BEYOND MEASURE

SCION INSTRUMENTS

Volatile Profiling in Red Wine using ChromSync

Application Note AN0032

INTRODUCTION

With a wide variety of wines available and the consumer market so large, it is vital that wineries test and monitor the volatile compounds, that contribute to the flavour profile, during the production process to ensure that the same flavours are consistently achieved. The volatile compounds that make up the flavour composition must therefore be profiled batch to batch. Strict controls are operated during the production of wine as varying levels of volatiles can vastly affect the flavour of the final product.

Gas chromatography (GC) is often the instrumentation of choice for the analysis of flavour active volatiles in wine. Compass Chromatography Data System (CDS) is a state of the art chromatography software platform that controls GC instruments whilst offering automated processing and reporting of results. ChromSync is an application add on specifically for the flavour and fragrance industry. Chromsync has the ability to determine the 'fingerprint' of volatile compounds in wine. The individual 'fingerprints' are then compared with a reference standard. ChromSync rapidly compares peak retention time as well as area% profiles of complex chromatograms, making processing volatile flavour profiles effortless. Additionally, ChromSync instantly confirms product batch to batch reproducibility whilst reporting any missing compounds and calculating the degrees of similarity.

This application note demonstrated the ease of using ChromSync with CompassCDS for the comparison of six Shiraz red wine samples analysed via headspace (HS) gas chromatography (GC) with flame ionisation detection (FID) and mass spectrometry (MS) for identification of volatile compounds. All samples were made from the same shiraz grape in order to identify both similar and varied volatiles in the wine.

EXPERIMENTAL

A SCION 456 GC-FID was coupled with the Teledyne Tekmar HT3 headspace autosampler. The same 456 GC was also coupled to a SCION single quad (SQ) mass spectrometer for peak identity confirmation. Six commercially available Shiraz red wines were prepared in 20mL HS vials; 5mL of each sample was added with 3g of NaCl. One of the red wine samples was used as the reference point for the ChromSync software. The samples were thoroughly mixed, ready for injection onto the analytical system. The analytical parameters used can be found in Tables 1 and 2.

The five beers analysed were labelled as Reference Sample, Sample A, Sample B, Sample C, Sample D and Sample E.

Table 1. Analytical conditions of the Teledyne Tekmar HT3 HS autosampler

Conditions	
Oven	80°C
Sample Loop	80°C
Transfer Line	100°C
Vial Equib Time	20 minutes
Fill Pressure	15psi at 50mL/min
Inject Time	0.5 minutes

Table 2. Analytical conditions of the SCION GC/FID/MS

Conditions	
Injector	S/SL 25:1, 200°C
Column	SCION-Wax 30m x 0.25mm x 0.25µm
Oven	40°C (3 mins), 10°C/min to 120°C , 20°C/min to 200°C
Carrier	Helium, 2mL/min
FID	250°C
Transfer Line	200°C
Source	250°C
Full Scan	40m/z to 350m/z

RESULTS

Mass Spec Work Station, the software used to control the GC-MS and acquire/report data, was used in order confirm the volatile identities obtained during the analysis. Using a library search of mass spectra (NIST) it was possible to confirm the individual profiles of each sample analysed. Figure 1 details the chromatogram of the GC-FID analysis whilst Figure 2 details the GC-MS chromatogram complete with peak identification. Peak identification numbers are representative to both chromatograms, which is the reference wine sample.

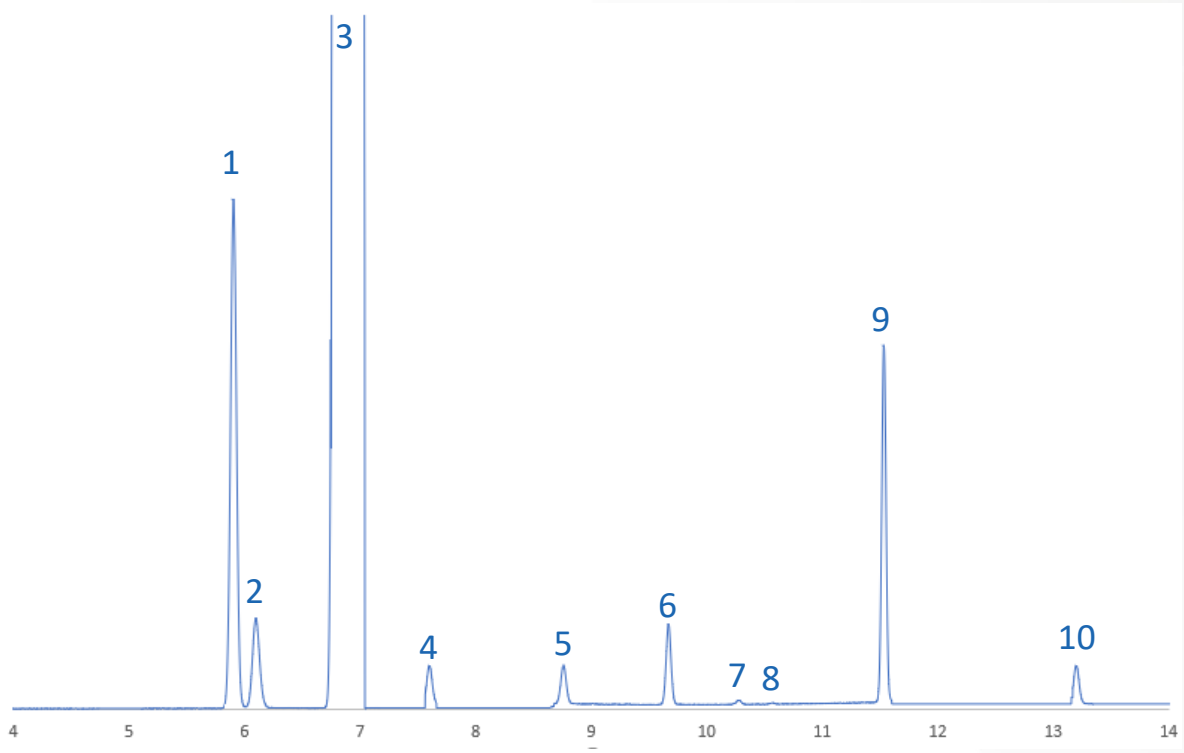

Fig 1. Reference sample: FID

Peak Number	ID
1	Acetic Anhydride
2	Carbohydrazide
3	Ethanol
4	Methylamine
5	1-propanol
6	Isobutane
7	1,4-butanediol
8	3-buten-1-ol
9	1-nitro-propane
10	Methyl nitrate

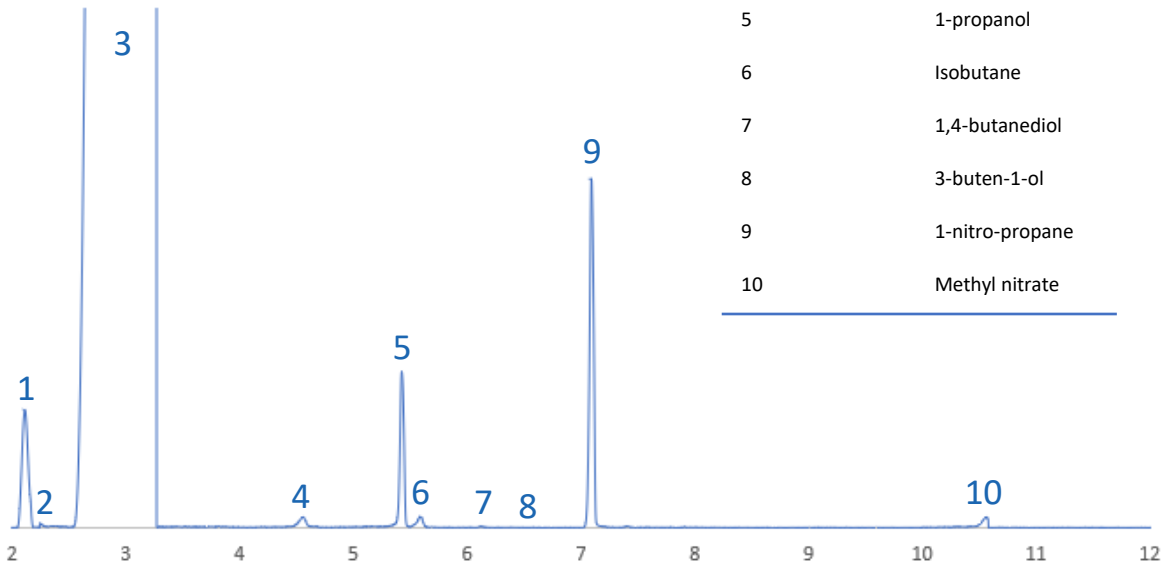

Fig 2. Reference sample: MS

Figure 3 details the overlay chromatogram of all five wine samples plus the reference sample. Figure 3 highlights the importance of ChromSync for comparing individual components across two chromatograms, as this gives a more detailed comparison profile.

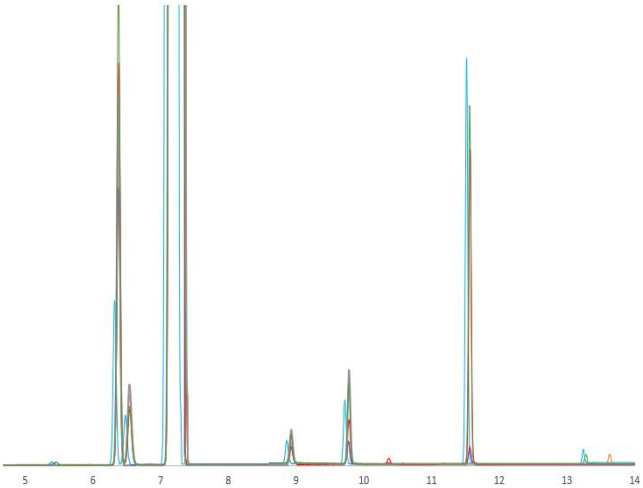

Fig 3. Sample and reference chromatogram overlay

Through CompassCDS it was possible to set the reference sample in ChromSync in which was used to compare the various wine samples. Utilising ChromSync eliminated the need for manual comparisons.ChromSync automatically determined tolerance levels for peaks set in the reference sample, however, users can set their own acceptable levels based upon their method requirements. Once the comparisons have taken place, users can easily identify similarities/differences between the samples. Table 3 details the results from the comparisons of the reference sample with all five wine samples.

ChromSync makes visualising all similarities and differences between the samples easy; not only between the different volatile compounds but the peak % that makes up each sample. ChromSync accurately performs comparisons between samples regardless of if there are issues surrounding peak distortion, scaling, column ageing, retention time shifts and even changes in experimental conditions.

Table 3 shows that there are a lot of volatile compounds that constitute the flavour profile of each wine. However, the amount of each volatile in the samples varies between the different wines. The higher priced wines; wine 4 and 5, have additional volatiles that are not present in the cheaper wines. Components such as acetaldehyde and methyl nitrate are produced during the fermentation process of wine, which indicates that the production process of the higher priced wines are different to those of the lower end products.

CONLUSION

ChromSync is an application add on for CompassCDS processing software. Chromsync allows easy comparisons between a reference sample and different analytical samples; making it the perfect tool for the flavour and fragrance industry. Monitoring batch to batch variances and highlighting any changes within the batches makes data processing of complex flavour chromatograms easy with ChromSync.

Table 3. Peak area % of each volatile, in each wine sample and reference sample

Peak Name	RT	Ref Sample (area %)	Wine 1 (area %)	Wine 2 (area %)	Wine 3 (area %)	Wine 4 (area %)	Wine 5 (area %)
Acetic Anhydride	5.90	0.41	0.30	0.42	0.34	0.26	0.24
Carbohydrazide	6.10	0.01	0.10	-	-	0.04	-
Methylglyoxal	6.48	-	-	-	-	0.10	0.10
Formic Acid	6.54	-	-	0.1	0.09	-	-
Ethanol	6.96	99.2	99.55	99.17	99.50	98.99	98.89
Methyl Amine	7.54	0.01	-	0.01	-	-	-
1-propanol	8.76	0.03	0.02	0.03	0.02	0.04	0.03
Isobutane	9.67	0.06	0.02	0.04	0.03	0.10	0.10
1,4-butanediol	10.28	0.006	-	-	-	-	-
3-buten-1-ol	10.57	0.07	-	-	-	-	-
2-nitro-propane	11.53	0.20	0.01	0.26	0.02	-	-
Methyl Nitrate	13.28	0.004	-	-	-	-	0.05
Acetaldehyde	13.68	-	-	-	-	0.47	0.59