

Identification of Key Odorants Related to the Typical Aroma of Oxidation-Spoiled White Wines

ANTONIO CÉSAR SILVA FERREIRA,* TIMOTHY HOGG, AND
 PAULA GUEDES DE PINHO

Escola Superior de Biotecnologia, Universidade Católica Portuguesa,
 R. Dr. António Bernardino de Almeida, 4200-072 Porto, Portugal

The oxidative degradation of white wines rapidly leads to a loss of their sensorial qualities. The identification of the most important descriptors related with oxidation-spoiled wine was performed by a trained sensory panel. The terms selected were “honey-like”, “farm-feed”, “hay”, and “woody-like”. By gas chromatography–olfactometry analysis three aromatic zones related to these descriptors in the oxidation-spoiled white wines could be determined. Comparison of the aroma extract dilution analysis aromagrams of oxidation-spoiled white wines and a nonspoiled wine showed the highest values of dilution factors were attributed to 3-(methylthio)propionaldehyde, phenylacetaldehyde, 1,1,6-trimethyl-1,2-dihydronaphthalene (TDN), and 4,5-dimethyl-3-hydroxy-2(5*H*)-furanone (sotolon). A “forced aging” experiment was implemented to simulate the typical oxidation-spoiled aroma. Samples rated with the highest score in the ranking test were also those that presented the highest concentration of these four molecules. To test the sensory impact of these substances, a normal wine (unspoiled) was spiked with these molecules (with the exception of TDN) singly and in combination, and the similarity value (SV) between samples and the oxidation-spoiled white wines was then determined. The highest value from the similarity tests was 5.4 when the three compounds were added simultaneously; 3-(methylthio)propionaldehyde alone was found to be responsible for 3.6, suggesting that, among the molecules studied, it is the most important contributor to the typical aroma of an oxidation-spoiled white wine.

KEYWORDS: White wine aroma; oxidative degradation; 3-(methylthio)propionaldehyde; phenylacetaldehyde; 1,1,6-trimethyl-1,2-dihydronaphthalene; 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone

INTRODUCTION

Oxidation/reduction (redox) phenomena play important roles in many parts of the wine-making process, causing profound alterations in wine composition. Such composition changes, occurring before, during, and after the fermentation process, influence greatly the color and aroma of the finished wine. Whether the specific alterations are themselves positive or negative, with regard to the quality of the final product, depends on the style of wine intended; a specific color and aroma profile that defines a certain wine style might be unacceptable in another. It is recognized that aromatic deterioration occurs prior to the chromatic changes (1–3).

Recent works presented information about the chemical compounds associated with “oxidative degradation” of white wines (3–7). The presence of “off-flavors” has been attributed to compounds such as 3-(methylthio)propionaldehyde (methylthional), 4-propenyl-2-methoxyphenol (eugenol), 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone (sotolon), and 2,4,5-trimethyl-1,3-dioxolane (4).

On the other hand, it has been shown that cyclic acetals are closely related to the progressive formation of ethanal and sotolon during oxidative aging of Port wines (8, 9). However, it was shown that the contribution of ethanal to the global aroma of oxidation-spoiled white wines is not so relevant as expected (6). 1,1,6-Trimethyl-1,2-dihydronaphthalene (TDN), a substance responsible for the “kerosene” aromas typical of old wines from the Riesling variety, has been reported as increasing in concentration during “accelerated” oxidation (10). Attempts were also made to predict the formation “off-flavors” of white wines based on a mathematical model using concentration data from different substances (6).

In the present study sniffing analysis is employed to screen for the presence of aromatic zones with odors that can be related with descriptors, previously selected by a trained panel as characteristic of “precocious aging”. The relative importance of each of the odor zones was ranked by the aroma extract dilution analysis (AEDA) technique (11). This technique is based on sniffing analysis and successive dilution of the extracts, enabling comparison of the respective aromagrams and selection of the substances of greatest interest for identification as potentially responsible for undesirable aromas.

* Author to whom correspondence should be addressed (e-mail ferreira@morango.esb.ucp.pt).

Table 1. White Wine Chemical Parameters

age (years)	pH	dissolved oxygen (mg/L)	free SO ₂ (mg/L)	combined SO ₂ (mg/L)
11	3.26	0.9	8	34
10	3.29	0.4	6	42
9	3.17	0.1	6	48
6	3.18	0.7	nd ^a	38
5	3.53	0.1	13	42
4	3.48	0.7	16	42
3 ^b	3.19	0.9	26	58
2	3.30	0.4	16	51
1	3.24	1.0	25	66

^a Not detected. ^b Oxidation-spoiled wine.

Previous works concerning the relationship between some technologic parameters and the rate of formation of off-flavors have shown that oxidative degradation is associated with low pH and too high temperatures of storage and high oxygen content (7). Thus, this work will attempt to correlate the molecules identified in oxidation-spoiled white wine as potentially responsible for off-flavors and the chemical contents and the sensorial notes gathered during the forced aging experiment.

MATERIALS AND METHODS

Wine Material. The wines used in this study were dry white wines, of the same commercial brand produced in the Dão region (Portugal) coming from Encruzado and Assario varieties, from nine different vintages (1–11 years old) following standard wine-making procedures and without wood contact. Wine chemical parameters are shown in **Table 1**. The ethanol content is close to 12%.

Forced Aging Experimental Protocol. This protocol is similar to that of previous work (7). A volume of 2000 mL of white wine, corresponding to a 6-year-old vintage (**Table 1**), a pH was divided into four portions; the parameters studied were adjusted as follows: (i) pH—one of the portions was kept at pH 3.2, whereas another was adjusted to pH 4 by adding K₂CO₃; (ii) oxygen content with (6.5 mg/L) by air bubbling (20:80; O₂/N₂) (Gasin, Portugal) and without saturation; (iii) free SO₂ levels absent [not detected by the analytical method (12)] and at 50 mg/L (free form by adding potassium metabisulfite); (iv) untreated wine (control). Each set was kept for 7 days at three different storage temperatures (15, 45, and 60°C) and then analyzed.

Sensory Studies. Descriptor Selection. The sensory panel employed in all sensorial measurements in this work was composed of 12 persons: university students, wine-makers, and laboratory personnel. The panel is a permanent wine evaluation resource, which receives weekly training sessions. Tests were performed in individual booths, using tulip glasses containing 30 mL of wine at room temperature (25 °C). The descriptor selection was effected using a white wine that was, in several sessions, unanimously considered to be “oxidation-spoiled”. The procedure used to select the most important descriptors was AFNOR NFV-09-021 (13). In a first set of sessions, every member of the panel was asked to freely describe the aroma of the spoiled wine. The hedonic and redundant terms, as well as the nonpertinent terms, were then disregarded, and a first group of descriptors was thus obtained. Then, the panel was asked to determine if the first series of descriptors were present or absent in the reference spoiled wine. Those descriptors considered as absent by 50% of the panel were eliminated, and a second group was obtained. The panel was then asked to rank each descriptor belonging to this group on a scale of 0–10. The descriptors that obtained the highest ratings were “honey-like”, “farm-feed”, “hay”, and “woody-like”.

Ranking Testing (Forced Aging). After 7 days of storage, the samples were presented simultaneously to the panel. The panel members were asked to order each of the 12 samples by its “degree of spoilage”, from the least to the most spoiled; equal ranks were not allowed. Data were treated according to a “ranking test” (14), and statistical significance was evaluated using “Basker tables” (15).

Similarity Testing. The identified aroma-active compounds were added in a single and combined manner to an unspoiled wine (a 4-year-old wine, **Table 1**). The similarity value (SV) was determined by a comparison test. Each supplemented sample (coded) was presented to the panel together with the oxidation-spoiled white wine (a 3-year-old wine, **Table 1**) as a pair, and the panel was asked to rate the similarity on a discontinuous scale from 0 (no similarity) to 10 (equal) of each sample with the oxidation-spoiled white wines. The data obtained were treated according to the ANOVA procedure.

Organic Extract Selection. Different organic solvents, hexane, ether, ethyl acetate, and dichloromethane, were used to obtain extracts from the oxidation-spoiled white wine. Similarity tests were performed between the aroma of the obtained extracts and the wine (16). Two milliliters of each organic extract was concentrated under a nitrogen stream to 0.5 mL. A drop was then put on a “perfume sampling paper”, and the aroma was compared with that of the original wine.

Gas Chromatography—Olfactometry (GC-O). Two microliters of the extract was injected into the GC equipped with an olfactometric detector. Chromatographic conditions were the following: Hewlett-Packard HP 5890 gas chromatograph; column BP-21 (50 m × 0.25 mm, 0.25 μm) fused silica (SGE); hydrogen (5.0, Gasin); flow, 1.2 mL/min; injector temperature, 220 °C; oven temperature, 40 °C for 1 min programmed at a rate of 2 °C/min to 220 °C, maintained during 30 min; splitless time, 0.5 min; split flow, 30 mL/min.

The makeup gas employed on the olfactometric device (SGE) was air (80% N₂; 20% O₂) (Gasin). Two streams were used; one was bubbled in water (nose moister) and the other was applied at the exit of the GC column in order to lower the temperature of the effluent.

Descriptor collection, on GC-O, was performed by five trained persons (laboratory students) and was repeated several times. The descriptors retained were those that obtained the higher number of citations, considering each member of the panel.

Aroma Extract Dilution Analysis (AEDA). The relative importance of each of the different odor zones was evaluated by AEDA (11). Two milliliters of the extract was concentrated to 1:10 under a nitrogen stream. Then, 2 μL of the concentrated dichloromethane extract was separated on a capillary column. The odor-active regions and the odor qualities were assigned by five assessors (GC-O). The extract was stepwise diluted with dichloromethane (1 + 1 by volume), and the odor zones were re-evaluated. The process stopped when no aromas were detected by the assessor.

Chemical Studies. Standards Preparation. The following molecules were purchased from Sigma-Aldrich: 3-(methylthio)propionaldehyde (27,746-0) (100% purity); 4,5-dimethyl-3-hydroxy-2(5H)-furanone (W36,340-5) (97%); phenylacetaldehyde (10,739-5) (90%); and 4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-2-buten-2-ol (W25,940-3) (96%). The 1,1,6-trimethyl-1,2-dihydronaphthalene (TDN) synthesis was attempted according to the method of ref 17. The degree of purity obtained was <30%, due to the presence of 1,1,6-trimethyl-1,2,3,4-tetrahydronaphthalene (TTN), which was not sufficient to use in sensorial analysis.

Quantification Methods. Extraction was performed according to the methodology published in ref 8. To 50 mL of white wine was added 50 μL of octan-3-ol in hydroalcoholic solution (1:1, v/v) at 466 mg/L as internal standard and 5 g of anhydrous sodium sulfate. The wine was extracted twice with 5 mL of CH₂Cl₂ (Merck). The two organic phases obtained were blended and dried over anhydrous sodium sulfate. Four milliliters of this organic extract was concentrated until 1/10 under a nitrogen stream with a 20 L/min gas flow. The quantification limits [LQ = [mean × 10 × standard deviation (SD)] (8) and the variation coefficients [CV % = (SD/mean) × 100] were calculated for the molecules studied. They were, respectively, for 3-(methylthio)propionaldehyde, 5.5 μg/L and 8.2%; for 4,5-dimethyl-3-hydroxy-2(5H)-furanone, 2.1 μg/L and 4.9%; and for phenylacetaldehyde, 7.9 μg/L and 5.3%.

Gas Chromatography—Mass Spectrometry (GC-MS). Extracts were analyzed using a Varian CP-3800 gas chromatograph equipped with a Varian Saturn 2000 mass selective detector and Saturn GC-MS workstation software version 5.51. The column used was a Stabilwax-DA (60 m × 0.25 mm, 0.25 μm) fused silica (Restek). The injector port was heated to 220 °C. The split vent was opened after 30 s. The carrier gas was helium C-60 (Gasin), at a 1 mL/min constant flow.

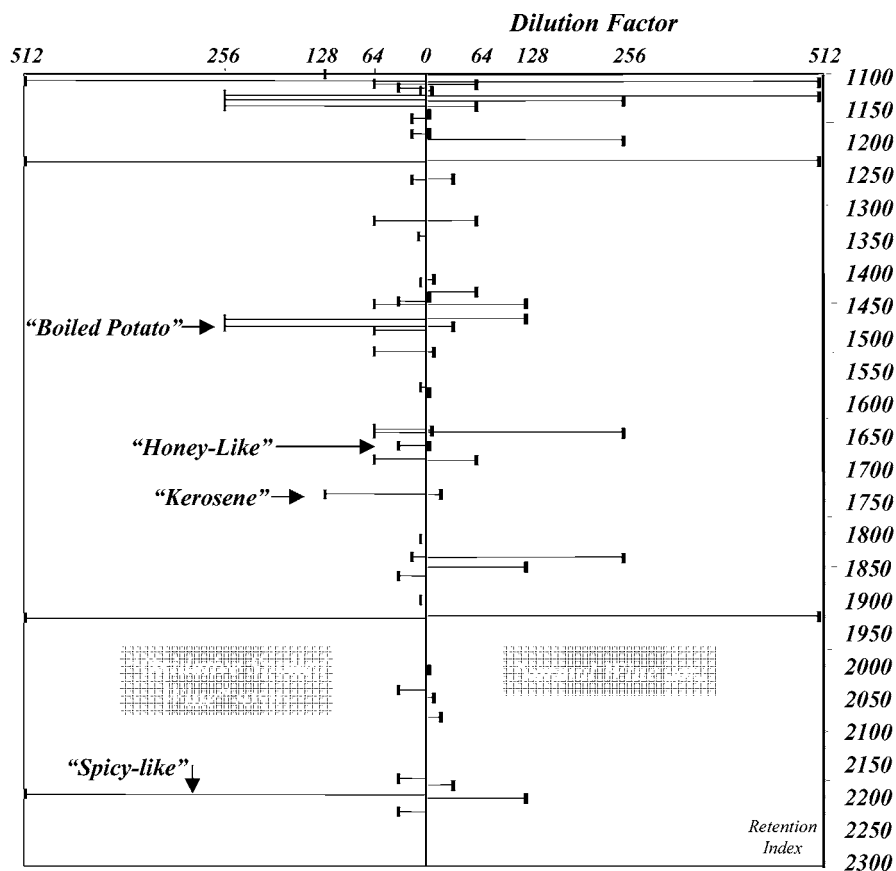


Figure 1. Aromagram obtained in AEDA.

The oven temperature was 40 °C (for 1 min), which was then increased at 2 °C/min to 220 °C and held for 30 min. All mass spectra were acquired in the electron impact (EI) mode. The ion trap detector was set as follows: The transfer line, manifold, and trap temperatures were, respectively, 230, 44, and 170 °C. The mass range was 33–350 *m/z*, with a scan rate of 6 scan/s. The emission current was 50 μ A, and the electron multiplier was set in relative mode to autotune procedure. The maximum ionization time was 25000 μ s, with an ionization storage level of 35 *m/z*. The injection volume was 1 μ L, and the analysis was performed in full scan mode.

Identification was achieved from comparisons of mass spectra obtained from the sample with those from pure standards injected in the same conditions by comparing the Kovats indices and the mass spectra present in the NIST 98 MS library database or in the literature.

Other Analytical Measurements. Measurements of redox potential, free SO₂ concentration, and chromatic index were performed (12). The concentration of dissolved oxygen was measured using a WTW 340 oxygen probe.

The Kovats index was calculated according to the literature (18).

RESULTS AND DISCUSSION

The identification of the most important descriptors related to the oxidative character of oxidation-spoiled white wines was performed according to the AFNOR 09-021 procedure by a trained panel. The terms selected were “honey-like”, “farm-feed”, “hay”, and “woody-like” (7).

Among the different organic solvents tested, the dichloromethane extract was the one that better represented the initial odor of a spoiled wine (3 years old, **Table 1**) containing the selected descriptors. GC-O analysis of the extract was performed to identify the odor zones in which aroma quality best relates with the selected off-flavor descriptors. It was possible to determine three aromatic zones related with the following

descriptors: “boiled-potato”, for a retention index (RI) of 1469; “honey-like”, RI = 1663; and “kerosene”, RI = 1753, which were the most frequently cited by the five members of the GC-O panel.

AEDA was used as a “screening methodology” to evaluate each of the aromatic zones selected by GC-O for its relative importance for identification (**Figure 1**).

From a comparison of the dilution factors (FD) obtained by AEDA analysis of the oxidation-spoiled wine and a normal wine (4 years old, **Table 1**) it could be seen that there were clear differences in the AEDA profiles for these two samples. This observation could be related with the overall perception of the two samples.

Nevertheless, the differences in FD that were retained were those related with the three selected odor zones. It could be seen that the intensity of these was greater in the spoiled wine (**Figure 1**). In fact, the FD values measured for spoiled wine and normal wine were, respectively, 256/32 for the boiled potato note, 32/2 for the honey-like note, and 128/2 for the kerosene note. The highest FD observed (FD = 512) corresponded to “spice-like” aroma with a retention index of 2186.

Using GC-MS with chemical standards it was possible to identify the corresponding molecules for the retention indices: 3-(methylthio)propionaldehyde (RI = 1469), phenylacetaldehyde (RI = 1663), TDN (RI = 1753), and 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone (RI = 2186).

To promote aroma degradation of white wine, similar to oxidation-spoiled white wines, a forced aging experiment was implemented, similar to previous works (7). Wine (6 years old, **Table 1**) was submitted to different regimes of oxygen and SO₂ contents at different pH values and stored at different temperatures. After 7 days, 3-(methylthio)propionaldehyde, 3-hydroxy-

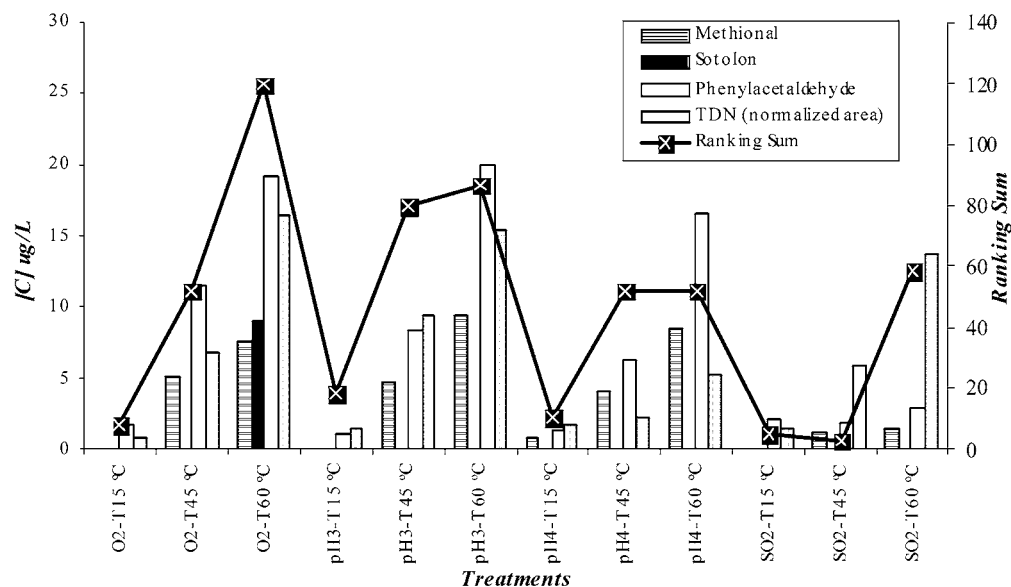


Figure 2. Variation of levels of 3-(methylthio)propionaldehyde, sotolon, phenylacetaldehyde, TDN (normalized area), and sensorial ranking value during experiment protocol.

4,5-dimethyl-2(5*H*)-furanone, phenylacetaldehyde, and TDN contents were determined.

The quantification of 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone, by GC, presents several limitations as mentioned by different authors (19, 20). Therefore, levels near the detection limits of the method were not considered (1.5 µg/L); only those above the quantification limits (2.1 µg/L) were taken into account.

As shown in **Figure 2**, the formation of 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone during the forced aging experiments was observed only for samples treated with oxygen and stored at 60 °C.

Samples were ranked according to their “degree of aroma degradation”. The results of sensorial and chemical evaluation are shown in **Figure 2**. Samples saturated with oxygen and those at pH 3.2, stored during 7 days at 60 °C, were considered to be the most spoiled, having the highest ranking values of, respectively, 120 and 87.

These results are in agreement with the difference observed in the FD values for these four molecules by AEDA between a nonspoiled wine and an oxidation-spoiled wine. Thus, the combined effect of substances seems to explain the higher frequency ranking obtained for “aroma-degraded” samples.

To investigate further the contribution of these molecules to the oxidation-spoiled aroma, they were added separately or in combination to a nonspoiled wine, at concentrations found in the oxidation-spoiled wine: 3-(methylthio)propionaldehyde (20 µg/L), phenylacetaldehyde (100 µg/L), and 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone (20 µg/L). These quantities are above threshold values of, respectively, 0.5 µg/L (3), 25 µg/L (7), and 15 µg/L (21).

A simple comparison test was carried out to rate the degree of similarity between each of the spiked samples and the oxidation-spoiled wine. The average of the similarity values as well as the standard deviation (SD) calculated for each pair is given in **Table 2**.

The ANOVA treatments of the data showed differences between samples and no significant differences between panelists ($p = 1.7e-12$) at the 95% level. The highest SV found was observed when the three molecules were added simultaneously to the wine (SV = 5.4).

Although TDN seems to be related to the spoiled character, in this work it was not tested due to its absence as a pure

Table 2. Results Obtained from Sensorial Analysis by Comparison Test

	similarity value (SV, av)	standard deviation (SD)
nW ^a	0.5	0.4
nW + [1]	3.6	2.5
nW + [2]	1.5	1.3
nW + [3]	1.9	1.5
nW + [1] + [2]	3.3	2.0
nW + [1] + [3]	3.9	2.2
nW + [2] + [3]	1.6	1.3
nW + [1] + [2] + [3]	5.4	1.0

^a nW, normal wine; [1], 3-(methylthio)propionaldehyde; [2], phenylacetaldehyde; [3], 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone.

standard commercially. The synthesis of TDN has been carried out from α -ionone. Due to the presence of TTN, the degree of purity was considered to be unsatisfactory for this kind of study. Thus, at the present we can only speculate that the SV could be increased if TDN was also added.

Comparing the SVs obtained by simple additions of 3-(methylthio)propionaldehyde (SV = 3.6), phenylacetaldehyde (SV = 1.5), and 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone (SV = 1.9) reveals the highest impact of the former molecule in the typical aroma of oxidation-spoiled wine, which is in agreement with previous work (3, 6). Furthermore, it is important to note that all combinations in which 3-(methylthio)propionaldehyde was included were rated with the highest similarity values.

Hence, these results suggest that, among the molecules studied, 3-(methylthio)propionaldehyde is the most important contributor to the typical aroma of an oxidation-spoiled white wine.

CONCLUSIONS

This work attempts to correlate the characteristic descriptors of the oxidation-spoiled aroma of white wines, “honey-like”, “farm-feed”, “hay”, and “woody-like”, with the presence of specific compounds.

GC-O and AEDA of a normal and a spoiled wine led to the identification of substances related with the typical oxidative

white wine aroma: 3-(methylthio)propionaldehyde, phenylacetaldehyde, TDN, and 3-hydroxy-4,5-dimethyl-2(5H)-furanone.

A forced aging experiment was implemented to simulate the typical oxidation-spoiled aroma, and four parameters were tested (temperature, O₂, pH, and SO₂). Samples supplemented with oxygen and kept at higher temperatures were rated with the highest score in the ranking test ("most spoiled"). Furthermore, it was observed that the concentrations of 3-(methylthio)propionaldehyde, phenylacetaldehyde, TDN, and 3-hydroxy-4,5-dimethyl-2(5H)-furanone seem to be related with the sensorial ranking score.

These molecules, with the exception of TDN, were spiked into a normal wine, and the degree of similarity with an oxidation-spoiled wine was evaluated. This procedure revealed that simultaneous addition of three compounds led to the highest SV.

It was observed that 3-(methylthio)propionaldehyde has the most aromatic influence in spoiled wine aroma, followed by 3-hydroxy-4,5-dimethyl-2(5H)-furanone and phenylacetaldehyde, respectively. The impact of 1,1,6-trimethyl-1,2-dihydronaphthalene needs further investigation.

Future work needs to be carried out to gather more information concerning the relationship between the chemical composition before and after "aroma spoilage" of white wine in order to contribute to the prediction the shelf life.

LITERATURE CITED

- (1) Singleton, V. L.; Kramling, T. E. Browning of white wines and accelerated test for browning capacity. *Am. J. Enol. Vitic.* **1976**, *27*, 157–160.
- (2) Singleton, V. L.; Trousdale, E.; Zaya, J. Oxidation of wines I. Young white wines periodically exposed to air. *Am. J. Enol. Vitic.* **1979**, *30*, 49–54.
- (3) Escudero, A.; Hernandez-Orte, P.; Cacho, J. E.; Ferreira, V. Clues about the role of 3-(methylthio)propionaldehyde as a character impact odorant of some oxidized wines. *J. Agric. Food Chem.* **2000**, *48*, 4268–4272.
- (4) Escudero, A.; Cacho, J.; Ferreira, V. Isolation and identification of odorants generated in wine during its oxidation: a gas chromatography–olfactometry study. *Eur. Food Res. Technol.* **2000**, *211*, 105–110.
- (5) Ferreira, V.; Escudero, A.; Fernández, P. E.; Cacho, J. Changes in the profile of volatile compounds in wines stored under oxygen and their relationship with the browning process. *Z. Lebensm. Unters. Forsch.* **1997**, *205*, 392–396.
- (6) Escudero, A.; Asensio, E.; Cacho, J.; Ferreira, V. Sensory and chemical changes of young white wines stored under oxygen. An assessment of the role played by aldehydes and some other important odorants. *Food Chem.* **2002**, *77* (3), 325–331.
- (7) 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.
- (8) Silva Ferreira, A. C. Caracterisation du Vieillissement du Vin de Porto. Approche Chimique et Statistique. Role Aromatique du Sotolon. These de Doctorat de L'Université Victor Segalen Bordeaux II, 1998; p 593.
- (9) Silva Ferreira, A. C.; Barbe, J. C.; Bertrand, A. Heterocyclic acetals from glycerol and acetaldehyde in Port wines: Evolution with aging. *J. Agric. Food Chem.* **2002**, *50*, 2560–2564.
- (10) Simpson, R. F. Aroma and compositional changes in wine with oxidation, storage and aging. *Vitis* **1978**, *17*, 274–287.
- (11) Ullrich, F.; Grosch, W. Identification of the most intense volatile flavour compounds formed during autoxidation of linoleic acid. *Z. Lebensm. Unters. Forsch.* **1987**, *184*, 277–82.
- (12) Ribereau-Gayon, J.; Peynaud, E.; Ribereau-Gayon, P.; Sudraud, P. *Traité d'Oenologie. Sciences et Techniques du Vin, Tome 1. Analyse et Contrôle des Vins*; Dunod: Paris, France, 1976.
- (13) AFNOR NFV09-021. *Recueil de Normes Françaises, Contrôle de la Qualité des Produits Alimentaires, Analyse Sensorielle*, 4th ed.; AFNOR: Paris, France, 1991; ISBN2-12-190843-9.
- (14) *Sensory Evaluation Techniques*; Meilgaard, M., Civille, G. V., Carr, T., Eds.; CRC Press: Boca Raton, FL, 1999.
- (15) Basker, D. Critical values of differences among rank sums for multiple comparisons. *Food Technol.* **1988**, *42* (2), 79.
- (16) Etievant, P. X.; Moio, L.; Guichard, E.; Langlois, D.; Leschaeve, P.; Schilich, P. E.; Chambellant, E. Aroma extract dilution analysis (AEDA) and the representativeness of the odour of food extracts. In *Trends in Flavour Research, Proceedings of the 7th Weurman Flavour Research Symposium*; Maarse, H., Van Der Heij, D. G., Eds.; Elsevier Science: Amsterdam, The Netherlands, 1995; pp 179–190.
- (17) Schneider, R.; Razungles, A.; Augier, C.; Baumes, R. Monoterpenic and norisoprenoidic glycoconjugates of *Vitis vinifera* L., cv. Melon B. as precursors of odorants in Muscadet wines. *J. Chromatogr. A* **2001**, *936*, 145–157.
- (18) Van Den Dool, H.; Kratz, P. D. A generalization of the retention index system including linear temperature programmed gas–liquid partition chromatography. *J. Chromatogr.* **1963**, *11*, 463–471.
- (19) Blank, I.; Sen, A.; Grosh, W. Potent odorants of the roasted powder and brew of Arabica coffee. *Z. Lebensm. Unters. Forsch.* **1992**, *195*, 239–245.
- (20) Martin, B.; Etievant, P.; Henry, N. The chemistry of sotolon: a key parameter for the study of a key component of flor sherry wines. In *Flavour Science and Technology, Proceedings of the 6th Congrès Weurman*; Thomas, A. F., Bessiere, Y., Eds.; Wiley: Chichester, U.K., 1990; pp 53–56.
- (21) Martin, B.; Etievant, P. X.; Le Quere, L.; Schlich, P. More clues about the sensory impact of sotolon in flor sherry wines. *J. Agric. Food Chem.* **1992**, *40*, 475–478.

Received for review July 25, 2002. Revised manuscript received October 22, 2002. Accepted October 27, 2002. A.C.S.F. is the recipient of a Postdoctoral Grant from the Portuguese Ministry of Science and Technology (PRAXIS XXI/ BPD/ 20181 /99).

JF0258470