Odour Port Dilution Assay for the Determination of the Aroma-Impact Components of Ciders

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SUMMARY

- Headspace extracts of cider volatiles were prepared and analysed by GC-MS to establish the identity of the major components
- Extracts were run by odour-port GC and the aroma descriptors recorded by three assessors
- Progressive serial dilutions of the extracts were re-assessed by odour-port GC until no more odour could be detected
- From this exercise, a relative ranking of the odour impact of individual cider volatiles could be established
- The most odour-active component was shown to be 2-methyl 4-pentyl 1,3-dioxane, the condensation product of acetaldehyde with 1,3 octanediol

Extraction of Cider Volatiles

A commercial UK cider (50 ml) was equilibrated at 35ºC and the headspace was sampled for 30 mins in a closed loop stripping apparatus using a 1.5 mg (Grob) charcoal trap. The extract was desorbed with 4 x 15µl of carbon disulphide.

GC-MS identification of cider volatiles

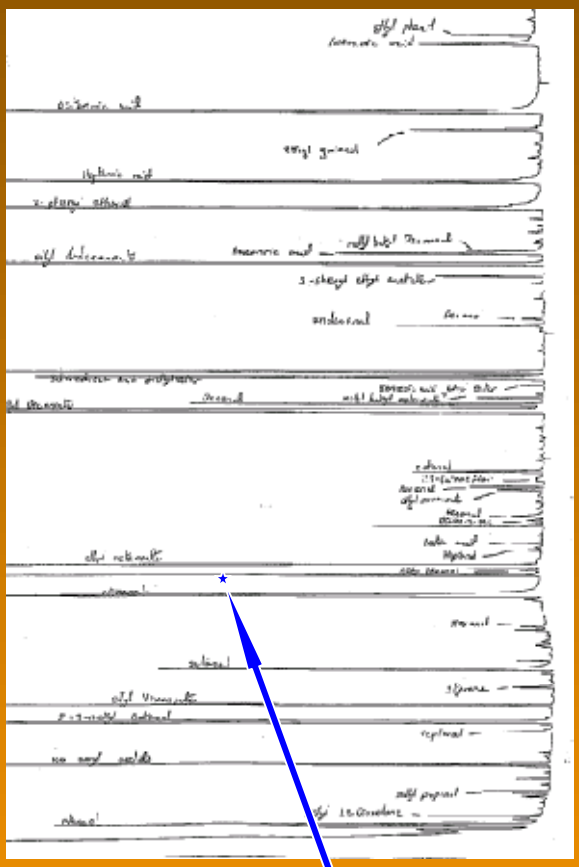
Separation of extracts (1 µl) was carried out on on a fused silica capillary coated with the polar phase OV 351 (50m x 0.32mm), temperature programmed from 55º - 200º at 2º/min. Peaks were identified using a VG 7070 MS in the EI mode.

Odour Port Analysis

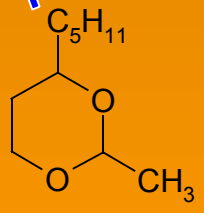
Separation of extracts (2 µl) was carried out on a polar (CP-Wax) 52CP column (25m x 0.53 mm) temperature programmed from 30º - 200º at 4º/min. The effluent was split to an FID detector and to a home-made odour-port assembly. Odour descriptors for each peak were recorded independently by three separate assessors.

Odour Port Dilution Analysis

The extracts were serially diluted 1:3 in carbon disulphide in six separate steps (final dilution 1:729). At each step an odour-port analysis was carried out to record the dilution at which the odour for each peak was no longer detectable. From this data the relative odour-impact of each component was calculated, at its natural concentration in the headspace of the cider under study.



Component	Descriptors	Relative Odour Value	
2-methyl 4-pentyl 1,3-dioxane	Green, sweet, fruity, cidery	1	
Ethyl 2-methyl butyrate	Banana, fruity, estery, fresh	3	High
2-phenyl ethanol	Roses, floral	3	
Octanal	Fatty, green, metallic	9	
Nonanal	Fatty, green, metallic, waxy	9	
Ethyl hexanoate	Banana, estery, fruity	9	
Iso-amyl acetate	Pear drops, estery	27	
2 and 3-methyl butanol	Fusel, green, grassy	27	
Eugenol + 4-ethyl phenol	Cloves, spicy	27	
2-phenyl ethyl acetate	Perfumed, floral, 'winey'	81	Low
Ethyl decanoate	Weakly fatty, leafy	> 243	
Ethyl octanoate, dodecanoate Hexanal, heptanal Hexanol, octanol Decanal, decenal Methyl butyl octanoate Diethyl succinate Heptanoic, octanoic, nonanoic acids 4-ethyl guaiacol	No detectable aroma on GC odour-port assessment	> 243	



2-methyl 4-pentyl 1,3-dioxane is the condensation product between acetaldehyde (from fermentation) and 1,3 octanediol (which occurs naturally in apples). It was reported at up to 24 mg/l in French cider by Dietrich et al. J. Agric Fd Chem 1997 **45** 3178 – 3182. *At the time our work was carried out (1989) the structure of this compound was still unknown.*