

Changes in the concentration of volatile oak compounds and esters in red wine stored for 18 months in re-used French oak barrels

T. GARDE CERDÁN, D. TORREA GOÑI and C. ANCÍN AZPILICUETA¹

Department of Applied Chemistry, Universidad Pública de Navarra, Campus de Arrosadía s/n, E-31006 Pamplona, Spain

¹ Corresponding author: Dr Carmen Ancín, facsimile +34 948 169 606, email ancin@unavarra.es

Abstract

Studies were made of changes in concentration of oak-wood-derived volatiles and the evolution of esters in red wine during storage in twice-used French oak barrels. Wine samples were taken after 8, 10, 12, 15, and 18 months maturation in the barrels. Results showed that most of the volatile compounds extracted from the wood (furanic compounds, volatile phenols, lactones) reached maximum concentration between 10 and 12 months of barrel storage. After 18 months of maturation many of the compounds showed concentrations similar to those found after 10 to 12 months. However, the concentrations of furfural, 5-methyl furfural, furfuryl alcohol, coniferaldehyde, acetovanillone and phenol in wines aged for 18 months were lower than those reached after 10 to 12 months. The concentration of the ethylphenols increased right up to 18 months of ageing, which can only have a negative impact on the quality of the wine. There were few modifications in the concentration of esters, except for ethyl lactate which reached peak concentration after 12 months maturation and decreased thereafter.

Keywords: maturation, wines, oak-wood, volatile composition, extraction, re-used barrels

Introduction

Wine stored in contact with oak-wood undergoes important modifications. Since the wood of an oak barrel is a porous material, barrel storage allows a wine to undergo processes associated with so-called 'low oxidation conditions' (Vivas and Glories 1993). Wood also confers on the wine numerous specific compounds which form during the toasting of wood (Vivas et al. 1991). The accumulation of volatile oak-derived compounds in wine depends on many factors, such as the origin and species of the oak (Miller et al. 1992), seasoning of the staves (Sefton et al. 1993a), toasting of the barrel (Chatonnet et al. 1989), the rate of release of these compounds from the wood, and the rate at which such compounds are consumed by further chemical or biochemical transformations (Spillman et al. 1998a).

During the last few years some investigations have been carried out on the accumulation of oak-wood-derived volatiles in wine. However, there is still little data available on the effects of long-term ageing of wine, and some of the studies made were based on synthetic wines. Puech (1987) studied the accumulation of vanillin and syringaldehyde in Cabernet Sauvignon and Merlot wines matured in four oak barrels. Chatonnet et al. (1990) furnished data on the evolution of various wood-derived volatile compounds in red wines aged in new oak barrels for 10 months. Towey and Waterhouse (1996) investigated

the evolution of 10 volatile substances from the wood in Chardonnay wines fermented and matured for 8 months in French oak barrels, as well as American and Bulgarian oak barrels, over three successive wine harvests. Likewise, Spillman et al. (1998a) studied the accumulation of volatile oak compounds in a synthetic wine stored in American and Limousin oak barrels over a longer period (93 weeks) than the above cases.

Another important group of compounds for the aroma of the wine is the ethyl esters, which provide pleasant fruity aromas (Ramey and Ough 1980). These compounds are mainly synthesised during fermentation by yeasts. At present, although little is known about the evolution of ethyl esters during the ageing of wine in wood, it seems that some of these compounds could be being synthesised through esterification of the corresponding acid, while some others could be undergoing hydrolysis (Jackson 1994).

This investigation studied changes in concentration of oak-wood-derived volatiles and the evolution of esters in a wine blend of Tempranillo, Cabernet Sauvignon and Garnacha [Grenache] aged for 18 months in French oak barrels. The barrels had been used for two years and our blend was thus the third wine to be stored in these barrels. It is important to know how these compounds evolve, in order to obtain more precise data on the ideal period of wine storage in re-used barrels. In this way it

may be possible to reduce excessively long periods of wine maturation and so help to reduce costs, time and product waste.

Materials and methods

Vinification of the wine blend and barrel storage

The wine used was a blend of Tempranillo (60%), Cabernet Sauvignon (20%) and Garnacha (20%). Vinification of the wine was carried out in a cellar and was similar for all the varieties. The grapes were destemmed and crushed before being put into stainless steel tanks. Maceration was carried out during the fermentation, with periodic pumping over. The alcoholic fermentation was made at 28°C and taken to dryness; all wines underwent malolactic fermentation. Afterwards, wines were blended in the proportions specified above; oenological parameters of the wine blend are shown in Table 1.

Table 1. Oenological parameters[†] of the wine (60% Tempranillo, 20% Cabernet Sauvignon and 20% Garnacha) that was stored in twice-used French oak barrels.

Alcohol level	12.6% (v/v)
Total acidity	5.66 g/L ^a
pH	3.40
Volatile acidity	0.51 g/L ^b
SO ₂ free	30.5 mg/L
SO ₂ total	88 mg/L
Fe	4.5 mg/L
Sugar	1.9 g/L
Dry extract	28.3 g/L
Malolactic fermentation	Yes

[†]Measured according to the methods described by the Office International de la Vigne et du Vin (1990).

^aAs tartaric acid.

^bAs acetic acid.

The wine blend was stored in three barrels from the same lot (triplicate treatments). The wine was aged for 18 months in barrels made of French oak (*Quercus sessilis*) from the Allier region. The wood was naturally seasoned for 36 months and during cooperage the wood was submitted to a medium toasting. The barrels were fabricated at the Radoux cooperage (Jonzac) in France. The barrels were two years old and our blend was the third wine to be stored in these barrels. The barrels had been treated after each wine storage by washing with high-pressure steam. Wine samples to be analysed were taken from each barrel after 8, 10, 12, 15 and 18 months of ageing.

Analysis of volatile compounds of wines by gas chromatography

The extraction of volatile compounds from the wines was made using a modification of the method of microextraction described by Ferreira et al. (1996). This modification involved the substitution of Freon 113 with a mixture of dichloromethane-pentane (2:3, v/v); both solvents were sourced from Panreac (Montcada and Reixac, Barcelona, Spain).

Chromatographic analyses were carried out with a GC-MS Finnigan (San Jose, CA, USA). A DB-WAX capillary column (30 m × 0.25 mm i.d. and 0.25 µm film thickness) was used. The chromatographic conditions were: He as carrier gas (40 cm/s); injector temperature, 230°C; temperature of the transfer line, 240°C. Two analyses were made on each extract, one for the major components (ethyl lactate, 2-phenylethanol, 4-ethylphenol and 4-ethylguaiacol), and a second for the minor components of the study, i.e. furfural, 5-methyl furfural, 5-hydroxymethyl furfural, furfuryl alcohol, *cis*- and *trans*-oak lactone, γ-nonalactone, γ-butyrolactone, ethyl butyrate, ethyl hexanoate, ethyl octanoate, ethyl decanoate, syringaldehyde, coniferaldehyde, vanillin, acetovanillone, β-ionone, eugenol, guaiacol, 4-methylguaiacol, phenol, *m*- and *p*-cresol.

Conditions for analysis of the major components were: split mode injection of 1 µL of extract at an initial column temperature of 60°C for 5 min, programmed at 7°C/min to 240°C, and held at that temperature for 10 min; detection was by 70 eV electron impact (EI) in full scan mode (*m/z* 50 to 300). The minor components were separated after splitless injection of 1 µL of extract at an initial column temperature of 40°C for 5 min, with a two-step program of 2°C/min to 150°C, then 4°C/min to 240°C, and held at that temperature for 10 min; detection was by 70 eV EI and selected ion monitoring (SIM) (see Table 2).

Standards were prepared from Aldrich reagents dissolved in synthetic wine at concentrations between

Table 2. Mass/charge (*m/z*) of ions monitored for compounds analysed by GC-MS in SIM mode.

Compound	<i>m/z</i>
Furfural	95, 96, 97
5-Methyl furfural	108, 109, 110
5-Hydroxymethyl furfural	96, 97, 98
Furfuryl alcohol	96, 97, 98
<i>cis</i> -Oak lactone	71, 99
<i>trans</i> -Oak lactone	70, 99
γ-Nonalactone	71, 99
γ-Butyrolactone	56, 86
Ethyl butyrate	88, 101, 117
Ethyl hexanoate	73, 99, 115
Ethyl octanoate	73, 88, 101, 115, 129
Ethyl decanoate	143, 157
Syringaldehyde	181, 182, 183
Coniferaldehyde	177, 178, 179
Vanillin	150, 151, 152
Acetovanillone	151, 166
β-Ionone	176, 177, 178
Eugenol	163, 164, 165
Guaiacol	109, 124
4-Methyl guaiacol	137, 138, 139
Phenol	66, 94
<i>m</i> -Cresol	107, 108, 109
<i>p</i> -Cresol	107, 108, 109

Table 3. Changes in concentration of some furanic and lactone compounds in a red wine during its maturation in twice-used French oak barrels.

Compound	Concentration ^a (µg/L) at the end of month:				
	8	10	12	15	18
<i>Furanic compounds</i>					
Furfural	110 ± 10	506 ± 13	771 ± 9	124 ± 3	81 ± 22
5-Methyl furfural	9.4 ± 0.5	15.3 ± 0.4	135 ± 3	7.1 ± 0.3	23.9 ± 0.1
5-Hydroxymethyl furfural	0.03 ± 0.01	0.04 ± 0.00	0.03 ± 0.01	0.05 ± 0.01	0.030 ± 0.001
Furfuryl alcohol	1995 ± 44	914 ± 12	3714 ± 63	2356 ± 47	1086 ± 18
<i>Lactone compounds</i>					
<i>cis</i> -Oak lactone	80 ± 5	100 ± 1	79 ± 10	85 ± 7	119 ± 3
<i>trans</i> -Oak lactone	46 ± 5	73 ± 5	73 ± 12	60 ± 4	68 ± 3
γ-Nonalactone	3.5 ± 0.2	3 ± 1	2.6 ± 0.4	1.9 ± 0.2	3 ± 2
γ-Butyrolactone	43605 ± 1829	48615 ± 1158	49930 ± 5070	63275 ± 8049	66729 ± 7283

^a The concentration figures are shown with standard deviations (n = 6).

0.34 µg/L and 31.1 mg/L. To these solutions and also to the wine samples were added internal standards 2-octanol (Aldrich, Gillingham, UK) and 3,4-dimethylphenol (Merck, Darmstadt, Germany) in the same concentrations. The synthetic wine (alcohol 13% v/v) was prepared by diluting 3 g of tartaric acid (Sigma Chemical Co., St Louis, MO, USA) and 65 mL of ethanol (Merck, Darmstadt, Germany) in deionised water up to 500 mL. The pH of the synthetic wine was adjusted to 3.2 with 3M NaOH.

The precision of the method was calculated from the coefficient of variation (CV) and the results fluctuated between 3 and 15% for the different quantified components. To calculate the CV, six extractions were taken from a sample of wine and the concentrations of all the components were determined in each extract in duplicate. To examine the accuracy of the method, the recovery index was used. A known amount of each analytical standard was added to a previously analysed wine sample and all the components were extracted and quantified. This procedure was carried out for two different concentrations of the added analytical standard. The recovered quantity was calculated from the difference between the measured concentration after adding the analytical standard and the initial, endogenous, concentration. The recovery index varied between 70 and 104% depending on the different components analysed.

The results shown in Tables 3, 4 and 5 are the arithmetic average of 6 analyses. One representative sample was taken from each of the three barrels, and the volatile compounds from each of the these samples were extracted and analysed in duplicate.

Results and discussion

Evolution of furanic compounds

Furanic compounds, with the exception of furfuryl alcohol, form during the toasting of the wood through degradation of carbohydrates; 5-methyl furfural and 5-hydroxymethyl furfural come from hexoses (rhamnose and glucose, respectively) and furfural comes from pentoses (xylose) (Chatonnet et al. 1989). Furfuryl alcohol is thought to be formed through reduction of furfural by means of biological mechanisms (Boidron et al. 1988,

Chatonnet et al. 1992a), although the possibility of a chemical reduction cannot be excluded. These compounds have an almond-like aroma except for 5-hydroxymethyl furfural which is practically odourless (Chatonnet et al. 1991). The threshold level in red wines of furfural is 20 mg/L and that of 5-methyl furfural is 45 mg/L (Boidron et al. 1988). Given that the perception thresholds are high it is believed that these two compounds do not have an important role in the aroma of wine, although they may strengthen the aroma of the lactones (Reazin 1981). The reduction of furfurals to the corresponding alcohols results in a quality shift from an almond-like to a hay/verbena-like aroma (Chatonnet et al. 1991).

In Table 3 the evolution of furfural, 5-methyl furfural, 5-hydroxymethyl furfural and furfuryl alcohol is shown. The first two of these showed maximum concentration at 12 months and diminished thereafter, probably due to the microbiological reduction of these aldehydes to the corresponding alcohols. 5-Hydroxymethyl furfural was found in a very low concentration; this is not surprising as the barrels were only subjected to a medium toast level. The concentration of this compound was not modified to any notable extent throughout the maturation of the wine. Furfuryl alcohol also reached its maximum concentration at 12 months of ageing and decreased during the final stage of ageing, although an increase might have been expected in its concentration since the levels of furfural decreased to a notable extent. The peaking of the concentration of furfuryl alcohol could have been due to the transformation of this compound into furfuryl ethyl ether. Furfuryl ethyl ether has been found in wine and beer, and seems to be a compound with a high chemical stability (Spillman et al. 1998b).

Evolution of lactone compounds

The two isomers of β-methyl-γ-octalactone (*cis*- and *trans*-oak lactones) are thought to be formed during the toasting of the barrels through dehydration of 2-methyl-3-(3,4-dihydroxy-5-methoxybenzo)-octanoic acid present in oak-wood (Otsuka et al. 1980). The *trans*-isomer has a threshold level of 460 µg/L, and the *cis*-isomer shows a

Table 4. Changes in concentration of phenolic aldehydes, acetovanillone, β -ionone, phenolic alcohols and 2-phenylethanol in a red wine during its maturation in twice-used French oak barrels.

Compound	Concentration ^a ($\mu\text{g/L}$) at the end of month:				
	8	10	12	15	18
Syringaldehyde	70 \pm 12	125 \pm 14	312 \pm 32	247 \pm 64	340 \pm 34
Coniferaldehyde	39 \pm 3	79 \pm 7	40 \pm 4	39 \pm 7	34 \pm 1
Vanillin	65 \pm 10	65 \pm 2	89 \pm 15	59 \pm 6	86 \pm 3
Acetovanillone	140 \pm 15	157 \pm 7	114 \pm 25	104 \pm 21	117 \pm 7
β -Ionone	0.18 \pm 0.02	0.28 \pm 0.06	0.16 \pm 0.01	0.21 \pm 0.01	0.16 \pm 0.02
Eugenol	21 \pm 2	21.68 \pm 0.04	20 \pm 1	20.25 \pm 0.08	21 \pm 1
Guaiacol	6 \pm 1	7.5 \pm 0.2	8.8 \pm 0.5	8 \pm 1	8 \pm 1
4-Methylguaiacol	0.03 \pm 0.00	0.02 \pm 0.01	0.06 \pm 0.04	0.05 \pm 0.01	0.03 \pm 0.01
Phenol	12 \pm 2	18 \pm 2	15.52 \pm 0.06	15 \pm 1	9 \pm 1
<i>m</i> -Cresol	0.9 \pm 0.1	0.8 \pm 0.1	0.8 \pm 0.2	1.30 \pm 0.01	1.8 \pm 0.1
<i>p</i> -Cresol	0.19 \pm 0.02	0.21 \pm 0.02	0.19 \pm 0.01	0.15 \pm 0.00	0.06 \pm 0.01
4-Ethylphenol	898 \pm 57	825 \pm 2	656 \pm 2	915 \pm 7	1064 \pm 66
4-Ethylguaiacol	111 \pm 6	108 \pm 2	87 \pm 1	116 \pm 1	140 \pm 9
2-Phenylethanol	1919 \pm 85	2243 \pm 75	2051 \pm 39	1900 \pm 52	2572 \pm 193

^a The concentration figures are shown with standard deviations (n = 6).

sensory threshold of 92 $\mu\text{g/L}$ (Chatonnet et al. 1990). This latter study showed how, at low concentrations, these lactones give off a woody aroma which adds quality to wines. γ -Nonalactone, present in oak-wood (Nishimura et al. 1983), may strengthen the aroma of oak lactones in wines (Nakamura et al. 1988). This compound shows an aroma threshold level of 30 $\mu\text{g/L}$ in wine, and confers a sweeter, more pleasant, fruitier impression (Singleton 1995). γ -Butyrolactone, which gives off a bitter and burnt odour (Maga 1987), is formed through dehydration of γ -hydroxybutyric acid during the toasting of the wood (Maga 1987) and also comes from alcoholic fermentation, being formed from glutamic acid or related compounds (succinic, 2-oxoglutamic or gamma-aminobutyric acids) (Muller et al. 1973).

In Table 3 the evolution of lactone compounds (*cis*- and *trans*-oak lactones, γ -nonalactone and γ -butyrolactone) in the wine samples aged for 18 months in barrels is shown. *cis*-Oak lactone showed concentrations higher than its sensory threshold at 10 months of maturation of the wine in the barrel. From then on, its concentration showed small modifications and the wine, after 18 months of maturation, had a somewhat higher concentration of the *cis*-oak lactone than that found after 10 months. *trans*-Oak lactone did not reach its threshold level and its concentration remained unaltered between the tenth and the eighteenth month of storage of the wine in barrel. The results for these two lactones concur with those obtained by Spillman et al. (1998a), who found that in a synthetic wine, the accumulation of these lactones was at a maximum after one year of ageing and the concentrations did not increase at any later period. The concentration of γ -nonalactone remained more or less constant throughout the period of maturation under study, and its levels were very low (Table 3). γ -Butyrolactone showed the highest levels from this group of compounds. The concentration of this substance

increased up to the 15 months point of ageing and then stayed constant.

Evolution of phenolic aldehydes, acetovanillone, β -ionone, phenolic alcohols and 2-phenylethanol

The phenolic aldehydes and acetovanillone arise from thermal degradation of lignin, although their extraction into wine occurs via different means (Nishimura et al. 1983). β -Ionone, a norisoprenoid, comes from terpenic precursors of the grape and is also a component of oak-wood (Nishimura et al. 1983). In general the contribution of vanillin and β -ionone to the aroma of wine is important due to their lower perception thresholds (Ohloff 1978, Sefton 1991, Singleton 1995). The phenolic alcohols have less influence on the quality of wine than the phenolic aldehydes. These former compounds form from the thermal degradation of lignin at high temperature, except eugenol and the ethylphenols. Eugenol is present in the wood before its toasting (Sefton et al. 1993b). The ethylphenols are responsible for unpleasant smells in wines; they form from yeast species (*Brettanomyces* and *Dekkera* genus) during the maturation of wine in barrels. High levels of these compounds were found in wine aged in used barrels (Chatonnet et al. 1992b). 4-Ethylguaiacol has a smoky, spicy and somewhat medicinal character, while 4-ethylphenol has been described as mainly 'medicinal' and 'horsey'. 2-Phenylethanol is formed during alcoholic fermentation (Towey and Waterhouse 1996).

Table 4 shows the evolution of phenolic aldehydes, acetovanillone, β -ionone, phenolic alcohols and 2-phenylethanol. Syringaldehyde increased constantly up to 12 months and, after fluctuating, the concentration in the wine after 18 months of ageing was similar to that at the end of one year. Coniferaldehyde reached maximum concentration after 10 months of ageing, and thereafter its concentration diminished. This decline was probably due to microbiological reduction of the aldehyde to the

Table 5: Changes in concentration of some esters in a red wine during its maturation in twice-used French oak barrels.

Compound	Concentration ^a (µg/L) at the end of month:				
	8	10	12	15	18
Ethyl butyrate	393 ± 11	475 ± 60	517 ± 11	521 ± 23	461 ± 17
Ethyl hexanoate	199 ± 4	224 ± 14	206 ± 3	259 ± 46	200 ± 33
Ethyl octanoate	258 ± 3	283 ± 4	246 ± 25	376 ± 73	195 ± 6
Ethyl decanoate	12.1 ± 0.5	11.7 ± 0.4	9 ± 2	16 ± 2	12 ± 1
Ethyl lactate	22689 ± 256	27890 ± 279	31198 ± 625	22948 ± 503	21155 ± 392

^a The concentration figures are shown with standard deviations (n = 6).

corresponding alcohol. High concentrations of vanillin were extracted from the barrels up to 12 months of wine ageing. The concentration of this compound after 18 months of maturation was the same as that found after one year. The evolution of vanillin is similar to that reported by Puech (1987) who observed that, in three out of four barrels studied, the accumulation of this substance in Cabernet Sauvignon and Merlot wines was at a maximum at around 12 months of ageing. However, Spillman et al. (1998a), found a linear increase in the concentration of this compound in a synthetic wine up to two years of maturation. This difference could be due to the fact that microbial activity in wine could transform the vanillin into vanillyl alcohol. Acetovanillone was extracted by the wine up to 10 months of ageing and afterwards its concentration diminished slightly. β -Ionone, which was always found at very low concentrations, reached its maximum after 10 months ageing, and thereafter diminished.

As far as the phenolic alcohols are concerned, the concentration of eugenol hardly changed between the eighth and the eighteenth month of ageing (Table 4). Guaiacol reached its maximum concentration after 12 months of ageing and then remained constant; 4-methylguaiacol showed very low concentrations, which were practically the same throughout the whole period of ageing. Phenol reached maximum concentration at 10 months of ageing and then diminished such that after 18 months it was present at half the maximum concentration. *m*-Cresol and *p*-cresol were found in low concentrations. *m*-Cresol increased up to 18 months of ageing of the wine in barrels, while *p*-cresol showed a maximum between 8 and 12 months of ageing. The concentrations of ethylphenols after 18 months of ageing were higher than those found at earlier stages. At all samplings 4-ethylphenol was present at a level above the value considered as the limit (620 µg/L) by Chatonnet et al. (1992b) and at which it has a negative influence on the quality of wine. 2-Phenylethanol, found in high concentrations in all the samples (and indeed in all wines), was still accumulating after 18 months of ageing. The concentration of this fermentation-derived alcohol would probably have been augmented through hydrolysis of its ester during ageing (Garofolo and Piracci 1994).

Evolution of ethyl esters

The ethyl esters with shorter hydrocarbon chains, such as butanoate, hexanoate and octanoate, are somewhat fruity in character. As the hydrocarbon chain becomes longer, the odour shifts to soapy and finally lard-like (Jackson 1994). Table 5 shows the evolution of ethyl butyrate, ethyl hexanoate, ethyl octanoate, ethyl decanoate and ethyl lactate. The concentrations of ethyl butyrate and ethyl hexanoate hardly changed between 8 and 18 months ageing of the wine. Similarly ethyl octanoate and ethyl decanoate did not undergo any important modifications in their concentrations, although they did show their highest values at 15 months and afterwards diminished slightly. Ethyl lactate was produced during the ageing period and reached its maximum concentration after 12 months of maturation of the wine. This coincides with reports in the literature, where it can be observed that the ethyl esters of the majority acids of wine are made during the ageing process (Jackson 1994).

Conclusions

The results show that a range of furanic compounds, volatile phenols, and lactones extracted from French oak barrels on their third fill with red wine reached maximum accumulation after the red wine had been 10 to 12 months in the barrels. Many of these compounds, after 18 months of maturation, showed similar concentrations to those reached within 12 months. Exceptions to this were furfural, 5-methyl furfural, furfuryl alcohol, coniferaldehyde, acetovanillone and phenol which, after 18 months of ageing, were present at significantly lower concentrations than after 10 to 12 months. The concentration of ethylphenols increased up to 18 months of ageing, which is negative for the quality of the wine. This continuous evolution of the ethylphenols is probably due to the fact that in used barrels microbial processes for the production of these compounds are favourable. The ethyl esters hardly showed any change in their concentrations during maturation of the wine, except ethyl lactate, the concentration of which increased up to 12 months.

References

- Boidron, J.-N., Chatonnet, P. and Pons, M. (1988) Influence du bois sur certaines substances odorantes des vins. *Connaissance de la Vigne et du Vin* **22**, 275–294.
- Chatonnet, P., Boidron, J.-N. and Pons, M. (1989) Incidence du traitement thermique du bois de chêne sur sa composition chimique. 2^e partie: évolution de certains composés en fonction de l'intensité de brûlage. *Connaissance de la Vigne et du Vin* **23**, 223–250.
- Chatonnet, P., Boidron, J.-N. and Pons, M. (1990) Maturation of red wines in oak barrels: evolution of some volatile compounds and their aromatic impact. *Sciences des Aliments* **10**, 565–587.
- Chatonnet, P., Dubourdieu, D. and Boidron, J.-N. (1991) Effects of fermentation and maturation in oak barrels on the composition and quality of white wines. *Australian and New Zealand Wine Industry Journal* **6**, 73–84.
- Chatonnet, P., Dubourdieu, D. and Boidron, J.-N. (1992a) Incidence des conditions de fermentation et d'élevage des vins blancs secs en barriques sur leur composition en substances cédées par le bois de chêne. *Sciences des Aliments* **12**, 665–686.
- Chatonnet, P., Dubourdieu, D., Boidron, J.-N. and Pons, M. (1992b) The origin of ethylphenols in wines. *Journal of the Science of Food and Agriculture* **60**, 165–178.
- Ferreira, V., Sharman, M., Cacho, J.F. and Dennis, J. (1996) New and efficient microextraction/solid-phase extraction method for the gas chromatographic analysis of wine volatiles. *Journal of Chromatography A* **731**, 247–259.
- Garofolo, A. and Piracci, A. (1994) Evolution of esters and fatty acids in the wine preservation. Equilibrium constants and activation energies. *Bulletin de l'OIV* **67**(757–758), 225–245.
- Jackson, R.S. (1994) Oak and cooperage. In: 'Wine Science. Principles and Applications'. Ed. S.L. Taylor (Academic Press: New York) pp. 299–311.
- Maga, J.A. (1987) The flavor chemistry of wood smoke. *Food Reviews International* **3**, 139–183.
- Miller, D.P., Howell, G.S., Michaelis, C.S. and Dickmann, D.I. (1992) The content of phenolic acid and aldehyde flavor components of white oak as affected by site and species. *American Journal of Enology and Viticulture* **43**, 333–338.
- Muller, C.J., Kepner, R.E. and Webb, A.D. (1973) Lactones in wines – a review. *American Journal of Enology and Viticulture* **24**, 5–9.
- Nakamura, S., Crowell, E.A., Ough, C.S. and Totsuka, A. (1988) Quantitative analysis of γ -nonalactone in wines and its threshold determination. *Journal of Food Science* **53**, 1243–1244.
- Nishimura, K., Ohnishi, M., Masuda, M., Koga, K. and Matsuyama, R. (1983) Reactions of wood components during maturation. In: 'Flavour of distilled beverages'. Ed. J.R. Piggott (Ellis Horwood: Chichester) pp. 241–255.
- Office International de la Vigne et du Vin (1990) 'Recueil des méthodes internationales d'analyse des vins et des moûts' (OIV: Paris).
- Ohloff, G. (1978) Importance of minor components in flavors and fragrances. *Perfumer and Flavorist* **3**, 11–22.
- Otsuka, K.-I., Sato, K. and Yamashita, T. (1980) Structure of a precursor of β -methyl- γ -octalactone, an ageing flavour compound of distilled liquors. *Journal of Fermentation Technology* **58**, 395–398.
- Puech, J.-L. (1987) Extraction of phenolic compounds from oakwood in model solutions and evolution of aromatic aldehydes in wines aged in oak barrels. *American Journal of Enology and Viticulture* **38**, 236–238.
- Ramey, D.D. and Ough, C.S. (1980) Volatile ester hydrolysis or formation during storage of model solutions and wines. *Journal of Agricultural and Food Chemistry* **28**, 928–934.
- Reazin, G.H. (1981) Chemical mechanisms of whiskey maturation. *American Journal of Enology and Viticulture* **32**, 283–289.
- Sefton, M.A. (1991) How does oak barrel maturation contribute to wine flavour? *Australian and New Zealand Wine Industry Journal* **6**, 69–72.
- Sefton, M.A., Francis, I.L., Pocock, K.F. and Williams, P.J. (1993a) The influence of natural seasoning on the concentrations of eugenol, vanillin, and *cis*- and *trans*- β -methyl- γ -octalactone extracted from French and American oakwood. *Sciences des Aliments* **13**, 629–643.
- Sefton, M.A., Spillman, P.J., Pocock, K.F., Francis, I.L. and Williams, P.J. (1993b) The influence of oak origin, seasoning, and other industry practices on the sensory characteristics and composition of oak extracts and barrel-aged white wines. *Australian Grapegrower & Winemaker No. 355*, 17–25.
- Singleton, V.L. (1995) Maturation of wines and spirits: comparisons, facts, and hypotheses. *American Journal of Enology and Viticulture* **46**, 98–115.
- Spillman, P.J., Iland, P.G. and Sefton, M.A. (1998a) Accumulation of volatile oak compounds in a model wine stored in American and Limousin oak barrels. *Australian Journal of Grape and Wine Research* **4**, 67–73.
- Spillman, P.J., Pollnitz, A.P., Liacopoulos, D., Pardon, K.H. and Sefton, M.A. (1998b) Formation and degradation of furfuryl alcohol, 5-methylfurfuryl alcohol, vanillyl alcohol, and their ethyl ethers in barrel-aged wines. *Journal of Agricultural and Food Chemistry* **46**, 657–663.
- Towey, J.P. and Waterhouse, A.L. (1996) The extraction of volatile compounds from French and American oak barrels in Chardonnay during three successive vintages. *American Journal of Enology and Viticulture* **47**, 163–172.
- Vivas, N., Glories, Y. and Francois, J. (1991) Mise au point sur l'élevage des vins rouges en fûts de chêne. Diversité des constituants du bois cédés au vin. *Revue des Oenologues* **17**, 17–21.
- Vivas, N. and Glories, Y. (1993) Les phénomènes d'oxydoréduction liés à l'élevage en barriques des vins rouges: aspects technologiques. *Revue Française d'Oenologie* **33**, 33–38.

Manuscript received: 8 November 2001

Revised manuscript received: 19 April 2002