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# Modification of the olfactory sensory characteristics of Chardonnay wine through the increase in sotolon concentration

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ABSTRACT

Among lactones formed during the oxidative maturation of wines, sotolon (3-hydroxy-4,5-dimethyl-2 [5H] furanone), with an odor reminiscent of nut and curry, and its low perception threshold, is known to have a significant impact on the aroma. Although recent research work has designated sotolon as one of the contributors to the specific Chardonnay olfactory space, sotolon is still mainly described for its sole nut and curry olfactory characteristics. The present work is aiming at evaluating the potential involvement of sotolon in small concentrations as a contributor to the complex sensory characteristics of Chardonnay wine. For this purpose, a Chardonnay wine was spiked with increasing amounts of a titrated sotolon solution, resulting in a concentration ranging from 0 to 60 μg $L^{-1}$. A sensory panel was then asked to describe the olfactory sensations associated with these incremental changes. The study demonstrated that several new olfactory attributes were generated by the panel in response to sotolon addition, and that these attributes were significantly different depending on the sotolon concentration range studied.



## 1. Introduction

While most wines undergo a classic maturation process in bottles under reductive conditions, some wines can undergo an oxidative maturation, which confers them specific aromas. By definition oxidative aging can only involve chemical reactions due to the presence of oxygen, as is the case with wines from Madera, Porto, and a few French fortified wines. This aging can also involve biochemical reactions, which result from the development of veil-forming oxidative yeasts, as is the case with wines from the Jerez area, with "Vin Jaune du Jura" (France), with "Vernaccia di Oristano" (Italy) and lastly with Tokaji wines (from Hungary).

During oxidative storage, aldehyde levels increase significantly, due either to the oxidation of alcohols by a coupled oxidation process involving the oxygen present in the air and the wine's di- and tri-hydroxyphenols (Wildenradt & Singleton, 1974); or to a biosynthesis process by veil-forming yeasts (Baro & Quiros, 1977; Otsuka, Iki, Nozu, Limura, & Totsuka, 1980). To date among lactones also formed during this type of maturing, only one is known to have an important impact on the aroma: sotolon (3-hydroxy-4,5-dimethyl-2[5H] furanone) (Guichard, Pham, & Charpentier, 1997). The smell of sotolon is reminiscent of nut and curry, and has a low perception threshold (1–5 μg $L^{-1}$) (Martin, Etievant, Le Quere, & Schlich, 1992). This compound is present in high amounts in wines matured under a veil of yeasts, i.e.: wines from the Jerez area, Tokaji wines from Hungary, and "Vin Jaune du Jura" from France (Pham, Guichard, & Charpentier, 1995). This compound is formed by the aldol condensation of acetaldehyde and α-ketobutyric acid (Aiken & Noble, 1984; Chatonnet, Boidron, & Pons, 1990). The latter molecule comes from the deamination of L-threonine by veil-forming yeasts.

Sotolon has been identified as the key aroma compound in the "perceived age" of barrel storage Port wine and consequently in the aroma quality of the product (Silva Ferreira, Barbe, & Bertrand, 2003). The odor threshold value was estimated at 19 μg $L^{-1}$ in Port wines (Silva Ferreira et al., 2003), which is close to the 15 μg $L^{-1}$ value reported for flor sherry (Martin et al., 1992). Let's now examine the perception thresholds of the different enantiomers of sotolon and their distribution in wines. The perception threshold of (*S*)-sotolon was set at to 0.8 μg $L^{-1}$ in the model wine solution, and was 100 times lower than that of the (*R*) form (89 μg $L^{-1}$). This indicates that (*S*)-sotolon is one of the main contributors to the characteristic aroma of the prematurely aged dry white wines (Pons, Lavigne, Landais, Darriet, & Dubourdieu, 2010). Both enantiomers are detected in white wines with different distribution

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patterns (Lavigne, Pons, Landais, Darriet, & Dubourdieu, 2008) which were partially explained by the slow racemization kinetics (20 months) of the optically active sotolon.

Some recent studies have designated sotolon as one contributor to the specific Chardonnay olfactory space, the latter being linked to the relative concentrations of 29 volatile possible aroma-impact compounds. This highlights the complexity of the odor quality of young unoaked Chardonnay wines (Jaffré et al., 2011). However, sotolon was mainly described through its sole nut and curry olfactory characteristics in oxidized wines (Pham et al., 1995).

Several internal sensorial surveys on oxidized and non-oxidized Chardonnay wines which differed mainly on their sotolon contents proved that very complex orthonasal olfactive notes were likely to be encountered. The aim of this work was thus to examine the potential involvement of sotolon in low concentrations as a contributor to the complex sensory characteristics of Chardonnay wine.

## 2. Materials & methods

### 2.1. Participants

Eighteen trained judges (four men and fourteen women aged 21–65 years-old) took part in this sensory study. They belonged to the permanent panel of the Sensory Laboratory of the Sciences for Enology unit (INRA) and were paid for their participation. All of them were selected on the basis of their sensory performances and their interests (ISO 8586-2, 1994) and were trained to perform a descriptive sensory analysis of wines. All of them had some previous experience in sensory analysis.

### 2.2. Wine samples and preparation

#### 2.2.1. Synthesis of 4,5-dimethyl-3-hydroxy-2,5-dihydrofuran-2-one $d_3$ (Sotolon-$d_3$)

The 4,5-dimethyl-3-hydroxy-2,5-dihydrofuran-2-one (Wako Pure Chemical, Osaka, Japan) (53.1 mg) was dissolved in a solution of NaOH (33 mg) in deuterium oxide (1.55 mL). The reaction was maintained at 80 °C during 4 h under nitrogen atmosphere. The mixture was first extracted with diethylether (2 × 10 mL). Then the aqueous phase was then acidified with a solution of $D_2SO_4$ (5 M) until pH = 1 before being extracted again with diethylether (2 × 10 mL) and concentrated to dryness in a vacuum. This resulted in an oily compound and its chemical characterization was performed as previously described (Dagan, Schneider, Lepoutre, & Baumes, 2006).

#### 2.2.2. Analysis of sotolon in wines by SIDA-GC–MS/MS

An accurate amount (40 µg) of the sotolon-$d_3$ previously synthesized was first added in a determined amount of wine (400 mL). Some of this wine (200 mL) was extracted under nitrogen with 80 ml of dichloromethane. The organic phase was brought to dryness, and then acidified with $H_2SO4$ (0.5 N). The wine extracts were analyzed by GC–MS/MS (Saturn 2000 Varian) on a DBWAX column (J&W, 30 m × 0.25 mm × 0.25 µm). The oven temperature started at 35 °C (during 3 min) and rose to 185 °C at a rate of 5 °C $min^{-1}$ then to 245 °C (during 5 min) at a rate of 20 °C $min^{-1}$. Helium was used as a vector gas at a flow rate of 1.2 mL $min^{-1}$. The injection volume was equal to 2 µL and the temperature of the injector was programed as follows: 45 °C (during 1 min), then 250 °C at 200 °C $min^{-1}$. The ionization process was performed under chemical ionization (using isobutene) and the detection was made in MS/MS mode. Natural and labeled sotolon were quantified by using respectively: the ions $m/z$ 129 and 132 as parent ions and the ions $m/z$ 83 and $m/z$ 86 as daughter ions. In this method, the detection limit is <1 µg $L^{-1}$.

#### 2.2.3. Wine

The wine matrix used was a Chardonnay wine with the "Indication Géographique Protégée" (IGP) Pays d'Oc and was produced in the year preceding the sensory analysis, then stored in a Bag-In-Box® before use (Table 1). This wine was not oaked and did not undergo malolactic fermentation. In this wine, no sotolon could be detected by chemical analysis using SIDA-GC–MS/MS with detection limit of 1 µg $L^{-1}$. On the day preceding the sensory analysis, the wine was spiked with titrated Sotolon, of which its (*R*)- and (*S*)- enantiomers had previously been equilibrated (from 1 to 60 µg $L^{-1}$, see Table 2). Two liters of wine were prepared for each sotolon concentration. Before and during the analysis, the wine samples remained at room temperature (between 20 and 22 °C).

### 2.3. Experimental procedure

This study was conducted in the INRA research center in Montpellier (France) during three sessions over two consecutive weeks. Each session lasted approximately an hour.

In the first part of the study, the judges attended two training sessions, in which they had the opportunity to familiarize themselves with the wine matrix, the wines spiked with sotolon and the methodology. No information was given to the judges about the purpose of the experiment and about the wines. They had to describe odor properties by orthonasal olfaction of 10 samples per session, with low sotolon concentrations samples during the first session (from 0 to 6 µg $L^{-1}$) and higher sotolon concentrations samples in the second session (from 5 to 20 µg $L^{-1}$). Three samples were duplicated during each training session (Table 2). During the training, the service was performed randomly in a monadic order (Latin square) in order to minimize carry-over effects (Macfie, Bratchell, & Greenhoff, 1989).

During the second part of the study only one range of sotolon concentrations was looked into (Table 2). Trained judges gave a description of 10 samples (from 0 to 60 µg $L^{-1}$). In this case, the sample presentation order was the same for all the panelists and followed an increasing order of concentration. This experimental protocol was chosen to analyze the olfactory modifications gradually induced by sotolon addition. The concentration was brought up to 60 µg $L^{-1}$, which exceeds the average perception threshold mentioned in wine literature (5–19 µg $L^{-1}$) (Martin et al., 1992; Silva Ferreira et al., 2003). The repeatability of the judges' answers was assessed by using two identical sotolon concentrations among the several concentrations used in the experiment (30 and 60 µg $L^{-1}$).

The samples (40 mL) were served in black glasses following a monadic order and were identified by a set of three digits random codes, which were different for each judge and each sample. The wine analysis was intended to be purely olfactory. In this purpose; the glasses were covered with a lid. The judges were requested to stir the glass before lifting the lid and smelling. The notation instruction communicated to the judges was formulated as follows: "describe the perception you have of the wine in question

**Table 1**
Enological parameters of Chardonnay wine.

| | | |
|---|---|---|
| TA (g $L^{-1}$ $H_2SO_4$) | Enzymatic determination | 3.79 |
| pH | pH-metric | 3.51 |
| Residual sugar (g $L^{-1}$) | Enzymatic determination | 0.84 |
| Alcohol (% vol/vol) | NIR determination | 12.79 |
| Free $SO_2$ (mg $L^{-1}$) | Ripper method with iodine standard | 29 |
| Total $SO_2$ (mg $L^{-1}$) | Ripper method with iodine standard | 140 |