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► **To cite this version:**

Angélique Villière, Gaëlle Arvisenet, Carole Prost, Thierry Serot. Assessment of ciders typicality characterisation through odorant volatile compounds. 12th International Weurman Flavour Research Symposium - Expression of Multidisciplinary Flavour Science, Jul 2008, Interlaken, Switzerland. hal-01943395

**HAL Id: hal-01943395**

**<https://hal.science/hal-01943395>**

Submitted on 13 Dec 2018

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# ASSESSMENT OF CIDERS TYPICALITY CHARACTERIZATION THROUGH ODORANT VOLATILE COMPOUNDS

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## **Abstract:**

This work intended to determine the compounds responsible for the typicality of ciders. In this aim, a method was developed to provide representative aromatic extracts of ciders. Then volatile compounds responsible for the odorant perception of three different ciders were determined by gas chromatography/olfactometry/mass spectrometry (GC/O/MS).

## **Introduction:**

Apple cider is an alcoholic beverage obtained by fermentation of apple must. Unless volatile compounds of apple and apple juice have been extensively studied over the last decades, less information is available on odorant volatile compounds of apple cider. Gas chromatography/olfactometry (GC/O) analysis is a powerful way to determine key compounds of food aroma on condition that the odor of the extract is close to that of the food submitted to extraction. This similarity in odor is not obvious since different classes of compounds are preferentially extracted according to the method chosen. Therefore prior to GC/O analysis, a comparison of the odor of the food with the corresponding extract should be made. The objective of this work was to develop a representative extraction method to analyse volatile compounds of ciders and to examine those responsible for the odorant perception of different ciders. Because of the undesirable odor of solvent and odors developed when heating to eliminate this solvent, only solvent-free extraction methods were tested.

## **Experimental:**

### *Chemicals and materials*

A commercial cider purchased in a supermarket was used for the development of a representative extraction method. Study of aroma compounds was conducted on three ciders selected among several according to their different organoleptic properties: a sweet cider from Normandy, a brut cider from the *pays d'Auge* and a pear cider. These ciders were verified to be perceived as different by discriminating tests performed with a panel of 36 naïve judges. Ciders were stored at  $-80\text{ }^{\circ}\text{C}$  until analyses in 125 mL brown flasks sealed with Teflon/rubber screw caps. Prior analyses, vials were put at  $37.5\text{ }^{\circ}\text{C}$  for 1h. Chemical standards ( $p\geq 98\%$ ) used for mass spectra identification of odorant compounds were purchased from Aldrich (St Quentin Fallavier, France).

### *Choice of a representative extraction method*

For all extractions, 20 mL of cider were sampled except when otherwise mentioned. For static headspace extraction, vials (60 mL) containing the cider were sealed using Teflon/Silicone septa plastic caps (Varian, France). After equilibration at  $37.5\text{ }^{\circ}\text{C}$  for

1 h, a 250  $\mu$ L sample of headspace was withdrawn using gastight syringe and injected in the gas chromatograph (GC).

Headspace SPME (HS SPME) extractions were conducted with DVB/CAR/PDMS, CAR/PDMS and PDMS fibres (Supelco, Inc., Bellefonte, PA). The sample in a 60 mL vial was equilibrated at 37.5°C in a thermostatic bath for 30 min, and then the fibre was introduced in the headspace for 10 min. Sample was maintained under stirring. The fibre was withdrawn and inserted into the injection port of the GC. Same fibres and procedure were used for aroma extraction by liquid SPME except that 60 mL of cider were placed in the 60 mL vial and the fibre was immersed in the cider.

Headspace purge and trap was conducted with a concentrator (model LSC 2000; Teckmar Inc., Cincinnati, OH) equipped with a capillary interface, for cryofocusing, connected to the GC. Cider was introduced into a flask (50 mL) containing a stir bar. Temperature was maintained at 37.5 °C with a heating ring and the headspace of the sample was purged with helium during 5 min at 60 mL.min<sup>-1</sup>. Volatile compounds extracted were swept into a Tenax trap at room temperature. After trap desorption, volatiles were cryofocused at -40 °C using carbon dioxide and thermally desorbed at 200 °C for 2 min to be transferred into the column. Same procedure was applied for bubbling purge and trap except that 60 mL of cider was introduced in the flask and the sample was bubbled with helium during 5 min.

#### *Recovery of the extracts and evaluation of the extracts representativeness*

Extracts were analyzed on a Varian Star 3400 GC (Varian, Palo Alto, CA) equipped with a DB Wax column (30 m length, 0.32 mm internal diameter, 0.5  $\mu$ m film thickness, J&W Scientific, Folsom, CA). Detector temperature was set at 260°C and oven temperature program was set from 50 °C (5 min) to 150 °C at 5 °C.min<sup>-1</sup> and from 150 °C to 250 °C (7 min) at 10 °C.min<sup>-1</sup>. Helium was used as carrier gas at 2 mL.min<sup>-1</sup> flow rate. The GC effluent was split 1:1 between the FID (T: 250 °C), to control the chromatographic pattern of the extract, and a 100mL glass syringe<sup>1</sup>.

Twelve trained judges were asked to evaluate, on a 10 cm-scale, the proximity between the odour of each extract collected in the syringes and the original cider odour (representativeness). The reference (right anchor of the scale) corresponded to 0.05 mL of the original cider in a syringe. A hidden reference, containing 0.05 mL of the original cider, was also presented with the extracts. All syringes were covered with aluminium foil paper to avoid judges to know if they contained cider or gaseous extracts. Results are the means of the 12 judges' evaluations.

#### *GC/O/MS analysis of 3 discriminated ciders*

The GC/O system consisted of a 6890N GC (Hewlett-Packard Co., Palo Alto, CA) equipped with a FID, a mass detector (5973-Network), and a sniffing port ODP2 (Gerstel, Baltimore, MD). The GC effluent was split 1:1:1 between the FID, the mass detector and the sniffing port.

Extracts obtained from the three ciders were injected into a DB Wax column (30 m length, 0.32 mm internal diameter, 0.5  $\mu$ m film thickness, J&W Scientific) and volatile compounds were separated according to chromatographic conditions similar to those described above except for flow rate of helium (3 mL.min<sup>-1</sup>) and oven temperature program: 50 °C (2 min) to 80 °C at 3 °C.min<sup>-1</sup>, from 80 °C to 105 °C at 5 °C.min<sup>-1</sup> and from 105 °C to 240 °C (1 min) at 10 °C.min<sup>-1</sup>. Mass spectra were recorded in electron impact mode (70 eV) between 33 and 300  $m/z$  mass range at a scan rate of 2.7 scan.sec<sup>-1</sup>. Compounds identification was based on a comparison of mass spectra with those of MS spectra database (Wiley 6) and of standard molecules injected in the same conditions. Coincidence of odour description by judges and

literature data could be used to confirm identification of odorant compounds. Olfactometry was conducted with 10 trained judges. GC effluent was carried to the sniffing port using deactivated capillary column heated at 200 °C and supplied with humidified air at 40 °C. Judges were asked to assign odor descriptor to each odorant area detected. Results were expressed in frequency detection and odorant areas that were not perceived by at least 3 judges were not taken into account.

## Results:

### *Representativeness of the extracts*

Representativeness scores of the extracts obtained by the different methods ranged from 2.5/10 for the extracts obtained by liquid SPME with a PDMS fibre, to 6.5/10 for those obtained by HS SPME with a CAR/PDMS fibre (Table 1).

**Table 1.** Representativeness mean scores of the extracts - Letters represented results of multiple comparison of means test.

Extraction method	Representativeness mean score
Liquid SPME PDMS fibre	2.5 <sup>e</sup>
Headspace purge and trap	2.9 <sup>de</sup>
Bubbling purge and trap	3.8 <sup>cde</sup>
Liquid SPME Car/PDMS fibre	3.9 <sup>cde</sup>
Liquid SPME DVB/Car/PDMS fibre	4.4 <sup>cde</sup>
Headspace SPME DVB/CAR/PDMS fibre	4.5 <sup>cd</sup>
Headspace SPME PDMS fibre	4.9 <sup>bc</sup>
Headspace SPME CAR/PDMS fibre	6.5 <sup>ab</sup>
Hidden reference	8.5 <sup>a</sup>

The extract obtained by HS SPME with a CAR/PDMS fibre was the most representative of the original cider odour. Multiple comparisons of means showed that, contrary to other extracts, no significant difference was perceived between the odour of this extract and the odour of the hidden reference. Thus, these extraction conditions were applied to investigate the aroma typicality of discriminated ciders.

### *Olfactometric analyses of 3 discriminated ciders*

More than 100 volatiles were detected in the ciders whereas only 50 were detected in previous studies<sup>2,3</sup>. A majority of them was esters (~60) and alcohols (~20).

43 odorant areas (OA) were pointed by judges in the sweet cider from Normandy and 24 in the pays d'Auge and pear ciders (Figure 1). These OA were either associated with a single volatile compound or with several coeluted volatile compounds.

15 OA, which were among the most potent, were common to the 3 ciders (numbered 1 to 15) however their frequency of detection differ from a cider to another. 7 OA were associated to esters (1, 3-5, 7, 10, 13), and were described with fruity or sweet notes. The others were described as floral, green, earthy or metallic.

Few OA were characteristic of one cider. Six were found in the sweet cider from Normandy. There were associated with ethanal (fruity), 1-pentanol (fruity, sweet), heptyl acetate (metallic, earthy), hexyl butanoate (cocoa) and hexyl hexanoate (sweet) and one OA (rancid) could not be associated with a chromatographic peak. Two OA were detected only in extract from the pear cider and were associated to ethyl crotonate (sweet) and  $\beta$ -citronellol (floral, fruity). No specific OA was found in the cider from the pays d'Auge.

These results suggest that both OA specific to a cider and the proportions of major OA common to all ciders, were involved in the ciders odorant characteristics.

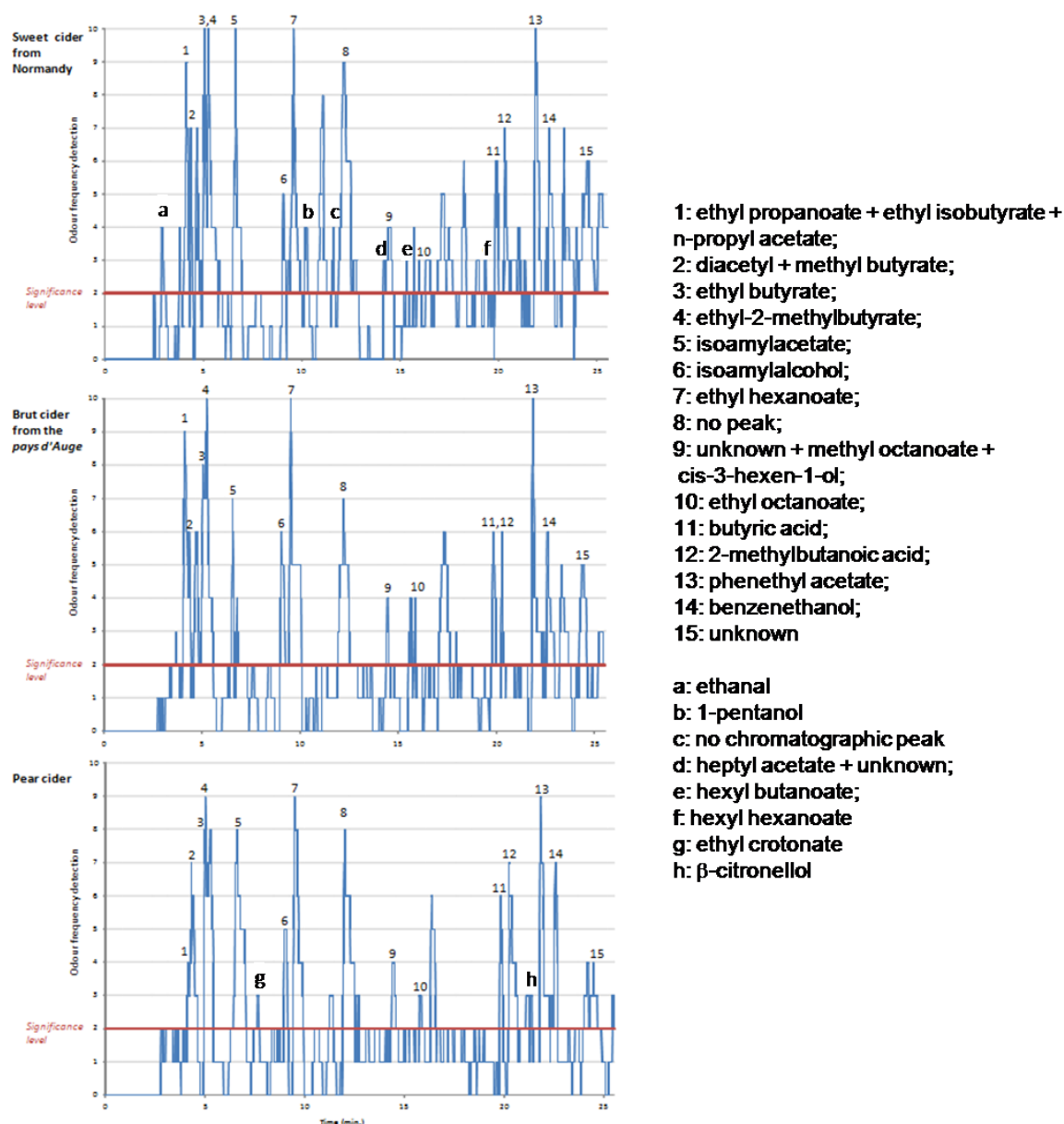


Figure 1. Olfactometric pattern of 3 discriminated ciders: a sweet cider from Normandy, a brut cider from the pays d'Auge and a pear cider.

## Conclusion

These results provide elements to understand the typicality of ciders. Further studies, with a larger selection of ciders, will now be necessary to confirm if these aromatic profiles can explain ciders odorant typicality. Then a connection with the soil and the know-how of a region would be interesting to investigate.

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