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# Thermal desorption–gas chromatography–mass spectrometry–flame ionization detection–sniffer multi-coupling: A device for the determination of odorous volatile organic compounds in air

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**Abstract**

A multi-coupling analytical system is described, consisting of a thermal desorber installed with a gas chromatograph, a flame ionization detector, a mass spectrometer and a sniffer. The sniffer was connected at the output of the GC column leading to the flame ionization detector. The retention times obtained by the flame ionization detector and the sniffer are well correlated, allowing the detection of odorous compounds, which can be then identified by mass spectrometry. This device is therefore able to identify and quantify volatile organic compounds (VOC) and odorous compounds in only one analytical run. This device appears to be very helpful for indoor air quality investigations. As an example, the VOC emissions from two types of wall covering and, in particular, the identification of the odorous compounds, are reported.

## 1. Introduction

Indoor air quality is of interest in terms of impact on public health as a result of the growing number of complaints linked to poor air quality and of the recognized influence of environmental factors on the appearance of some pathological affections. The characterization of air pollution sources is mainly based on physico-chemical analytical methods for assessing emissions of pollutants such as volatile organic compounds (VOC). Few epidemiological studies concerning these compounds at low levels have been conducted.

Alternative assessment procedures can be envisaged, and in particular reactions linked to the odour due to the presence of some VOC may be used. In particular, owing to its high sensitivity, the human nose is able to detect odours, which can be interpreted as a poor air quality parameter. Indeed, sensory evaluation procedures using human panels allow the assessment of a potential source of odour in terms of intensity [1] or acceptability [2], but the link between olfactometric and physico-chemical analyses is far from being well understood.

Only a few studies on the determination of odorous compounds linked to particular pollution sources have been conducted. The analytical methods generally used in such studies consisted of a sniffer connected at the capillary column outlet. The identification of odorous compounds

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is usually carried out by implementing two or three separate analytical systems [gas chromatography (GC)–sniffer, GC–flame ionization detection (FID) and GC–mass spectrometry (MS)], thus making the determination (identification, quantification and evaluation of odorous compounds) sometimes complex [3–5]. Therefore, thermal desorption, GC, MS and sniffer (TD–GC–MS–sniffer) have been coupled for the identification of odorous compounds but quantification is much more difficult than using FID [6]. Another configuration has been proposed with two systems, headspace–GC–FID–sniffer and headspace–GC–MS, working in parallel [7], but this approach has the drawbacks of doubling the analytical procedures and making the identification of odorous compounds sometimes difficult.

Our purpose was therefore to develop a method making possible both the identification and quantification of VOC and odorous compounds using a TD–GC–MS–FID-sniffer multi-coupling system. This approach allows a decrease in analysing time and costs and eliminates problems of sampling reproducibility. In order to illustrate the potential of the device for indoor air quality investigations, we present here some preliminary results for VOC and odours emitted by wall coverings.

## 2. Experimental

### 2.1. Analytical equipment, procedure and method

The analytical system (Fig. 1) is composed of a Chrompack "purge-and-trap" thermal desorber, which serves as injector for an HP 5890 gas chromatograph. Before analysis, VOC are trapped on 100 mg of adsorbent contained in a glass tube. We used Tenax TA [8], a 2,6-diphenylene oxide-based polymer of porosity 20–35, which traps a wide variety of $C_2$–$C_{18}$ VOC [9].

The thermal desorption sequence is as follows: precooling, −100°C for 2 min; desorption, 250°C for 5 min; and injection, 220°C for 2 min. After

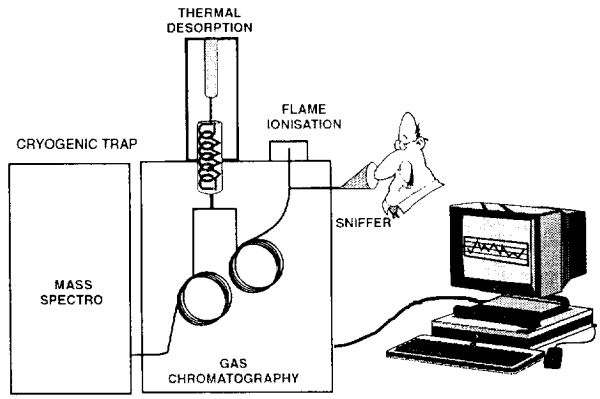


Fig. 1. Schematic diagram of TD–GC–MS–FID–sniffer analytical system.

desorption, VOC are injected into the GC system and eluted into two capillary columns (SGE). The stationary phase is a modified siloxane polymer with 5% phenylsiloxane. Column A (50 m × 0.32 mm, 1 μm) is connected to the flame ionization detector and to the SGE sniffer, while column B (50 m × 0.22 mm, 1 μm) is connected to the HP 5971A mass spectrometer fitted with an electron impact ionization source and a quadrupole (mass limit, 650 Th). The analysed sample is therefore divided twice, first at the output of the Chrompack injector and then before the flame ionization detector and sniffer (Fig. 1). Helium is used as the carrier gas at 0.21 MPa pressure at the head of the columns.

The sniffer consists of a glass cone where the operator inhales the separated compounds at the outlet of the chromatographic column. Further, an air humidifier is installed to prevent the olfactory mucous membrane from rapid drying. The operator manually records the retention times of each inhaled odorous compound using an HP 3396 integrator. All detectors and integrators start at the same time when the gas sample is injected into the capillary column.

After analysis, retention times of odorous compounds determined by the operator are compared with those of the chromatographic peaks obtained by FID in order to locate the odorous peaks. The retention times obtained by FID and by MS cannot be directly compared because of the different diameter columns used,

leading to retention time differences. Nevertheless, the order of elution of the separated compounds is the same since both columns have identical stationary phases. The relationship between FID and mass peaks can therefore be established and the compounds identified by means of the mass spectrometer's NIST Database HP spectral library.

The analytical system is thus made up of three detection systems working in parallel: FID for quantification, MS for identification and the sniffer for the detection of odorous organic compounds. This is, to our knowledge, the first device allowing the characterization of odorous VOC in one analytical run.

### 2.2. Calibration of the sniffer via FID

As column A is connected to the flame ionization detector and to the sniffer, the carrier gas flow-rates at the outlets of the two detectors must be identical in order to obtain equivalent retention times.

We measured the flow-rates at the outlet of the flame ionization detector and the sniffer using an HP digital flow meter for different temperatures (50–250°C) of the gas chromatograph. We obtained a good correlation between the FID and sniffer flow-rates (correlation coefficient = 0.995, slope = 1.057, intercept = 0.009), suggesting that the retention times should be comparable. In fact, only a slight deviation occurred in favor of the sniffer compared with FID (52 and 48% of the initial flow-rate, respectively).

A standard solution containing *m*-xylene (345.6 ng/$\mu$l), cyclooctane (166.6 ng/$\mu$l), propylbenzene (344.8 ng/$\mu$l), 4-ethyltoluene (344.4 ng/$\mu$l), 1,2-dichlorobenzene (522 ng/$\mu$l), linalool (584.6 ng/$\mu$l) and geraniol (355.6 ng/$\mu$l) was prepared in methanol from Aldrich or Fluka standards in order to determine odorous compounds. The concentrations were chosen so that the compounds could be detected by sniffing without any ambiguity. Then, retention times obtained by FID were compared with those determined by sniffing, and a study of the reproducibility was conducted with six experiments.

A 1-$\mu$l volume of this solution was injected with a syringe into an adsorbent tube. This tube was then swept with 100 ml of pure nitrogen in order to remove methanol and placed in the thermal desorber for analysis.

The GC temperature programme used for these tests was initial temperature: 60°C; increased at 3°C/min to 250°C.

Standard deviations of the retention times for each of the compounds determined by sniffing range from 1.3 to 2.1 s (Table 1). The average standard deviation is 1.6 s, which corresponds to the approximate time required by normal inhalation [10]. This is in reasonable agreement with same kind of studies comparing FID and sniffer

Table 1
Comparison of the FID and sniffer retention times

| Compound | FID | | Sniffer | | $\delta = T_{FID} - T_{sniffer}$ (s) |
|---|---|---|---|---|---|
| | Average retention time, $T_{FID}$ (min) | $\sigma$ (s) | Average retention time, $T_{sniffer}$ (min) | $\sigma$ (s) | |
| *m*-Xylene | 10.00 | 0.33 | 10.00 | 2.07 | 0.09 |
| Cyclooctane | 12.46 | 0.36 | 12.49 | 1.55 | −1.71 |
| Propylbenzene | 13.77 | 0.37 | 13.76 | 1.53 | 0.15 |
| 4-Ethyltoluene | 14.28 | 0.37 | 14.34 | 1.26 | −3.36 |
| 1,2-Dichlorobenzene | 18.26 | 0.41 | 18.38 | 1.38 | −7.03 |
| Linalool | 20.89 | 0.42 | 20.89 | 1.31 | −0.16 |
| Geraniol | 28.70 | 0.45 | 28.86 | 2.02 | −9.52 |

retention times. For example, using different installations but the same kind of data processing, the standard deviations on the retention times measured by Sävenhed et al. [4] and Khiari et al. [5] were 1.1 and 9.7 s, respectively.

The differences in the FID and sniffer average retention times range from −9.5 to 0.15 s (Table 1). These differences indicate that the odorous compounds chosen in this study are generally detected after the FID peak maximum, but they remain lower than the integration interval of the corresponding FID signal (24 s on average). This phenomenon was also reported [4,5]. This cannot be explained by the difference in the carrier gas flow-rates, which is slightly higher at the outlet of the sniffer than at the outlet of the flame ionization detector, because the phenomenon should have been the reverse. This difference could be explained by the fact that the least volatile compounds are somehow delayed at the output of the column by a kind of cooling effect [4], and by the longer pathway from the outlet to the sniffer. Nevertheless, we observed an excellent correlation between the FID and the sniffer average retention times as attested by the parameters of the regression line (slope = 1.0074, intercept = −0.074, $r^2 > 0.9999$), indicating that our device is reliable in terms of the characterization of odorous compounds.

### 2.3. Sample preparation

In order to illustrate the potential of our multicoupling system for indoor air quality investigations, we present here some preliminary results for VOC and associated odours emitted by wall coverings. We used two different types of wall coverings: R1, polyester plastic backed with PVC; and R2, PVC plastic backed with cotton. R1 and R2 were sampled at the factory and sent to our laboratory in hermetically sealed polyethylene packages to avoid any exchange between the materials and ambient air during transport.

R1 and R2 were successively inserted into a chemically inert 3.5-l glass chamber (Fig. 2) designed for the simulation of a room in a building and to isolate the tested material from external pollution sources. For each test, the chosen loading rate (surface area of the sample in $m^2/m^3$ volume of the chamber) was 1 $m^2/m^3$, a value slightly above that commonly used (0.4 $m^2/m^3$) [11]. The temperature and the relative humidity inside the chamber were measured but it was not possible to control these physical parameters [12,13]. The chamber was ventilated with air from the laboratory previously filtered using activated carbon, and with an air exchange rate (air flow-rate in $m^3/h$ per $m^3$ volume of the chamber) of 1 $h^{-1}$. Before each test, a blank chamber test was performed by sampling 1 l of indoor air on a Tenax tube to ensure its cleanliness.

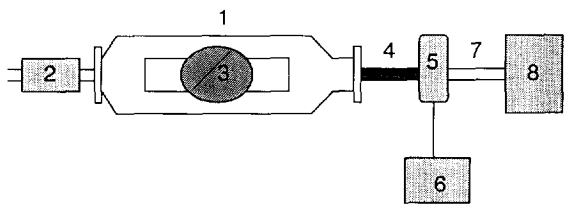


Fig. 2. Schematic diagram of environmental test chamber. 1 = 3.5-l glass chamber; 2 = carbon filter; 3 = wall covering sample; 4 = Tenax TA tube; 5 = mass flow regulator; 6 = mass flow controller; 7 = PTFE tubing; 8 = pump.

The sample was left in the chamber for 24 h so that the VOC emission profile of the material was balanced. A 1-l sample of air was then sampled by pumping 100 ml/min through a Tenax tube, which was subsequently introduced into the TD–GC–MS–FID–sniffer system for analysis.

The GC temperature programme used for these analyses was initial temperature 40°C, increased at 3.5°C/min to 250°C.

## 3. Results and discussion

The FID and the total ionic current (TIC) mass chromatograms are presented in Figs. 3 and 4 for R1 and R2, respectively. Compounds were identified by MS by means of the spectral library included in the data processing. This library generally allows us to characterize 50% of the

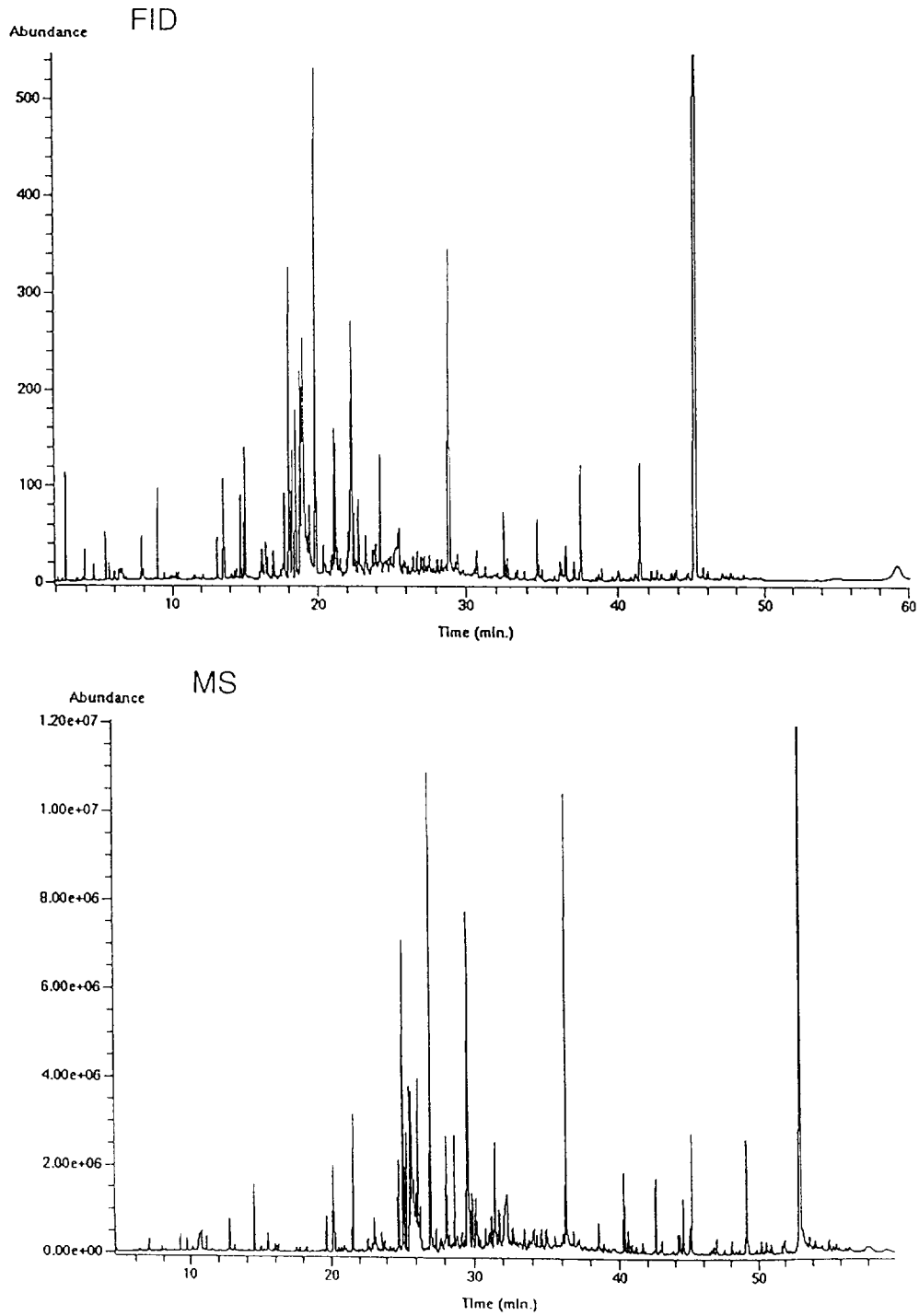


Fig. 3. FID and mass chromatograms for R1.

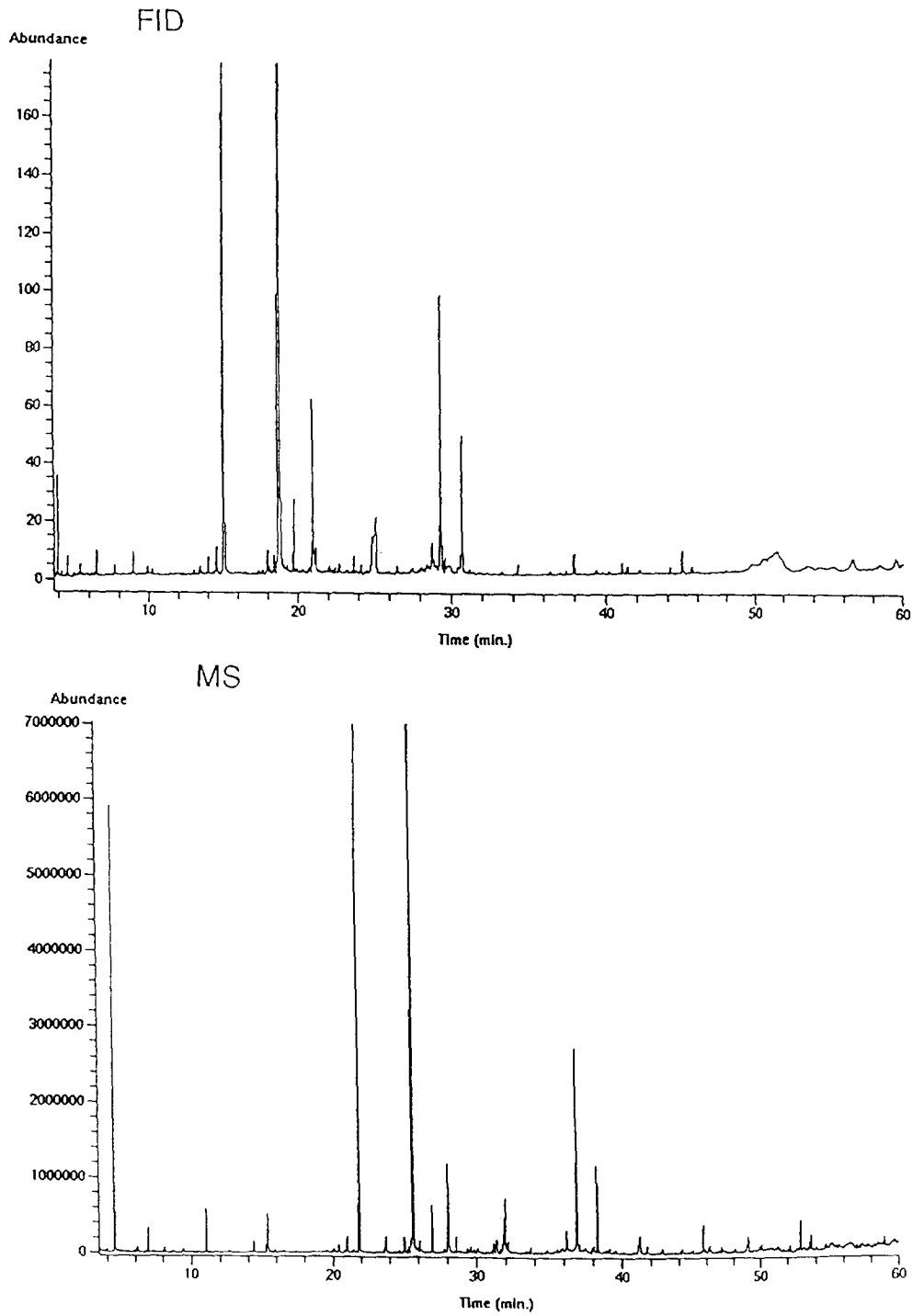


Fig. 4. FID and mass chromatograms for R2.

Table 2
Concentrations of VOC identified by MS for R1

| Compound identified | Concentration ($\mu g/m^3$) | | Compound identified | Concentration ($\mu g/m^3$) | |
|---|---|---|---|---|---|
| | External standard | Toluene equiv. | | External standard | Toluene equiv. |
| Formamide | | 12.5 | Cymene | | 61 |
| 1,2-Propanediol | | 24 | Propyltoluene | | 34 |
| Toluene | 1.8 | 1.4 | Decahydronaphthalene | | 24 |
| *m*- or *p*-xylene | 11.8 | 12.5 | Ethyldimethylbenzene | | 34 |
| 2-Methyl-2,4pentanediol | | 20 | | | |
| 2-Butoxyethanol | 168.2 | 58 | Undecane | | 75 |
| Cumene | | 15.5 | Ethyldimethylbenzene | | 29 |
| 4-Methylhexanol | | 13 | | | |
| Propylbenzene | 20.2 | 22 | 2-Ethylhexanoic acid | | 283 |
| 3-Ethyltoluene | | 103 | Tetramethylbenzene | | 27 |
| 4-Ethyltoluene | 41.9 | 42 | Tetramethylbenzene | | 36 |
| 1,2,5-Trimethylbenzene | | 69 | 2-(Butoxyethoxy)ethanol | | 629.5 |
| Phenol | | 730 | (1-Ethyl-1-methylbutyl)benzene | | 22 |
| 1,2,4-Trimethylbenzene | | 290 | | | |
| 2-Ethylhexanol | | 113 | Undecane | | 8 |
| 1,2,3-Trimethylbenzene | | 88 | (1-Methylheptyl)benzene | | 10 |
| 1-Methyl-2-pyrrolidinone | | 641 | Tetradecane | | 145.8 |
| Propyltoluene | | 35 | | | |

total number of chromatographic peaks with a presence probability exceeding 80%.

The concentrations of compounds identified by MS are summarized in Tables 2 and 3. R1 emits many aromatic hydrocarbons, in addition to phenol, 2-(butoxyethoxy)ethanol and 1-methyl 2-pyrrolidinone (Table 2), whereas R2 mainly releases phenolic and ketonic derivatives (Table 3). Some studies on the analysis of volatile phases for wall paper have also shown the existence of families and main chemical compounds such as aromatic hydrocarbons (toluene, xylene) and chlorinated hydrocarbons, in addition to phenol and ketonic compounds [14,15].

Table 3
Concentrations of VOC identified by MS for R2

| Compound identified | Concentration ($\mu g/m^3$) | | Compound identified | Concentration ($\mu g/m^3$) | |
|---|---|---|---|---|---|
| | External standard | Toluene equiv. | | External standard | Toluene equiv. |
| 2-Butanone | 0.7 | 3 | Phenol | | 696 |
| Trichlorethylene | 24.3 | 4.5 | 2-Ethyltoluene | | 2 |
| Toluene | 6.5 | 6 | 1,2,4-Trimethylbenzene | | 21 |
| 3-Heptanone | | 3 | 2-Ethylhexanol | | 52 |
| 3-Heptanol | | 9 | 1,2,3-Trimethylbenzene | | 8 |
| Cyclohexanone | | 402 | 2-Ethylhexanoic acid | | 40.5 |
| 3-Ethyltoluene | | 7 | 2-(Butoxyethoxy)ethanol | | 11 |
| 4-Ethyltoluene | 2.6 | 2.5 | 2-Isopropylphenol | | 85 |
| 1,2,5-Trimethylbenzene | | 5 | 4-Isopropylphenol | | 41 |
| | | | 2,4-Bisisopropylphenol | | 6 |

These phenolic derivatives may originate from the PVC layer of R2. Indeed, branched phenolic compounds are often included in polymers in order to delay thermal oxidation during their transformation or use. R2 is also a significant source of cyclohexanone. This compound, which often serves as a PVC solvent, may come from the printing process.

2-Ethylhexanol, which is typical of PVC covering emissions, is emitted by both R1 and R2. It has been shown that this compound comes from the chemical degradation of the plasticizer dioctyl phthalate (DOP) contained in the PVC layer [16].

The concentrations reported in Tables 2 and 3 were calculated by the flame ionization detector, either by external calibration or in toluene equivalents using the toluene response factor. As it is not possible to calculate all concentrations by external calibration because of the numerous compounds present, the concentrations are often expressed in toluene equivalents [17,18]. In this case, we assume that the behaviour of the volatile compounds on the chromatographic column is similar to that of toluene. This assumption is valid when the compounds belong to the same chemical class as toluene, i.e., aromatic hydrocarbons, or even, to a second approximation, hydrocarbons saturated or not. If the concentrations obtained by external calibration or expressed in toluene equivalents are compared (Tables 2 and 3), a good correlation is observed as far as aromatics are concerned (e.g., ethyltoluene, xylenes). In contrast, a large deviation in the concentrations is observed for oxygenated or chlorinated compounds such as trichloroethylene or 2-butoxyethanol, and to a lesser extent for some aldehydes (crotonaldehyde, hexanal) and alcohols (propylene glycol) [18].

As a recent study has shown that the compounds emitted by floor and wall coverings used for building decoration are mainly hydrocarbons [19], the expression of their concentrations in toluene equivalents applies correctly to indoor air quality investigations (same chemical classes).

Further, the TVOC (total volatile organic compounds), which represents the total mass of VOC per unit volume, is calculated as the sum of all individual VOC concentrations [20–24]. In our case, the R1 and R2 TVOC concentrations (3.7 and 1.4 $mg/m^3$ toluene equivalents, respectively) indicate that R1 gives off almost three times more VOC in mass than R2. The ratio between the sum of the concentrations of the identified products and TVOC shows that 77% of the chromatogram has been identified for R1 and 95% for R2. Therefore, most of the compounds have been identified by MS and will be quantified by FID.

A biological model based on the sensory perception, the weak inflammatory reactions and the stress reactions due to the environment was proposed by Molhave [25]. This model links up discomfort reactions and exposure to VOC expressed in TVOC. According to this classification, exposure to R1 would probably result in irritation and headaches, whereas R2 could cause irritation and discomfort.

The odorous compounds from these two wall coverings were identified through the sniffer. The results are summarized in Table 4 for each odorous VOC, including their concentration and odour intensity.

The odour of R1 is a priori mainly due to aromatic hydrocarbons, such as 1,2,4-trimethylbenzene and cymene. The compounds at the origin of the R2 odour are likely to belong to several chemical classes. For instance, phenol and cyclohexanone are detected with a strong intensity. Therefore, the nature of the odours induced by these two different materials is likely to be different.

2-Ethylhexanoic acid seems to be a special compound. Indeed, it is perceived from R2 as odorous with a medium intensity whereas it is not identified as an odorous compound from R1 even though its emission from R1 is greater than that from R2 (283 and 40.5 $\mu g/m^3$, respectively) (Table 4).

We have included in Table 4 the odour thresholds reported [26] for some of the VOC that we have identified as odorous. We can note that the measured odorous VOC concentrations are generally much lower than the concentrations assumed to represent the odour threshold. For

Table 4
Intensities and measured concentrations of odorous VOC emitted by R1 and R2 (reported odour thresholds are from [26])

| Material | Odorous compound | Odour intensity (sniffer) | Concentration ($\mu g/m^3$) | Concentration ($\mu g/m^3$) (in toluene equiv.) | Odour threshold ($\mu g/m^3$) |
|---|---|---|---|---|---|
| R1 | Toluene | Low | 1.8 | 1.4 | 5890 |
| | Propylbenzene | Low | 20.2 | 22 | |
| | 4-Ethyltoluene | Low | 41.9 | 42 | |
| | 1,2,4-Trimethylbenzene | Strong | | 290 | 776 |
| | Cymene | Strong | | 61 | |
| R2 | Toluene | Low | 6.5 | 6 | 5890 |
| | 1,2,4-Trimethylbenzene | Low | | 21 | 776 |
| | Cyclohexanone | Strong | | 402 | 2880 |
| | Phenol | Strong | | 696 | 426 |
| | 2-Ethylhexanoic acid | Medium | | 40.5 | |

example, toluene is perceived in low intensity from R1 and R2 with concentrations of 1 and 6 $\mu g/m^3$, respectively, whereas its reported odour threshold is 5890 $\mu g/m^3$.

## 4. Conclusions

TD–GC–MS–FID–sniffer multi-coupling appears to be a reliable method making possible the identification and quantification of VOC and also of odorous compounds resulting, for example, from indoor air pollution. Compared with other methods, the main advantage of this device is that this goal is achieved in only one analytical run. Since the relationship between the sniffer/FID retention times and the FID/mass retention times can be established, it is possible to identify almost simultaneously VOC and odorous compounds.

In particular, using the same capillary column to supply the sniffer and the flame ionization detector allows us to minimize the gaps between the respective retention times, thus resulting in a reliable identification of the odorous compounds.

The VOC (odorous or not) released by two different wall coverings have been identified to illustrate the potential of this device for indoor air quality investigations. The analysis of the volatile phases of the wall coverings has shown the existence of the main chemical classes and main chemical compounds (aromatic, phenolic and ketonic) emitted.

Typical VOCs such as 2-ethylhexanol, phenol derivatives and cyclohexanone have been identified. 2-Ethylhexanol emitted by the two materials and phenol derivatives given off by R2 probably originate from the PVC layer, whereas cyclohexanone emitted by R2 probably results from the manufacturing process (printing process).

VOC were quantified either by external calibration or with respect to the toluene response factor. Good agreement between these two modes of concentration calculation was found for hydrocarbons, whether saturated or not. The comparison of the TVOC calculation with health effects suggests that exposure to these two coverings could cause discomfort such as irritation or headaches.

Finally, the odorous compounds emitted by the two coverings were determined by sniffing. The odorous compounds assumed to have a strong intensity are different for R1 and R2, suggesting that the odour emitted by each covering can be qualitatively different. The identified odorous VOC were generally present at con-

centration levels much lower than the commonly reported odour threshold.

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