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Characterization of Beer Samples, Using SPME/Capillary GC Analysis

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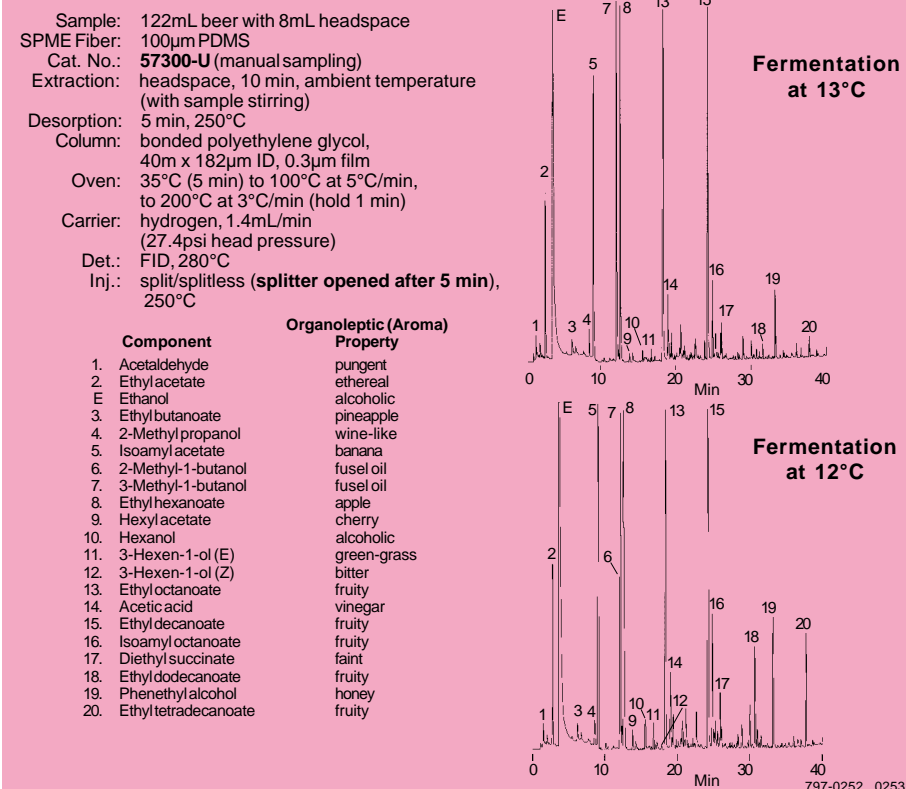
Solid phase microextraction (SPME)* is a new technique for concentrating samples prior to analysis. Its main advantages are that it is very simple, requires little sample manipulation, and is very fast. SPME is a solvent-free technique that can be used either for headspace analysis or for direct extraction of analytes from liquids. SPME with capillary GC or GC-MS has been reported previously for the analysis of wine aromas (*Reporter* 15, No. 5). Some important fragrance compounds, like ethyl esters and alcohols, can be enriched selectively by SPME, depending on the type of extraction fiber used.

Headspace GC-MS is an excellent technique for aroma characterization: it is selective, sensitive, fast, simple, and relatively inexpensive. Under the experimental conditions employed, detection limits for some components, using headspace extraction, were in the low ng/L level (ethyl octanoate, ethyl decanoate, β -phenethyl alcohol) or low ppb level (ethyl acetate, alcohols). Needless to say, this technique can be used for aroma characterization not only of wines, but of spices, fruits, and other food products as well. The purpose of the present work is to demonstrate the utility of SPME coupled to GC analysis for characterizing beer aromas.

Capillary gas chromatography (CGC) has been the main instrumental method in flavor analysis of beer. The development of more selective and sensitive analytical methods will make it possible to detect additional minor compounds in beer. The effect of minor compounds on the overall flavor of the product cannot always be demonstrated. However, a human measuring instrument, the flavor panel, will always maintain its position in the determination of the overall flavor and palatability of the beer.

In the beer industry the headspace GC technique is used in beer quality control, as a tool with a two-fold function. First, it has a watch-dog function, in that variations and differences in peak height/shape and appearance of new small peaks in the chromatograms act as warning signals for possible defects, or as a technology fingerprint at a point of time in the processing stage before substandard organoleptic quality occurs. Second, headspace chromatograms serve as

Figure A. Beer Samples by Headspace SPME/CGC



reference tools for small-scale fermentation with a pure yeast culture every time a pure culture must be tested. This can be considered a link to quality control. Examples of problems detected with headspace GC are:

- significant increases of ethyl formate, acetone, and/or methanol in cases of contamination caused by inadequately disinfected tanks or leaks in cooling systems
- an abnormal increase in ethyl hexanoate, seemingly related to yeast and fermentation disfunctions
- appearance of ethyl lactate indicates malolactic fermentation or addition of lactic acid to the base material

A decrease in heavy esters, such as ethyl octanoate, ethyl decanoate, ethyl dodecanoate, and ethyl tetradecanoate, is a fingerprint of higher fermentation temperature (Figure A).

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Ordering Information:

Description	Cat. No.
SPME Holder**	
For manual sampling	57330-U
For Varian 8100/8200 AutoSampler	57331
SPME Fiber Assemblies	
100 μ m polydimethylsiloxane, pk. of 3	
For manual sampling	57300-U
For Varian 8100/8200 AutoSampler	57301
SUPELCOWAX™ 10 Capillary Column	
40m x 0.20mm ID, 0.3 μ m film	
prepared on request	

*Technology licensed exclusively to Supelco. US patent #5,691,206; European patent #0523092.

**Initially you must order both holder and fiber assembly. Holder is reusable indefinitely. Use with AutoSampler requires Varian SPME upgrade kit (available from Varian).

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Fused silica columns manufactured under HP US Pat. No. 4,293,415.