Evaluation of Odour Representativeness of HS-SPME Extracts of Fruit Yoghurt by Direct-GC-Olfactometry

K. BREME[a], H. Schlichtherle-Cerny[a], E. Eugster-Meier[a], A. Dubois[b], and M. Braun[b]

[a]Agroscope Liebefeld-Posieux Research Station ALP, Switzerland, [b]Nestlé PTC Konolfingen, Switzerland
Contact: katharina.breme@alp.admin.ch

Introduction

Headspace Solid-phase MicroExtraction (HS-SPME) sampling followed by GC separation is routinely used for the study of volatile odorant compounds in foodstuffs and dairy products [1, 2]. An **accurate representation of the sample's odour** is important for GC-Olfactometry (GC-O) studies. **Direct-GC-Olfactometry (D-GC-O)** consists in injecting an extract into a GC-O setup equipped with a deactivated capillary instead of a coated analytical column. The extract's global odour without chromatographic separation is smelled at the sniffing port and compared to the odour of the original sample [3−6]. The DVB/CAR/PDMS 50/30 µm fibre is suitable for the extraction of volatile and semi-volatile flavour and trace compounds [7], and it is often used due to its "universal" coating and because it is the only available 2 cm-fibre.

Objectives

- study of the suitability of the DVB/CAR/PDMS 50/30 µm fibre for dairy product analysis by GC-O
- investigation of the odour representativeness of a fruit yoghurt drink extracted with 1 cm-fibres:
  - CAR/PDMS 85 µm
  - DVB/CAR/PDMS 50/30 µm
  - PDMS 100 µm
  - Polyacrylate (PA) 85 µm

Direct-GC-O set-up

- **evaluation** of the **global odour** of the **SPME-extract compared to the original sample** by rating the **extract's similarity with the original product** on a scale from 0 (completely different) to 3 (identical) and its **odour intensity** (1 = weak, 2 = medium, 3 = strong)
- data processing with SYSTAT12®-software: general linear model

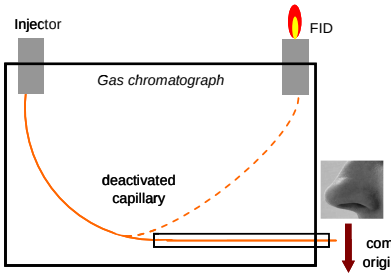


Fig. 1 D-GC-O setup
Absence of chromatographic separation was checked by directly connecting the capillary to the FID instead of the sniffing outlet - - - - (**Fig. 2** and **3**)

- commercial red berries yoghurt drink
- 10 g in 20 mL SPME-HS-vial
- sample conditioning: 10 min at 40°C
- $t_{adsorption}$ = 5 min
- all GC temperatures set at 240°C
- helium flow 12.7 mL/min
- seven trained panellists

Results

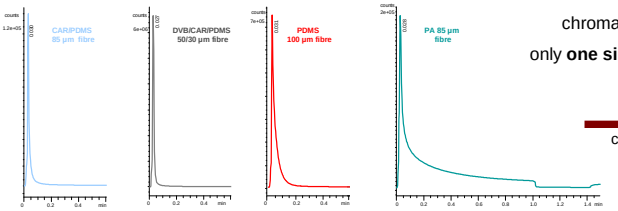
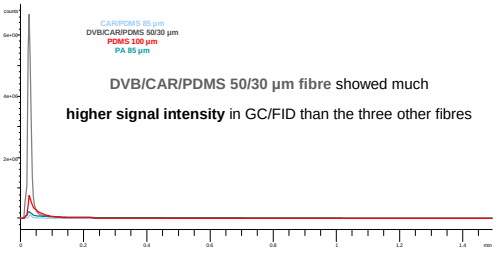


Fig. 2 GC/FID signals of red berries yoghurt drink headspace extracted with four different SPME fibres: no chromatographic separation

Verification of absence of chromatographic separation: only **one single signal by GC/FID**

overlay of chromatograms



DVB/CAR/PDMS 50/30 µm fibre showed much **higher signal intensity** in GC/FID than the three other fibres

Fig. 3 Overlaid GC/FID signals of red berries yoghurt drink headspace extracted with four different SPME fibres as presented in **Fig. 2**. The DVB/CAR/PDMS 50/30 µm fibre gives a much higher signal than the other three.

Four analyses per panellist and fibre are not statistically relevant, more analyses are needed!
Final number of **eight analyses per panellist and fibre** leads to **statistically relevant results**

Table 1. Resume of similarity results

	Fibre coating	Presentation order	Panellists (human factor)	Repetitions
Significant influence on similarity	**YES**	**YES**	**YES**	**NO**
Confidence interval	0.90	0.95	0.95	0.95
p-value	0.055	0.006	< 0.001	0.929
Conclusion	best choice DVB/CAR/PDMS 50/30 µm fibre	decrease	uncontrolled despite training	**good repeatability**

Table 2. Resume of intensity results

	Fibre coating	Presentation order	Panellists (human factor)	Repetitions
Significant influence on intensity	**YES**	**YES**	**NO**	**NO**
Confidence interval	0.95	0.95	0.95	0.95
p-value	< 0.001	0.039	0.248	0.198
Conclusion	best choice DVB/CAR/PDMS 50/30 µm fibre	decrease	no influence	**good repeatability**

The two repetitions were evaluated over six panellists.

Conclusion

It is highly useful to determine the most suitable fibre coating for odour compound analysis for each individual product prior to extensive GC-O analyses. For the studied product, the DVB/CAR/PDMS 50/3 µm fibre is the best choice in the employed conditions. Realising GC-O with a 2 cm-fibre should even increase intensity. The fibre coating, but also the order of presentation showed significant influence on similarity and intensity ratings. Since both decrease from the first to the last presented fibre, doing enough injections in order to analyse each fibre twice in each presentation position is crucial for finding the most suitable one.

Literature

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