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Changes in the Sotolon Content of Dry White Wines during Barrel and Bottle Aging

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GC-MS in electron ionization mode (EI) was used as a simple, sensitive method for assaying sotolon [4,5-dimethyl-3-hydroxy-2(5)*H*-furanone] in various dry white wines. The impact of barrel-aging conditions, that is, whether yeast lees were present or not, on the formation of sotolon in dry white wines was then studied. The sotolon content was highest in dry white wines aged in new barrels without lees, often exceeding the perception threshold (8 μg/L). These results demonstrated that yeast lees were capable of minimizing the formation of sotolon in dry white wines during aging. The sotolon and oxygen contents of several bottle of the same white wine were also compared 7 years after bottling. At the range of dissolved oxygen concentrations generally measured, between 5 and 100 μg/L, the sotolon content remained below its perception threshold in wine. The perception threshold was exceeded only in wines with oxygen concentrations above 500 μg/L. The presence of dissolved oxygen in the wine samples analyzed also resulted in a decrease in their free sulfur dioxide content.

KEYWORDS: Sotolon; white wines; aging; oxygen

INTRODUCTION

Sotolon [4,5-dimethyl-3-hydroxy-2(5)*H*-furanone] is a volatile compound with an intense odor of curry. A great deal of research has been done in recent years on the sensory properties of this compound and its contribution to the aromas of various foods (*1–6*). Sotolon contributes to the aromas of "vins jaunes" from the Jura and sherries (*7, 8*), as well the "dried fig" and "rancio" nuances in French fortified wines [Vins doux Naturels (VDN)] and port (*9, 10*). Concentrations in white wines made from grape varieties such as Savagnin increase during aging with yeast "flor", as well as during the barrel aging of sweet wines (*9, 11, 12*). Wines of this type over 20 years old may contain up to 1 mg/L sotolon. This furanone has also been detected in white wines made from botrytized grapes (*13*). More recently, several authors (*14–16*) determined the contribution of sotolon to the oxidation aromas of prematurely aged dry white wines. Oxidation phenomena are involved in generating sotolon in wine (*9, 14, 17–19*). According to Cutzach (*10*), this defect is accentuated in bottles of fortified wine with imperfect seals due to faulty corks.

Assaying sotolon in as complex a dilute-alcohol matrix as wine requires a specific, highly sensitive method. A great deal of work has been done in recent years on developing separation methods capable of assaying sotolon concentrations in the micrograms per liter range (*8, 10, 20–22*).

The physicochemical properties of sotolon are now well-known. This thermolabile, polar compound is highly soluble in dilute-alcohol solution (*5, 23*). Sotolon is stable at the pH of wine, so assay results are not disturbed by short-term exposure to a neutral or slightly alkaline pH (*20*). The most sensitive assay methods described in the literature use gas-phase chromatography coupled with mass spectrometry (GC-MS). Volatile compounds are usually extracted from the wine using organic solvents. According to Ferreira (*24*), certain macroreticular resins have a greater capacity for extracting sotolon from wines than standard polar solvents (ether, dichloromethane, or ethyl acetate). This author also proposes a solid/liquid extraction method, by which sotolon is fixed on a resin and then desorbed by dichloromethane and the extract is analyzed by GC-MS (*22*). Assay methods using marked sotolon (^{13}C, deuterated) as an internal standard are the most sensitive (*20*). Repeatability of the assay was also satisfactory. However, it may sometimes be difficult to synthesize this analogue (*25*), making it tedious to prepare and assay the sample.

This paper describes a simple, sensitive method for assaying sotolon in wine by GC-MS. We used this method to monitor changes in the sotolon content of a white wine during barrel aging.

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MATERIALS AND METHODS

Reagents. 3-Octanol (>99%), 4,5-dimethyl-3-hydroxy-2(5)*H*-furanone (>99%), and dichloromethane (grade) were from Sigma-Aldrich (St Quentin Fallavier, France). Anhydrous sodium sulfate (99%) and sodium sulfite (99%) were supplied by Prolabo (France).

Wine Samples. Commercial white wines from several vintages (2–32 years old) were all analyzed in 2005. The grape varieties and appellation are presented in **Table 3**. For each wine, duplicate sotolon analyses took place immediately after bottle opening.

Commercial active dry yeast, *Saccharomyces cerevisiae* (ZYMAFLORE VL3 strain) was provided by Laffort (Floirac, France). Yeast cells were rehydrated according to the manufacturer's instructions. White Sauvignon Blanc wine was made from grapes harvested at Chateau Reynon in 2002.

Sotolon was assayed in wines that had been fermented and aged in new or used French oak barrels, with or without lees, for 8 months after the end of alcoholic fermentation. When Sauvignon Blanc was aged without lees, the lees were removed just after the end of alcoholic fermentation. Each assay was carried out in triplicate. The sulfur dioxide concentration was maintained constant (25 mg/L) throughout the experiments. Lees were maintained in suspension by stirring once per week. After 8 months of barrel aging, 200 mL samples were taken from each wine. Samples were clarified by centrifuging (Beckmann, 5000 rpm, 20 min) prior to analysis.

Assaying Dissolved Oxygen in Bottle. Dissolved oxygen was measured using an oxygen electrode. This polarographic probe, patented by Orbisphere (model 31120), was equipped with a Derlin circulation chamber and connected to a piercing-sampling system capable of measuring the dissolved oxygen content directly in a sealed bottle by creating a nitrogen counterpressure (Linde gas). The system was calibrated daily in saturated air at room temperature. A test, consisting of measuring very low oxygen concentrations, was carried out in deoxygenated water, made by adding sodium sulfite. The electrode response was considered to be optimum in this test when an oxygen content below 10 μg/L was measured in under 10 min (manufacturer's recommendations). The liquid flow rate was maintained at 130 mL/min during measurement. Each measurement was made in duplicate.

Sotolon Extraction. The extraction procedure was based on the method described by Cutzach (*10*). Wine samples (100 mL) were spiked with 100 μL of 3-octanol in a 100 mg/L alcohol solution as an internal standard and 15 g of anhydrous sodium sulfate (its higher ionic strength increases extractability). Wines were extracted three times with 10, 5, and 5 mL of CH_2Cl_2 (magnetic stirring, 10, 5, and 5 min; 750 rpm). The three organic phases obtained were blended, dried over anhydrous sodium sulfate, and concentrated to 0.5 mL under a nitrogen stream. Two microliters of the extract was injected into the GC with an MS detector.

Gas Chromatography−Mass Spectrometry (GC-MS) Conditions. A Star 3400CX gas chromatograph fitted to a Saturn 2000 electronic ion trap mass spectrometer from Varian was used to analyze the organic extract. We used two types of capillary columns: The first was a fused silica column coated with SPB1 (apolar) from Supelco (Saint Quentin Fallavier, France), 30 m × 0.25 mm; film thickness = 1 μm. The second column was coated with BP-20 (polar) from SGE (France), 50 m × 0.22 mm; film thickness = 0.25 μm. The carrier gas was He (Linde gas, Bordeaux), N55 grade, with a flow rate of 0.9 mL/min (10 psi for SPB1, 20 psi for BP20). A Varian 1078 temperature programmable injector was used to inject the 2 μL sample. The injector was initially set at 180 °C for 0.3 min, and then the temperature was raised to 230 at 180 °C/min for 30 min. Oven temperature (SPB1) was initially set at 45 °C for 1 min, then raised to 200 °C at 3 °C/min and to 270 °C at 15 °C/min and held at that temperature for a further 10 min. In the case of the BP20 column, the oven temperature was initially held at 45 °C for 1 min, then raised to 230 °C at 3 °C/min, and maintained at this temperature for a further 30 min. The temperature-programmable injector was the same as previously described. The transfer line and manifold were maintained at 210 and 80 °C, respectively. Trap temperature was maintained at 170 °C. Axial modulation was 3.5 V. Injection (2 μL) was in splitless mode (closure time = 0.75 min). Data acquisition was segmented. The first detection segment corresponded to the internal standard (t_R = 19–23 min) and the second to sotolon (t_R = 24–28 min). Current intensity was 10 μA for the first acquisition phase and 20 μA during the acquisition of sotolon. The voltage value (offset multiplier) was 10 V for the first segment and 100 V for the second. The mass spectra were acquired in electron impact (SIS mode, ionization energy = 70 eV) between masses 57 and 85 for the first segment and masses 81 and 130 for the second segment. Ions 59 and 83 were used to quantify octanol-3 and sotolon, respectively. Ion 128 confirmed the presence of sotolon.

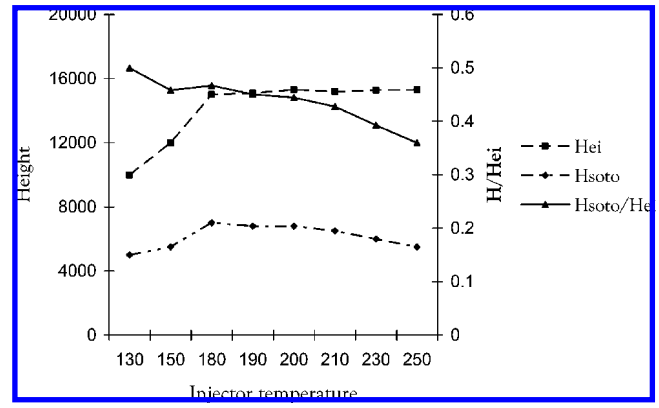

Figure 1. Impact of injector temperature on the sotolon and octanol-3 responses in GC-MS.

Table 1. Impact of the Type of Column on the Quantification (Height of the Chromatogrpahic Peak) of Sotolon and Octanol-3

	BP20	SPB1
sotolon	10243 (912)	26808 (884)
octanol-3	52111 (2605)	50112 (1504)

Table 2. Desorption Temperature of Sotolon and Octanol-3 According to the Type of Column

	BP20	SPB1
sotolon	205 °C	120 °C
octanol-3	117 °C	108 °C

Measuring Free SO_2. The free SO_2 content was determined by iodometry as described by Ribereau-Gayon (*26*).

Statistical Analysis. The StatBox 6.22 statistical package (Grimmer Logiciel, Paris, France) was used for all statistical calculations. Experimental results are expressed as means (SD).

RESULTS AND DISCUSSION

This method for assaying sotolon in wine does not require any preliminary extraction of the sample and uses a commercially available internal standard.

Impact of Injection Temperature. The thermodegradation of sotolon is one of the known properties applied in this on-column assay mode (*20, 23*). We determined the optimum injector temperature, under our assay conditions. A model solution (EtOH 12% vol; 4 g/L tartaric acid, pH 3.5) supplemented with 10 μg/L sotolon was extracted, as previously described. The organic extract obtained was analyzed repeatedly by GC-MS, with the injector temperature varied from 130 to 250 °C. The results obtained are shown in **Figure 1**. We demonstrated that, above 180 °C, a fraction of the sotolon was degraded in the injector and the octanol-3 was completely volatilized. Injection temperature was, therefore, programmed at 180 °C.

Impact of the Type of Column. Polar capillary columns (grafted with polyethylene glycol) are most commonly used for

Table 3. Examples of Sotolon Concentrations in Several Sauvignon Blanc, Semillon, Chardonnay, and Savagnin Wines from Different Vintages Made under Reducing (R) or Oxidizing (O) Conditions

code	grape variety	appellation	winemaking conditions	vintage	sotolon (μg/L)
1	Sauvignon Blanc	Pessac Leognan	R	1997	5.2 (0.2)
2	Sauvignon Blanc	Pessac Leognan	R	1997	1.1 (1.1)
3	Sauvignon Blanc	Pessac Leognan	R	1997	traces
4	Sauvignon Blanc	Pessac Leognan	R	1992	10 (0.4)
5	Sauvignon Blanc	Entre-Deux-Mers	R	2003	7 (0.3)
6	Sauvignon Blanc	Entre-Deux-Mers	R	2001	0.9 (0.2)
7	Sauvignon Blanc and Semillon	Pessac Leognan	R	1983	1.7 (0.3)
8	Sauvignon Blanc	Graves	R	1985	1.4 (0.3)
9	Sauvignon Blanc	Graves	R	1980	1.1 (0.2)
10	Sauvignon Blanc and Semillon	Pessac Leognan	R	1975	6.1 (0.7)
11	Sauvignon Blanc	Pessac Leognan	R	1973	5.8 (0.5)
12	Sauvignon Blanc	Sancerre	R	1999	2.3 (0.2)
13	Chardonnay	Pouilly Fuissé	R	2001	4.7 (0.1)
14	Chardonnay	Pouilly Fuissé	R	2001	3.1 (0.1)
15	Chardonnay	Pouilly Fuissé	R	2002	3.0 (0.1)
16	Chardonnay	Saint Veran	R	2001	1.1 (0.1)
17	Chardonay	Vin de pays d'Oc	R	2001	2.8 (0.3)
18	Roussette	Roussette de Savoie	R	2001	1.9 (0.2)
19	Roussette	Roussette de Savoie	R	2002	4.3 (0.2)
20	Roussette	Roussette de Savoie	R	2000	1.9 (0.3)
21	Colombelle	Côte de Gascogne	R	1999	2.5 (0.5)
22	Colombelle	Côte de Gascogne	R	1998	6.9 (2.2)
23	Colombelle	Côte de Gascogne	R	2000	2.2 (0.4)
24	Colombelle	Côte de Gascogne	R	1998	4.1 (0.3)
25	Savagnin	Château Chalon	O	1995	52 (3.3)
26	Savagnin	Château Chalon	O	1997	46 (1.1)
27	Savagnin	Château Chalon	O	1989	43 (0.9)
28	Savagnin	Château Chalon	O	1996	41 (1.5)
29	Savagnin	Château Chalon	O	1987	140 (3.4)

assaying sotolon in wine (*16, 19, 22*). They are suited to appropriate temperature programming, separation, and quantification of sotolon in wine. However, the oven temperature at which sotolon is desorbed from the column generally exceeds 180 °C. Under these conditions, it is highly probable that some of the sotolon is degraded in the column. We tested this hypothesis by injecting the same organic wine extract supplemented with 20 μg/L sotolon into polar (BP20) and nonpolar columns (SPB1). The height of the chromatography peak associated with sotolon on the nonpolar column was over twice as high as it was on the polar column (**Table 1**). Apparently, the sotolon was degraded in the capillary analysis column when it was desorbed at high temperatures: 205 °C on BP20 (**Table 2**). These results contradict the findings of Blank (*27*), thus justifying the use of polar phases to assay sotolon by GC-MS. In this way, we showed that the use of a nonpolar phase with a low affinity for sotolon was particularly well-suited to assays in wine.

Calibration Graphs, Repeatability, Sensitivity. The standard range was prepared by adding increasing concentrations of sotolon, from 1 to 20 μg/L, to a dry white wine. The height of the sotolon peak (*m*/*z* 83) compared to the internal standard peak (*m*/*z* 59) correlated with the sotolon concentration in the sample in a linear manner. The regression equation is as follows: [sotolon] (μg/L) = 43.59(h/hei) − 0.57 (R^2 = 0.998). Repeatability of the analysis for a series of five measurements in a single wine sample containing 6 μg/L sotolon was 5.2%. The detection threshold for sotolon in dry white wine was 0.8 μg/L, with a signal/noise ratio of three. This value is adequate to evaluate the sensory contribution of sotolon to white wines, because it is well below the corresponding threshold value (8 μg/L).

Application to Sotolon Assays in Wines. *Assaying Sotolon in Several Dry White Wines.* The sotolon content was assayed in dry white wines of different origins and vintages (**Table 3**). All of the wines analyzed were assessed by a training panel and chosen for their oxidative flavors. Wines made under reducing conditions were considered to have nontypical oxidative aging flavors, whereas these aromas were normally associated with wines made under oxidizing conditions.

Sotolon concentrations varied not only from one wine to another but also according to the vintage. The amount of sotolon in the 29 wine samples analyzed ranged from 0.9 to 140 μg/L. The sotolon content of most samples was below the perception threshold (8 μg/L). The distribution of sotolon concentrations in the wines in **Figure 2** shows the log transform of sotolon content in wine samples from the different grape varieties. Due to the distribution and low number of samples per grape variety studied, nonparametric tests, that is, Kolmogorov–Smirnov and Kruskal–Wallis, were used to check differences. The Savagnin wines (aged in barrels with a yeast flor) differed from those of the other grape varieties, according to all of the tests used. No other differences were observed between wines (reducing conditions group) made from different grape varieties. These differences correlated with the presence of oxidation conditions during winemaking.

The 1992 Sauvignon Blanc sample had an odor reminiscent of "rancio" wines and a very high sotolon content for a dry white wine (10 μg/L), although it had not been aged under oxidation conditions. In contrast to the results obtained by Camara (*12*) for Madeira wine, there was no apparent correlation between the sotolon concentration of the dry white wines analyzed and the vintage (R^2 = −0.192). Its formation in dry

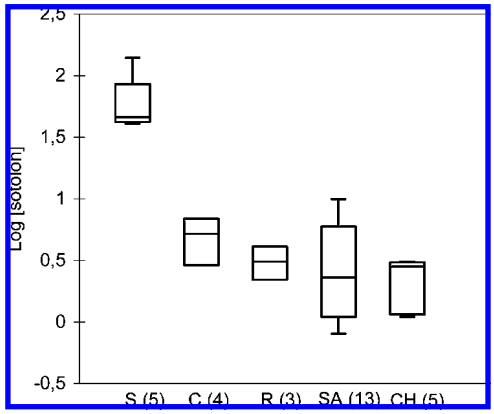

Figure 2. Box plots of the logarithm of sotolon content in prematurely aged wine samples from different French grape varieties. The thick horizontal lines are the medians. The upper and lower edges of the boxes enclose 50% of the data. S, Savagnin; C, Colombelle; R, Roussette; SA, Sauvignon Blanc; CH, Chardonnay. Numbers of samples are given in parentheses.

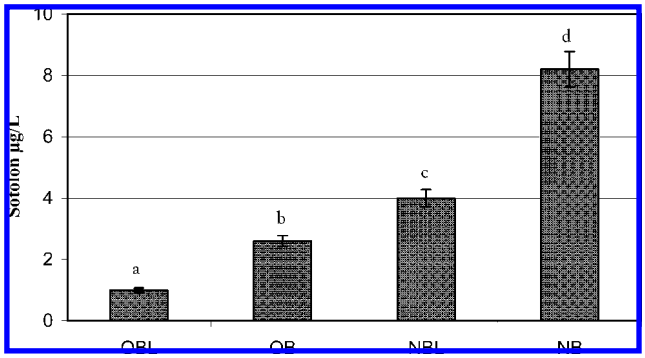

Figure 3. Changes in the sotolon content of a white wine after 8 months of barrel aging, $n = 3$. OBL, old barrels with lees; OB, old barrels without lees; NBL, new barrels with lees; NB, new barrels without lees. Letters a−d indicate significant differences between sotolon concentrations ($p < 0.05$).

white wines is apparently promoted or even triggered by unknown parameters.

Changes in the Sotolon Content during the Barrel-Aging of Dry White Wines. As sotolon is a marker for defective aging in dry white wines, we monitored changes in the sotolon content of a white Sauvignon Blanc wine, fermented and aged in new or used barrels, with or without total lees. During aging, the free sulfur dioxide content was maintained at 25 mg/L and the lees were kept in suspension by stirring once per week.

The presence of lees in dry white wines during 8 months of barrel-aging (**Figure 3**) delayed the formation of sotolon, considered to be an aging defect in this type of wine. It was detected in all of the wines, irrespective of the aging method. The nonparametric Kruskal–Wallis test was used to detect significant differences among assays, with a significance level of 5%. When statistically significant differences were found for sotolon, wine aging conditions were compared using the Mann–Whitney test ($p < 0.05$). The sotolon content was highest in new barrels without lees, often exceeding the perception threshold in dry white wine (8 μg/L). These results demonstrated that yeast lees were capable of minimizing the formation of sotolon in dry white wines during aging. The lees' capacity to combine oxygen (*28*) probably explains their protective effect in preventing sotolon formation.

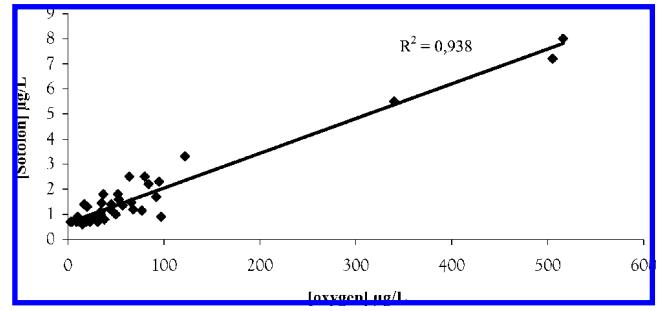

Figure 4. Correlation between dissolved oxygen concentrations and sotolon content measured in bottle.

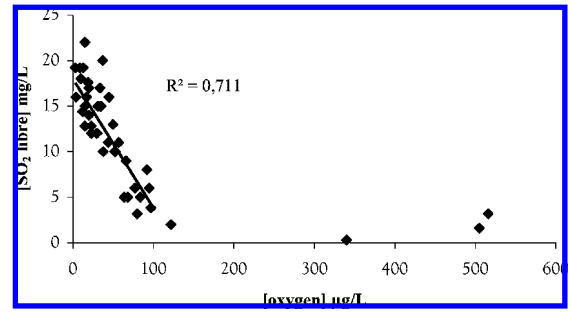

Figure 5. Correlation between dissolved oxygen content and free SO_2 concentrations measured in bottle.

Sotolon Changes in Dry White Wines during Bottle Aging: Impact of Dissolved Oxygen Content. The chemical mechanism responsible for the formation of sotolon in wine involves oxygen. This explains the high sotolon content found in wine aged under oxidation conditions, for example, *vin jaune* from the Jura (*18*), port, and *vins doux naturels* (French fortified wines) (*10*). According to Cutzach (*10*), sotolon concentrations are considerably higher in VDN bottles with defective corks (leaky bottles).

Dry white wines are traditionally protected from oxygen during the winemaking process. During barrel aging, the presence of yeast lees and sulfur dioxide minimizes the attenuation of varietal aromas as well as prevents sotolon formation (*15*). It is relatively common for the aromas of white wines aged in bottle to age abnormally rapidly and develop defects. According to several authors, the variability of this aromatic deterioration is due to considerable differences in permeability to oxygen among cork stoppers (*29, 30*). For all of these reasons, we felt it was useful to monitor the involvement of oxidation phenomena in sotolon formation during the bottle aging of dry white wines.

Forty bottles of dry white Pessac Léognan wine, from the same 1997 vintage and bottling batch, were analyzed after 7 years in bottle. The bottles were sealed with top-grade natural corks. The dry white wine samples analyzed had variable dissolved oxygen contents, which were very highly correlated with the sotolon concentration ($R^2 = 0.938$) (**Figure 4**). Dissolved oxygen concentrations were usually between 5 and 100 μg/L. At this range of oxygen concentrations, the sotolon content remained below its perception threshold in wine (8 μg/L), exceeding this value only when the oxygen content was above 500 μg/L. The presence of dissolved oxygen in the wine samples analyzed also resulted in a decrease in their free sulfur dioxide content (**Figure 5**). These results clearly demonstrated the role of low oxygen concentrations in the formation of sotolon in dry white wines aged in bottle.

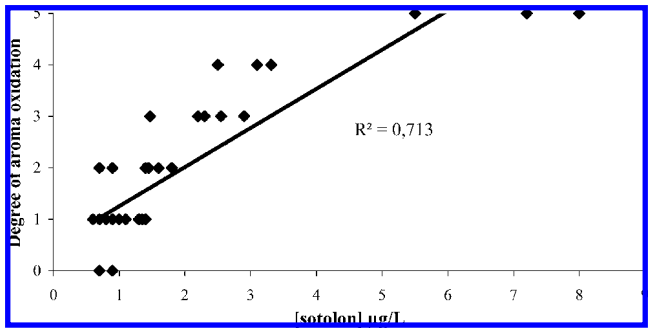

Figure 6. Correlation between sotolon concentration and degree of oxidation of dry white wines analyzed.

In addition to this chemical analysis of defective aging of white wines, a trained jury made a sensory evaluation of the correlation between sotolon content and the degree of oxidation (**Figure 6**). The correlation coefficient calculated ($R^2 > 0.7$) clearly indicated that the perception of oxidation aromas in aged wines was dependent on the sotolon concentrations present. These findings indicate that this furanone is a good "marker" for the defective aging phenomenon which affects white wines during bottle aging.

Conclusion. A conventional GC-MS method for assaying high concentrations of sotolon in VDN was optimized to develop a sensitive method for analyzing traces of sotolon in dry white wines. This technique was used to monitor the sotolon concentrations in several wines. We demonstrated that the sotolon content of these wines was much lower than that of other alcoholic beverages: fortified wines, port, and sherry. These differences are due to the specific composition of dry white wines and the winemaking methods used (reducing conditions). We showed that barrel-aging conditions had a significant impact on the formation of sotolon in dry white wines: higher concentrations were produced under conditions more conducive to oxidation. We also showed that the uncontrolled permeability to oxygen of cork stoppers during bottle aging had a significant impact on the formation of sotolon and the perception of oxidation flavors in dry white wines.

LITERATURE CITED

(1) Blank, I.; Sen, A.; Grosch, W. Potent odorants of the roasted power and brew of Arabica coffee. *Z. Lebensm. Unters. Forsch.* **1992**, *195*, 239–245.

(2) Blank, I.; Schieberle, P. Analysis of the seasoning-like flavour substance of a commercial lovage extract (*Levisticum officinale* Koch). *Flavour Fragrance J.* **1993**, *8*, 191–195.

(3) Karagül-Yüceer, Y.; Drake, M. A.; Cadwallader, K. R. Aroma-active components of nonfat dry milk. *J. Agric. Food Chem.* **2001**, *49*, 2948–2953.

(4) Kirchhoff, E.; Schieberle, P. Determination of key aroma compounds in the crumb of a three-stage sourdough rye bread by stable dilution assays and sensory studies. *J. Agric. Food Chem.* **2001**, *49*, 4304–4311.

(5) Girardon, P.; Sauvaire, Y.; Baccou, J.-C.; Bessière, J.-M. Identification de la 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone dans l'arôme des graines de fenugrec (*Trigonella foenum graecum* L.). *Lebensm. -Wiss. -Technol.* **1986**, *19*, 44–46.

(6) Kobayashi, A. Sotolon identification, formation, and effect on flavor. *Flavor Chemistry Trends and Development*; ACS Symposium Series 388; American Chemical Society" Washington, DC, 1989; pp 49−59.

(7) Dubois, P.; Rigaud, J.; Dekimpe, J. Identification de la Diméthyl-4,5-tertrahydrofuranedione-2,3 dans le Vin Jaune du Jura. *Lebensm.-Wiss. -Technol.* **1976**, *9*, 366–368.

(8) Guichard, E.; Pham, T. T.; Etievant, P. Quantitative determination of sotolon in wines by high-performance liquid chromatography. *Chromatographia* **1993**, *37*, 539–541.

(9) Silva Ferreira, A. C.; Barbe, J. C.; Bertrand, A. 3-Hydroxy-4,5-dimethyl-25*H*)-furanone: a key odorant of the typical aroma of oxidative aged port wine. *J. Agric. Food Chem.* **2003**, *51*, 4356–4363.

(10) Cutzach, I.; Chatonnet, P.; Dubourdieu, D. Rôle du sotolon dans l'arôme des vins doux naturels, influence des conditions d'élevage et de vieillissement. *J. Int. Sci. Vigne Vin* **1998**, *32* (4), 223–233.

(11) Cutzach, I.; Chatonnet, P.; Dubourdieu, D. Influence of storage conditions on the formation of some volatiles compounds in white fortified wines (vins doux naturels) during the aging process. *J. Agric. Food Chem.* **2000**, *48*, 2340–2345.

(12) Camara, J. S.; Marques, J. C.; Alves, M. A.; Silva Ferreira, A. C. 4,5-Dimethyl-3-hydroxy-2(5*H*)-furanone levels in fortified Madeira wines: relationship to sugar content. *J. Agric. Food Chem.* **2004**, *52*, 6765–6769.

(13) Masuda, M. Identification of sotolon and ethyl 9-hydroxynonanoate in botrytised wine and evaluation of the roles of compounds characteristic of it. *Agric. Biol. Chem.* **1984**, *48,* (11), 2707–2710.

(14) Escudero, A.; Cacho, J.; Ferreira, V. Isolation and identification of odorants generated in wine during its oxidation: a gas chromatography−olfactometric study. *Eur. Food Res. Technol.* **2000**, *211*, 105–110.

(15) Lavigne-Cruège, V.; Dubourdieu, D. Role of glutathione on development of aroma defects in dry white wines. In *13th International Enology Symposium*, Trogus, H.; Gafner, J.; Sutterlin., Eds. International Association of Enology: Montpellier, France, 2002; pp 331−347.

(16) Silva Ferreira, A. C.; Hogg, T.; Guedes de Pinho, P. Identification of key odorants related to the typical aroma of oxidation-spoiled white wines. *J. Agric. Food Chem.* **2003**, *51*, 1377–1381.

(17) Cutzach, I.; Chatonnet, P.; Dubourdieu, D. Study of the mechanisms of some volatile compounds during the aging of sweet fortified wines. *J. Agric. Food Chem.* **1999**, *47*, 2837–2846.

(18) Pham, T. T.; Guichard, E.; Schlich, P.; Charpentier, C. Optimal conditions for the formation of sotolon from α-ketobutyric acid in the French Vin Jaune. *J. Agric. Food Chem.* **1995**, *43*, 2616–2619.

(19) Silva Ferreira, A. C.; Guedes de Pinho, P.; Rodrigues, P.; Hogg, T. Kinetics of oxidative degradation of white wines and how they are affected by selected technological parameters. *J. Agric. Food Chem.* **2002**, *50*, 5919–5924.

(20) Dagan, L.; Schneider, R.; Lepoutre, J. P.; Baumes, R. Stability of sotolon in acidic and basic aqueous solutions. Application to the synthesis of deuterated analogue for its quantitative determination in wine. *Anal. Chim. Acta* **2006**, *563*, 365–374.

(21) Martin, B.; Etievant, P. Quantitative determination of solerone and sotolon in flor sherries by two-dimensional-capillary GC. *J. High Resolut. Chromatogr.* **1991**, *14*, 133–135.

(22) Ferreira, V.; Jarauta, I.; Lopez, R.; Cacho, J. Quantitative determination of sotolon, maltol, and free furaneaol in wine by solid-phase extraction and gas chomatography-ion-trap mass spectrometry. *J. Chromatogr., A* **2003**, *1010*, 95–103.

(23) Martin, B.; Etievant, P.; Henry, R. The chemistry of sotolon: a key parameter for the study of a key component of flor sherry wines. In *Flavour Sciences and Technology*; Bessière, Y., Ed.; Wiley: Chichester, U.K., 1990; pp 53−56.

(24) Ferreira, V.; Ortega, L.; Escudero, A.; Cacho, J. A comparative study of the ability of different solvents and adsorbents to extract aroma compounds from alcoholic beverages. *J. Chromatogr. Sci.* **2000**, *38*, 469–476.

(25) Blank, I.; Schieberle, P.; Grosch, W. Quantification of the flavour compounds 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone and 5-ethyl-3-hydroxy-4-methyl-2(5*H*)-furanone by stable isotope dilution assay. In *Progress in Flavour Precursor Studies* Schreier, P., Winterhalter, P., Eds.; Allured: Wheaton, IL, 1992; pp 103–109.

(26) Ribereau-Gayon, P. Connaissance de la nature des combinaisons de l'anhydride sulfureux dans les vins. *Bull. O.I.V.* **1972**, *507*, 406–413.
(27) Blank, I., Gas-chromatography−olfactometry in food aroma analysis. In *Techniques for Analyzing Food Aroma*; Marsily, R., Ed.; Dekker: New York, 1997.
(28) Salmon, J. M.; Fornaison, C.; Moutounet, M. Modification chimique des lies consécutive à l'apport d'oxygène pendant l'élevage des vins sur lies. *Oenologie 99, 6eme Symposium International d'oenologie*; Tec et Doc: Paris, France, 1999; pp 428−432.
(29) Sanchez, J.; Aracil, J. M. Gaseous permeability of different obturators. *Bull. O.I.V.* **1998**, *71*, 279–283.
(30) Lopez, P. *L'étude des phénomènes oxydatifs pendant le vieillissement des vins en bouteille. Rôle de l'obturateur*; Victor Ségalen Bordeaux 2: Bordeaux, France, 2005.

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