCs in indoor and outdoor air samples using passive sampling in connection with dual-column gas chromatography and tandem ECD–FID detection. The method permits routine monitoring of these VOCs in indoor and outdoor air at the environmental concentration level. Using the dual-column system a simultaneous confirmation of the results is achieved within a single chromatographic run.

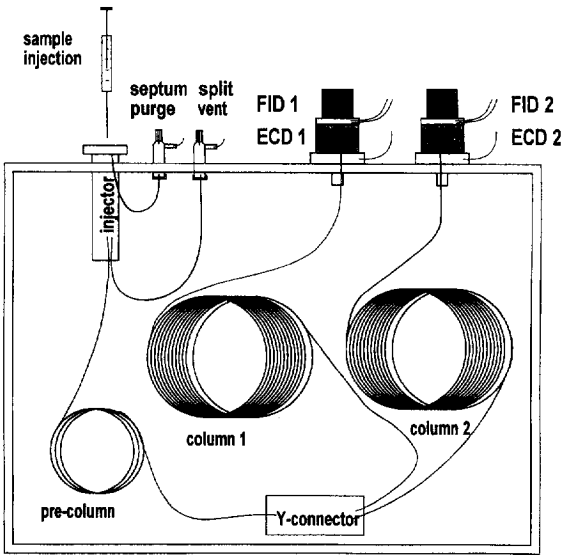

Fig. 1. Illustration of the dual-column gas chromatographic system with tandem ECD–FID.

2. Experimental

2.1. Passive sampling

Indoor and outdoor air samples were collected using passive (diffusive) samplers (OVM 3000, available from 3M, Neuss, Germany). Due to the small amounts that are collected onto the charcoal pads of the monitors at environmental concentrations, it is important to determine the background levels of the unexposed samplers. Unexposed monitors are therefore measured on a regular basis. As the blank levels of the unexposed monitors can vary from lot number to lot number ([1,7], this work), the unexposed and the sampling monitors must come from the same lot number. For outdoor

sampling, the monitors were placed in an aluminium box attached to lamp-posts near the pedestrian walk at a height of about 2 m [1]. To protect them from the influence of weather (rain, snow, dust etc.) they were housed in an aluminium box with a height of 10.5 cm and a diameter of 11.5 cm, which had to slits of 0.5×6 cm aside and an opening at the bottom. Inside the box had a device to fix 2 passive samplers. Indoors the monitors were placed at breathing height at a distance of at least 1 m from walls to guarantee sufficient air circulation. In both cases the passive samplers were exposed for 4 weeks. At the end of the exposure period they were closed with an impermeable cap and stored at 4–8°C until analysed.

Storage stability of the passive samplers was tested by exposing 8 monitors in the same room at a distance of about 20 cm from each other. In 4 monitors, the 28 VOCs were determined immediately after exposure and 4 monitors were analysed after a storage period of 6 weeks at 4–8°C. Storage losses were not observed.

2.2. Sample preparation

All reagents and materials coming into contact with the samples and standards were randomly tested for contamination.

Before use, all glassware was washed with nitric acid (1+1, 95°C, 30 min), rinsed with ultrapure water and dried at 150°C for 12 h. Plastic material was cleaned with Extran solution (60°C, 30 min) and ultrapure water and heated for 20 h at 80°C. All cleaned materials were stored in a laminar flow cabin additionally equipped with a charcoal filter.

The VOCs collected on the charcoal pads of the monitors were desorbed with 1.5 ml carbon disulphide ("low benzene" grade, Promochem, Wesel, Germany). After addition of carbon disulphide, the

Table 1
Gas chromatographic conditions for the determination of 28 VOCs in air samples

Gas chromatograph	HRGC 5300 (Fisons Instruments) with autosampler AS 200 and PC data station with software "Maxima" (version 3.3)
Injector	Split-splitless temperature programmable multi-injector MFA 515
Detectors	2 ECD and 2 FID system; the ECD and FID systems were switched in series
Precolumn	2.5 m methyl silicone deactivated capillary column, 0.32 mm inner diameter (Chrompack)
Capillary column 1	60 m DB-5 (J&W Scientific) 1 μm film thickness, 0.32 mm inner diameter
Capillary column 2	60 m DB-1701 (J&W Scientific) 1 μm film thickness, 0.32 mm inner diameter
Carrier gas	Helium, purity: 5.0, flow rate: 2 ml/min for each column
Make-up gas	nitrogen, purity: ECD grade flow rate: 30 ml/min
Split	10 ml/min
Temperatures	
ECD 1 and ECD 2	330°C
FID 1 and FID 2	330°C
Injector (PTV)	50°C for 1 s, then ballistically heated to 250°C
Columns	5 min at 35°C, 4°C/min to 150°C, 30 min at 150°C
Sample size	2 μl
Data evaluation	Peak area

Table 2
Retentions times (t_R) on the two columns of different polarity and detection limits (DL) related to an exposure interval of four weeks

Compound	t_R (min)		DL ($\mu g/m^3$)
	DB-5	DB-1701	
Benzene	10.99	11.79	0.1
2-Butanone	7.81	10.51	0.8
n-Butyl acetate	19.19	20.85	0.1
n-Decane	29.16	27.17	0.2
Ethyl acetate	8.60	10.01	0.2
Ethylbenzene	22.12	22.86	0.2
2-Ethyltoluene	28.50	29.21	0.3
3-Ethyltoluene	27.49	28.05	0.4
4-Ethyltoluene	27.58	28.20	0.5
n-Heptane	12.87	11.09	0.1
n-Hexane	8.07	6.85	1.3
Naphthalene	38.85	42.32	0.2
n-Nonane	24.01	22.00	0.1
n-Octane	18.51	16.52	0.1
n-Propylbenzene	27.11	27.69	0.1
Pyridine	15.27	18.56	0.2
Tetrachloroethene	19.24	18.56	0.01
Tetrachloromethane	10.99	10.56	0.01
Toluene	16.74	17.67	0.2
1,1,1-Trichloroethane	10.13	10.56	0.1
Trichloroethene	12.88	13.51	0.01
Trichloromethane	8.78	11.10	0.01
n-Tridecane	44.22	41.46	0.2
1,2,4-Trimethylbenzene	29.16	29.96	0.2
1,3,5-Trimethylbenzene	27.84	28.45	0.3
n-Undecane	33.90	31.97	0.2
m,p-Xylene	22.56	23.38	0.4
o-Xylene	23.89	24.87	0.1

monitors were closed and mechanically shaken for 30 min.

2.3. Gas chromatographic analysis

The system used is schematically shown in Fig. 1. The in-series connection of ECD and FID was achieved by means of an adapter with built-in lines for the supply of the fuel gases required by FID. This detector combination is equipped with an additional heater electrically connected to the ECD controller to prevent condensation of high boiling substances. A kit to connect both detectors in series is commercially available from Fisons Instruments. The precolumn was coupled via a "Y" connector (by SGE, Weiterstadt, Germany) to the two capillary columns of different polarity switched in parallel splitting the gas phase by a ratio of 1:1. The gas chromatographic conditions are given in Table 1. Prior to injection the split–splitless temperature programmable multi-injector was held at 50°C. After injection the solvent was removed via the split, while the VOCs remain in the insert of the injector filled with glass wool. After 1 s the split was closed and the injector was rapidly heated to 250°C to evaporate the VOCs and to transfer them to the capillary columns. At the same time the oven temperature program was started.

2.4. Calibration and calculation

Calibration was performed by analysing a blank and three standards before and after each analytical section (25 samples). The means of the two runs were used for calibration. The calibration curves were set up between 8 $\mu g/l$ and 35 $\mu g/l$ (FID, non-halogenated hydrocarbons) and between 0.3 $\mu g/l$ and 1.6 $\mu g/l$ (ECD, halocarbons). Standards were prepared using reagents with the highest purity available. The stock solution prepared in methanol can be used for at least 6 months. Standards made by diluting the stock solution with carbon disulphide were freshly prepared just before use.

The air concentrations C_a of the individual VOCs were calculated according to the following equation:

$$C_a\,(mg/m^3) = 1.01\, m\, t^{-1}\, r^{-1}\, A$$

where m = mass of substance adsorbed on the sampler (μg), A = constant, including uptake rate (10^{-3} min m^{-3}), r = recovery coefficient, t = sampling interval (min).

The constant A for each individual VOC was adopted from [8]. The recovery coefficients of the desorption procedure to adjust for incomplete desorption of the compounds from the charcoal pad were determined according to the phase equilibrium method described by Rodriguez et al. [9]. A standard solution of the analytes (in carbon disulphide) was pipetted through the center port of the cap onto the charcoal pad of a monitor. The port was closed and the monitors were allowed to elutriate for 30 min, decanted into glass vials and analysed.

The reproducibility between different monitors was investigated by exposing 10 monitors simul-

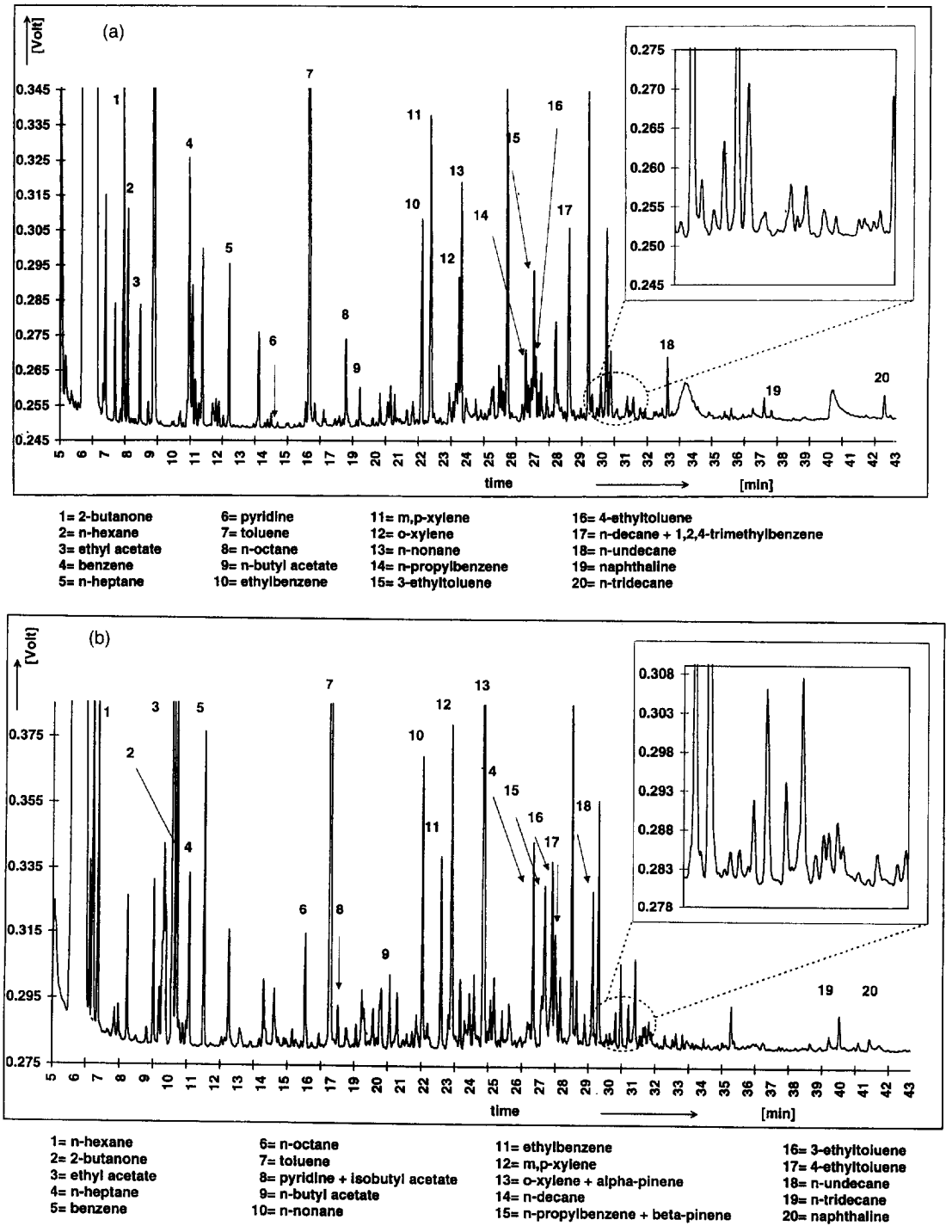

Fig. 2. Typical gas chromatograms of an indoor air sample obtained by FID detection. (a) DB-5 column, (b) DB-1701 column.

Table 3
Background levels of unexposed passive samplers ($n=5$) from two different lots (related to an exposure interval of four weeks)

Compound	Background level ($\mu g/m^3$)	
	Lot A	Lot B
Benzene	0.07	0.06
2-Butanone	0.16	0.07
n-Butyl acetate	n.d	n.d.
n-Decane	n.d	n.d.
Ethyl acetate	n.d	n.d.
Ethylbenzene	n.d	0.11
2-Ethyltoluene	n.d	n.d.
3-Ethyltoluene	n.d	n.d.
4-Ethyltoluene	n.d	n.d.
n-Heptane	n.d	n.d.
n-Hexane	1.09	1.07
Naphthalene	n.d	n.d.
Nonane	n.d.	n.d.
n-Uctane	n.d.	n.d.
n-Propylbenzene	n.d.	n.d.
Pyridine	n.d.	n.d.
Tetrachloroethene	0.001	0.001
Tetrachloromethane	0.001	0.001
Toluene	0.09	0.05
1,1,1-Trichloroethane	0.024	0.01
Trichloroethene	0.003	0.005
Trichloromethane	0.003	0.004
n-Tridecane	0.13	n.d.
1,2,4-Trimethylbenzene	n.d.	n.d.
1,3,5-Trimethylbenzene	n.d.	n.d.
n-Undecane	n.d.	0.14
m,p-Xylene	n.d.	0.24
o-Xylene	n.d.	0.12

n.d. = not detectable.

taneously in a living room at a distance of about 20 cm from each other.

In a former study [1] we have demonstrated by comparative testing that the passive sampler procedure is equivalent to the conventional active pump/solid sorbent tube method.

Table 4
Interferences due to peak overlap

Column	Analyte	Interference
DB-5	Benzene	*n*-Butanol, cyclohexane
	n-Decane	1,2,4-Trimethylbenzene
	2-Ethyltoluene	β-Pinene
	1,2,4-Trimethylbenzene	*n*-Decane
DB-1701	*n*-Propylbenzene	β-Pinene
	Pyridine	Isobutyl acetate
	Tetrachloromethane	1,1,1-Trichloroethane
	1,1,1-Trichloroethane	Tetrachloromethane
	o-Xylene	α-Pinene

2.5. Detection limit and reliability criteria

The detection limit was calculated as the three-fold standard deviation of replicate measurements ($n=10$) of the monitor blanks. For analytes with blank values too low to be registered the three-fold standard deviation of a low level standard solution was used for calculation. The detection limits are read from the individual calibration curves and are given in $\mu g/m^3$ related to an exposure interval of four weeks.

For internal quality control a standard pooled from real air samples in the environmental concentration range was used. The quality control standard was analysed within each analytical section.

2.6. Applications

Outdoor air concentrations of 28 VOCs were determined in the context of a field study performed in three areas with different traffic density. The study was carried out in two quarters of Wuppertal (Elberfeld and Oberbarmen), an industrialized city in Northrhine-Westphalia, Germany. Borken (Northrhine-Westphalia), a small town located in a rural area, served as reference area.

Sampling was performed by exposing monitors at five measurement points per study area during five 4-week periods. According to this, 25 samples per study area were available.

3. Results and discussion

The dual capillary column combination of DB-5 and DB-1701 switched in parallel permits the separation and determination of 28 VOCs in indoor and outdoor air samples at environmental concentrations. Retention times for the 28 VOCs obtained on the two different columns are summarized in Table 2. Fig. 2 shows two typical chromatograms of an indoor air sample obtained by FID on columns of different polarity. The detection limits referring to a sampling interval of four weeks are also given in Table 2. The

Table 5
Reproducibility of the gas chromatographic procedure and variation from monitor to monitor

Compound	Conc. ($\mu g/m^3$)	Within-series precision $n=10$ (%)	Day-to-day precision $n=7$ (%)	Monitor-to-monitor precision $n=10$ (%)
Benzene	5.8	3.0	13.0	2.0
2-Butanone	0.8	3.9	34.9	2.1
n-Butyl acetate	1.1	20.2		1.3
n-Decane	0.7	5.2	20.3	3.3
Ethyl acetate	0.7	8.4	7.2	2.0
Ethylbenzene	4.4	4.0	16.2	5.2
2-Ethyltoluene	1.3	4.1	18.6	4.9
3-Ethyltoluene	3.5	3.6	17.4	2.9
4-Ethyltoluene	1.6	3.8	16.9	3.3
n-Heptane	1.5	3.3	14.9	2.3
n-Hexane	n.d.			
Naphthalene	1.1	6.2	20.0	21.1
n-Nonane	0.5	4.7	17.4	1.6
n-Octane	0.6	7.3	19.4	4.8
n-Propylbenzene	0.5	5.0	17.6	2.0
Pyridine	0.3	10.4	19.1	22.4
Tetrachloroethene	1.6	5.2	14.6	3.0
Tetrachloromethane	0.8	3.1	12.1	2.6
Toluene	16.8	3.9	13.3	1.9
1,1,1-Trichloroethane	1.5	3.1	10.5	1.5
Trichloroethene	0.4	3.4	9.3	2.9
Trichloromethane	0.1	3.5	13.6	
n-Tridecane	0.2	19.2	35.2	
1,2,4-Trimethylbenzene	4.9	3.8	18.4	
1,3,5-Trimethylbenzene	1.3	4.0	17.8	
n-Undecane	0.3	5.2	25.1	
m,p-Xylene	10.3	4.2	18.0	
o-Xylene	3.3	4.2	25.1	

n.d. = not detectable.

background levels in unexposed samplers from two different lots related to an exposure interval of 4 weeks are listed in Table 3. The results show that *n*-hexane is most seriously affected by blank levels which are responsible for its relatively poor detection limit. Aromatic and chlorinated hydrocarbons are also normally found in unexposed samplers. As already mentioned above, unexposed samplers that come from the same lot as the sampling monitors have to be analysed within each analytical series to permit blank correction, if necessary.

Interferences due to peak overlap occurring by using either the DB-5 or the DB-1701 column are summarized in Table 4. For each of the 28 VOCs under investigation interference-free determinations are possible with at least one of the two columns. The non-specific FID and less-specific ECD used in this method are easier to use, less expensive and in some cases (e.g. ECD for the determination of halogenated compounds) more sensitive than specific detectors, such as MS or ITD. Because in the case of non-specific detectors the identification of the analyte is solely determined by its retention time, the described dual-column system is applied using sample coinjection onto two columns of different polarity. This procedure provides an increased resolution and specificity resulting in an increased number of compounds which were detectable within a single gas chromatographic run and can be used for confirmation and identification purposes. According to Beaumier and Leavitt [10], it is unlikely that a specific interference would coelute with the analyte

Table 6
Recovery of the desorption procedure

Compound	Recovery (%)±standard deviation	
	Column 1 ($n=3$)	Column 2 ($n=3$)
Benzene	–	102±3
2-Butanone	101±2	100±2
n-Butyl acetate	101±5	104±5
n-Decane	–	109±4
Ethyl acetate	98±2	100±1
Ethylbenzene	106±5	106±5
2-Ethyltoluene	–	104±5
3-Ethyltoluene	106±5	106±5
4-Ethyltoluene	107±4	106±5
n-Heptane	108±3	107±3
n-Hexane	105±1	–
Naphthalene	42±9	43±9
n-Nonane	109±4	109±5
n-Octane	108±4	108±5
n-Propylbenzene	107±10	–
Pyridine	50±12	–
Tetrachloroethene	102±2	102±3
Tetrachloromethane	102±1	–
Toluene	104±5	104±5
1,1,1-Trichloroethane	108±3	–
Trichloroethene	100±2	99±2
Trichloromethane	95±1	93±2
n-Tridecane	107±4	107±4
1,2,4-Trimethylbenzene	–	103±4
1,3,5-Trimethylbenzene	105±4	105±4
n-Undecane	108±4	103±4
m,p-Xylene	105±5	105±5
o-Xylene	102±5	–

on both columns. It is thus generally accepted as sufficient for identification when an analyte and a standard substance have identical retention times on two columns of different polarity.

The tandem configuration of ECD and FID offers several additional advantages: since all the analytes pass through both detectors, sensitivity is better than a parallel or split configuration, in which the column effluent is divided in a definite ratio between the two detectors, and thus only a part of the effluent reaches each detector [11].

In real samples the within-series precision of the dual-column/four-detector configuration ranged between 3.0 and 20.2% for the individual VOCs, while day-to-day precision was between 7.2 and 35.2% (Table 5). The variation between 10 monitors ranged between 1.3 and 22.4% (Table 5) and was thus in the same order as the within-series precision of the gas chromatographic procedure. Compared to the within-series and day-to-day precision of the one-column-dual-detector system used in a previous work [1] ranging between 7.1–10.9% and 9.3–18.4%, respectively, the precision of the dual-column system is slightly worse.

Desorption efficiencies for each VOC obtained by the phase equilibrium method according to Rodriguez et al. [9] are summarized in Table 6. Except for pyridine and naphthalene, desorption rates were around 100% with standard deviations between 1 and 10%. According to Rodriguez et al. [9], this procedure is applicable for less polar to non-polar VOCs. They compared desorption efficiencies obtained by the phase equilibrium method and the vapor-state spike and found a close agreement between the two methods, except for isobutanol and chloroethane.

The results of the field study carried out in Wuppertal and Borken are summarized in Table 7 a–c giving for each aromatic (a), aliphatic (b) and halogenated (c) hydrocarbon the geometric mean, geometric standard deviation, range, 95th percentile and the number of samples below the detection limit. With the exception of the halocarbons, outdoor air concentrations were generally higher in the industrialized study areas (Wuppertal-Oberbarmen, Wuppertal-Elberfeld) than in the rural study area (Borken). This is an indication for the assumption that traffic is the main source of exposure. Regarding BTX and the halocarbons this study confirms the results of a previous study [1] carried out in 1991 an urban (Essen) and a rural area (Borken) of Germany. Besides this, outdoor VOC concentrations obtained by passive sampling are to our knowledge only available from Porstmann et al. [4], who determined outdoor benzene and toluene concentrations in the context of an epidemiological study in Duisburg, Germany. Their study design was somewhat different, because they placed the monitors outside at the window of the child's bedroom. The benzene and toluene levels found by Porstmann et al. [4] were similar to those which we obtained in Borken, but lower than those obtained from Wuppertal-Oberbarmen and Wuppertal-Elberfeld. This may be a result of the lower traffic density in the study area in Duisburg.

Table 7
Outdoor VOC levels in the three study areas ($\mu g/m^3$)-geometric means (GM), standard deviations (GSD), ranges, 95th percentiles (P95) and number of samples below the detection limits ($n<DL$)

Substance	Area	GM	GSD	Range	P95	$n<DL$
(a)						
Tetrachloroethene	Wuppertal-	0.7	1.3	0.5–1.2	1.0	-
Tetrachloromethane	Elberfeld	0.6	1.1	0.5–0.8	0.7	-
1,1,1-Trichloroethane		0.9	1.1	0.7–1.2	1.1	-
Trichloroethene		0.5	1.3	0.4–0.8	0.7	-
Trichloromethane		0.08	1.9	0.03–1.1	0.1	-
Tetrachloroethene	Wuppertal-	0.7	1.5	0.4–1.8	1.5	-
Tetrachloromethane	Oberbarmen	0.7	1.1	0.5–0.9	0.8	-
1,1,1-Trichloroethane		0.9	1.2	0.7–1.2	1.1	-
Trichloroethene		0.6	1.7	0.3–1.9	1.4	-
Trichloromethane		0.06	1.6	0.04–0.4	0.08	-
Tetrachloroethene	Borken	0.5	2.9	0.2–4.8	4.2	-
Tetrachloromethane		0.7	1.1	0.5–0.8	0.8	-
1,1,1-Trichloroethane		0.9	1.2	0.6–1.2	1.1	-
Trichloroethene		0.2	1.8	0.07–0.4	0.3	-
Trichloromethane		0.07	1.9	0.04–1.1	0.08	-
(b)						
Benzene	Wuppertal-	6.4	1.6	3.0–16.8	14.5	-
Ethylbenzene	Elberfeld	5.4	1.7	2.1–13.4	12.3	-
2-Ethyltoluene		1.3	1.7	0.5–3.6	2.9	-
3-Ethyltoluene		3.4	1.7	1.3–9.5	7.9	-
4-Ethyltoluene		1.6	1.7	0.6–4.6	3.7	-
Naphthalene		0.5	1.8	<0.2–1.5	1.2	3
n-Propylbenzene		0.5	1.7	0.2–1.4	1.1	-
Pyridine		0.3	1.7	<0.2–1.0	0.9	4
Toluene		17.1	1.7	8.0–50.2	22.1	-
1,2,4-Trimethylbenzene		4.6	1.7	1.0–13.2	11.3	-
1,3,5-Trimethylbenzene		1.3	1.8	0.5–3.8	3.2	-
m,p-Xylene		11.0	1.8	3.9–30.1	25.1	-
o-Xylene		3.5	1.7	1.4–9.2	8.1	-
Benzene	Wuppertal-	5.0	1.5	2.7–9.7	8.7	-
Ethylbenzene	Oberbarmen	3.5	1.5	1.8–7.9	6.3	-
2-Ethyltoluene		0.9	1.5	0.5–1.8	1.7	-
3-Ethyltoluene		2.3	1.5	1.1–4.7	4.5	-
4-Ethyltoluene		1.1	1.5	0.5–2.2	2.1	-
Naphthalene		0.4	1.6	<0.2–0.9	0.6	3
n-Propylbenzene		0.4	1.5	0.2–0.7	0.7	-
Pyridine		0.3	1.6	<0.2–0.6	0.6	4
Toluene		12.8	1.5	6.6–24.4	22.1	-
1,2,4-Trimethylbenzene		3.3	1.5	1.8–6.6	6.1	-
1,3,5-Trimethylbenzene		0.9	1.5	<0.5–1.8	0.6	10
m,p-Xylene		7.1	1.5	3.7–19.3	12.4	-
o-Xylene		2.4	1.5	1.3–4.7	4.4	-
Benzene	Borken	2.5	1.6	0.9–4.3	4.3	-
Ethylbenzene		1.5	1.6	0.6–3.9	2.7	-
2-Ethyltoluene		0.3	1.5	<0.2–0.6	0.6	8
3-Ethyltoluene		0.9	1.7	<0.3–1.8	1.6	2
4-Ethyltoluene		0.5	1.5	<0.3–1.0	0.8	12
Naphthalene		0.2	1.4	<0.2–0.4	0.4	15
n-Propylbenzene		0.2	1.6	<0.1–0.4	0.3	8

Table 7 (Contd.)

Substance	Area	GM	GSD	Range	P95	n<DL
Pyridine		0.2	1.2	<0.2–0.3	0.2	22
Toluene		5.0	1.6	2.0–9.9	8.8	-
1,2,4-Trimethylbenzene		1.2	1.6	0.4–2.2	2.1	-
1,3,5-Trimethylbenzene		0.3	1.6	<0.2–0.7	0.6	10
m,p-Xylene		2.9	1.7	1.0–11.1	5.4	-
o-Xylene		1.0	1.6	0.4–1.9	1.8	-
(c)						
n-Butanone	Wuppertal-	1.2	1.4	<0.8–2.8	1.9	1
n-Butyl acetate	Elberfeld	1.8	2.6	0.6–20.5	9.6	-
n-Decane		0.6	1.4	0.3–1.0	1.0	-
n-Ethyl acetate		1.5	1.5	0.9–4.2	2.7	-
n-Heptane		1.6	1.5	1.0–3.9	3.7	-
n-Hexane		4.6	1.5	2.2–9.3	7.8	-
n-Nonane		0.4	1.5	0.2–0.8	0.7	-
n-Octane		0.5	1.7	0.2–1.5	1.2	-
n-Tridecane		0.2	1.5	<0.2–0.4	0.4	6
n-Undecane		0.3	1.7	<0.2–0.7	0.5	6
n-Butanone	Wuppertal-	1.4	1.5	<0.8–3.1	2.5	1
n-Butyl acetate	Oberbarmen	1.3	1.7	0.4–3.2	2.7	1
n-Decane		0.5	1.4	0.2–0.8	0.8	-
n-Ethyl acetate		1.6	1.8	0.6–5.9	4.2	-
n-Heptane		1.7	1.4	1.0–3.2	2.6	-
n-Hexane		5.0	1.8	1.9–28.2	15.6	-
n-Nonane		0.3	1.4	0.2–0.5	0.5	-
n-Octane		0.5	1.6	0.2–0.9	0.9	-
n-Tridecane		<0.2	1.4	<0.2–0.3	0.2	14
n-Undecane		<0.2	1.4	<0.2–0.3	0.3	18
n-Butanone	Borken	0.8	1.6	<0.8–1.6	1.5	12
n-Butyl acetate		0.4	1.6	0.2–1.0	0.8	-
n-Decane		0.2	1.5	<0.2–0.61	0.4	6
n-Ethyl acetate		0.5	1.3	0.4–0.9	0.8	-
n-Heptane		0.5	1.6	0.2–0.9	0.9	-
n-Hexane		<1.3	1.5	<1.3–2.7	2.1	12
n-Nonane		0.2	1.6	<0.1–0.3	0.3	7
n-Octane		0.2	1.5	<0.1–0.4	0.3	4
n-Tridecane		<0.2	1.2	<0.2–0.2	0.2	22
n-Undecane		<0.2	1.2	<0.2–0.3	<0.2	24

4. Conclusions

The dual-column tandem ECD–FID configuration described here is valuable for routine analysis, particularly in trace and ultratrace analysis. It is a simple and cost-effective way for screening analysis and provides an appropriate procedure for the qualitative identification and reliable quantitative determination of trace amounts of substances in complex matrices. Within one analytical run optimal resolution and detection power including validation of the results can be obtained.

At the same time sample throughput is enhanced drastically since several items of information are obtained simultaneously.

The GC configuration used here is easily transferable to other applications. Other multi-detector combinations, such as the photoionization detection (PID)–Hall tandem detection described by Butler [12], also seem to be promising. The connection of non-destructive detection methods like ECD or PID in series with the nitrogen and phosphorous selective NPD as described by Gagliardi and Verga [11] seem to be advantageous for pesticide screening analysis.

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