Phenomena in GC-O Analysis – Training and Validation of a Sensory Panel

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Summary

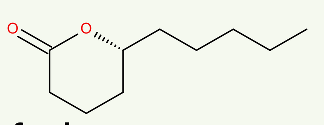
- A **training mixture of odorants** from different substances was used to **reveal factors** influencing **validation** in **GC-Olfactometry** (GC-O). Each panelist had to recognize the odorants and evaluate the perceived intensity in 7 dilution levels. Individual recognition thresholds have been determined for all substances.
- The **individual differences** of recognition thresholds among the panel participants varied from factor 16 for 2-pentylfuran to factor 4096 for 2-acetyl-pyrazine. This underlines the need for at least a small group of panelists for GC-O investigations.
- Within GC-O dilution analysis the decrease of intensity perception and of the share of recognition within the panel revealed to be **substance specific**. That demonstrates the **limitations** of the concept of **aroma extract dilution analysis** (AEDA).

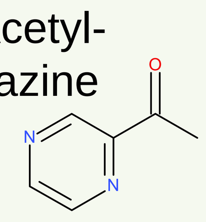
Introduction & Aim of the study

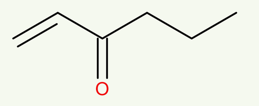
- The **GC-Olfactometry (GC-O)** method is not just relevant for the analysis of olfactory **active volatile organic substances (oVOC)** in food, but also for the analysis of odours and off-odours from food contact materials (FCM) and consumer goods.
- This study deals with the scientific presentation of factors influencing the validation in **GC-O analysis**, as well as the training of a sensory panel.

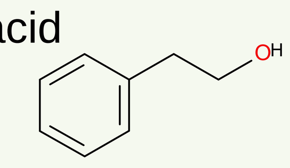
Methodology

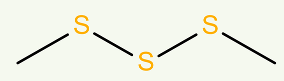
- In this study, a panel was **initially trained** for GC-O [1] and a **mixture of 10 oVOC** was developed for training purposes. The substances are relevant for both consumer goods and food [2] and represent **different structure types**. The mixture was successively **diluted by a factor of 4** in the test setup.

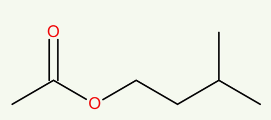
Dec
δ-Deca-lactone

fruity, coconut

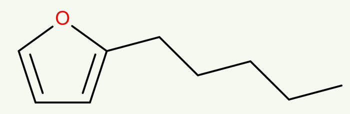
Ace
2-Acetyl-pyrazine

roasty, popcorn

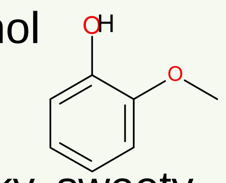
Hex
Hexen-3-one

burnt, plastics

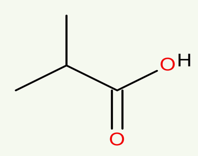
Phe
Phenylacetic acid

honey, soapy

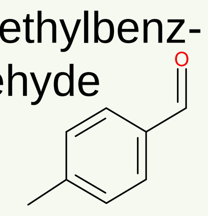
Dim
Dimethyl trisulphide

sulphurous, onion

Iso
Isoamyl acetate

banana

Pen
2-Pentylfuran

vegetative, fruity

Oxy
2-Methoxy-phenol

smoky, sweety

But
Isobutyric acid

cheesy, sweaty

Met
4-Methylbenz-aldehyde

marzipan-like

- The sensory panel consisted of **11 persons** (2 male, 9 female, aged from 23 to 61). Prior to each GC-O measurement, each panellist had to pass a short **ranking test** consisting of 3 different concentrations of 2-methylnaphthalene to check their olfactory ability due to nasal health.

Focus in investigation

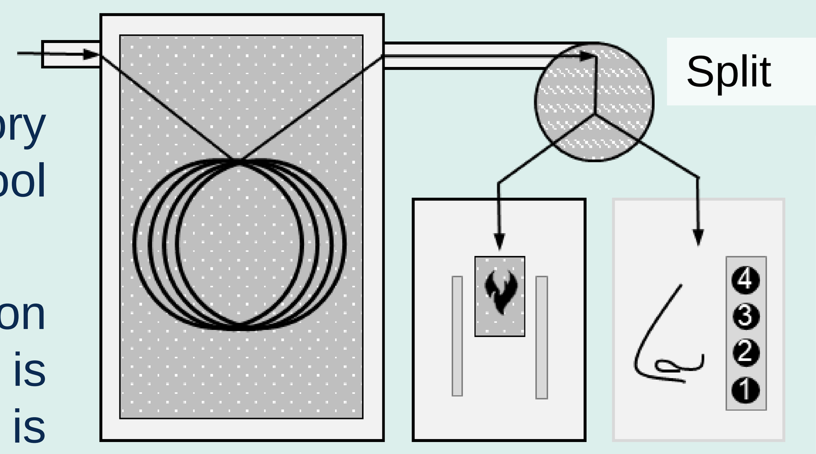
A **Sensitivity** human nose vs. device detector

B Substance specific dependence of **recognition** during **dilution analysis**

C **Variance** of sensitivity of panel participants for the substances due to individual differences

GC-FID/ODP in general

- The oVOC are analysed using a **GC-FID/ODP** (olfactory detection port). GC-MS is also used as a supporting tool e.g. to ensure the purity of the reference substances.
- The **intensity** of the odour impressions is evaluated on a **scale from 0 to 4**. A **description of the odour** is recorded via a headset as soon as an intensity button is pressed on the control panel.



Scale
4 intense odour
3 clear odour
2 recognition threshold
1 sensitivity threshold
0 no odour impression

Results

A Sensitivity human nose vs. device detector

- For all substances, the concentration was set that the **odour intensity** on the GC-O was rated as **3 on average** for the panel.
- However, the FID responses differed significantly.

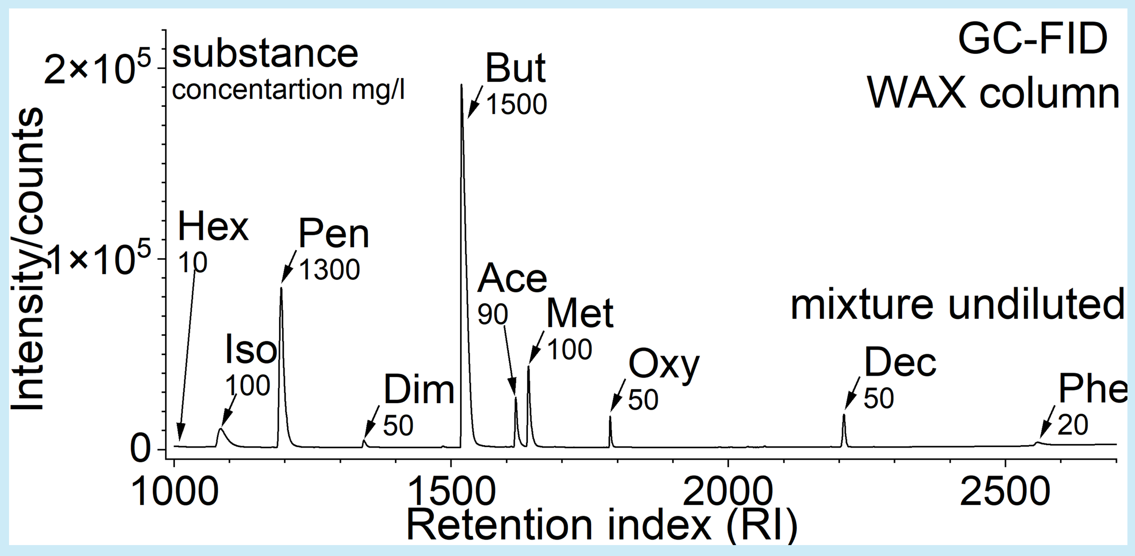


Fig. 1 **Chromatogram** of the tranings mixture (see [3] for displaying the RI on the abscissa)

- Not all VOC that are detectable in GC-FID/MS are also detectable in GC-O (become oVOC) (see *Fig. 1*). On the other hand the **human olfactory** sense is for some substances much **more sensitive** than the common GC detectors: A smell is perceived without a visible peak.

B Substance specific dependence of recognition during dilution analysis

- Different substances showing **different decreases** in recognition levels when diluted by the same factor (see *Fig. 2*). Some substances are no longer perceptible after just a few dilution steps (e.g. isoamyl acetate), while others remain perceptible over many dilution steps (e.g. phenylacetic acid). This reveals the **limitations of** the concept of aroma extract dilution analysis (**AEDA**).

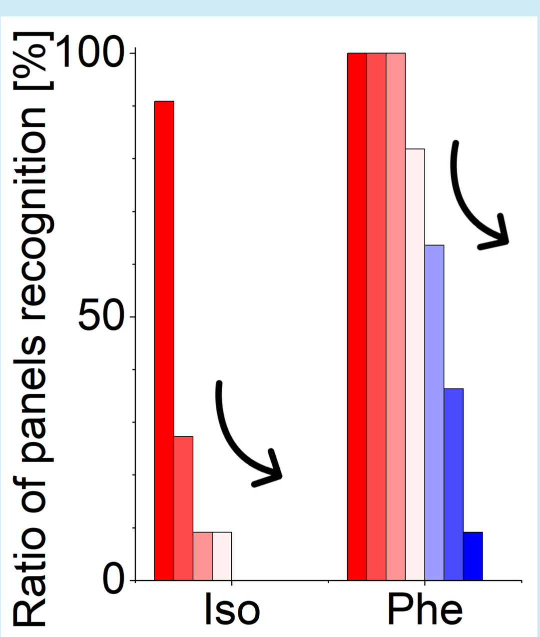


Fig. 2 **Concentration-responce realtionship** of selected substances, see *Fig. 3* for dilution steps

C Variance of sensitivity of panel participants for the substances due to individual differences

- The **heat map** shows the **dilution level** of the training mix at which a panel participant could **recognise** the given substance when sniffing the GC-O (see *Fig. 3*). It shows both **individual differences** between panellists and **substance-specific differences**.

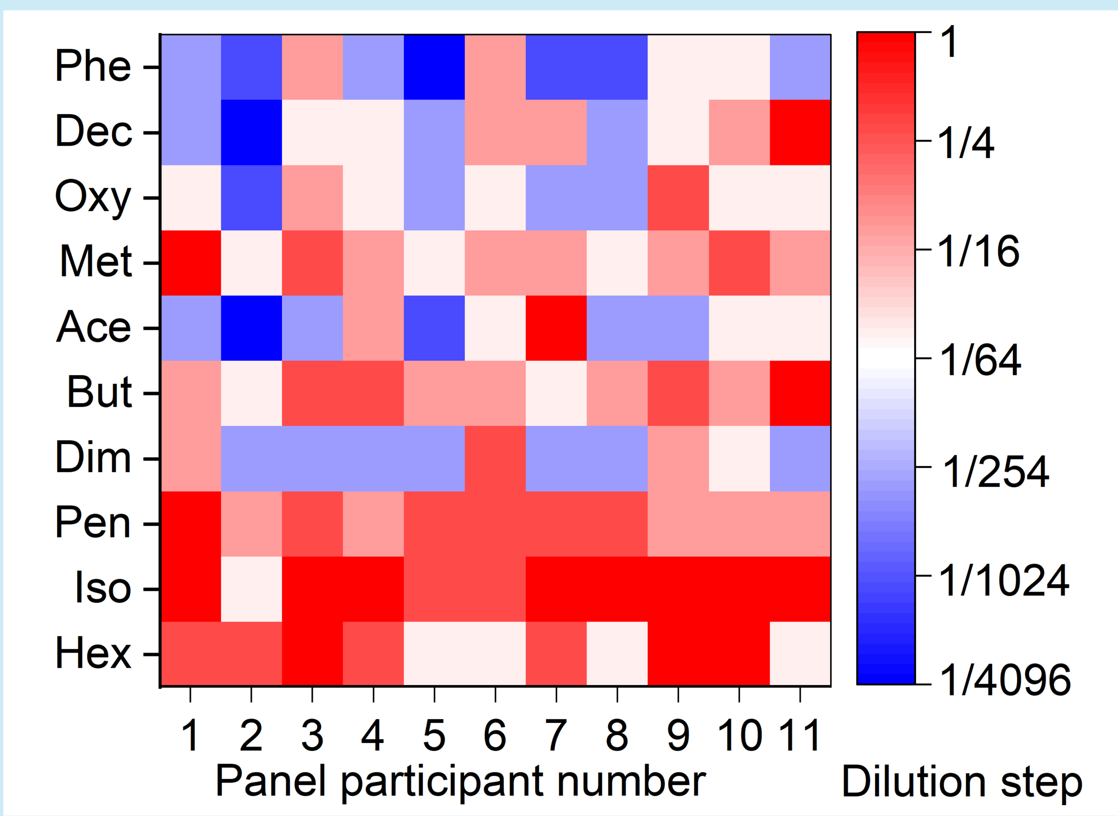


Fig. 3 **Heat map** showing dilution levels of the training mix at which the substances were recognisable on the GC-O

- *Fig. 4* visualizes sensitivity differences between panellists by comparing their individual recognition thresholds with the panel median.

Panellist 2 (P2) is for most odorants more sensitive than the panel median (→ **general sensitive type**).

P3 tends to be less sensitive than the panel median (→ **in-sensitive type**).

P7 reveals an average sensitivity except for 2-acetyl-pyrazine (Ace) (→ **specific in-sensitive type**).

P11 is **more or less sensitive** in relation to the panel median dependent on the individual substance (→ **variance type**). This type of variance occurred most frequently in this panel. The variance type was the most occurring type in this panel (6 out of 11).

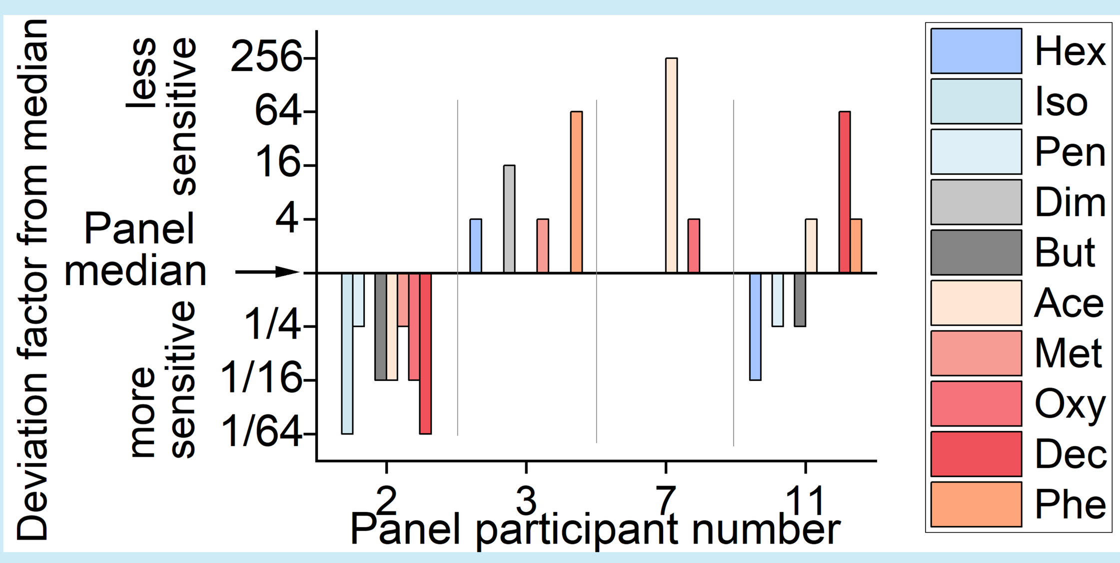


Fig. 4: **Differences** in individual sensitivities in recognition of substances from the panel median, exemplified by selected panellists

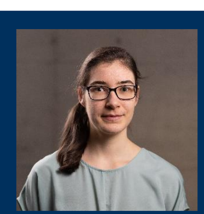
Conclusion

- Partly huge **individual differences in sensitivity** to specific substances occurred in the panel. In consequence, a panel of **at least three** persons is required for a **comprehensive analysis** of odorants in a sample. However, for identification of taints, participants can be **selected** based on the intensity of perception of the off-flavor directly from the sample in order to achieve a maximum homogeneity and sensitivity within the panel.

References:
[1] Vene et al. (**2013**) A Method for GC-Olfactometry Panel Training, *Springer*, Volume 6, page 179-189
[2] Hoffman et al. (**2014**) Natures Chemical Signatures in Human Olfaction: A Foodborne Perspective for Future Biotechnology, *Wiley VCH*, Volume 126, page 2–24
[3] L. Müller, J. M. Zimmermann, T. J. Simat (**2025**), Rt-to-RI Python-Tool [Software], https://github.com/mue-li/Rt-to-RI

GitHub Rt-to-RI

Link zum Poster



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