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Indoor Air 1993, 3: 283–290
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Indoor Air
ISSN 0905-6947

Characterization of Office Dust by VOCs and TVOC Release - Identification of Potential Irritant VOCs by Partial Least Squares Analysis

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Abstract

Floor dust from nine city hall office buildings was separated into fiber and particulate fractions and analyzed for volatile organic compounds (VOCs) and total VOC (TVOC) by thermal desorption/high resolution gas chromatography (HRGC). Components were identified by HRGC/mass spectroscopy (MS). Principal component analysis was applied to VOC emission profiles revealing similarities between buildings and correlations between profiles and SBS symptoms of mucous membrane irritation and "concentration difficulty". While the dominant pattern in emission profiles was not correlated with SBS irritation complaints, partial least squares analysis in latent variables (PLS analysis) identified VOCs for which peak areas were correlated with SBS irritation complaints and the CNS complaint, "concentration difficulty".

KEY WORDS:
Dust, Principal component analysis, Partial least squares analysis, Volatile organic compounds (VOCs), Total VOC (TVOC) release, Thermal desorption

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Introduction

Several field studies indicate that particle concentration could be associated with increased sick building syndrome prevalence in the form of mucous irritation and odour annoyance (Wallace et al., 1991). It is also known that domestic dust contains hundreds of volatile and semi-volatile organic compounds (VOCs and SVOCs, respectively) which can be released (Wolkoff and Wilkins, 1993). In addition, marcromolecular organic dust, as described in the Danish town hall study (Skov et al., 1990), and fleecy surfaces are associated with increased SBS prevalence.

Most field studies have failed to correlate concentrations of VOCs, or total VOC (TVOC), with symptom prevalence. One study, however, in which symptom reports and VOC measurements were simultaneous, showed a strong correlation with SBS prevalence (Hodgson et al., 1991). Other studies have tried to correlate selected VOC GC peak areas and number of VOCs to distinguish between sick and healthy buildings by use of multivariate analysis techniques (Sundell et al., 1993; Baird et al., 1987; Noma et al., 1988).

In a multidisciplinary study, dust was collected from 12 city halls in the Copenhagen metropolitan area (Gyntelberg et al., 1993). In this study several physical, chemical, and biological factors of nine dust samples were measured and compared with symptom prevalence among 870 persons, the data being obtained by a questionnaire (Gyntelberg et al., 1993).

The purpose of this work was to identify and measure VOCs and TVOC desorbed from office dust and correlate common patterns in the dust VOC emission profiles with symptom prevalence. In another paper the VOCs from household dust have been characterized (Wolkoff and Wilkins, 1993).

Methods

Sampling

Floor dust, collected by a specially designed vacuum cleaner (HVS-3, Cascade Stack Sampling Systems, Oregon) (Roberts et al., 1991), was analyzed from nine city hall buildings (Gyntelberg et al., 1993). The questionnaires were distributed to workers in the vacuum cleaned locations that were selected as being representative of the buildings. The collected dust was divided into particle and fiber fractions with a DIN 6 sieve (1.0 mm). The fraction which did not pass through the sieve was called fiber. Samples were stored in vials at ambient temperature.

Analysis of VOCs and TVOC

The thermal desorption/HRGC and HRGC/MS analyses of VOCs and TVOC were performed as described previously (Wolkoff and Wilkins, 1993). SVOCs with retention times eluting after nicotine and nonanoic acid were not included in the TVOC determination due to systematic memory effects. For multivariate analysis, peak areas of substances eluting before and including dodecanoic acid were utilized. Areas of peaks smaller than 250 area units (ca. 160 ng/g n-decane) were estimated using peak height to a minimum of 100 area units.

Principal Component Analysis (PCA) and Partial Least Square Analysis (PLS)

Principal component analysis is a mathematical analysis method based on multiple linear correlation concepts which is used to find dominant patterns in data sets. The linear models which most effectively reduce group variance are chosen to represent the data. There are usually serveral patterns in complex data sets. The patterns of interest are both relationships between objects and those between variables. SIMCA (a type of PCA) is an acronym for soft independent modeling of class analogy in which the number of statistically significant components is determined by cross validation. PLS refers to partial least squares in latent variables in which PCA type projections for independent and dependent variable data of objects are fitted to each other to allow predictions from independent variable sets of new objects (Wold et al., 1984, 1985).

The SIMCA 3B program was supplied by Sepanova AB, Ostrandsvagen 14, S-112 43 Enskede, Sweden. The data were normalized to eleminate the dominance of large chromatographic peaks. Missing peaks were given the arbitrary value 10.

Tables 3 and 4 list the VOCs and SVOCs identified in the PLS analysis having modeling power (degree of contribution to the model) greater than 0.300 and 0.350, respectively. Data for mucous membrane irritation symptoms and "concentration difficulty" were taken from the joint report (Gyntelberg et al., 1993).

Results and Discussion

Using GC/MS, 188 VOC and SVOC were identified from thermal desorption of office dust at 120 °C. Many of the compounds were also identified in desorption experiments at lower temperatures and in the headspace analysis of household dust (Wolkoff and Wilkins, 1993). A list of the compound classes is given in Table 1 and the identity and occurrence of the identified substances is shown on Table 2. Saturated aldehydes (C_{4-11}), carboxylic acids (C_{2-14}), saturated hydrocarbons (C_{6-21}) and phthalate esters were dominant peaks in GC/FID analyses. Nicotine, 2-pentylfuran and 2-methylpyrrole were also dominant dust volatiles in some buildings.

The aldehydes and carboxylic acids can be formed by microbiological degradation of lipids although the branched aldehydes (2-methylpropanal and 3-methylbutanal) can also result from microbiological de novo synthesis (Rivers et al., 1992; McJilton et al., 1990). Autooxidation of fatty acids or the corresponding alcohols can produce most of the aldehydes, carboxylic acids, and 2-pentylfuran, which has also been identified as a fungal metabolite (Wilkins and Scholl, 1989). Sidestream cigarette smoke has been shown to contain, among many other compounds, 2-methylpyrrole, pyrrolidine and 2-furanmethanol (Bayer and Black, 1987), which were identified in this investigation. Nicotyrine and

Table 1 Number of desorbed (S)VOCs from office dust (120 °C)

Group	VOCs	Group	VOCs
saturated hydrocarbons	16	carboxylic acids	13
aromatic hydrocarbons	23	esters	12
unsaturated hydrocarbons	4	phthalates	8
chloroorganics	5	furans	4
miscellaneous aromatics	9	γ-lactones	4
miscellaneous heterocycles	7	amides	4
alcohols	15	mono-terpenes	7
phenols	11	sesquiterpenes	6
saturated aldehydes	11	siloxanes	5
2-alkanones	13	miscellaneous	6
unsaturated ketones	4		

Table 2 Compounds from thermal desorption of City Hall dust identified by GC/MS

	A	B	C	D	E	F	G	H	I
acetone ■	+	+	+	+	+	+	+		+
hexane ■		+	+	+		+	+	+	
t-butanol									+
2-methylpropanal ■	+	+	+	+	+	+		+	+
butanal ■	+	+	+	+		+		+	+
2-butanone ■	+	+	+	+		+	+	+	+
2-methylfuran ■	+		+	+	+	+	+	+	+
3-buten-2-one ■	+	+	+	+	+	+	+	+	+
heptane	+	+	+	+		+		+	+
benzene	+	+	+	+		+	+	+	
1,2-dichloroethane					+				
3-methylbutanal ■	+	+	+	+	+	+			+
2,4-dimethylfuran	+								+
methyl methacrylate				+	+			+	+
2-methyl-1-propanol ■	+	+		+	+	+		+	+
1-butanol ■					+		+	+	+
pentanal ■	+	+	+	+		+			
2,3-pentanedione								+	
acetic acid ■	*	+	+	+	+	+	+	+	+
pyrrolidine						+			+
octane ■	+	+	+	+	+		+	+	+
toluene ■						+	+		
hydroxyacetone							+		
4-methyl-2-pentanone			+						
perchloroethylene		+	+		+	+	+	+	+
ethyl methacrylate				+					+
hexanal ■	*	*	#	*	+	#	+	+	+
nonane ■	+	+	+	+			+		+
1-nonene						+			
propionic acid	+	+	+		+	*	+	+	+
pyrrole				+			+	+	+
ethylene glycol							+	+	+
p-xylene	+	+							
5-methyl-3-methylene-5-hexen-2-one? ■	+	+	+	+	+		+	+	+
α-pinene								+	
1,2-propandiol ■	#	+	+	+	+	+	+	+	+
dimethylformamide ■	*	+	+	+	+	+	+		+
C_6H_9N?	+							+	
styrene ■	+			+	*		+		+
MW 142 (2-alkanone) ■	+	+	+	+	+	+		+	+
4-hydroxy-4-methyl-2-pentanone	+								
5-methyl-2-hexanone		+							
1-pentyl acetate?				+					
heptanal ■	+	+	+	+	+	+	+	+	+
butanoic acid ■	+	+	+	+	+	+	+	+	+
4-methylanisole?									+
2-(1-butoxy)ethanol								+	+
1-butyl-2-chloroethylether									+
2-methylpyrrole	#	+	+	#	#	+	+	+	+
octamethylcyclotetrasiloxane	+	+	+						
sabinene	+								
cyclohexanone						+			
ethyltoluene						+			
2-furanmethanol								+	
trimethylbenzene (3 isomers)		+				+		+	
Δ3-caran ■	+								
methyl-2-heptanone			+	+				+	+
2-pentylfuran ■	+	+	+	+	+	+	*	#	+
1-butylmethacrylate		+					+		

Table 2 Cont

	A	B	C	D	E	F	G	H	I
N,N-dimethylacetamide ■	+	+	+			+	+	+	+
limonene	+								
2-heptenal		+							
octenone (isomer)		+							+
pentanoic acid ■	+	+	+	+		+	+		
6-methyl-5-hepten-2-one?				+		+			
benzaldehyde ■	+	+	+	+	+	+	+	+	+
2-ocetanone								+	
octanal ■	+	+	+	+	+	+	+	+	+
N,N-diethylformamide					+				
1-chlorooctane					+	+	+	+	+
dihydro-2(3H)-furanone								+	
2-ethylhexanol ■		+	+	+	+	+	+	+	+
indene					+				
decamethylcyclopentasiloxane	+	+	+						
5-ethyldihydro-2(3H)-furanone								+	+
hexanoic acid ■	#	#	#	*	*	#	*	*	*
1-octanol			+				+	+	
dichlorotoluene (2 isomers)						+			
C_{11} alkene				+					+
nonanal ■	+	*	*	+		*	+	*	+
2-(2-hydroxyethoxy)ethanol	+	+			+				
acetophenone ■	+	+		+	*	+	+	+	+
benzyl alcohol								+	+
phenol ■	+	+		+	+	+	+	+	+
1,1,3trimethylindene					+				
2,6-heptadione					+				
trichlorobenzene (3 isomers) ■ - 25.2 and 27.1 min	+	*	+	+	+	+	+	+	+
camphor			+						
heptanoic acid ■	+	*	*	+	*	+	+	+	+
3-isopropyl-α-methylstyrene					+				
phenylcyclopentane					*				
glycerol								+	
dodecamethylcyclohexasiloxane	*	*	*						
2-ethylhexanoic acid ■									+
decanal ■				+				+	+
menthol	+							+	
benzothiazole ■	+	*	*	*	*			+	+
2-(2-butoxyethoxy)ethanol ■					*	+	+	+	+
2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one								*	
1-hydroxynaphthalene ■					+				+
monoterpene acetate MW 196	+								
octanoic acid ■	+	#	#	#	#	#	#	#	*
tetradecene(isomer)	+								
1-decanol		+				+	+	+	
methyl-2-hydroxy-3-methyl-benzoate								+	+
2-decenal									+
isopropylphenol	+								
tetradecane						+			+

Table 2 Cont

	A	B	C	D	E	F	G	H	I
1-methylnaphthalene ■						+			+
2-undecanone ■						+			
benzoic acid	+	*			+	+			
2-phenoxyethanol ■					*	+			+
sesquiterpene $C_{15}H_{24}$							+		
2-methylnaphthalene ■									+
nonanoic acid ■	#	#	#	#	#	#	#	#	#
nicotine ■	#	#	#	#	#	*	#	#	#
widdrene $C_{15}H_{24}$?	#								
2-(2-butoxyethoxy)ethyl acetate							+		+
tetradecamethylcycloheptasiloxane		+	+						
biphenyl		+			+	+		+	+
sesquiterpenes $C_{15}H_{24}$ - 3 compounds	+								
caprolactam ■	+	+	+	+	+	+	+	+	+
Texanol (2,2,4-trimethyl-1,3-dihydroxypentane,isobutyrate), 2 isomers		+	+		+			+	+
pentadecane	+	+				+	+		+
2-(1,1-dimethylethyl)-phenol		+			+	+			
1-chlorodecane					+	+			+
BHT 2,6-bis-(1,1-dimethylethyl)-4-methylphenol		+			+		+		
cuparene ■	+								
hexadecane	+	+	+		+	+	+		+
decanoic acid ■	+	+	+	+	+	+	+	+	+
3-methyl-4-chlorophenol									+
6,10-dimethyl-5,9-undecadien-2-one								+	
dimethyl phthalate ■			+	+	+	+		+	+
2-decyloxyethanol		+							
BHA ■	+	+	+	+	+			+	+
hexadecene (isomer)							+	+	
5-octyldihydro-2(3H)furanone		+							
β-nicotyrine								+	
1,4-diacetylbenzene									+
$C_{19}H_{40}$ branched isomer	+				+				
heptadecane ■	+	+	+	+	+	+	+	+	+
TXIB (2,2,4-trimethyl-1,3-pentandiol,diisobutyl ester) ■						+		+	+
2-hydroxybiphenyl		+	+	+	+	+	+		+
dodecanoic acid	+	+	+	+	+	+	+	+	+
2,4-diisopropylphenol	+								+
C_{12} alkylbenzene (3 isomers)	+			+					+
diethyl phthalate	+	+	+	+	+	+	+	+	+
octadecane	+		+	+	+	+	+	+	+
methoxybenzophenone (isomer)	+				+				
1,1,3-trimethyl-3-phenyl-2,3-dihydro-1H-indene						+		+	+
tributyl phosphate		+	+						
$C_{20}H_{42}$ (isomer)	+			+	+		+	+	
dibutyl adipate						+	+		+
isopropyl tetradecanoate	+	+	+	+	+		+		+
octylphenol (isomer)							+		
nonadecane	+		+	+	+	+	+	+	+
$C_{14}H_{10}$ anthracene or phenanthrene							+		+
4-nonylphenol	+	+							
tetradecanoic acid	+	+	+		+				
13-methyl-17-norkaur-15-ene	+								
eicosan	+	+	+	+	+	+	+	+	+
terphenyl?							+		
tris(2-chloroethyl)phosphate					+		+		
diisobutyl phthalate	+	+	+		+		+	+	+
2,6-di-t-butyl-2,5-cycohexadien-1,4-dione	+								
butylpentylphthalate (isomer)		+							
4-phenylbicyclohexyl	+				+		+		
heneicosane	+	+	+		+	+	+	+	+
butylisobutylphthalate		+			+		+	+	
dibutyl phthalate	+	+	+			+			+
dipentyl phthalate (2 isomers)					+				
manool			+						
docosane	+		+			+	+	+	+
fluoranthene							+		
tricosane						+			

* ≥5 µg/g, # ≥10µg/g, ■ used in PLS

an isomer of nicotine as well as 2,5-dimethylfuran, which has been found in smoker's breath, (Gordon, 1990), were also observed. Many of the compounds identified are problably derived from polymers and their additives (methyl, ethyl and butyl methacrylates, styrene, 2-(1-butoxy)-, 2-(2-butoxyethoxy)- and 2-phenoxyethanol, 1,1,3-trimethylindene, 3-isopropyl-α-methylstyrene, benzothiazole, 2-(2-butoxyethoxy)-ethyl acetate, caprolactam, Texanol and Texanol isobutyrate, BHT, BHA, 1,1,3-trimethyl-3-phenyl-2,3-dihydro-1H-indene, tributyl phosphate, dibutyl adipate, 2-hydroxybiphenyl, 2,6-di-t-butyl-2,5-cyclohexadiene-1,4-dione, methoxybenzophenone, isopropyl tetradecanoate and the phthalate esters). The sesquiterpene hydrocarbons may be derived from cleaning products or be emitted by wood products. The γ-lactones may either be of microbiological origin (Labows et al,. 1979) or may be formed by non-microbial fatty acid oxidation. Siloxanes are constituents in carpet anti-dirt products and personal care products (Shields and Fleischer, 1993). 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one is a well known Maillard reaction product, which has been found in tobacco smoke (Yeo and Shibamoto, 1991; Ishiguro et al,. 1976).

TVOC measurements for the nine city hall office buildings, estimated from FID response, are listed in Table 3 with an average mucous membrane irritation index obtained from the questionnaire (Gyntelberg et al,. 1993). Additionally, Table 3 lists the number of VOCs/SVOCs in fibers released in concentrations greater than 5μg per gram dust. There is no relationship between fiber or particle TVOC and irritation symptoms. However, it was found that TVOC released from the fibers and the number of VOCs therein greater than 5 μg/gram dust were correlated with the CNS effects "concentration difficulty" and "heavy head", 0.90 and 0.67 respectively (Gyntelberg et al., 1993). There was no apparent correlation between dust adsorbed VOCs ($\mu g/m^2$) [dust weight (g) × VOC concentration (μg/g) area cleaned (m^2)] and SBS symptoms.

The FID response data of 71 GC peaks, selected on the basis of their appearance in five or more samples, were utilized for principal component analysis (see Table 2). Of the 71 peaks, 14 represented unidentified compounds. The dominant VOC patterns represented in the PC dimensions did not correspond to the irritation indices from the nine buildings, however. PLS analysis of the GC data combined with percent complaints for six mucous membrane irritation types allowed 83% of the variance in GC data to be explained by two dimensions while 80% of the variance in the complaint data was explained. The compounds with the highest modeling power are listed in Table 4. While several of these compounds are probably irritating or odorous, they may not be the causative agents of SBS, but may be reaction products derived from the causative agents. They may thus give clues about the primary processes which generate irritants (the action of O_3, singlet oxygen, or microbiological growth). Most of the compounds with high modeling power could arise either from (bio)-degradation of fatty acids or microbiological de novo synthesis (Wolkoff and Wilkens, 1993). Methyl ketones are known fatty acid degradation products by both bacteria and fungi (Cailleux et al., 1992; Kindelerer, 1987; Zechman and Labows, 1985; Lee et al., 1979). The unsaturated compound, 5-methyl-3-methylen-5-hexen-2-one could be an α- or β-pinene oxidative degradation product similar to the previously identified 6-methyl-2-heptene-2-one (Ciccioli et al., 1992). It is also reasonable that oxidative processes are dominant in the indoor atmosphere (Sundell et al., 1993) since only low concentrations of isoprene have been measured indoors (Cailleux et al., 1993), while it has been shown to be one of the major constituents of human VOCs (Ellin et al., 1974). Possible oxidation products, α-methacrolein and methylvinyl ketone have been identified in hair headspace (Dmitriev et al., 1985), while methylvinyl ketone was found in domestic dust by the same group (Dmitriev et al., 1987) as well as in all of our dust samples. It is noteworthy that both TVOC released from fibers and the number of VOCs therein correlated (Spearman rank correlation) with the macromolecular organic dust (MOD) and fungi (colony forming units) (Gyntelberg et al., 1993).

PLS analysis using the prevalence of the CNS complaints, "concentration difficulty", allowed for explanation of 66% of the GC peak area variance with two dimensions. The VOCs with high modeling power are shown in Table 5. Here carboxylic acids and aldehydes are also dominant. These compounds can be formed by the same oxidation processes mentioned above.

It is important to establish the sources and processes responsible for the formation of the compounds with high modeling power. Although the relationships between the compounds with high

Table 3 TVOC desorbed from particle fractions (μg/gram dust calculated as decane equivalents), standard error in parentheses, average irritation index (%) Gyntelberg et al. (1993), and number of VOCs ≥ 5 μg/gram fiber fraction (up to nonanoic acid)

Town hall	Fiber fraction			Particle fraction
	TVOC (std.error)	No. of VOCs	Average irritation index	TVOC (std.error)
A	206 (1)	18	39.0	105 (8)
B	210 (12)	16	14.6	147 (7)
C	230 (12)	16	34.4	141 (18)
D	126 (8)	11	13.9	91 (9)
E	238 (8)	20	17.8	260 (6)
F	121 (3)	9	14.6	51 (18)
G	119 (9)	7	6.8	239 (10)
H	152 (12)	11	17.7	157 (10)
I	116 (11)	8	13.4	139 (7)

Table 4 Results of partial least square analysis of VOC desorption data from fiber fraction. Dependent variable is "average mucous irritation". VOCs with modeling power > 0.30

Modeling power	VOCs	Modeling power	VOCs
0.640	2-methylpropanal	0.412	octane
0.586	hexanoic acid	0.375	pentanoic acid
0.568	2-alkanone $M^+ =$ 142	0.362	heptanoic acid
0.498	3-methylbutanal	0.332	2-undecanon
0.420	35.8 min not identified	0.305	5-methyl-3-methylene-5-hexene-2-on $M^+ = 124$

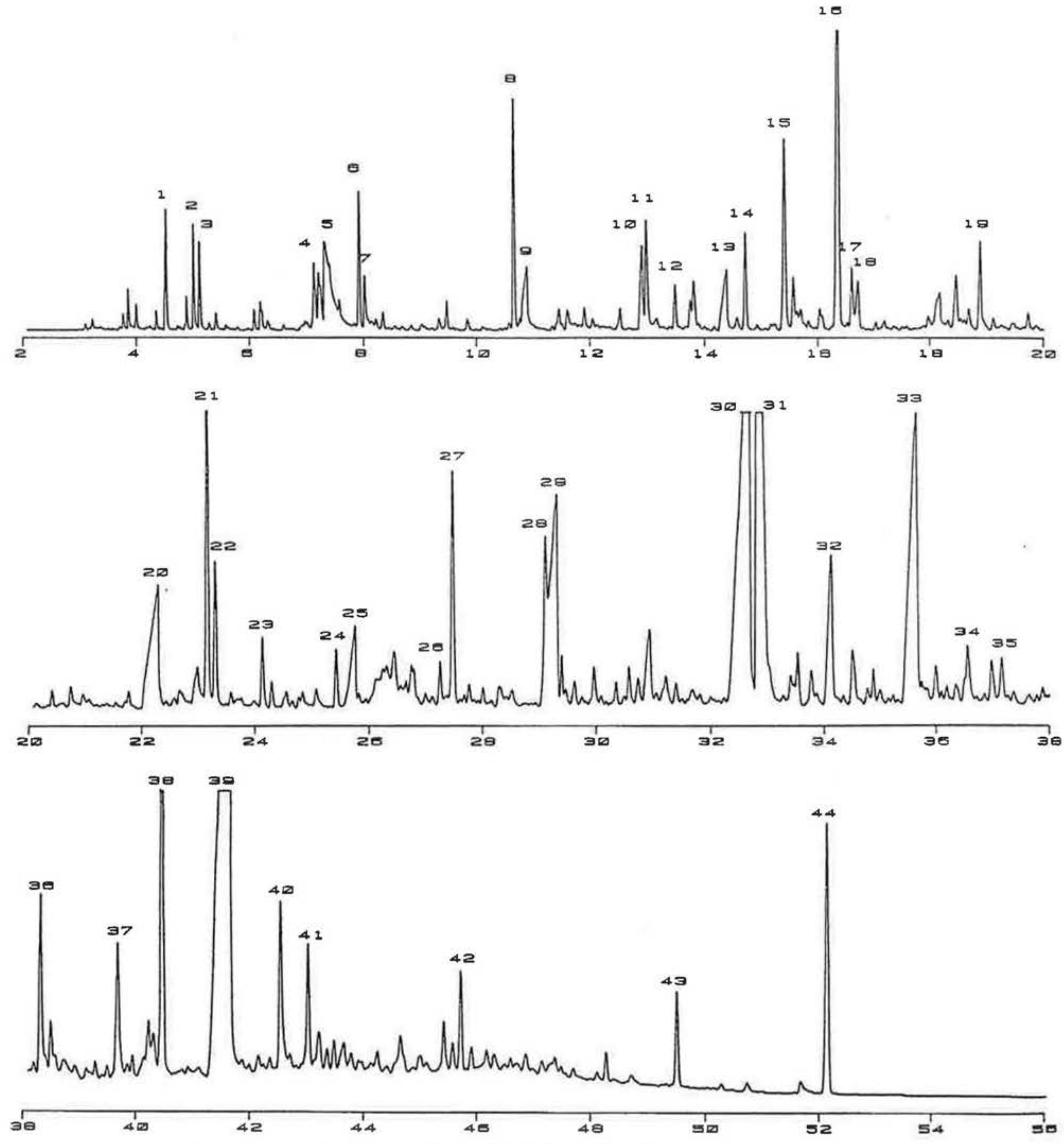

Fig. 1 FID response of GC separation of VOCs desorbed at 120 °C from office (H) dust

1. 2-methylfuran
2. 3-buten-2-one
3. heptane
4. butanol
5. acetic acid
6. hydroxyacetone
7. perchloroethylene
8. hexanal
9. propionic acid
10. propandiol
11. N,N-dimethylformamide
12. 2-alkanone (MW 142)
13. butanoic acid
14. heptanal
15. 2-methylpyrrole
16. methyl-2-heptanone
17. 2-pentylfuran
18. N,N-dimethylacetamide
19. octanal
20. hexanoic acid
21. nonanal
22. acetophenone
23. phenol
24. trichlorobenzene
25. heptanoic acid
26. 2(2-butoxyethoxy)ethanol
27. 2,2-dihydro-3,5-dihydroxy-6-methyl-4-H-pyran-4-one
28. benzothiazole
29. octanoic acid
30. nonanoic acid
31. nicotine
32. caprolactam
33. decanoic acid
34. BHT
35. BHA
36. dimethyl phthalate
37. hexadecane
38. 2-hydroxybiphenyl
39. dodecanoic acid
40. diethyl phthalate
41. octadecane
42. nonadecane
43. eicosane
44. diisobutyl phthalate

Table 5 Results of partial least square analysis of VOC desorption data from fiber fraction. Dependent variable is "concentration difficulty". VOCs with modeling power >0.35

Modeling power	VOCs	Modeling power	VOCs
0.562	pentanoic acid	0.422	heptanoic acid
0.558	hexanoic acid	0.418	2-methylbutanal
0.526	hexanal	0.380	butyric acid
0.492	35.8 min not identified	0.336	benzaldehyde

modeling power to irritation and CNS complaints is uncertain, it is possible that some of the correlations found in this study can lead to the identification of causative agents of indoor air complaints. We believe that multivariate analysis will prove to be useful in SBS studies.

Acknowledgement

This work was supported by a grant from the National Agency of Housing and Building. We thank Mr. K. Larsen for GC/MS analysis, Mrs. B. Kvamm for GC assistance and Mrs. S. H. Nielsen for sample fractionation.

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