Sensory Evaluation of consumer products by gas chromatography-olfactometry

Jennifer Bartsch 1, Nicole Schulz 1, Erik Uhde 1
Fraunhofer Institute WKI

Keywords: odour, perception, sensory, VOC

1 Introduction
Gas chromatography with olfactometric detection (GC-O) is a commonly used sensory evaluation technique typically done to determine odour-active and odour-inactive compounds in odorous mixtures, materials and products. Generally, the perception of the individual compounds can be completely different from the chemical composition of the mixtures and may result in a varying odour character. Sometimes the smell of the odour-active substances can even be masked or hidden by other fragrances.

This study describes the sensory evaluation of certain consumer products. Particularly product off-odours are of special interest. With the use of thermal desorption GC-O/FID the different odorous organic ingredients of the consumer products were detected by the human nose and ideally identified and compared with the results of GC-MS analysis. In some cases the concentration of the odour-active substances is too low (<1µg/m³) to be detected by routine emission analysis using GC/MS but can be measured by a sensitive detector, the human nose (Mayer and Breuer, 2006).

The odour character of the individual compounds was determined as a basic verbal description during analysis.

2 Materials/Methods
Several different consumer products (scented and unscented products) with considerable odour emissions were placed in a desiccator at room temperature. Around one hour later, air samples were collected using stainless steel desorption tubes packed with Tenax TA and analysed on a thermal desorption GC-O with flame ionization detection (FID). The analysis was done with a HP-5MS column (30m x 0,25mm; film thickness 0,25µm). After separation, the gas flow was split at the end of the GC column into two parts. The smaller portion flowed into a FID and the bigger one to an olfactory detection port (ODP).

To reduce subjectivity the sniffing on this port was done repeatedly by two trained persons. The run of the analysis was limited to about 30 minutes in order to avoid fatigue of the panelists. They marked the odour active substances individually by use of voice recognition software and described the odour quality and intensity with basic specifications as far as it was possible.

Subsequently, the identification of the resolved odorants was done with respect to the odour effect and the retention data of pure reference substances. The temperature-programmed retention indices of the numerous volatile organic (odorous) compounds were calculated before with standardized parameters in accordance to the van den Dool and Kratz equation (Van den Dool and Kratz, 1963). The same was done to estimate the retention indices of the odorous substances using n-alkanes as external references.

In addition, the characterization of the samples was performed by GC/MS to compare the results of the odour analysis.

3 Results and Discussion
As there is a great variety of consumer products with a diversity of odour-active substances, the GC-O analysis concentrated on differently scented (e.g. scented wood, candles) and unscented (e.g. mouse pad, bath duck, massage ball, wallpaper) products with a noticeable fragrance or even an off-odour. Moreover, typical substances that could be sniffed were categorized into substance groups to have an overview which compounds were relevant: alcohols, aldehydes (saturated and unsaturated), carboxylic acids, esters, ketones, naphthalenes,
sulphur-containing compounds, terpenes, other special odorants.

Generally, strong and unpleasant smells determined in some products correlated with the three following substance groups. The carboxylic acids like propanoic, butanoic, pentanoic, hexanoic, isoheptanoic acid, dimethyl butyric acid or isovaleric acid came up with a pungent, acidic, sweaty smell. Unsaturated aldehydes (e.g. heptenal, octenal, nonenal, decenal) had a fatty/green smell. In particular the identified sulphur-containing compounds (e.g. dimethyltrisulfide or carbon disulfide) with their strong cabbage-like and foul smell could lead to the off-odour of the product.

Figure 1 gives an excerpt of a GC-O/FID run with a scented candle that had a strong off-odour.

![GC-O/FID excerpt of a scented candle](image)

Some significant substance groups being present in the products and contributing to a similar obvious odour (mostly fruity, green-grassy and/or pungent) were the saturated aldehydes, esters and ketones. n-Hexanal was typically present in scented and unscented products. The ester compounds (e.g. ethyl-2-methyl butyrate, isopentyl acetate, 2-methyl butyl acetate, benzyl acetate) could be especially detected in high concentration levels in the scented products.

Typical odorous alcohols identified were the butanols with their characteristic smell. Others were, for example, cis-3-hexen-1-ol, 2-ethyl-1-hexanol or 1-methoxy-2-propanol.

Tetralin and the naphthalene compounds such as 2,6-dimethylnaphthalene, 1-methylnaphthalene, 2-methylnaphthalene and various naphthalene derivates could be detected particularly in elastomeric products (e.g. bath duck, mouse pad, massage ball) and lead to their characteristic odour.

Terpenes and terpenoids (especially limonene, alpha-pinene, myrcene, linalool) were typically found in high concentration levels in scented products like candles or scented wood which contains essential oils. Amongst others, different sesquiterpenes were detected being responsible for the main fragrances of these products.

Additionally, other odorants including substance groups and single substances occurred in the products. For instance, lactones (e.g. gamma-decalacton, gamma-undecalacton), phenols, terpineols, borneols, phenylpropenes (e.g. eugenol), nopol, linalylacetate, decaline were determined, mainly in the scented products.

4 Conclusions
It could be shown that the off-odour or unpleasant smell of different consumer products correlates with the presence of unsaturated aldehydes and carboxylic acids but also with sulphur-containing compounds. In contrast, various other substance groups can lead to the pleasant or unpleasant odour of the consumer products depending on the concentration, quantity of substances and the personal sensation.

5 References
Mayer F. and Breuer K. 2006. Material odors – odor active compounds identified in different materials the surprising similarities with certain foods, possible sources and hypotheses on their formation. Indoor Air, 16(5), 373–382.