Analysis of Aroma Compounds in White Wines Using Solid Phase Micro Extraction

Application Note

Food/Flavor

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Abstract:

Every wine has a distinct aroma profile. The major components in the aroma profiles can be very similar; however it is the subtleties that can make all the difference. The seemingly small components in the aroma profile of a wine can often have a large impact on the overall flavor. There are an enormous variety of wine brands, flavors, etc… This application note will focus on three types of white wine and their respective aroma profiles using Headspace Solid Phase Micro Extraction sampling.

Introduction:

Solid Phase Micro Extraction (SPME) consists of a fiber with an extraction phase being exposed to a sample for a set time, temperature, and agitation. The efficiency of the extraction is based on how quickly the analytes in the sample come to equilibrium with the fiber coating. The most effective SPME coating varies with the sample matrix, the sampling technique and the analytes of interest. In this case, the best fiber extraction coating for the wine matrix was found to be a 50/30μm Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS).

This application will compare Chardonnay, Pinot Grigio and Sauvignon Blanc for the similarities and differences in their respective aroma profiles. Using headspace SPME in conjunction with Gas Chromatography/Mass Spectrometry (GC/MS), the aroma compounds will be sampled, separated and analyzed.

Experimental:

The FLEX Autosampler was used in order to automate the SPME extraction process. The FLEX was coupled to an Agilent 7890 GC configured with a Restek Rxi-624 Sil MS 30m x 0.250mm x 1.4μm column for compound separation and a 5975 MS was employed for aroma analysis. As stated earlier, a 50/30μm DVB/CAR/PDMS was chosen for the extraction phase of the SPME fiber.

The ease of the FLEX drag and drop method development software allowed sampling parameters to be enhanced in order to finalize the most efficient sampling and analysis parameters. Ultimately, it was found that one gram of sodium chloride and five milliliters of wine sealed in 20ml headspace vials along with the sampling and analysis parameters listed in Table 1 and 2 were the optimum parameters for this analysis.

Autosampler	FLEX
General	
Method Type	SPME
GC Ready	Continue
GC Cycle Time	25min
Constant Heat Mode	Yes
Sample Incubate Agitate	
Incubation Temp.	40°C
Incubation Time	5.1min
Agitation Speed	100%
Agitation Delay	0.1min
Agitation Duration	5.0min
Extraction	
Fiber Guide Depth	50%
Sample Vial Fiber Depth	1cm
Extraction Time	60min
Agitate	No
Wait	
Wait on Input	Yes
Wait Input	GC Ready
Desorbtion	
Injection Port	A
Fiber Guide Speed	20%
Fiber Guide Depth	60%
Fiber Insertion Speed	50
Fiber Insertion Depth	1cm
Fiber Desorbtion Time	3min
Injection Start Output	Start

Table 1: Flex Autosampler Experimental Parameters

GC/MS	Agilent 7890/5975
Inlet	Split/Splitless
Inlet Temp.	290°C
Inlet Head Pressure	11.905 psi
Mode	Pulsed Splitless
Injection Pulse Pressure	30psi until 3.0min
Purge Flow to Split Vent	10ml/min at 3.01min
Desorbtion	3min at 290°C
Column	Rxi-624 Sil MS 30m x 0.25mm I.D. 1.4μm film thickness
Oven Temp. Program	40°C hold for 2.0 min., ramp 10°C/min to 220°C, hold for 1min, total run time 21 min
Column Flow Rate	1.0ml/min
Gas	Helium
Total Flow	14.0ml/min
Source Temp.	230°C
Quad Temp.	150°C
MS Transfer Line Temp.	180°C
Scan Range	m/z 31-265
Scans	3.07 scans/sec
Solvent Delay	0.7 min.

Table 2: GC/MS Experimental Parameters

The three wines were purchased from a local market. Each wine was sampled and analyzed four times in order to ensure reproducibility of the results. The compound responses for each wine were then averaged in order to best represent the complete aroma profile. Finally, the individual compound responses were divided by the overall compound response in order to see the percent of each flavor compound in the respective wine class. A chromatogram of each wine is displayed in Figures 1, 2, and 3. Figure 4 shows the aroma profile as a percentage of each aroma in graph form. Finally, Table 3 lists the percent of each aroma versus the wine type.

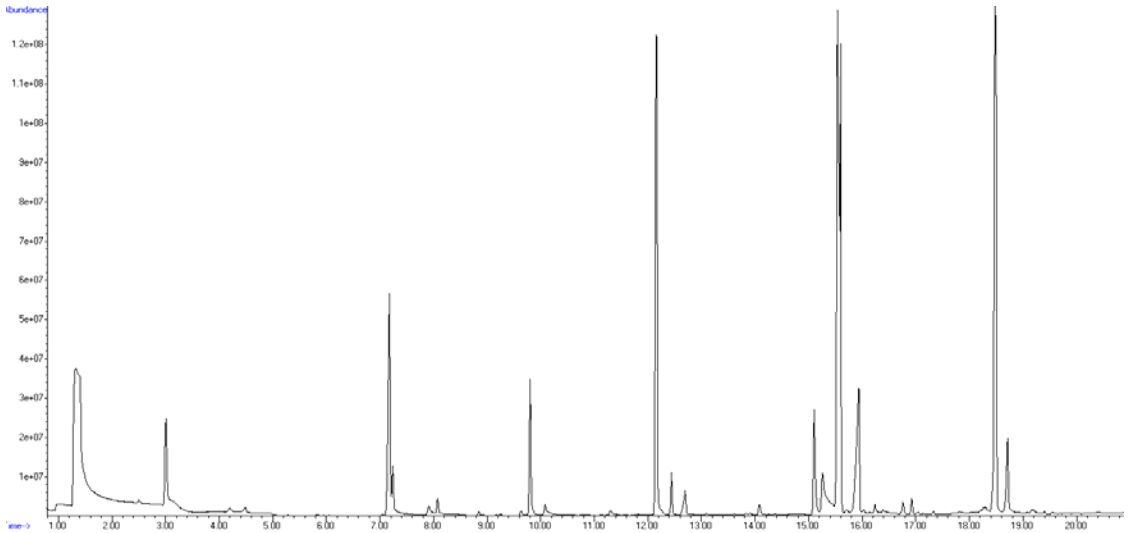

Figure 1: Chromatogram of Pinot Grigio

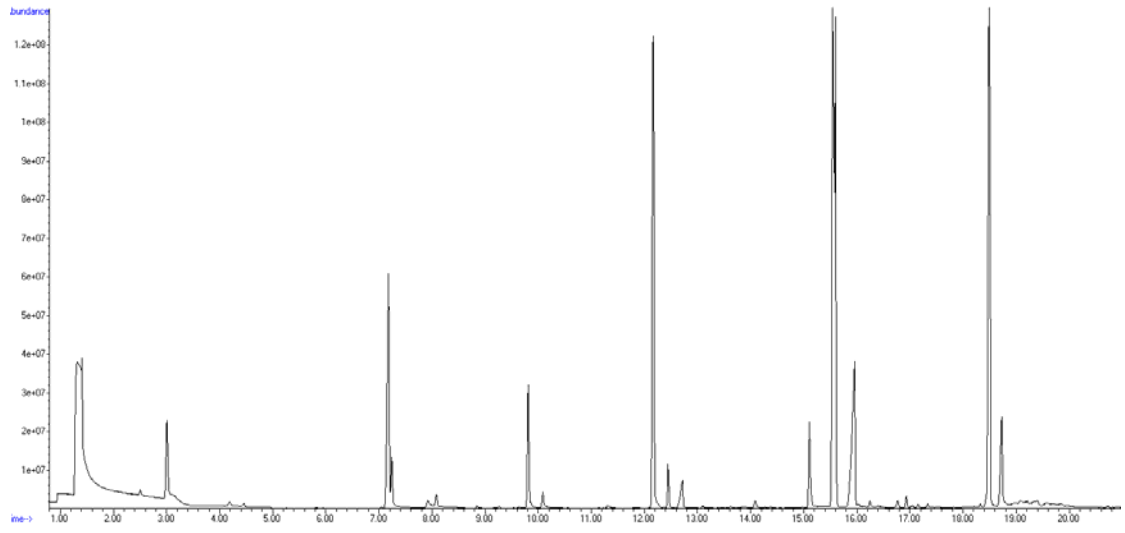

Figure 2: Chromatogram of Sauvignon Blanc

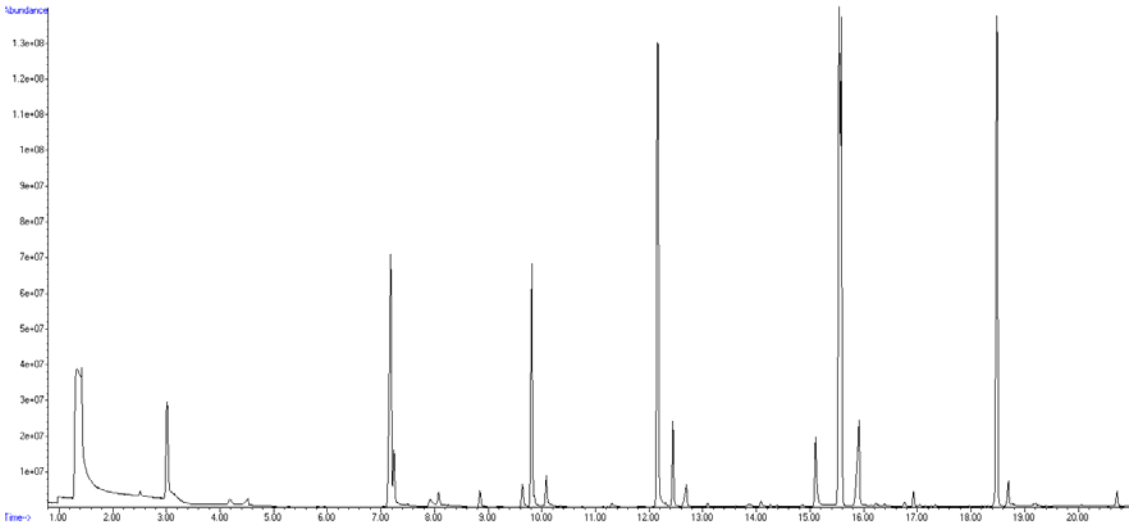

Figure 3: Chromatogram of Chardonnay

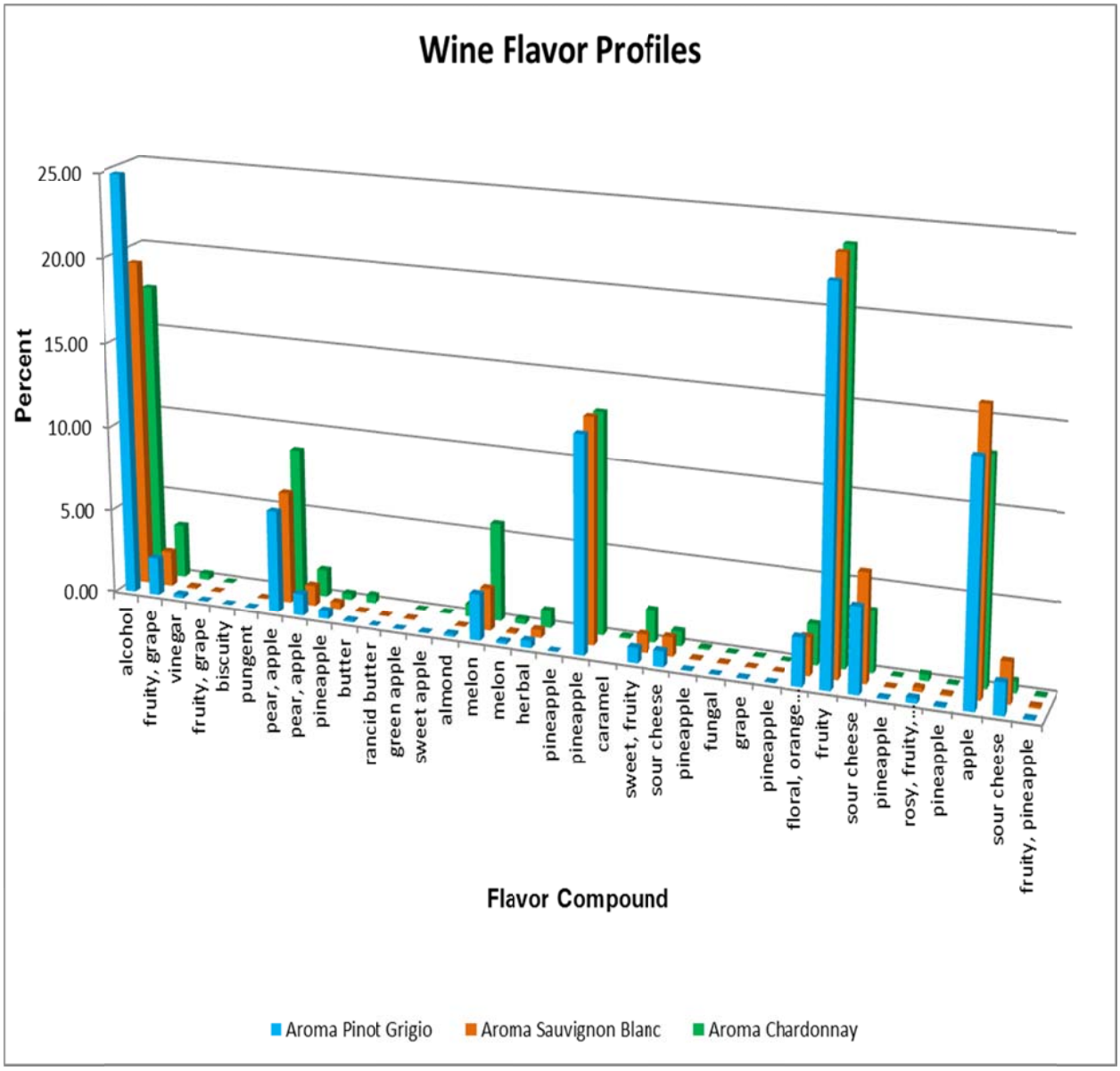

Figure 4: Wine Aroma Profile Graph

Compound	Aroma	Compound Percent		
		Pinot Grigio	Sauvignon Blanc	Chardonnay
ethanol	alcohol	24.86	19.34	17.50
ethyl Acetate	fruity, grape	2.22	2.07	3.15
acetic acid	vinegar	0.20	0.11	0.35
ethyl propionate	fruity, grape	0.02	0.03	0.03
acetal	biscuity	0.01		
isobutyric Acid	acid smell, pungent	0.03	0.04	0.03
isoamyl alcohol (fusel oil)	pear, apple	6.02	6.60	8.68
amyl alcohol (fusel oil)	pear, apple	1.28	1.21	1.61
ethyl butyrate	pineapple	0.46	0.40	0.36
ethyl lactate	butter	0.10	0.07	0.46
butyric acid	rancid butter	0.02	0.02	
ethyl-2-methyl butyrate	green apple	0.02	0.06	0.01
ethyl isovalerate	sweet apple	0.04		0.04
furfural	almond	0.15	0.05	0.75
isoamyl acetate	melon	2.75	2.51	5.76
amyl acetate	melon	0.15	0.12	0.28
1-hexanol	herbal	0.42	0.46	1.01
methyl hexanoate	pineapple	0.01		
ethyl caproate	pineapple	12.72	13.23	13.03
5-methyl furfural	caramel			0.10
acetic acid, hexyl ester	sweet, fruity	0.89	1.06	1.90
carproic acid	sour cheese	0.92	1.12	0.87
ethyl hexanoate	pineapple	0.04	0.07	0.10
methyl 2-furoate	fungal	0.02	0.06	0.03
ethyl caprylate	grape	0.04	0.04	0.04
butyl caproate	pineapple	0.01	0.01	0.01
phenylethyl alcohol	floral, orange blossom	2.80	2.22	2.36
ethyl caprylate	fruity	22.47	23.61	23.67
caprylic acid	sour cheese	4.98	6.33	3.55
pentyl caproate	pineapple	0.06	0.04	0.04
2-phenethyl-acetate	rosy, fruity, honey like	0.29	0.24	0.36
ethyl-3hydroxyhexanoate	pineapple	0.07	0.09	0.05
capric acid ethylester	apple	13.97	16.30	13.12
capric acid	sour cheese	1.87	2.39	0.68
isoamyloctanoate	fruity, pineapple	0.07	0.12	0.07

Table 3: Aroma Percent Summary

Conclusions:

The three types of wine had many similarities in their respective flavor profiles. For some of the aroma compounds such as ethyl caproate, phenyl ethyl alcohol and ethyl caprylate, the aroma percentages were very similar. For others, such as capric acid ethyl ester, isoamyl acetate and isoamyl alcohol the percentages varied with the wine type. Overall, the differences in the wines could be discerned in the small percentages of the aroma compounds found in some and not in others or analytes observed in a larger proportion in one of the wines versus another. SPME proved to be a powerful tool in examining aroma compounds in wine. The FLEX automated the entire extraction process from the method development to the final sampling making the examination of the wine flavors a simple reliable process.

References:

1. Pawliszyn, Janusz. *Handbook of Solid Phase Microextraction.* P.R. China: Chemical Industry Press of China, 2009
2. Cannavan, Tom. *Wine-pages*, 21 Aug. 2014, http://www.wine-pages.com/

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