

Determination of Volatile Organic Compounds in Residential Buildings

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ABSTRACT

Contamination of indoor air in residential buildings by volatile organic compounds (VOCs) is of great concern to public health. Measurement of these contaminants at progressively lower sub-ppb levels is required so that the health impacts of toxic VOCs at very low concentrations can be more completely assessed and understood. A complete GC/MS system with a two-stage gas desorption (TM-ATD) has been used to analyze VOCs in air samples taken from over 150 residential houses or apartments as part of a study aimed at developing baseline data on inhalable indoor pollutants and their contributions to adverse respiratory health effects in infants and children. Using the analytical procedure developed, the VOCs were separated and analyzed in about one hour, with detection limits lower than 0.05 ppb for most target components. Library searches were performed to evaluate the integrity of analyte mass spectra over the range of calibration measurements performed. Measurement results were observed on analytes in air samples at levels down to approximately 0.002 mg/m³. This paper summarizes the analyses of more than 254 air samples, including over 30% duplicates. A representative TIC chromatogram of VOCs for a field sample is illustrated.

INTRODUCTION

Volatile organic compounds (VOCs) are major indoor air contaminants, which easily emanate from a range of sources including building materials, furnishings, and occupant activities. VOCs may also be transported from outdoor air by infiltration or HVAC systems.^[1,2] Exposure to VOCs can potentially lead to a variety of adverse health outcomes such as irritation of the eyes and respiratory tract, and reactions involving the eyes, skin, and lungs. Several VOCs (benzene, 1,1-dichloroethylene, 1,4-dichlorobenzene, chloroform, ethylene dibromide, methylene chloride and carbon tetrachloride) commonly found in indoor air have been associated with an increased risk of cancer.^[3,4]

Indoor air pollutants are often difficult to quantify because they are present in relatively low concentrations and their sources are diverse. Therefore, methods to collect and analyze, air samples for VOCs have been actively studied.^[5] Traditionally, VOCs have been measured by static or dynamic headspace GC/MS and purge-and-trap-GC/MS. The static headspace method is relatively simple and requires little sample preparation, but sensitivity is limited; hence, the method has been restricted to samples with relatively high VOC concentrations. Higher sensitivity for VOC analysis is often achieved by more demanding dynamic headspace, purge-and-trap-GC/MS methods as well as MIMS.^[6]

The aim of this investigation is to measure VOCs in the general environment of residential buildings using a solid adsorbent contained in a stainless steel tube to collect the air sample followed by automatic thermal desorption/gas chromatography/mass spectrometry analysis.

EXPERIMENTAL

The Syracuse AUDIT Study (Assessment of Urban Dwellings for Indoor Toxics) is a U.S. EPA-sponsored 18-month study investigating the relationship between respiratory health in a group of 100 infants at high risk for development of asthma and numerous indoor (home) environmental parameters (including VOCs, particulate matter, carbon monoxide, allergens, etc.). The study dwellings are mostly lower socio-economic homes in inner city Syracuse, with a high prevalence of occupants who smoke: >80% of the homes have one or more smokers indoors at least part of the time. Nearly all the dwellings have natural draft ventilation as the only fresh air source.

Sampling The samples were collected in over 150 residential buildings. The measurements were conducted during all seasons. The VOCs were collected with stainless steel tubes (150 mm × 4 mm i.d., Supelco) packed with multisorbent, which consists of 20/40 mesh Carbopark B (100 mg) and 20/40 mesh Carbopark C (200 mg) or 60/80 mesh Tenax TA (~350 mg) supplied by Supelco, Bellefonte, PA. Tubes were conditioned at 350°C for at least 8 h while passing 30 ml/min of carrier gas (99.999%, helium) through them. Prior to use, the tubes were tested until their cleanness was confirmed by GC/MS output. Each tube was sealed with caps and placed in a tube container. The active samples were collected with sampling pumps (Catalog No. 224-PCXR8, SKC Inc.) for ~24 hrs. Batteries were removed and sampling pumps were adapted to run using line voltage in order to operate over the full 24-hour period. Adjustable, low flow dual tube holders (Catalog No. 224-26-02, SKC, Inc.) were used to collect duplicate samples at flow rates of ~0.007 L/min. Flow rates were measured immediately before and after sampling using an electronic soap film calibrator. The average flow rate was used to calculate sample volume. To prevent significant loss of VOCs, the samples stored in a refrigerator at ~3° C until analysis, which was typically within one week of collection. Over 30% of the samples were duplicates.

Chemicals All chemicals used for calibration and analyses were purchased from Supelco (Bellefonte, PA), Fluka, Aldrich (Milwaukee, WI) and Acros (NJ), and are p.a., HPLC or

mass spec grade, except R-(+)-Limonene (97%, Sigma-Aldrich): Benzene (HPLC grade 99.9%, Aldrich); Toluene (mass spec grade 99.0%, Supelco), Dichloromethane, Cyclohexanone, Ethylbenzene, Tetrachloroethylene, n-Butanol, n-Hexane, 1,4-Dichlorobenzene; Cyclohexane (mass spec grade 99.9%, Supelco), n-Octane (mass spec grade, 99.4%, Supelco); Dodecane (mass spec grade, 99.6%, Supelco), Decane, 1-Octane, n-Undecane, 1,2-Dichlorobenzene, 2-Butanone; 1-Butanol (spec grade 99%+, Acros); Benzaldehyde (p.a. 99%+, Fluka); 2-Butanol (p.a. Acros); o-Xylene (HPLC grade 99%, Sigma-Aldrich), p-Xylene. VOC mixture solutions were purchased from Supelco: EPA 524.2 VOC Mix (Cat. No. 47932), VOC Calibration Standard Kit (Cat. No. 48804) and JMHV VOC Mix (Cat. No. 47938).

Calibration standards Calibration curves were obtained by injecting 4 μ l, 6 μ l and 8 μ l standard solution (methanol as solvent) ranging from 320 ng/ μ l to 2000 ng/ μ l into Tenax tubes following clean air drawn through the tubes. For VOCs different from the above target compounds, concentrations were calculated as toluene equivalents.

Analysis The air samples were desorbed in a TurboMatrix-ATD (Perkin-Elmer Instruments, Norwalk, CT) and analyzed by an AutoSystem XL gas chromatograph equipped with a TurboMass Gold EI detector and accompanying TurboMass v4.5 software and NIST98 Library.

For the ATD, a two-stage desorption was adopted to reduce the component bandwidths and improve the efficiency of the chromatographic separation. For the primary desorption of ATD, the sample tube was heated to 300 °C for 10 min under a stream of He. The desorb flow was adjusted to 45 mL/min to extract VOCs from the sampling tube by thermal desorption and sweep them onto a secondary trap filled with Texane. The second trap was cooled with a Peltier cooler to temperatures -20 °C. The cold trap refocused all the components eluting from the sample tube. When the primary desorption was complete, the cold trap was electrically heated at a rate of 40 °C/s to 300 °C and held for the secondary desorption for about 30 min to elute all the retained components in a vapor band as narrow as possible into the GC column. The valve and transfer-line temperatures were maintained at 175 °C and 200 °C, respectively. The flow rates of the outlet split and inlet split were adjusted to 30 and 40 mL/min, respectively.

An Elite 624 capillary column (30 m \times 0.25 mm ID \times 1.4 μ m film thickness, Perkin-Elmer Instruments, Norwalk, CT) was used for the GC separation of analytes. A carrier gas flow (He, 99.999%, Airgas) was split before entering the column such that a low flow rate was controlled to enter the GC and other was vented. The oven temperature was programmed for the tests as follows: 35 °C for 2 min, a 10 °C/min ramp to 150 °C, and a 8 °C/min ramp to 250 °C and 2 min hold at 250 °C, for total analysis running times of ~28 min.

The performance of the system was verified with the experimental configuration and conditions. The MS was tuned to meet EPA method requirements with PFTBA (Heptacosafuorotributylamine) by using the auto tune program or setting suitable tune parameters. The mass spectrometer was scanned from m/z 45 to 350 at a cycle of 0.8 s.

The ion source temperature was 150 °C, and the electron ionization potential (EI) was 70 eV. The tune factors are outlined in Table 1. Full scan EI data were acquired under these conditions.

Table 1. Instrument Conditions

MS Operating Conditions	
Ionization mode	EI+
Scan range (m/z)	45-350
Scan rate (sec/scan)	0.8
GC line temperature (°C)	200
Electron energy (eV)	70
Source temperature (°C)	150
Ion energy	1.9
Emission current	100 μ A
Multiplier (V)	334

Data process Analyte responses were integrated with automatic quantitation software. The NIST98 spectral library and user library were used for qualitative confirmation. Automatic quantitation identified analyte peaks based on the calibration file reference spectrum and the retention time by setting each of these parameters. For this study, a library fit parameter of 700/1000 was required for a good spectral match and a retention window of ± 12 sec was required for chromatographic integrity.

RESULTS AND DISCUSSION

Thermal desorption The starting ATD conditions used for the optimization of extraction, separation, identification and quantification of VOCs under this investigation were presented in the experimental section. To determine if there was an eventual memory effect of the system for heavy components, 320 ng of each standard was deposited at the end of a tube followed by one empty tube (the tube without adsorbent), which was analyzed following the spiked tube. Analytical results show that the memory effect hardly occurs under the given conditions.

Calibration To examine linearities of the responses versus concentration for the 18 components by the ATD/GC/MS system, duplicate standard samples were analyzed at three amounts over a range from 480 to 2000 ng. As Figure 1 (a result for toluene) exemplifies, a linear regression analysis of the resulting data was calculated for each compound. Good linearity was observed for all compounds without solvent methanol, which did not chromatograph well with the analytical system. Correlation factors ranged from 0.9881 – 0.9995 for the linear regression analysis.

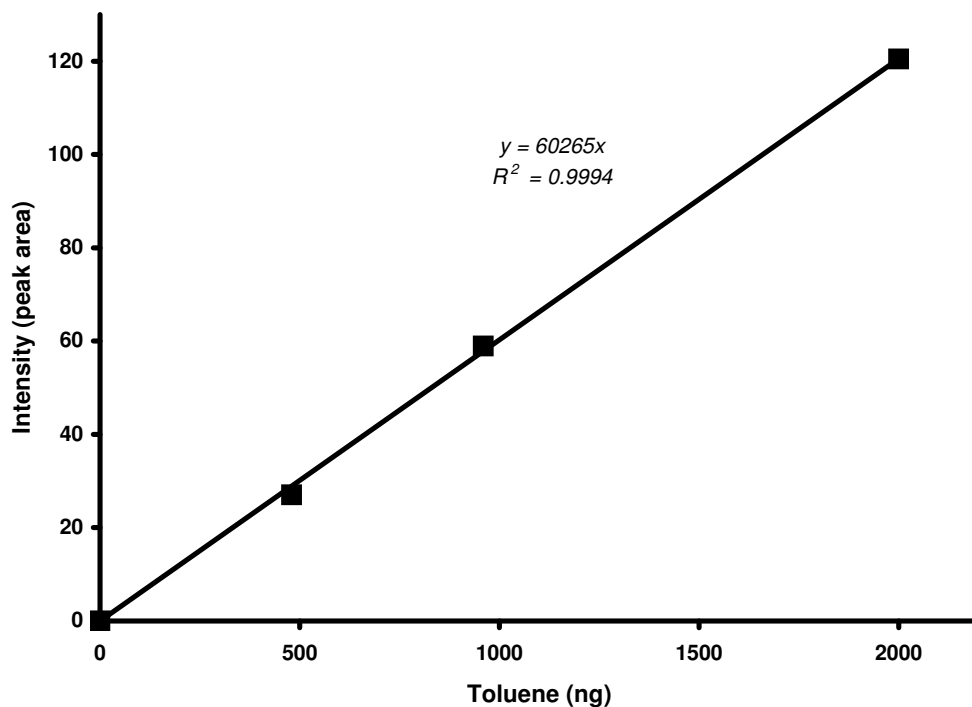


Figure 1. Linearity of Toluene, up to 2000 ng

Application to indoor air samples. The MS system used electron impact ionization full scan as a universal detector at the optimal detection level to identify and estimate the quantities of target and non-target analytes. The reconstructed total ion current gas chromatogram for a representative air sample containing VOCs is presented as one segment in Figure 2.

Tube#B02726, 126B, PUMP#3, 10.0 L, 2/3/03#B02726
au0224-1

24-Feb-2003 + 14:32:18

Scan EI+
TIC
1.59e9

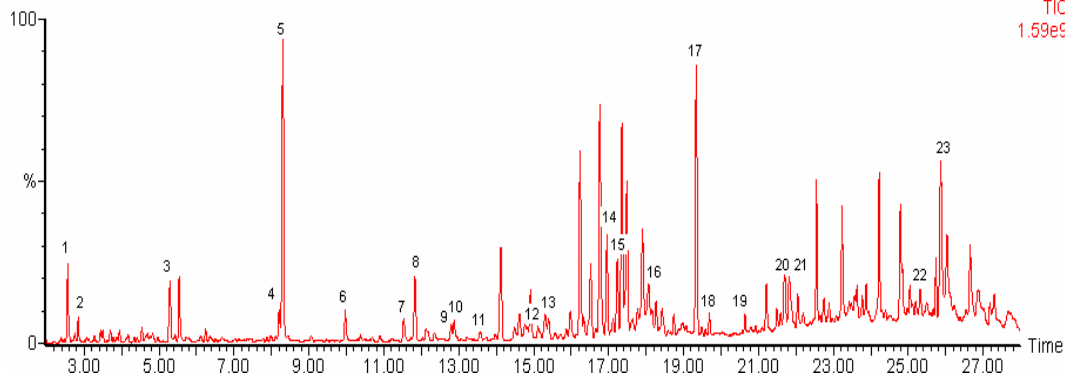


Figure 2. TIC Chromatogram of Volatile Organic Compounds for a Representative AUDIT Sample by ATD/GC/MS.

Gaussian peak shapes are exhibited by most of the identified compounds. Peak numbering is according to compound order in Table 2. m- and p-Xylenes were analyzed as a sum, as is generally done. Co-eluting VOCs (e.g., pyridine: peak 4 and toluene: peak 5; o-xylene: peak 9 and styrene: peak 10) shown in Figure 2 were re-analyzed by changing the test conditions if a duplicate sample was available (30% duplicate samples were required in this investigation).

As shown in Figure 2, some characteristic mass spectra of the sample have been identified by ATD/GC/MS. Benzene (MW 78) from tobacco smoke or gasoline products, toluene (MW 92), limonene (MW 136) and C₈H₁₀ isomer: ethylbenzene (r.t. 11.53), m, p-xylene (r.t. 11.83), and o-xylene (r.t. 12.80) were observed, these were also frequently detected in other samples. Peak 1, 4, 10, 11, 12, 20, and 23 were identified as 1,3-pentadiene (MW 67), pyridine (WM 79), styrene (104), α -pinene (WM 136), 1,3,5-trimethylbenzene (WM 120, r.t. 15.12), diethyl phthalate (WM 222) and dibutyl phthalate (WM 278), respectively. Peaks 15 and 16 were confirmed as a C₆H₄Cl₂ isomer: 1,4-dichlorobenzene (r.t. 17.24) and 1,2-dichlorobenzene (r.t. 18.07). Peaks 2 and 17 were assigned as MW 60 of isopropyl alcohol and MW 370 of decamethylcyclopentasiloxane, a likely emission compound from personal care products such as deodorants. Peaks 6, 13, 19, were identified as n-hexanal, which emanates from pressed wood products, and as decane and dodecane by comparing with standard compounds. Other compounds were identified based on the library search software and confirmed with fit parameters over 700/1000 for a good spectral match as shown in Table 2.

Table 2. Analytical Results for House No. 126B

Peak No.	Compound Identified	Ret. Time (min)	Concentration (mg/m ³)
1	1,3-Pentadiene	2.56	0.0071
2	Isopropyl Alcohol	2.84	0.0018
3	Benzene	5.29	0.0073
4	Pyridine	8.20	0.0035
5	Toluene	8.30	0.0345
6	Hexanal	9.97	0.0086
7	Ethylbenzene	11.53	0.0027
8	m-, p-Xylene	11.83	0.0085
9	o-Xylene	12.80	0.0019
10	Styrene	12.88	0.0026
11	α -Pinene	13.57	0.0015
12	1,3,5-Trimethylbenzene	15.12	0.0018
13	Decane	15.40	0.0031
14	D-limonene	16.96	0.0110
15	1,4-Dichlorobenzene	17.24	0.0080
16	1,2-Dichlorobenzene	18.07	0.0047
17	Decamethylcyclopentasiloxane	19.34	0.0324
18	Nonanal	19.69	0.0021
19	Dodecane	20.64	0.0014
20	Diethyl Phthalate	21.70	0.0067
21	3-Methyl-2-(2-oxopropyl) Furan	21.82	0.0079
22	2-Methyl-3-hydroxy-2,4,4-trimethylpentyl Ester Propanoic Acid	25.32	0.0028
23	Dibutyl Phthalate	25.87	0.0249

Others	0.2698
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*: Non-target compounds were calculated as toluene equivalents.

A summary of the VOC concentrations in different houses are illustrated in Figure 3. Volatile organic compounds ranged from 0.002 to 2.72 mg/m³. Several cases exhibited relatively high total VOC levels, up to 3.171 mg/m³. Further, if all compounds were completely identified, even higher VOC concentrations would likely be expected. Some VOCs with high occurrence ratios (toluene, benzene, ethylbenzene, xylene, limonene, decamethylcyclpentasiloxane) are typical air contaminants of residential buildings^[7]. VOCs with occurrence ratios larger than 0.06 out of the 150 sampled houses are listed in Table 3 including 28 compounds with values of median, mean, minimum and maximum concentrations. All compounds are listed in the order of their retention times.

Table 4 compares the VOC concentrations detected in the AUDIT study with the data reviewed by Hodgson and Levin^[7]. Their comparisons focused on the median, mean, maximum, 90th percentile and 95th percentile concentrations in ppb. Most VOC concentrations found in existing houses are significantly higher than Hodgson and Levin's review data. As mentioned earlier, >80% of the houses measured in this study had evidence of smoking activities. This may have contributed to the high concentration values of some VOCs. For example, cigarette smoking has been found to emit about 300-500 ug/cigarette of benzene (a carcinogenic compound)^[8]. The sites measured were mostly lower socio-economic homes. The significantly higher VOC concentrations in these homes than those reported previously may indicate a higher indoor pollutant exposure in the lower socio-economic homes. Further analyses will be conducted to determine relationships among the measured VOCs to potential sources identified in walk-through surveys of the residential buildings, and observed health effects.

Table 3. VOC concentrations (ppb) with occurrence ratios larger than 0.06 (25°C)

Compound	Ret. Time, min	MW	CAS No.	Chem. Class	Occurrence Ratio	Median	Mean	Min	Max	90%ile	95%ile
Hexane	3.5	86.2	110-54-3	Alkane HC	0.15	2.12	5.12	0.53	43.25	7.77	19.19
Benzene	5.28	78.1	71-43-2	Aromatic HC	0.41	2.06	4.01	0.18	73.73	5.89	11.62
Toluene	8.3	92.1	108-88-3	Aromatic HC	0.92	2.65	6.63	0.11	133.88	11.79	20.70
Hexamethylcyclotrisiloxane	9.04	222.5	541-05-9	R-siloxane	0.09	0.91	2.68	0.29	20.84	4.01	10.34
n-Hexanal	9.97	100.2	66-25-1	Aldehyde	0.12	1.58	2.31	0.32	8.34	4.27	5.05
1,1-dimethylethyl-cyclohexane	10.55	112.2	590-66-9	Cyclic HC	0.41	6.33	15.92	0.81	63.39	58.17	60.78
Ethylbenzene	11.51	106.2	100-41-4	Aromatic HC	0.30	0.60	3.80	0.12	132.86	3.28	5.05
m-xylene, p-xylene	11.81	106.2	106-42-3	Aromatic HC	0.78	1.48	8.46	0.24	626.21	8.78	16.67
o-xylene	12.77	106.2	95-47-6	Aromatic HC	0.29	0.86	7.48	0.29	261.53	5.78	8.24
Styrene	12.86	104.2	100-42-5	Aromatic HC	0.06	0.63	2.16	0.31	12.38	4.20	8.29
α -Pinene	13.54	136.2	7785-70-8	Terpene HC	0.21	1.07	2.71	0.16	20.34	5.35	10.48
2-butoxy-ethanol	13.97	118.2	111-76-2	Glycol ether	0.07	6.33	15.92	0.81	63.39	58.17	60.78
Octamethylcyclotetrasiloxane	14.88	296.6	556-67-2	R-siloxane	0.15	1.36	2.64	0.10	20.56	4.41	6.61
Decane	15.41	142.3	124-18-5	Alkane HC	0.08	1.34	2.52	0.40	15.83	3.50	8.52
1,2,4-Trimethylbenzene	16.31	120.2	95-63-6	Aromatic HC	0.24	1.46	2.32	0.19	16.18	4.11	6.42
Benzaldehyde	16.44	106.1	100-52-7	Aldehyde	0.06	2.12	4.59	1.13	20.26	8.38	14.32
D-limonene	16.96	136.2	5989-27-5	Terpene HC	0.85	2.80	8.22	0.18	217.91	17.84	27.10
1,4-Dichlorobenzene	17.22	147	106-46-7	Cl-aromatic HC	0.15	5.69	35.68	0.11	218.87	112.63	193.47
1,2-Dichlorobenzene	18.07	147	95-50-1	Cl-aromatic HC	0.06	1.15	2.39	0.76	12.15	3.92	8.04
Undecane	18.36	156.3	1120-21-4	Alkane HC	0.06	3.44	4.82	0.36	14.36	9.93	12.14
Decamethylcyclopentasiloxane	19.33	370.8	541-02-6	R-siloxane	0.85	2.78	8.98	0.13	102.92	20.85	36.41
Nonanal	19.67	142.2	124-19-6	Aldehyde	0.45	1.23	1.47	0.26	4.63	2.97	3.37
Dodecane	20.64	170.3	112-40-3	Alkane HC	0.10	1.08	1.48	0.21	6.65	2.61	3.56
Napthalene	21.83	128.2	91-20-3	Aromatic HC	0.07	2.84	9.52	0.30	44.70	19.87	32.28
$\alpha,\alpha,4$ -trimethyl-3-cyclohexene-1-methanol	21.85	154	98-55-5	Miscellaneous	0.36	1.10	4.44	0.29	56.38	9.08	26.11
Dodecamethylcyclohexasiloxane	21.9	444.9	540-97-6	R-siloxane	0.15	0.84	1.39	0.10	5.80	3.55	4.91
2-methyl-3-hydroxy-2,4,4-trimethylpentyl ester propanic acid	25.32	216	74367-34-3	Acid	0.09	1.47	2.81	0.30	9.64	7.24	8.63
2-methyl-2-ethyl-3-hydroxyhexyl ester propanic acid	25.38	216	74367-31-0	Acid	0.09	2.14	2.58	0.50	5.56	5.00	5.22

Table 4. Comparison of VOC concentrations (ppb, 25°C) in dwelling from current study with the data reviewed by Hodgson and Levin^[7]

Compound	CAS No.	Median		Mean		Max		90%ile		95%ile	
		This Study	H & L	This Study	H & L	This Study	H & L	This Study	H & L	This Study	H & L
2-Methylpentane	107-83-5	0.94	0.56	0.94		1.09		1.06		1.08	
Hexane	110-54-3	2.12	0.51	5.12	0.28	43.25		7.77		19.19	
3-Methylpentane	96-14-0	1.02	0.33	1.02		1.02		1.02		1.02	
Chloroform	67-66-3	3.37	0.19(5)	3.37	0.37(5)	3.37	1.2-4.3(4)	3.37	0.8, 1.3	3.37	1.2
Benzene	71-43-2	2.06	0.87(7)	4.01	1.5(7)	73.73	8.4-41	5.89	1.9-4.1(4)	11.62	4.0
Methylcyclohexane	108-87-2	1.96	0.40	2.63		4.75		4.19		4.47	
Trichloroethylene	79-01-6	1.31	0.08(8)	1.31	0.23(7)	2.13	0.58-5.0(7)	1.97	0.21-0.42(4)	2.05	0.13, 0.26
Pyridne	110-86-1	1.09	0.17(2)	1.09	0.54(2)	1.09	1.5, 2.7	1.09		1.09	
Toluene	108-88-3	2.65	3.3(6)	6.63	6.7(5)	133.88	12-240(5)	11.79	7.8, 13	20.70	
Octane	111-65-9	0.64	0.24(2)	0.71	0.70	1.10	3.6	0.97	2.4	1.03	
Tetrachloroethylene	127-18-4	1.34	0.15(7)	1.34	0.41(6)	1.34	0.76-65(7)	1.34	0.34-1.0(3)	1.34	0.72,1.0
Ethylbenzene	100-41-4	0.60	0.53(4)	3.80	1.4(4)	132.86	5.9-4.0(3)	3.28	3.0	5.05	
m-, p-xylene	106-42-3	1.48	1.4(3)	8.46	2.9(3)	626.21	28-130	8.78	2.8,13	16.67	5.0
o-xylene	95-47-6	0.86	0.53(5)	7.48	1.1(6)	261.53	7.9-43(5)	5.78	1.3, 3.7	8.24	1.6
Styrene	100-42-5	0.63	0.25(5)	2.16	0.35(6)	12.38	1.7-33(5)	4.20	0.5,0.89	8.29	0.56
α-Pinene	7785-70-8	1.07		2.71	4.1	20.34		5.35		10.48	
1,2,3-Trimethylbenzene	526-73-8	1.63	0.20	1.76	0.42	3.06	2.2,7.9	2.73		2.90	
1,3,5 -Trimethylbenzene	108-67-8	0.81	0.25	1.91	0.51	8.81	3.1,14	4.05		6.43	
Decane	124-18-5	1.34	0.44(2)	2.52	0.97(2)	15.83	7.9,20	3.50		8.52	
1,2,4-Trimethylbenzene	95-63-6	1.46	0.79	2.32	2.4	16.18		4.11		6.42	
Benzaldehyde	100-52-7	2.12		4.59	0.38	20.26	1.3	8.38		14.32	
D-limonene	5989-27-5	2.80		8.22	3.6	217.91		17.84		27.10	
1,4-Dichlorobenzene	106-46-7	5.69	0.08(4)	35.68	1.3(6)	218.87	16-50(5)	112.63	0.34, 4.7	193.47	0.57
1,2-Dichlorobenzene	95-50-1	1.15		2.39	11	12.15	0.09	3.92	0.04	8.04	
Undecane	1120-21-4	3.44	0.28(2)	4.82	1.3(2)	14.36	9.2, 39	9.93		12.14	
Dodecane	112-40-3	1.08	0.17(2)	1.48	0.55(2)	6.65	4.1,18	2.61		3.56	
Naphthalene	91-20-3	2.84	0.09	9.52	0.35(2)	44.70	0.95	19.87	0.41	32.28	

CONCLUSIONS

An analytical procedure developed here demonstrates that thermal desorption and gas chromatography-mass spectroscopy method appears to be rapid, sensitive, and reliable for the detection of VOCs. Additionally this method offers convenient analytical techniques for trace gas determination of environmental samples. The survey was conducted throughout over 150 residential houses to provide accurate data for elucidating the health effects potentially caused by the air pollutants.

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