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Fast Screening of Wines Using SPME/GC

G. Vas, Research Institute for Viticulture & Enology of Agricultural Ministry, Eger, Hungary

The ability to accurately determine the origin of a wine or detect the presence of additives is important to the wine industry. A new method of analysis was tested, consisting of sampling by headspace SPME and separation by GC using a PAG capillary GC column. This method characterized aroma patterns of two muscat-type wines from different vineyards, and revealed the presence of low concentrations of cold-pressed coriander seed oil in wine and distilled water. The resulting chromatograms showed noticeable differences between the two wines and were used to detect a 1ppm spike of additive.

Aroma compounds of wines originate from grapes and are formed during fermentation. Their production is influenced by various factors: environment (soil, climate), grape variety, ripeness, fermentation conditions (pH, temperature, yeast flora), the wine production process (enological methods, treatment substances), and aging (bottle maturation). Wines contain aroma compounds in a wide concentration range — some components at high ppm level, but most at low ppb or ppt levels. The low concentration of most volatile components in wine makes extraction and concentration necessary before analysis by gas chromatography (GC) or gas chromatography/mass spectrometry (GC/MS). The purpose of this study was to characterize aromas of muscat-type wines and to detect extraneous flavor addition, using the solid phase microextraction (SPME)/GC sample preparation and analysis method. Two wines from the same muscat grape variety — originating from the Eger wine region, Hungary, and the Trento wine region, Italy — were analyzed.

SPME■ requires no solvents or complicated apparatus. It can be used to concentrate volatile and nonvolatile compounds in both liquid and gaseous samples. An SPME unit consists of a length of fused silica fiber coated with a phase film. The fiber is attached to a stainless steel plunger in a protective holder.

A 100µm PDMS (polydimethylsiloxane) SPME fiber was exposed to the headspace of the wine for 10 minutes at ambient temperature. After sampling, the fiber was retracted into the SPME needle, then inserted into a GC injection port and exposed for 5 minutes to desorb the analytes of interest.

Figure A. Comparison of Muscat Wines by SPME/GC

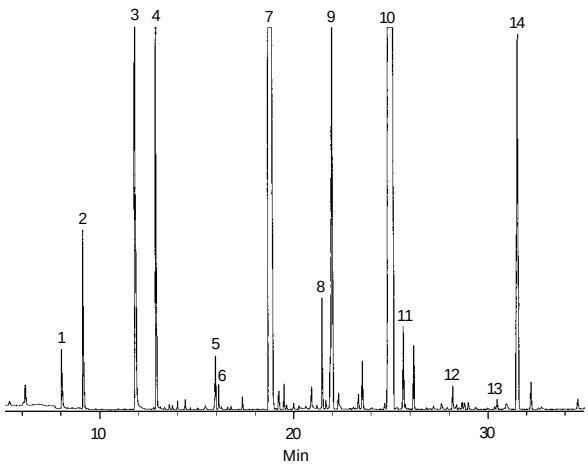
Sample: 125mL
SPME Fiber: **100µm polydimethylsiloxane film**
Cat. No.: **57300-U**
Extraction: 125mL, 10 min headspace (stirring)
Desorption: 5 min

Column: **PAG, 30m x 0.25mm ID, 0.25µm film**
Cat. No.: **24223**
Oven: 35°C (5 min)
5°C/min to 100°C (0 min)
3°C/min to 200°C (1 min)
10°C/min to 220°C (0 min)
Carrier: hydrogen, 45cm/sec
Det.: FID, 250°C
Inj.: 5 min, splitless, 250°C

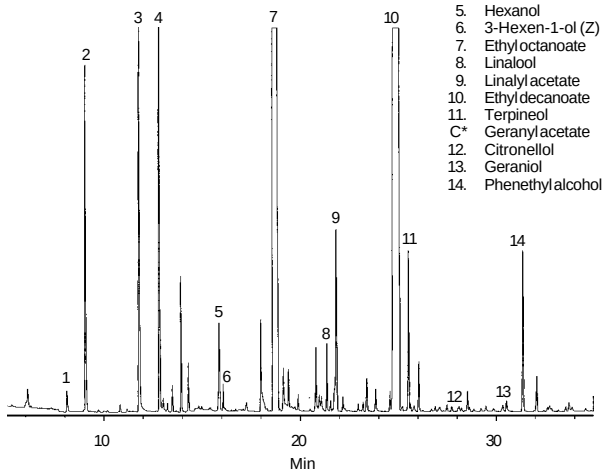
Callouts for Figures A-C

	Compound	Retention Index
1.	Isobutanol	1044
2.	Isoamyl acetate	1079
3.	3-Methyl-1-butanol	1158
4.	Ethyl hexanoate	1190
A*	Myrcene	1201
B*	Limonene	1215
5.	Hexanol	1299
6.	3-Hexen-1-ol (Z)	1302
7.	Ethyl octanoate	1390
8.	Linalool	1474
9.	Linalyl acetate	1486
10.	Ethyl decanoate	1588
11.	Terpineol	1619
C*	Geranyl acetate	1684
12.	Citronellol	1695
13.	Geraniol	1763
14.	Phenethyl alcohol	1802

Eger Region (Hungary)



Trento Region (Italy)



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*Peaks A, B, and C are characteristic of cold-pressed coriander seed oil (see Figures B and C).

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Figure B. Muscat Wine from Eger Region Spiked with 1ppm Cold-Pressed Coriander Seed Oil

Conditions and callouts: see Figure A.

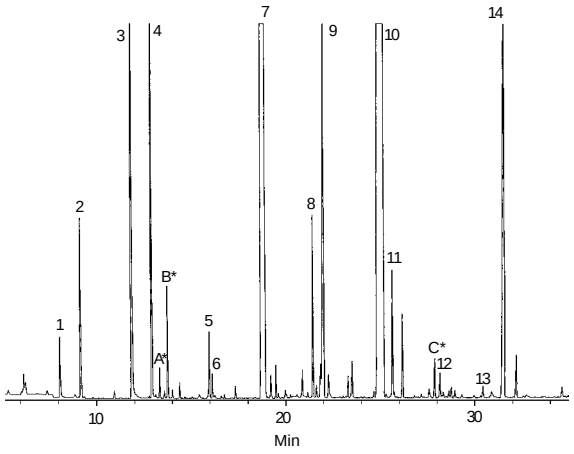
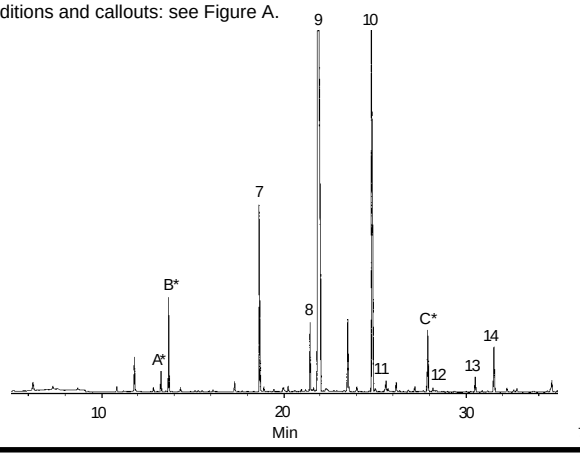


Figure C. Distilled Water Spiked with 1ppm Cold-Pressed Coriander Seed Oil

Conditions and callouts: see Figure A.



A polyalkylene glycol (PAG) capillary GC column separated the analytes. The PAG phase has similar characteristics to the polyethylene glycol (PEG) phase, but retention indices are somewhat different. This can be advantageous for separating components that are not well resolved on a PEG column. Our results show that the PAG column is suitable for headspace SPME wine aroma characterization. Chromatograms of the two muscat wine samples show significant, characteristic differences between the respective wine regions (Figure A). This distinction is of major importance in wine analysis, providing a basis for analytical determination of the origin of a wine sample.

Using some additives (such as cold-pressed coriander seed oil) in wines is considered adulteration in many countries. Cold-pressed coriander seed oil is occasionally added to increase the muscat flavor of wine. The oil is typically used in the 10 to 20ppm concentration range — enologists easily recognize it by taste in this range. At lower concentrations it may still add a slight muscat flavor, but is difficult to identify by taste. Sampling by headspace SPME with GC analysis, using a PAG column, can easily identify such adulteration. Figure B shows wine from the Eger region spiked with 1 ppm cold-pressed coriander seed oil. The peaks marked with an asterisk (*) are characteristic of this oil, but normally are found only in small amounts in wine. The chromatogram of pure, cold-pressed coriander seed oil in distilled water, studied under identical conditions, is shown in Figure C.

The headspace SPME sampling technique, in combination with GC analysis, easily distinguishes muscat wines of different origins and detects small concentrations of cold-pressed coriander seed oil additive. Use of this efficient and accurate sample preparation method will benefit analysts trying to ascertain or prove the origin of wines and the use of additives in wine.

Ordering Information:

Description	Cat. No.
SPME Fiber, 100µm polydimethylsiloxane, pk. of 3	
For manual sampling	**57300-U**
For Varian 8100/8200 AutoSampler or HPLC	**57301**
SPME Holder■■	
For manual sampling	**57330-U**
For Varian 8100/8200 AutoSampler or HPLC	**57331**
PAG Fused Silica Capillary Column	
30m x 0.25mm ID, 0.25µm film	**24223**

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

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

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

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