Influence of Storage Conditions on the Formation of Some Volatile Compounds in White Fortified Wines (*Vins doux Naturels*) during the Aging Process

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Sweet fortified wines, traditionally aged under strong oxidation conditions, have a characteristic aroma. An experimental study investigated the aging of white sweet fortified wines under various conditions. The development of various molecules, previously identified as characteristic of the aroma of this type of wine, was monitored by analysis. The development of these compounds during accelerated aging was affected by oxidation and the color of the wine. Aging in oak containers, variable storage temperatures, and variable modes of oxidation affect the formation of many of the volatile compounds responsible for the aging aroma of *vins doux naturels* (fortified wines). Furfural (**1**), 5-ethoxymethylfurfural (**2**), and sotolon (**3**) always reach concentrations above their perception thresholds in wood-aged wines, especially when new oak is used. The isomers of methyl-γ-octalactone (**4** and **5**) are reliable indicators of aging in oak. Among the many identifiable volatile phenols, only the vanillin (**7**) content increases with aging, particularly if the container is made of wood and there is a high degree of oxidation. This molecule frequently reaches and even exceeds its perception threshold. We show here that vanillin and isomers of methyl-γ-octalactone have a major impact on the aromas of fortified wines.

Keywords: *Fortified wine (vin doux naturel); aromas; oxidation during aging; oak*

INTRODUCTION

Sweet fortified wines have traditionally been aged in used barrels, often partly empty, in cellars with variable temperatures. Due to their alcohol content (15−18%), yeast is not involved in the aging process of sweet fortified wines, unlike sherry or Vin Jaune from the Jura (Martin et al., 1992; Dubois et al., 1976). Oxygen is assumed to play a major role in the many chemical reactions occurring during aging. The wine is put into used barrels (20−80 years old) of various sizes (225, 660, and 1500−5000 L) to facilitate the diffusion of oxygen, whereas the aromatic contribution of the oak wood was often considered relatively unimportant.

A great deal of research has already been carried out into wine aging and oxidation (Ribéreau-Gayon, 1933). Oxidation results in organoleptic modifications, generally described as “oxidized” in dry wines, “rancio” in sweet fortified red wines, and “maderized” in sweet fortified white wines. Berthelot (1863, 1864) was the first to report the presence of acetaldehyde and associate it with the phenomenon of oxidation. Cantarelli (1967) showed the importance of the carbonyl group of aldehydes as well as the role of amino acids in the formation of the “rancio” odor in maderized wines. In the case of dry wines, these phenomena lead to the deterioration of the bouquet during aging. On the other hand, oxidation phenomena are generally considered to be favorable, or even indispensable, for the proper development of the aroma of sweet fortified wines.

In our recent research (Cutzach et al., 1999) into the formation mechanisms of the molecules responsible for the characteristic aroma of old sweet fortified wines, we drew inspiration from the work of Deibner and Bernard (1956). These authors already mentioned the role of Maillard reactions in the formation of the aroma of sweet fortified wines following heat treatment, but they were unable to identify the compounds responsible. This study investigated both white and red wines, as the phenolic compounds present in red wines have well-known antioxidant properties (Ribéreau-Gayon, 1933) and were thus likely to affect the development of the aroma during aging.

Using modern techniques, such as gas-phase chromatography combined with olfactory detection, mass spectrometry, and infrared spectrometry (Cutzach et al., 1998a,b), we have recently identified various volatile and odorous compounds found in sweet fortified wines during aging. In a recent work (Cutzach et al., 1999), we have studied the formation of some volatile compounds during the experimental laboratory aging of sweet fortified wines. We have shown that the development of aroma components may sometimes be affected by the presence of the phenolic compounds responsible for the color of red wines.

Their formation mechanisms under controlled laboratory conditions have also been studied (Cutzach et al., 1999). The results obtained have made it possible to identify the molecules whose formation depends on oxidation and/or the color of the wine.

This current work prosecutes our last research (Cutzach et al., 1999), which we studied the formation

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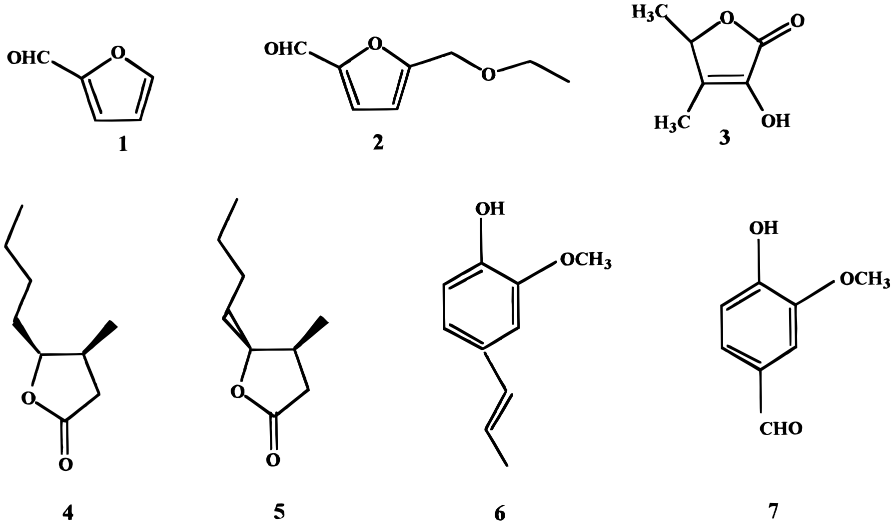

Figure 1. Structures of volatile compounds cited in this work. **1**, furfural; **2**, 5-ethoxymethylfurfural; **3**, sotolon; **4**, *cis*-methyl-γ-octalactone; **5**, *trans*-methyl-γ-octalactone; **6**, eugenol; **7**, vanillin.

of these same volatile compounds under normal cellar aging conditions, according to the type of container and the mode of oxidation only in white sweet fortified wines.

MATERIALS AND METHODS

Reference Volatile Compounds. The following substances (Figure 1), supplied by Sigma-Aldrich Chimie (F-38297 Saint Quentin Fallavier), were always at least 97% pure, as measured by gas-phase chromatography: furfural (**1**), sotolon (**3**), *cis*- and *trans*-methyl γ-octalactone (**4** and **5**), vanillin (**7**), and eugenol (**6**). The 5-ethoxymethylfurfural (**2**) was obtained by organic synthesis in the laboratory, using the method previously described (Cutzach et al., 1998b).

Olfactory Perception Threshold Determination. For each substance assessed, a threshold for tasting perception is determined. This threshold limit corresponds to the minimum concentration level below which 50% of involved tasters are statistically no longer in a position to make out the difference with a reference glass solutions are set up in AFNOR glasses filled up with 60 mL. The assessing jury, trained yet not selected for the present test, is made up of 30 people of both sexes aged between 20 and 50. They meet at 11:00 am, in a room air-conditioned to 19 °C and soundproofed. Each solution to be assessed finds itself tried in a 3-fold items discrimination test. Starting from the lowest, five increasing solutions are successively assessed.

The individual threshold of each taster is considered to be equal to the minimum level from which he shall always give positive answers. The increasing cumulative threshold frequency curve is being traced, and the frequency from which 50% of involved tasters shall give positive answers is determined on this curve.

Wine is a complex and varying environment, hence, is it difficult to base one's work on any standard wine which could possibly be considered as reference item? Consequently, a reference environment is used, the composition of which is as follows: glycerol, 8 g; sodiumdihydrogenatephophate, 0.3 g; calcium chlorate, 0.1 g; magnesium chlorate, 0.1 g; potassium hydrogenatetatrate, 2.5 g; potassium sulfate, 1 g; sodium chlorate, 0.5 g; citric acid, 0.4 g; succinic acid, 1 g; lactic acid, 2 g; ethanol at 99.5%, 120 mL; water qsp 1000 mL. The solution pH is raised to 3.5 thanks to a KOH addition. Its use enables the correct assessment, as well as in a reproducible way, of the perception threshold of each volatile and olfactory substance liable to be met in wine.

Assay of the Aromatic Impact of a Substance. The contribution of a volatile compound to a wine's aroma is estimated from its aromatic index: $I = c/s$, where c is the concentration of the substance in the wine considered and s its perception threshold, determined in a synthetic reference solution. This estimation does not take into account the depressive and synergetic effects resulting from the interaction of different smelling molecules with one another. Nevertheless, it well accounts for a potential for participation to the global aroma. Though I may be a number having no proper unit of its own, it is taken into account in terms of olfactory units (OU). Olfactory perception threshold determination: The perception thresholds of the various molecules considered were determined in previous research (Cutzach et al., 1998b; Chatonnet, 1995). A molecule is considered to contribute actively to a wine's aroma if its aromatic index, I, has a value of at least 1.

Gas-Phase Chromatography Assay of Volatile Phenols. The volatile phenols in the fortified wines were assayed using the method described by Boidron (1988). The ionic strength of the medium was adjusted and an internal standard was incorporated (3,4-dimethylphenol). Liquid−liquid extraction was then carried out using dichloromethane. The extract obtained was purified by washing it several times at selected pHs (pH 8.5 and 13) to isolate the phenolic fraction. The final extract was concentrated to 500 μL under nitrogen and analyzed by capillary gas phase chromatography and FID detection. Only the eugenol (**6**) and vanillin (**7**) content of the wines was studied in this work.

Assay of Volatile Compounds by Gas-Phase Chromatography Coupled with Mass Spectrometry. The volatile compounds in fortified wines were assayed using the method described by Cutzach et al. (1998b). One ml of internal standard (100 mg/L 3-octanol in absolute alcohol) then 5 g of $(NH4)_2SO_4$ were added to a 100 mL sample. Ammonium sulfate was used to increase the ionic strength of the solution and reduce the solubilization of the compounds in water. Three extractions were then carried out, using 10, 5, and 5 mL of dichloromethane, respectively (with magnetic stirring at 700 rpm for 10, 5, and 5 min.). The organic phases were decanted, collected, and dried on sodium sulfate, then cold (ambient temperature) concentrated to 500 μL under nitrogen. The final extract was analyzed by gas-phase chromatography coupled with mass spectrometry (Cutzach et al., 1998a,b). The coefficients of determination were obtained from the assay linearity for each molecule. Ten analyses (extraction and measurement) were carried out on each wine to determine the variation coefficient for measuring each component. The GC-SM analysis conditions were identical to those described by Cutzach et al. (1998a,b, 1999). We used a Carbowax 20M column (50 m × 0.25 mm, 0.25 μm, BP-20, SGE) installed on a HP5890 chromatograph connected to a (MSD HP5970b and 5972)

Table 1. Repeatability and Linearity of Quantitative Analysis of Volatile Compounds That Take Part in Sweet Fortified Wines

volatile compounds column BP20 (polar)	variation coefficient[a] (%)	mean overlap of the additions assayed[a] (%)	assay linearity (coefficient of determination R2)
furfural	3.7	98	0.9984
ethoxymethylfurfural	9.7	99	0.9995
sotolon	9.8	95	0.9922
trans-γ-methyloctalactone	15	104	0.9986
cis-γ-methyloctalactone	12.3	94	0.9996

[a] Into a set of 10 analysis of a same fortified wines.

Table 2. Influence of the Nature of Containers and of the Temperature Regulation on the Evolution of the Volatile Compounds of a White Fortified Wine[a]

	air-conditioned winery (16−18 °C)						non-air-conditioned winery (8−33 °C)						
	concrete vats		2-year-old barrels		new barrels		concrete vats		2-year-old barrels		new barrels		
volatile compounds	6 month	30 month	6 month	30 month	6 month	30 month	6 month	30 month	6 month	30 month	6 month	30 month	sensory threshold (μg/L)
furanic derivatives (μg/L)													
furfural	270	1200	2700	7700	**34 000**	**97 000**	366	5200	2500	9000	**24 000**	**85 000**	15 000
5-ethoxymethylfurfural	17	47	54	38	**302**	**207**	17	14	54	88	**350**	**330**	90
enolic derivatives (μg/L)													
sotolon	0	3	3	6	2	**94**	2	**13**	5	11	7	**105**	10
lactons (μg/L)													
trans-methyl-γ-octalactone	0	0	25	3	63	**287**	0	45	50	105	58	**397**	122
cis-methyl-γ-octalactone	0	0	**160**	**296**	**359**	**464**	0	**93**	**213**	**361**	**332**	**755**	35
volatile phenol (μg/L)													
eugenol	na	0.3	na	**35**	na	**96**	na	0.3	na	**45**	na	**82**	65
vanillin	na	3	na	**79**	na	**209**	na	4.5	na	**111**	na	**210**	15

[a] Boldface, concentration higher than perception threshold. na, not assayed at sixth month.

quadripolar mass spectrometer with electronic impact (ionization energy, 70 eV; source temperature, 250 °C). The extracts were injected under the previously described chromatographic conditions. Identification was carried out after the mass spectrums and retention indexes were compared to those of control compounds. These are shown in Table 1, together with the mean overlap of the additions assayed and the variation coefficients.

Influence of Container Type and Storage Temperature. The wine (1995 white Rivesaltes) was fermented in three different containers: concrete vats, 2-year-old barrels, and new barrels. When fermentation was completed, the wines were kept in an air-conditioned or non-air-conditioned winery, in one of the three types of storage containers: vats, 2-year-old barrels, and new barrels. The temperature in the air-conditioned winery was maintained between 16 and 18 °C, whereas temperatures in the non-air-conditioned winery fluctuated from 8 °C in winter to 33 °C in summer. Samples were taken after 6 and 30 months' aging. After 29 months, wine from the vat in the non-air-conditioned winery had to be transferred to 2-year-old barrels. The wines were tasted during aging, by a panel of 12 wine tasters, who were asked to rate the fortified wines on a scale of 1−6.

Influence of Container Type on the Development of Aromas in the Wine during Long-Term Aging. The wine studied was a white Rivesaltes (1974) from the village of Terrats, made from white Grenache and Macabeu grapes, stored for 24 years using three different methods: (i) aging in vat only, (ii) aging in vat for 14 years and 600-L barrels for 10 years, (iii) and aging in wood only (600-L barrels).

These samples showed marked differences on tasting. They were then assayed for volatile and odorous compounds as described above.

RESULTS AND DISCUSSION

Influence of Container Type and Storage Temperatures on the Volatile Compounds in Fortified White Wines Aged 2 Years. The formation of the volatile compounds identified and investigated in this work was affected by the type of container and variations in temperature (non-air-conditioned winery). Wooden containers promoted the formation of these molecules (Tables 2 and 3).

Table 3. Influence of Conservation in Wood and of Temperature Variations on the Accumulation of Volatile Compounds in a White Fortified Wine

volatile compounds	effect of wood	effect of temperature variation
furfural	f*[a]	f*
5-ethoxyméthylfurfural	f*	f*
sotolon	f*	f*
cis-methyl-γ-octalactone	f*	f*
trans-methyl-γ-octalactone	f*	f*
vanillin	f*	a[b]
eugenol	f*	a

[a] f, favorable on the formation of the compound considered. (*) Significatif effect on the aroma of fortified wines. [b] a, any effect on the formation of the compound considered.

The accumulation of the majority of volatile compounds was promoted both by variations in temperature and wood aging. Concentrations of furfural, isomers of methyl-γ-octalactone (**4** and **5**), ethoxymethylfurfural (**2**), and sotolon (**3**) increased in wines aged in barrel, especially if the wood was new. Among these compounds, furfural, isomers of methyl-γ-octalactone (**4** and **5**), and sotolon (**3**) are actively involved in the aromas of these wines ($I > 1$).

The volatile phenol concentrations of these white fortified wines were also analyzed. Wood aging facilitates an increase in the volatile phenol content of fortified wines. Those white fortified wines that were fermented had brandy added to stop fermentation and were then aged in new barrels in a non-air-conditioned winery always had the highest volatile phenol content. White fortified wines that were fermented and aged exclusively in oak always had eugenol (**6**) and vanillin (**7**) concentrations above the perception threshold.

On tasting, the white fortified wines fermented and aged in 2-year-old barrels in an air-conditioned winery

Table 4. Influence of Wood and the Temperature Variations of the Taste of a White Fortified Wine[a]

	notes of the taste					
	non-air-conditioned winery (8−33 °C)			air-conditioned winery (16−18 °C)		
tasters ($n = 12$)	(A) vats	(B) 2-year-old barrels	(C) new barrels	(A) vats	(B) 2-year-old barrels	(C) new barrels
1	4	1	3	1	2	5
2	6	4	5	3	1	2
3	6	4	2	5	3	1
4	6	2	4	3	1	5
5	6	4	5	1	3	2
6	6	3	2	1	4	5
7	5	3	6	1	2	4
8	5	2	3	1	4	6
9	6	1	3	5	2	4
10	5	2	3	4	1	6
11	6	5	2	2	1	4
12	6	2	5	4	1	3
sum of ranges	**67**	33	43	31	**25**	47

[a] Kramer test: [28−56] $a = 5\%$. E is sample preferred. A was the sample judged the worst and discarded.

was judged to be the best (Kramer test). The wine fermented and aged in vat in a non-air-conditioned winery was judged inferior than the others (Table 4).

Influence of the Type of Container on the Aging of a White Rivesaltes Aged for 24 Years in a Non-Air-Conditioned Winery. Table 5 shows the results for the various compounds assayed in a white fortified wines aged for 24 years using three different methods. The development of volatile compounds was monitored according to the type of container and oxidation level (partly empty). The wine aged exclusively in vat was only slightly oxidized, while oxidation was moderate after the mixed aging program (14 years in vat then 10 years in 600-L barrels), and high in the wine aged only in wood.

The development of almost all the volatile compounds studied in this work was promoted both by wood aging and variations in temperature. Concentrations of furfural (**1**), sotolon (**3**), isomers of methyl-γ-octalactone (**4** and **5**), and vanillin (**7**) increased in wines aged in wooden containers in the presence of oxygen. The 5-ethoxymethylfurfural (**2**) content varied little from one aging method to another.

Used wooden containers provide more limited amounts of volatile phenols (Table 5). The barrels were very old—over 20 years. Among the many volatile phenols in the old white Rivesaltes, only the vanillin (**7**) content was above the perception threshold in the samples aged in oak (Table 5). The wine aged for 24 years in 600-L barrels had a vanillin (**7**) content of 890 μg/L, significantly above its olfactory perception threshold. This molecule makes an active contribution to the wine's aroma.

Changes in the bouquet and flavor of fortified wines are largely due to oxidation during aging (Cutzach et al., 1998a−c, 1999).

The use of oak containers and variable storage temperatures accelerate the chemical changes of fortified wines, promoting the formation of volatile compounds that affect the wine's aroma as it ages (Tables 2, 3, and 5). The results described in this study are identical to those obtained in a laboratory study of the formation mechanisms of volatile compounds (Cutzach et al., 1999). The sotolon concentration in fortified wines, for example increases as they become more and more oxidized. All these volatile compounds are indicative of oxidation in white *vins doux naturels* (fortified wines) (Cutzach et al., 1999).

Among the furanic derivatives, furfural (**1**), a molecule produced by the degradation of sugars, and 5-ethoxymethylfurfural (**2**), which results from the conversion of hydroxymethylfurfural, reach concentrations higher than their perception thresholds in wood-aged wines, especially when new oak is used. The formation of 5-ethoxymethylfurfural (**2**) during aging in the cellar is similar to that observed in laboratory tests (Cutzach et al., 1999).

New wood does not add sotolon (Cutzach et al., 1997). It may, however, promote the alcohol oxidation reactions required for the formation of this molecule by releasing ellagitannins that are involved in the oxidation process (Chatonnet, 1991). This molecule always reaches and often exceeds its perception threshold in old white fortified wines.

Aging in 600-L barrels promotes oxidation of the wine, as oxygen seeps in through the bunghole and "barrel walls". This aging method may be compared with laboratory aging tests carried out in an oven, in the presence of air, but without the contribution from the wood. Aging in a vat is similar to laboratory test conditions in the absence of air (Cutzach et al., 1999). The same phenomena that had been identified in the laboratory were also observed under normal winemaking conditions (Cutzach et al., 1999).

Table 5. Influence of the Type of Containers on the Formation of the Volatile Compounds of a White Fortified Wine (Aging for 24 Years in a Winery without Air-Conditioning)[a]

volatile compounds (μg/L), oxidation	vat (24 years), weak	vat (14 years) and 600-L barrels (10 years), middle	600-L barrels (24 years), strong	sensory threshold (μg/L)
furanic derivatives				
furfural	8500	**17 000**	**23 000**	15 000
5-ethoxymethylfurfural	**150**	**186**	**131**	90
enolic derivatives				
sotolon	**95**	**254**	**413**	10
lactons				
trans-methyl-γ-octalactone	0	6	20	122
cis-methyl-γ-octalactone	0	17	**157**	35
volatile phenol				
eugenol	0	2	2.5	65
vanillin	**60**	**190**	**890**	15

[a] Boldface, concentration higher than perception threshold.

Furthermore, wood aging for 2−7 years, a traditional practice in making *vins doux naturels*, allows the wine to develop “vanilla” and “oaky” aromas which are generally identifiable on tasting (Torres et al., 1991). These aromas are due to the vanillin (**7**) and methyl γ-octalactone (**4** and **5**) released into the wine during wood aging.The older the wood, the smaller the amount of volatile phenols released into the *vins doux naturels*. However, only the vanillin content, responsible for the “vanilla” aromas very frequently found in old *vins doux naturels*, continues to increase with aging time. This substance plays a significant organoleptic role in the aroma of *vins doux naturels*. Its accumulation seems to be promoted by the highly oxidizing conditions in *vins doux naturels* and the use of very old oak barrels (Table 5). The oxidative breakdown of coniferaldehyde and terminal units of monomethoxylated lignin may produce vanillin (**7**). This is similar to the reactions that occur when spirits are aged in barrel (Puech, 1978).

Methyl-γ-octalactone (**4** and **5**), typical of oak (Masuda and Nishimura, 1981), was not present in control wines aged in vat. Considerable concentrations accumulated, however, in *vins doux naturels* aged in oak barrels. These compounds made an active contribution to the wines' aromas (I, 1). Extraction of methyl-γ-octalactone (**4** and **5**), seems to be facilitated when wine is aged in a non-air-conditioned winery. On tasting, the wine aged only in new wood was not preferred, as it was considered to be “overoaked” (Table 4). Unlike the vanillin content, which continues to increase with oxidation during aging in used barrels, concentration of *cis*- and *trans*-methyl-γ-octalactone (**4** and **5**) are much lower when wine is aged in used barrels.

CONCLUSIONS

The results obtained in this research have made it possible to interpret the development of seven molecules that contribute to the aroma of white *vins doux naturels* and the factors that affect their formation. This study provides additional information on the influence of storage and aging conditions.

Wooden containers and variations in temperature facilitate the formation of the volatile compounds investigated. Furfural (**1**), 5-ethoxymethylfurfural (**2**), and sotolon (**3**) always reach concentrations above their perception thresholds in wood-aged wines. This effect is enhanced in new wood. The development of the volatile compounds studied in this research is identical to that which took place in a model medium during laboratory tests.

From a practical standpoint, identifying and assaying isomers of methyl-γ-octalactone (**4** and **5**) may be a reliable indicator of wood aging in *vins doux naturels*. Among the many volatile phenols, only the vanillin (**7**) content increases with aging, particularly in wooden containers where there is a high level of oxidation. Concentrations of this molecule are very often above its perception threshold. The aromatic contribution of vanillin (**7**) and methyl-γ-octalactone (**4** and **5**) is important for the typical aging aroma in the *vins doux naturels* in our study.

The results obtained in this research have contributed toward a better understanding of the formation of sotolon (**3**), a vital molecule for the aroma of *vins doux naturels*. In particular, it is responsible for their “rancio” character. The experimental data obtained will contribute toward more effective control of the aging and oxidation conditions that are so important for *vins doux naturels*.

LITERATURE CITED

Berthelot. Action de l’oxygène sur les vins. *C. R. Acad. Sci.* **1863**, *57*, 398, 795, 963.

Berthelot. Action de l’oxygène sur les vins. *C. R. Acad. Sci.* **1864**, *57*, 80, 292.

Boidron, J. N.; Chatonnet, P.; Pons, M. Influence du bois sur certaines substances odorantes des vins. *Connaiss. Vigne Vin* **1988**, *22* (4), 275−294.

Cantarelli, C. Etude de la madérisation et de sa prévention dans les vins blancs. Fermentations et vinifications. *2nd Symposium International d’œnologie*; Bordeaux-Cognac, 1967; pp 391−407.

Chatonnet, P. *Incidences du bois de chêne sur la composition chimique et les qualités organoleptiques des vins--Applications technologiques*; Thèse de D. E. R, Université de Bordeaux II, 1991; No. 2, 224 p.

Chatonnet, P. *Influence des procédés de tonnellerie et des conditions d’élevage sur la composition et la qualité des vins élevés en fûts de chêne*; Thèse de Doctorat, Université de Bordeaux II, 1995; No. 338, 268 p.

Cutzach, I.; Chatonnet, P.; Henry, R.; Dubourdieu, D. Identification of volatile compounds with a “toasty” aroma in heated oak used in barrelmaking. *J. Agric. Food Chem.* **1997**, *45*, 2217−2224.

Cutzach, I.; Chatonnet, P.; Dubourdieu, D. Etude de l’arôme des vins doux naturels non muscatés. 1ére Partie: Analyse qualitative de l’arôme des vins doux naturels de Banyuls et Rivesaltes rencontré au cours de leur vieillissement. *J. Int. Sci. Vigne Vin.* **1998a**, *32*, 99−110.

Cutzach, I.; Chatonnet, P.; Henry, R.; Pons, M.; Dubourdieu, D. Etude de l’arôme des vins doux naturels non muscatés. 2ème Partie: Dosage de certains composés volatils intervenant dans l’arôme des vins doux naturels au cours de leur vieillissement. *J. Int. Sci. Vigne Vin.* **1998b**, *34*, 211−221.

Cutzach, I.; Chatonnet, P.; Dubourdieu, D. Role du sotolon dans l’arôme des vins doux naturels. Influence des conditions d’élevage et de vieillissement. *J. Int. Sci. Vigne Vin.* **1998c**, *34*, 223−233.

Cutzach, I.; Chatonnet, P.; Dubourdieu, D. Study of the formation mechanisms of some volatile compounds during the aging of sweet fortified wines. *J. Agric. Food Chem.* **1999**, *45*, 2837−2846.

Cutzach-billard, I. *Etude de l’arôme des vins doux naturels non muscatés au cours de leur élevage et de leur vieillissement*; Thèse de Doctorat, Université de Bordeaux II, 1999; No. 364, 150 p.

Deibner, L.; Benard, P. Recherche sur la maturation des vins doux naturels. I. Influence du traitement thermique prolongé à l’abri de l’air. *Ann. Technol.* **1956**, *3*, 357−376.

Dubois, P.; Rigaud, J.; Dekimpe, J. Identification de la diméthyl-4,5 tetrahydrofuranedione-2,3 dans le vin Jaune du Jura. *Lebensm.-Wiss. Technol.* **1976**, *9*, 366−368.

Martin, B.; Etievant. P. X.; Le Quere, J. L.; Schlich, P. More clues about sensory impact of sotolon in some flor sherry wines. *J. Agric. Food Chem.* **1992**, *40*, 475−478.

Masuda, M.; Nishumira, K. Absolute configuration of *Quercus* lactones, (3S,4R)-3-methyl-4-octanolide, from oak wood and chiroptical properties of monocyclic-*g*-lactones. *Chem. Lett.* **1981**, 1333−1336.

Puech, J. L. *Vieillissement des eaux de vie en fûts de chêne. Extraction de la lignine et de ses produits de dégradation*; Thèse, Université Paul Sabatier de Toulouse, 1978, No. 208, 768 p.

Ribereau-Gayon, J. Contribution à l’étude des oxidations et réductions dans les vins. *Application à l’étude du vieillissement et des casses*; Delmas: Bordeaux, France, 1933, 2nd ed.

Torres, P.; Brugirard, A.; Fanet, J.; Seguin, A. *La dégustation et le service des vins doux naturels à appellations d'origine contrôlées*; Université des vins du Roussillon, station vitivinicole du Roussillon, France, 1991.

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