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Determination of the Sources Responsible for Odour Complaints in an Enclosed Working Space via the Use of Sensorial Analysis, GC-MS Analysis and GC-Sniffing

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People spend an estimated 90% of their time indoors in homes, offices, shops and public buildings; thus, indoor air quality (IAQ) may considerably affect human health and well-being. If this applies, it is essential that one can work without interference of for example noise, smell, bad lighting, … In this examination focus is on the aspect of smell. Of the different categories of compounds found in indoor environments, volatile organic compounds (VOC) for example aldehydes, alcohols, aromatic hydrocarbons, carboxylic acids and ketones can often reach higher indoor concentrations compared to the corresponding outdoor values. The odour thresholds of several VOC are low enough to affect perceived air quality, resulting in a number of acute effects of reported sensory irritation of eyes and respiratory tract and deterioration of performance. The smell experience (air quality) depends on a number of factors that can influence the odour impact. An odorous VOC generally induces a sense of smell at concentrations at and above the odour threshold. In addition, at a concentration, generally several orders of magnitude higher, the compound can cause sensorial irritation. The aim is always to improve the IAQ and promoting in this way the workers' comfort and protecting human health. In that context, an accurate identification of sources and other determinants of exposure to VOC in indoor microenvironments is essential for the development of successful prevention strategies in building design, operation and maintenance.

1. Introduction

The research is carried out in a company which has a couple of offices where there have been odour complaints for quite some time. In some offices the complaints are so severe that they are no longer used. In a study of indoor offices a lot of the identified indoor sources VOC were linked to building materials (e.g., carpets, floorings, paints, and wood-based materials) (Campagnolo et al., 2017). This study aims at analysing samples taken from possible sources of odour and linking them with chemical compounds detected via the use of sensorial analysis, GC-MS analysis and GC-sniffing.

The odour character of the air observed in the studied office can be described as chemical, pungent, textile, glue and plastic. If one stayed in the office for a limited time, the first signs of a headache were observed.

There are a lot of possible causes for odour complaints in indoor building spaces as is described in a research by Kagi N. et al. in 2008 (Kagi et al., 2008). The possible odour sources that could be identified in the studied office are the ventilation, the old wall and ceiling insulation, the present furniture and the floor carpet. With this knowledge in mind the possible sources of the odour complaints were investigated on site. The functioning of the ventilation present in the office was checked. It worked properly and did not bring any extra smell into the office. Subsequently, the furniture present in the office was examined for smell, but again no aberrant odour was detected. Thereafter, the insulation material present in the walls and ceiling was cleared and subjected to a sensorial examination. A distinct musty smell could be detected, which can contribute to the odour character present in the office, but was certainly not the main source of the odour nuisance as the odour character differed from the one perceived in the office. Finally, the carpet was examined. The whole surface including pieces of cut-out carpet attached against the wall by an adhesive, consisted of the same type floor carpet. It gave off a strong chemical odour very similar to the odour present in the office.

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The on-site sensorial examination showed that the smell of the carpet most closely resembled the ambient air. This does not rule out the possibility that the other possible odour sources do contribute to the odour in the office. Based on these findings, it was decided to conduct research on the carpet to link the perceived odour to chemical components via TD-GC-MS and GC-sniffing.

When assessing the health risks of floor coverings, two factors are important. Carpets can act as a repository for indoor air pollutants such as dirt, dust particles, allergens, and other biological contaminants that can accumulate in the carpets. The other factor is that carpets can emit volatile organic compounds that can cause odour and irritation of the mucous membranes (Becher et al., 2018). This research is focused on the emission of VOC by the carpet and linking these compounds to the odour complaints. The VOC that are present in the material when the installation takes places are referred to as a primary source of VOC and are emitted indoors. A carpet consists of adhesives, underlayment and the carpet itself. All these compounds can contribute to the emission of hundreds of VOC and semi-volatile organic compounds (SVOC). Some of these identified components are known to produce smell. These include 4-phenylcyclohexene which has the smell of new carpet, aromatic hydrocarbons (styrene, benzene, toluene, xylenes) and formaldehyde (Haines et al., 2020). In general, carpets are a relatively low source of VOC (Hodgson et al., 1993). SVOC used for the manufacture of carpets include per- and polyfluoroalkyl substances (PFAS), triclosan used as antimicrobial, phthalate plasticizers used as backing for carpets and flame retardants consisting of polyurethane foam (Haines et al., 2020). This research focuses on the VOC.

2. Materials and methods

2.1 Sampling

Following an on-site sensorial examination, similarities in terms of smell between the different offices could be determined. The office with the highest intensity of smell perceived, was chosen for further analysis. To further investigate the chosen office, samples of the ambient air, a headspace of carpet on-site and headspace of pieces of carpet with and without adhesive were taken for further analyses. A Nalophan sampling bag was used for its inert properties. It does not significantly interact chemically with odorants, ensuring that the collected samples remain unaffected during storage (Eusebio et al., 2017). The bags were filled with air by creating an under pressure in the receptacle according to the lung principle (Guillot, 2012). A 10 Liter Nalophan bag was used for the samples taken on site. The headspaces were prepared in the lab. For the headspace of the carpet with adhesive, a piece was taken from the office for examination. To examine a piece the carpet without adhesive, a spare carpet tile was used and was cut so that the shape matched the piece of carpet with adhesive. Both pieces were put in a separate 40 litre Nalophan bag, which was afterwards filled with odourfree air. After 12 hours samples were taken from the bags for analysis. In the NOLAB laboratory, the air samples were transferred onto a Carbon Graphitised sorbent tube for the GC-MS and GC-sniffing analysis. Sulficarb adsorption tubes (Markes Ltd) were used for adsorption of the gases.

2.2 Sensorial analysis

To determine the sensorial differences between the samples a sensorial analysis was carried out. On the collected air and product samples a sensorial analysis is performed in an odour free room at NOLAB. The sensorial odour analysis lab offers a novel approach for the direct, undiluted evaluation of an odour sample. This method aims to generate relevant information about the odour character that complements the data obtained from traditional dynamic olfactometry, which primarily focuses on odour concentration. The direct evaluation technique is particularly effective in resolving key aspects such as odour character, perceived intensity, and hedonic tone.

An automated sample presentation method is employed, which involves a pressurized chamber and a sniffing cup. This setup allows for the presentation of identical aliquots of the air sample bag to each panelist, ensuring consistency in the evaluation process. The controlled flow rate of 18 L/min, combined with specially designed sniffing cups, ensures a homogeneous air flow for each sample. To comply with the EN 13725 standard, all instrument parts that come into contact with the sample are constructed from stainless steel (316L), PTFE, or borosilicate glass, which ensures the integrity and purity of the sample.

The evaluation process involves an odour panel consisting of at least six individuals who meet the criteria outlined in EN 13725. Furthermore, the panelists are specifically trained to recognize odours and quantify the intensity using an n-butanol reference scale. These panelists assess the intensity and hedonic tone of the sample. The hedonic tone refers to the degree of pleasantness or unpleasantness of the odour. To facilitate a comprehensive analysis, the panelists use a custom interactive odour wheel for categorization. This odour wheel allows for a structured and detailed characterization of the odour profile, enhancing the accuracy and depth of the evaluation.

Overall, the direct sensory evaluation of odour samples in this lab setting provides valuable insights that are not achievable through concentration measurement alone. By focusing on the nuanced aspects of odour perception, such as character and hedonic tone, this method enriches our understanding of how odours are experienced and interpreted. The evaluation of these parameters is done by using the scores in the following Table 1. The members of the panel are also asked to describe the odour in their own words and to attribute a certain predetermined odour class to the sample.

Table 1: Score scaling of odour intensity and odor (un)pleasantness

Odour intensity	Odour (un)pleasantness
Undetectable (0)	Neutral to pleasant (0)
Very weak (1)	Slightly unpleasant (-1)
Weak (2)	Unpleasant (-2)
Clear (3)	Very unpleasant (-3)
Strong (4)	Extremely unpleasant (-4)
Very strong (5)	
Extremely strong (6)	

The sensorial analysis is by preference performed on the same day as the sampling, with a maximum of 30 hours between sampling and analysis. After a longer time some compounds might react or diffuse through the sample bag which may cause differences in the odour character. (odour calibration in accordance to EN 13725).

2.3 GC-MS analysis

The sorbent tubes were analysed directly with the TD-GC-MS (UNITY-xr, Shimadzu GC 2010-plus and Shimadzu MS GP2010 SE). The analysis is based on several international standards (ISO-16006-6, ISO-16017, MDHS-72, TO-1). The sampled air is screened for the different volatile organic compounds present in the sample. A wide range of VOC can be measured in the lab, e.g. hydrocarbons (aromatic, aliphatic and cyclic) alcohols, ketones, aldehydes, esters, ethers, furans, organic acids, organic sulphur compounds (sulphides, thiophenes), chlorinated organic compounds and organic nitrogen compounds (amines, nitriles, pyridines). Using gas chromatography (Shimadzu GC 2010) the compounds are separated. Because it is impossible to calibrate every single VOC, a selection was made of the most common and odour related compounds.
For each compound, which is not calibrated, the following method is applied. First the identified compounds are divided in the groups (e.g. aromatic hydrocarbons, aldehydes, ketones, alcohols, organic acids) For each group about five compounds have been calibrated. Using the mean response factor of these five compounds, the concentration for other identified, but not calibrated compounds from the same group, is calculated.
As a result of the analyses, the composition in terms of specific compounds and their concentration was determined.

2.4 GC-Sniffing

For identification and quantification of the compounds a mass spectrometer is used (Shimadzu GCMS-QP 2010 Plus) to carry out very sensitive analyses. A well-trained human assessor is deployed as an additional detector and can sniff the eluate to detect the presence of odour active compounds through a specially designed odour gate (sniffing port) which is parallelly connected to a mass spectrometer. When an analysis is started, each individual compound, eluted by the GC, can be detected by a human evaluator (odour present or not), who is able to measure the start of odour activity, to describe the character of the odour perceived, and to quantify its intensity. The time of detection will be registered automatically by the audio software. In this way the sample is split between the detector and the human assessor thus allowing a comparison of both signals. Thus evaluation of odour compounds, but also their identification with mass spectral information is achieved (Hayes et al., 2023).
Each sample is measured three times by at least two different people to avoid, among other things, false positive perceptions as well as missing an odour. The GC-sniffing in this particular case was performed by 3 different evaluators. In this way, the scent perception of different people is addressed. This is essential, because scent is interpreted differently by everyone and is not an exact science. Each odour detected by an evaluator is given an odour description and intensity (1-6) with 1 being weak and 6 being extremely strong. It is expected that the peaks experienced with the highest intensity will contribute the most to the olfactory character of the sample.
In this way, the link is made between sensorial and chemical research. Using GC-Sniffing it is possible to indicate the most relevant odour compounds and to identify the cause of the odour nuisance. The collection of all the different odours gives an odour pattern which can then be used to indicate the specific source of the odour nuisance. The main objective of GC-Sniffing is to classify all compounds which have an important role in the general odour formation (Odournet, 2020).

3. Results and discussion

3.1 Sensorial analysis

The results of the sensorial examination are listed in Table 2 and Figure 1 and show that the sample from ambient air was almost undetectable, despite the fact that the air on site was perceived as strong. The on-site headspace sample taken from the carpet gave a different olfactory character than observed in the office. The results of the two headspaces made from pieces of carpet with and without adhesive showed very similar odour descriptions. They were sensorially the most correlated with the situation in the office. It is also important to note that the carpet tile itself did not really have a pungent odor, but when the carpet tile was cut so that the shape matched the piece of carpet with adhesive, a strong scent was released. A scent that is very similar to the smell present in the office.

Table 2: Description samples sensorial analysis

Sample	Description by panel members
Ambient air	musty
Carpet under plastic foil	musty, musty clothes and carpet
Headspace carpet	carpet, textile, chemical, glue and molten plastic
Headspace carpet + adhesive	carpet, textile, chemical, glue and molten plastic

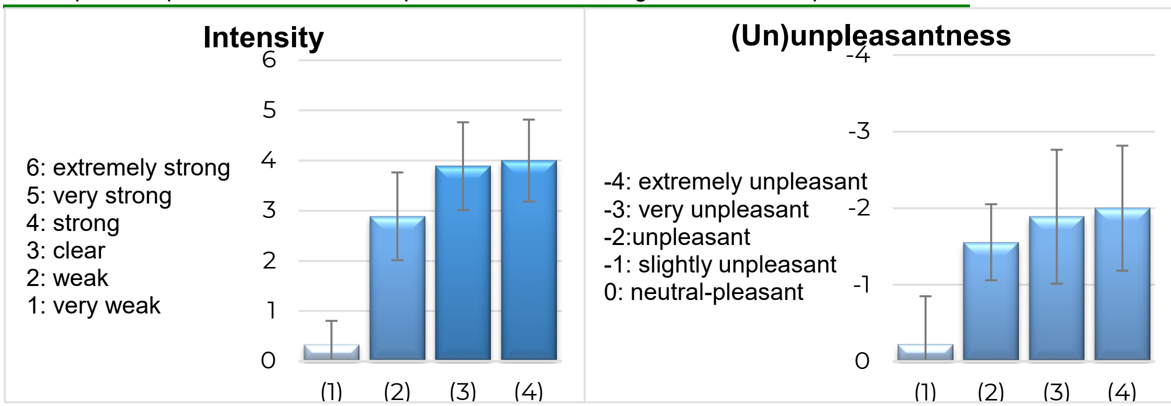

Figure 1: Results of intensity and (un)pleasantness sensorial analysis air samples (1) ambient air, (2) Carpet under plastic foil, (3) Headspace carpet and (4) Headspace carpet + adhesive.

3.2 GC-MS analysis

The summary results of the GC-MS analysis are listed Table 3. The compounds which an exceedance in odour threshold value were 2-butanon, 2-pentanon and 2-methyl-2-propenal. It is important to point out that not all compounds have a known odour threshold value. 4-phenylcyclohexene could not be found which indicates that the carpet is present in the office for a sufficient length of time since this compound is generally found in VOC emissions of newer carpets. GC sniffing is an aid to determine the odour threshold value of components of which there exists no odour threshold value in the literature. Some details of the comparison of the different chromatograms can be found in Figure 2. It can be clearly deduced that the components detected with the TD-GC-MS of the samples “ambient air” and “Carpet under plastic foil” were present in much smaller quantities. The same peaks were found in both samples of the headspaces, but the sample with the adhesive had generally higher peaks in the chromatogram so was chosen for further analysis via GC-sniffing.

Table 3: Total measured VOC concentrations based on the GC-MS analysis

Sample	Total measured VOC concentration (µg/m3)
Ambient air	324
Carpet under plastic foil	6,314
Headspace carpet	15,321
Headspace carpet + adhesive	17,802

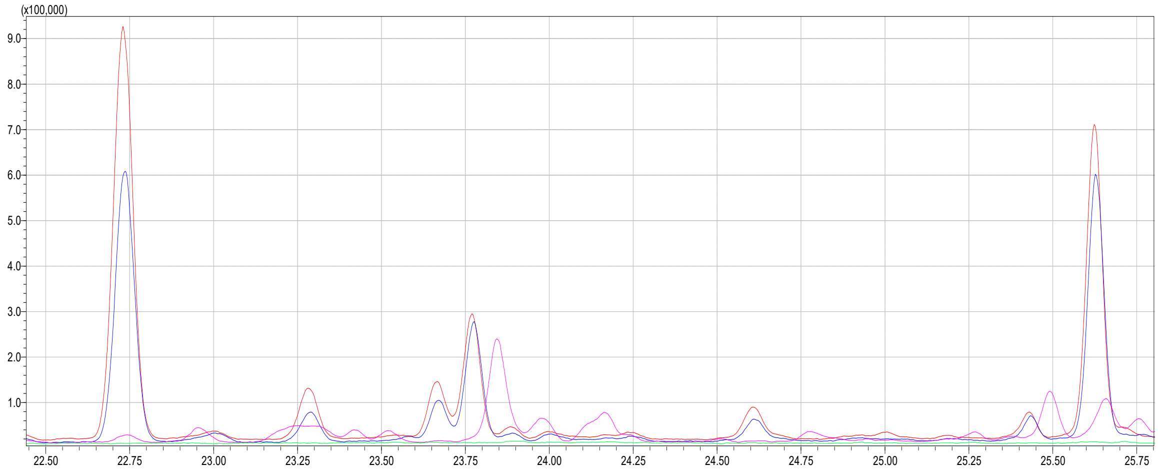

Figure 2: Zoomed-in part of the Chromatogram (retention time 22,5 – 25,75 min) of ambient air (green), carpet under plastic (pink), headspace carpet (blue) and headspace carpet + adhesive (red) with 3,4-dimethyl-2-pentanon (RT 22.73), 3-methyl-2-hexanon (RT 23.77) and a C10 alkane (RT 25.62).

3.3 GC-Sniffing

The components with the highest perceived intensity are listed in Table 4 with the assigned descriptions of the evaluators.

Table 4: Odour description of the detected components

Retention time (min)	Detected Component	Odour description by evaluators	Average intensity
2,33	acetaldehyde	molten plastic, sweet	1,5
6,27	2-methyl-2-propenal	fruity, musty	1
8,05	2-butanone	musty	1
11,93	3-methyl-2-butanone	burnt, musty, carpet, bitter, sweet, glue	2
19,47	3-penteen-2-one	solvent, burned, coal	1
21	3-pentanol	sharp, solvent, wood, grass, weed	2,5
21,47	4,4-dimethyl-2-pentanone	glue, amine, sharp	2,5
22,35	2-hexanone	solvent, chemical, hot plastic	1
24,3	C10 alkane	plastic, textile	1,5
24,47	3-ethyl-2-pentanone	hot plastic, vanilla	1,5
24,97	3-methyl-2-hexanone	chemical, solvent, sweaty feet	2
25,55	nonane	solvent, musty, hot plastic	1
27,87	hexanoic acid	melted, burnt plastic, musty, sharp	2,5
29,63	C11 alkene	polyethylene, burnt, greasy, plastic, mushrooms	3,5
30,98	2-ethyl-1-hexanol	solvent, chemical	3

There is a large amount of components that cause an odour that can be linked to the odour problem. The components 2-hexanon, 4,4-dimethylpentanon, 2-ethyl-1-hexanol and 3-methyl-2-hexanone had an odour that most closely matched the odour perceived in the office. Hodgson's article also refers to 2-ethyl-1-hexanol, acetaldehyde and different alkanes among others compounds as compounds that are linked to VOC emissions from carpet (Hodgson et al., 1993).There are also a number of components that create a smell that cannot be immediately linked to the smell present in the office, which shows that certain odours present are more strongly perceived, but the collection of all odours contributes to the overall olfactory character.

4. Conclusions

The research shows that ambient air can be assessed very strongly on site, but sampling this air does not always result in the expected results. This was confirmed in both the sensorial examination and the GCMS analysis by the low perception of smell and the small amount of peaks present in the chromatogram. Sensorial research appears to be a great help in making decisions about which samples are interesting to analyse further via GC-sniffing. The adhesive is not the biggest cause of odour, but certainly contributes to enhancing the odour present in the office. GC-sniffing is an interesting tool to link peaks to components that cause odour nuisance. Certain peaks in the chromatogram could be linked via GC-sniffing to the smell perceived in the office during the on-site sensorial examination and the perceived odour of the headspace samples. During the GC-sniffing a lot of different odour characters were observed from which only a few could be linked to the perceived odour in the room. Four compounds could be indicated to be dominating the overall odour character although other compounds will also add to it. Based on the analyses carried out, there can be concluded that the carpet certainly gives the most important contribution to the odour problem in the office. And especially if the carpet was also cut a number of times to cover the entirety of officefloor and the lower part of the wall. In this way, the smell of the glue is released a lot stronger.

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