Fast and Reliable Multiresidue Analysis of Aromas in Wine by Means of Gas Chromatography Coupled with Triple Quadrupole Mass Spectrometry †

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Abstract: The paper would like to show a direct injection into GC-MS/QqQ for the determination of secondary aromas in white wine samples fermented in two different ways. The procedure has been compared with more traditional methods used in this field, i.e., headspace analysis and liquid–liquid extraction. The application of such direct injection, for the first time in the literature, allows us to analyze Volatile Organic Compounds (VOCs) in the range 0.1–100 µg mL⁻¹, with Limits of Detection (LODs) and Limits of Quantification (LOQs) between 0.01–0.05 µg mL⁻¹ and 0.03–0.09 µg mL⁻¹, respectively, intraday and interday below 5.6% and 8.5%, respectively, and recoveries above 92% at two different fortification levels. The procedure has been applied to real wine samples: it evidences how the fermentation in wood (cherry) barrel yields higher VOC levels than ones in wine fermented in steel tank, causing production of different secondary aromas and different relative flavors.

Keywords: wine; Fiano; aromas; fermentation; headspace; liquid–liquid extraction; direct injection; comparison; VOC; GC-MS/QqQ

1. Introduction

Aroma is one of the important organoleptic characteristics of wine [1]. Nowadays, about 800 volatile organic compounds (VOCs) are identified [2]. Basically, the distinction in primary, secondary and tertiary aromas is nothing more than an old simplifying approach that dates to about sixty years ago. Today, it is an anachronistic and above all non-exhaustive classification, born in a historical period in which knowledge on the composition of the volatile fraction of wine was very limited. In fact, in those years only a few tens of volatile molecules had been identified, while today we know few hundreds of them. The primary, secondary and tertiary sequence is based on the origin of the odorous molecules during the grape–wine transformation process. The first studies on the volatile fraction of wine were conducted on Moscato. In fact, the grapes of this variety and the respective wine turned out to be an ideal study model due to their high content of odorous substances. The easy perception of precise smells during the tasting of these grapes and their wines led the first scholars to talk about primary, secondary and tertiary aromas, meaning by primary those of the grape [3–5], secondary those produced during alcoholic...
fermentation [6] and tertiary those deriving from the aging of wine (regardless of the container used for storage) [7]. Subsequently, this classification was erroneously generalized and applied to most wines without considering the advances in scientific knowledge of the sector, so today this subdivision into “watertight compartments” is simplistic and incomplete because the phenomena related to the biogenesis of volatile components in wine and the quality of the odor emanating from them are much more complex and interconnected.

Secondary aromas essentially refer to esters and higher alcohols of fermentative origin [8,9]. The volatile components belonging to these two chemical classes are typical secondary products of alcoholic fermentation, regardless of the starting food matrix. The esters (amyl and ethyl of short-chain fatty acids) are characterized by fruity notes such as banana, apple, melon, pineapple, etc.; the higher alcohols have historically been always associated with the so-called vinosity [10]. In practice, they are responsible for the aromatic background of any beverage of fermentation origin [11]. Thus, when they are clearly perceived in a wine, it means that the wine in question is extremely poor in odorous molecules. Furthermore, the quantitative balance between esters and alcohols of fermentation origin is very important, it greatly influences the olfactory quality of young wine and in particular that of white wines obtained from non-aromatic grapes [12].

Many variables, even more than the yeasts themselves that conduct alcoholic fermentation (indigenous or selected) significantly affect the balance between esters and alcohols. Some of them are turbidity of the medium, availability of nitrogenous nutrients, pH, oxygen level of the medium and fermentation temperature.

The free fraction consists of volatile compounds, directly accessible to the olfactory mucosa and therefore odorous, while the bound fraction consists of aromatic components present in the form of non-odoriferous precursors of a glycosidic nature [2,13,14]. The latter are gradually released by hydrolysis during winemaking, especially during the aging of the wine, thus increasing the complexity and aromatic specificity of the wine [15]. With the progress of aging, in fact, the aromatic character of the wine changes from an aroma of a mainly fermentative nature to a more complex one, strongly influenced by compounds of a varietal nature and therefore attributable to the grape of origin.

The grape variety used in the production of a particular wine characterizes the aroma of that wine [16–18]. However, other factors such as degree of ripeness of grapes, climate, viticultural practices, aging, winemaking techniques, yeasts and region are known to affect the aroma of wine [19–21].

Volatile aroma compounds are very important to grape wine quality and include varietal flavor (flavor compounds originating from the grapes), pre-fermentative flavor (originated during grape processing), fermentative flavor (produced by yeast and bacteria during alcoholic and malolactic fermentations) and post-fermentative flavor, due to transformations that occurred during conservation and aging of wine. Alcohols, acids, esters and terpenes are among the main substances that define the sensory properties and quality of wine [22,23]. Particularly, for a particular wine the aroma depends fundamentally on the grape variety used in the production [16,17] but also on some parameters such as region, climate, ripeness degree of the grape, yeasts and vinemaking techniques, viticultural practices, aging, affect the aroma of wine [19,21,24–29]. Further, most of the wine flavor compounds are produced or released during wine fermentation due to microbial activities of Saccharomyces and non-Saccharomyces yeast genera (Brettanomyces, Candida, Debaryomyces, Hanseniaspora, Hansenula, Kloeckera, Kluyveromyces, Lachancea, Metschnikowia, Pichia, Saccharomyces, Schizosaccharomyces, Torulaspora and Zygosaccharomyces). Both in spontaneous and inoculated wine fermentations, non-Saccharomyces are important in early stages of the fermentation, before Saccharomyces becomes dominant in the culture, and contribute meaningfully to the global aroma profile of wines [30–32]. Organoleptic characteristics of wine is the result of interactions of several compounds. The knowledge of these compounds are important factors in assessing the quality of wine [33,34].

To understand the wine composition, a multitude of scientific investigations was carried out and several appropriate analytical tools were developed in the past few decades.
In fact, the VOC characterization of these aromas is an important issue in analytical chemistry. Basically, the papers are mainly addressed to investigate the characteristics of each wine and/or an identification of possible regionality/falsification of a wine product [35–39]. This paper would like to investigate the possibility to characterize the wine aromas by means of different analytical methods and compare their ability to separate and quantify different compounds simultaneously. Three different analytical protocols have been tested followed by gas chromatography coupled with triple quadrupole mass spectrometry (GC-MS/QqQ): (i) direct injection; (ii) microextraction with hexane; (iii) headspace analysis. These three methods have been tested and compared according to the analytical parameters and applied to real wine samples.

2. Materials and Methods

2.1. Materials

All chemicals were of analytical reagent. Ultrapure water was obtained with a Milli-Q system (Millipore, Bedford, MA, USA). NaCl and hexadeuterobenzene (Benzened-\textsubscript{6}, perdeuterated benzene) were purchased from Sigma (Milan, Italy). Alcohol standard solutions, namely ethyl acetate, isopropanol, glycerol, 1-butanol, pentanol, 2,3-butanediol, propylene glycol, ethyl lactate, furfural, hexanol, lactic acid, hydroxymethylfurfural, succinic acid, tyrosol, were from Sigma. A stock solution (100 \(\mu\)g mL\(^{-1}\) of each compound) was prepared from the relative standard solutions and stored at \(-21^\circ\text{C}\). Solutions at different concentrations were obtained diluting stock standard solutions at the desired concentration.

2.2. Wine Sampling

In this study, white grape of \textit{Vitis vinifera} cv Fiano and the commercial yeast \textit{Saccharomyces cerevisiae} (Fermol Elegance, AEB, Brescia, Italy; indicated below with the abbreviation FE) were used. The grapes were harvested during the 2020 vintage and transported to an industrial winery (Mastroberardino s.p.a.) located in the Campania region (Italy). The grape must show the following chemical composition: pH 3.10, sugar 204 g L\(^{-1}\), total acidity (expressed as tartaric acid) 8.2 g L\(^{-1}\), and YAN (yeast assimilable nitrogen) 156 mg L\(^{-1}\). Before the use, the grape juice was clarified at 0 \(^\circ\text{C}\) for 48 h, then heated to 18 \(^\circ\text{C}\), added of potassium metabisulphite (50 mg L\(^{-1}\)). Finally, the grape must with 20 g hL\(^{-1}\) of the commercial yeast \textit{S. cerevisiae} FE was inoculated, in according to the producer’s instructions and portioned into two aliquots using stainless steel tanks (AISI 304) (Fiano 1) and cherry barrels of 2 hL\(^{-1}\) (Fiano 2).

Fermentations were performed at 18 \(^\circ\text{C}\) (\(\pm\)2 \(^\circ\text{C}\)). To maintain an optimal YAN level for yeasts, after 72 h of fermentation all the batches were added with 25 mg L\(^{-1}\) of ammonium phosphate. No further treatment was applied to the wine. The fermentation process was monitored assessing the reducing sugar. The samplings for chemical analysis were performed after the alcoholic fermentation.

2.3. Preliminary Chemical Analysis on Wine Samples

The pH, total acidity (g L\(^{-1}\) as tartaric acid) and reducing sugar (g L\(^{-1}\)) were analyzed according to the official methods established by the European Commission (EC) [40]; acetic acid, L-malic acid, L-lactic acid and catechins were enzymatically determined using commercial kit (Boehringer Mannheim GmbH, Ingelheim, Germany).

2.4. Extraction Methods

Three different extraction procedures have been tested and compared for achieving the best possible results: (a) headspace analysis, (b) liquid–liquid extraction and (c) direct injection.

2.4.1. Headspace Analysis

In the 20 mL glass vials, 2 g of NaCl (analytical grade NaCl purified in an oven at 400 \(^\circ\text{C}\) for 2 h) was inserted and 10 mL of wine were then introduced and capped with
a screw cap and a perforable PTFE membrane/Silicone. The wine was spiked with 2 ng of perdeuterated benzene in methanol by piercing the PTFE/Silicone septum. The vials were placed in a GC oven and brought to 80 °C for 10 min (preconditioning), after which helium 5.5 was bubbled at a flow of 40 mL min\(^{-1}\) for 5 min with a needle that reached the bottom of the vials. The outgoing helium, together with the vapors from the headspace, was collected by means of a short needle connected to a 40 cm-long air-cooled glass tube in order to condense the spray and partially the water vapors. An adsorbent trap was connected to the end of the tube by means of a swagelok union for the subsequent analysis in thermodesorber/GC-MS (thermodesorber CP4020 TCT Thermal Desorber, Chrompack, Gas Chromatograph Thermo Trace GC-Ultra, mass spectrometer Thermo DSQ Single Quadrupole MS, Thermo Fisher Scientific Inc., Waltham, MA, USA). The adsorbent trap consisted of a 4 mm I.D., 140 mm long glass tube filled with 240 mg of Activated Carbon Fiber (ACF) adsorbent. The adsorbent trap was then back flushed for 5 min with a helium flow of 100 mL min\(^{-1}\) at room temperature to reduce the amount of residual water. The adsorbent trap was thermodesorbed at 300 °C for 10 min with a flow of 20 mL min\(^{-1}\) of helium. The thermodesorbed vapors were cryofocused on a cryogenic trap consisting of a 0.53 mm I.D. and 15 cm long silica capillary. The cryogenic trap during heat absorption was maintained at −180 °C with liquid nitrogen. After the thermal desorption of the ACF trap, the cryofocused Volatile Organic Compounds (VOCs) were ballistically desorbed bringing the capillary temperature from −180 °C to 300 °C in 1 min.

### 2.4.2. Liquid-Liquid Extraction

Perdeuterated benzene (as standard) and 2 g of NaCl, for increasing the ionic strength, were added to 10 mL of wine sample (or ultrapure water, MilliQ, and alcohols for the blank procedure). The solution was debated for 10 min with 500 µL of pentane. A portion of the pentane was poured into a vial and injected into a GC-MS/QqQ system.

### 2.4.3. Direct Injection

After the addition of the perdeuterated benzene standard, the wine sample was directly injected into the GC-MS/QqQ system.

### 2.5. GC-MS/QqQ Analysis

The instrumental analyses were performed by a triple quadrupole gas chromatograph/mass spectrometer (Trace 1310 GC/TSQ 8000 Evo, Thermo Fisher Scientific), the chromatographic separation was performed by a DB-624 column (60 m × 0.25 mm, 1.40 µm I.D., Agilent Technologies, Santa Clara, CA, USA). The injected volume is always 1 µL in PTV injector operating in splitless mode (SL time 0.6 min), the injection temperature is 200 °C for 0.05 min and at 14.5 °C s\(^{-1}\) up to 300 °C for 0.6 min. Gas carrier is He, the temperature program starts at 2.0 mL min\(^{-1}\) and, after 20 min at 1.1 mL min\(^{-1}\) rate, up to 3.5 mL min\(^{-1}\) for 25 min. The optimized chromatographic run is 50 °C (hold time 1 min), ramp 7 °C min\(^{-1}\), 100 °C (0 min), ramp 10 °C min\(^{-1}\), 240 °C (30 min). Each analysis was performed with MS acquisition in scan mode (29–350 m/z) with 0.2 s scan time and emission current of 50 µA and an electron energy of 70 eV in EI+ mode. The source temperature is set up at 260 °C and the transfer line temperature at 240 °C. Data acquisition, processing and handling are performed using XCalibur software (Thermo Fisher Scientific). Before proceeding to the GC-MS/QqQ analyses, 2 ng of perdeuterated benzene in methanol were added as internal standard (I.S.) in all the samples.

### 3. Results

The paper would like to show an easy, rapid, and effective method for analyzing the VOCs fraction, which is responsible of the wine aroma, in comparison with traditional procedures. Before approaching this evaluation among the three different extraction methods, the authors reported the main chemical parameters for identifying the wine samples.
3.1. Preliminary Chemical Analysis

White grapes of the same varieties, i.e., Fiano, were subjected to alcoholic fermentation in two different containers: Fiano 1 in steel tank and Fiano 2 in cherry barrel. Under both conditions the fermentations were completed after 10 days as evidence of regular fermentative activity of the added starter yeast. Table 1 shows the main chemical characteristics of the two wine samples investigated in this study after fermentation.

Table 1. Main chemical characteristics of the wine investigated in this study.

| Wine   | Alcohol Content (% vol) | Reducing Sugar (g L\(^{-1}\)) | pH   | Total Acidity (g L\(^{-1}\)) | Volatile Acidity \(^2\) (g L\(^{-1}\)) | Malic Acid (g L\(^{-1}\)) | Lactic Acid (g L\(^{-1}\)) | Catechin (mg L\(^{-1}\)) |
|--------|-------------------------|-------------------------------|------|------------------------------|----------------------------------------|---------------------------|---------------------------|-------------------------|
| Fiano 1| 12.7                    | < 2.0                         | 3.48 | 6.6                          | 0.54                                   | 0                         | 1.23                      | 52                      |
| Fiano 2| 12.9                    | < 2.0                         | 3.40 | 7.2                          | 0.75                                   | 0                         | 1.26                      | 73                      |

\(^1\) Fiano 1 fermented in steel tank, Fiano 2 in cherry barrel; \(^2\) Measure related to acetic acid (g L\(^{-1}\) of acetic acid).

The data are quite similar between them, showing that the alcoholic fermentation in the two different containers does not significantly affect the chemical properties.

3.2. Comparison and Choice of the Extraction Procedure

Three different extraction procedures have been studied and compared: specifically, the comparison occurred among (a) headspace analysis, (b) liquid–liquid extraction and (c) direct injection. For this goal the authors would like to show the chromatograms obtained in the three different situations for better evidencing their strengths and weaknesses. Figure 1 shows the GC-MS/QqQ chromatograms obtained following the three different procedures applied to standard alcohol solutions and real wine samples (i.e., Fiano 2), respectively.

The chromatograms are quite different among them. The headspace procedure seems to be more effective than the others, the peaks are shaped and no artifacts are present or “produced” during the analysis (Figure 1a,b for standard solution and real wine sample, respectively), but, according to our experiments, it requires some precautions that make its operation not easy. There is a high risk of the cryogenic trap becoming clogged after each injection: the advantage of getting good chromatographic performances is balanced by strenuous operations for setting the instrument. On the other hand, the direct injection (the relative chromatograms are reported in Figure 1e,f, respectively) is quite powerful: even in this case, no artifacts are formed, whereas a saturated peak in the real sample can be revealed at around 20.5 min due to glycerin, but it does not interfere with the VOCs determination. Using this procedure, the precautions are relatively less than the previous procedure: the lines should be changed every 10 injections and the use a precolumn of 5 m is highly recommended for avoiding column problems. Basically, it means that the wine can be injected with no pretreatment operation. Finally, the liquid–liquid extraction does not give satisfactory results for overall the compounds, just few of them can be detected with high resolution.

Following this statement, the determination of the analytical parameters has been focused on the procedure based on the direct injection.

3.3. Study of the Analytical Parameters

In addition to the fact that no pretreatment is necessary, another advantage in using the direct injection method is that the analytical parameters to be controlled are almost zero. In this study, only the possible influence of adding NaCl to increase the ionic strength of the solution was verified to achieve higher recoveries \([41,42]\). It should be noted that the analytical parameters have been studied on the stock solution (and its relative diluted solutions) containing 13 VOCs. Table 2 shows the results (in terms of% recoveries) obtained spiking a wine sample with a standard solution of some alcohol (5 ng µL\(^{-1}\) of each) and perdeuterated benzene as I.S. (2 ng) and adding different amounts of NaCl.
The presence of low concentration salt both stabilizes the VOC measurements (salting out), especially as regards the standard deviation, and gives excellent recoveries whereas for NaCl quantities higher than 5 g L\(^{-1}\) the VOC solubility decreases. This occurrence justifies the authors’ choice regarding direct injection without the addition of NaCl due to the minimum advantage that would be obtained.

Figure 1. Chromatograms obtained after the three different procedures: (a,b) following headspace analysis for blank solution and real wine sample, respectively; (c,d) following liquid–liquid extraction; (e,f) following direct injection.
Table 2. Effect of different NaCl amounts on the VOCs recoveries (%). The conditions are as follows: real white wine sample spiked with VOCs (5 µg mL⁻¹ of each) and 2 ng of I.S. In brackets are reported the RSDs (%); each analysis was in triplicate.

| Compound          | Recovery (%) |
|-------------------|--------------|
|                   | 0 g L⁻¹      | 2 g L⁻¹      | 5 g L⁻¹      | 10 g L⁻¹     | 15 g L⁻¹     |
| Ethyl acetate     | 98.8 (7.1)   | 96.7 (5.9)   | 94.7 (3.8)   | 91.2 (3.5)   | 85.5 (3.2)   |
| Isopropanol       | 101.2 (6.9)  | 98.3 (5.7)   | 95.9 (4.0)   | 92.9 (4.1)   | 88.1 (3.0)   |
| 2,3-Butanediol    | 102.4 (8.1)  | 97.9 (7.1)   | 95.1 (5.1)   | 90.2 (3.0)   | 87.3 (2.6)   |
| 1-Butanol         | 99.5 (6.1)   | 98.2 (4.9)   | 94.8 (3.4)   | 89.9 (3.5)   | 85.2 (2.1)   |
| Pentanol          | 99.8 (7.2)   | 97.7 (6.5)   | 94.3 (4.2)   | 89.5 (2.8)   | 88.1 (2.4)   |
| Propylene glycol  | 101.4 (6.6)  | 98.5 (5.1)   | 97.2 (4.2)   | 93.6 (3.2)   | 89.3 (2.6)   |
| Ethyl lactate     | 99.5 (6.1)   | 97.2 (4.5)   | 91.6 (3.9)   | 91.8 (2.9)   | 90.1 (2.5)   |
| Furfural          | 98.7 (6.4)   | 96.3 (4.6)   | 94.7 (3.7)   | 92.8 (3.1)   | 89.5 (2.9)   |
| Hexanol           | 98.9 (5.7)   | 97.1 (4.2)   | 94.2 (3.5)   | 91.5 (2.7)   | 88.3 (2.1)   |
| Lactic acid       | 99.5 (5.7)   | 97.6 (5.2)   | 94.5 (4.8)   | 93.0 (3.8)   | 91.2 (2.3)   |
| Hydroxymethylfurfural | 101.0 (6.0) | 98.2 (4.8)   | 96.1 (4.1)   | 91.2 (3.3)   | 90.8 (1.8)   |
| Succinic acid     | 99.7 (6.4)   | 97.5 (4.0)   | 95.2 (3.1)   | 91.3 (2.8)   | 91.5 (1.7)   |
| Tyrosol           | 100.1 (7.8)  | 97.9 (6.1)   | 94.2 (4.2)   | 92.2 (2.9)   | 89.9 (2.3)   |

Following the optimized parameters all the analytical parameters have been studied. Table 3 reports the correlation coefficients ($R^2$) evaluated in the range 0.1–100 µg mL⁻¹ along with the limits of detection (LODs) and limits of quantification (LOQs) determined according to the Knoll’s definition (i.e., a chromatographic peak equal to three times and seven times, respectively, the standard deviation of the baseline noise) [43,44], and repeatability and reproducibility of VOCs present in the standard solution. As it could be seen, the linearity ($≥$ 0.9944) is good for all the compounds in the investigated range as well as LODs (between 0.01–0.05 µg mL⁻¹) and LOQs (between 0.03–0.09 µg mL⁻¹) and repeatability (as intraday precision; ≤ 5.6) and reproducibility (as interday precision; ≤ 8.2) are effective for determining and characterizing aromas in wine samples. Finally, the recoveries have been studied, spiking the real wine samples with two different VOC concentrations (0.5 µg mL⁻¹ and 20 µg mL⁻¹). Table 3 shows such recoveries ranging between 92 and 102% with RSDs ≤ 5.9. Finally, Figure 2 shows the GC-MS/QqQ chromatogram of a real white wine sample: the peaks appear well-solved as well as each determination is precise and accurate. Thus, the authors decided to proceed with the direct injection into the GC-MS/QqQ for analyzing the wine samples.

Table 3. Correlation coefficients ($R^2$) evaluated in the range 0.10–100 µg mL⁻¹, limit of detection (LOD) and limit of quantification (LOQ) inter and intraday precision as Relative Standard Deviation (RSD) and recoveries at two different concentrations of some VOCs determined by GC-MS/QqQ.

| Compound             | $R^2$   | LOD (µg mL⁻¹) | LOQ (µg mL⁻¹) | Intraday Recovery (%) | Interday Recovery (%) |
|----------------------|---------|---------------|---------------|-----------------------|-----------------------|
| Ethyl acetate        | 0.9968  | 0.04          | 0.06          | 6.5 (3.9)             | 94.1 (2.1)             |
| Isopropanol          | 0.9952  | 0.03          | 0.07          | 3.4 (3.1)             | 93.1 (2.1)             |
| 2,3-Butanediol       | 0.9981  | 0.04          | 0.08          | 4.5 (3.1)             | 96.2 (2.9)             |
| 1-Butanol            | 0.9989  | 0.03          | 0.05          | 2.8 (3.1)             | 95.9 (2.1)             |
| Pentanol             | 0.9979  | 0.03          | 0.06          | 3.9 (3.1)             | 96.2 (2.7)             |
| Propylene glycol     | 0.9985  | 0.02          | 0.05          | 3.6 (3.1)             | 92.3 (2.7)             |
| Ethyl lactate        | 0.9977  | 0.01          | 0.03          | 4.2 (3.1)             | 93.1 (2.7)             |
| Furfural             | 0.9991  | 0.03          | 0.06          | 5.6 (3.1)             | 94.5 (2.7)             |
| Hexanol              | 0.9968  | 0.02          | 0.04          | 4.8 (3.1)             | 97.3 (2.7)             |
| Lactic acid          | 0.9985  | 0.02          | 0.05          | 4.5 (3.1)             | 94.2 (2.7)             |
| Hydroxymethylfurfural| 0.9982  | 0.02          | 0.06          | 5.1 (3.1)             | 95.8 (2.7)             |
| Succinic acid        | 0.9979  | 0.04          | 0.07          | 4.2 (3.1)             | 94.5 (2.7)             |
| Tyrosol              | 0.9944  | 0.05          | 0.09          | 3.7 (3.1)             | 96.9 (2.7)             |
Table 3. Correlation coefficients (R²) evaluated in the range 0.10–100 µg mL⁻¹, limit of detection (LOD) and limit of quantification (LOQ) inter and intraday precision as Relative Standard Deviation (RSD) and recoveries at two different concentrations of some VOCs determined by GC-MS/QqQ.

| Compound             | R²      | LOD (µg mL⁻¹) | LOQ (µg mL⁻¹) | Intraday RSD (%) | Interday RSD (%) | Recovery (%) |
|----------------------|---------|---------------|---------------|------------------|------------------|--------------|
| Ethyl acetate        | 0.9968  | 0.04          | 0.06          | 6.5              | 7.5              | 94.1 (3.9)   |
| Isopropanol          | 0.9952  | 0.03          | 0.07          | 3.4              | 6.4              | 93.1 (4.1)   |
| 2,3-Butanediol       | 0.9981  | 0.04          | 0.08          | 4.5              | 6.3              | 96.2 (2.9)   |
| 1-Butanol            | 0.9989  | 0.03          | 0.05          | 2.8              | 5.2              | 95.9 (4.1)   |
| Pentanol             | 0.9979  | 0.03          | 0.06          | 3.9              | 5.5              | 96.2 (4.7)   |
| Propylene glycol     | 0.9985  | 0.02          | 0.05          | 3.6              | 5.7              | 92.3 (3.6)   |
| Ethyl lactate        | 0.9977  | 0.01          | 0.03          | 4.2              | 6.7              | 93.1 (4.5)   |
| Furfural             | 0.9991  | 0.03          | 0.06          | 5.6              | 8.5              | 94.5 (5.9)   |
| Hexanol              | 0.9968  | 0.02          | 0.04          | 4.8              | 7.2              | 97.3 (4.1)   |
| Lactic acid          | 0.9985  | 0.02          | 0.05          | 4.5              | 6.6              | 94.2 (4.3)   |
| Hydroxymethylfurfural| 0.9982  | 0.02          | 0.06          | 5.1              | 8.2              | 95.8 (3.8)   |
| Succinic acid        | 0.9979  | 0.04          | 0.07          | 4.2              | 5.9              | 94.5 (4.7)   |
| Tyrosol              | 0.9944  | 0.05          | 0.09          | 3.7              | 6.3              | 96.9 (5.3)   |

Figure 2. GC-MS/QqQ chromatogram in TIC mode of real wine sample fermented in cherry barrel. For experimental conditions, see text. For peak list, see Table 4.

Table 4. VOC levels (µg mL⁻¹; n.d.: not detected) determined in white wine samples fermented in cherry barrel and steel tank. The determinations were performed by means of direct injection followed by GC-MS/QqQ.

| Compound                                 | tᵣ (min) | Fiano White Wine (µg mL⁻¹) | Cherry Barrel | Steel Tank |
|------------------------------------------|----------|---------------------------|---------------|------------|
| Ethyl acetate                            | 9.68     | 4.08                      | 1.94          |            |
| Isobutanol                               | 10.58    | 8.40                      | 3.76          |            |
| Acetic acid                              | 10.74    | 11.27                     | n.d.          |            |
| Ammonium acetate                         | 10.94    | 45.06                     | n.d.          |            |
| Diglycerol                               | 11.17    |                           |               |            |
| 1-Hydroxypropan-2-one                    | 12.54    | 2.02                      | 0.28          |            |
| 1,3-Butanediol                           | 13.24    | 25.87                     | 20.76         |            |
| Pentanol                                 | 13.29    | 7.28                      | 5.42          |            |
| 1-Heptene-4-ol                           | 14.17    | 0.35                      | 0.12          |            |
| Dioxirane                                | 14.40    | 0.26                      | n.d.          |            |
| Propylene glycol                         | 14.54    | 2.10                      | 1.75          |            |
| Ethyl lactate                            | 15.14    | 1.52                      | 1.22          |            |
| 2,3-Butanediol                           | 15.32    | 8.59                      | 7.21          |            |
| 1,3-Butanediol                           | 15.50    | 5.67                      | 4.38          |            |
| Furan-2-carbaldehyde (or Furfural)       | 16.07    | 3.38                      | 0.47          |            |
| Hexanol                                  | 16.22    | 0.25                      | 0.19          |            |
| 2-Furannethanol                          | 16.53    | 2.13                      | 0.10          |            |
| Lactic acid                              | 16.96    | 0.19                      | 0.06          |            |
Table 4. Cont.

| Compound | $t_r$ (min) | Fiano White Wine (µg mL$^{-1}$) |
|----------|-------------|---------------------------------|
|          | Cherry Barrel | Steel Tank |
| Pyruvic acid | 17.10 | 0.64 | 0.07 |
| 1-Methoxybutan-2-ol | 17.39 | 0.12 | 0.25 |
| 1,3-Dioxane-2-methyl-4-methyl | 17.58 | 0.41 | n.d. |
| 4-Acethylpyrazole | 17.67 | 0.45 | 0.08 |
| 2,4-Dihydroxy-2,5-dimethyl-3(2H)-furanone | 18.53 | 1.73 | 0.16 |
| Furfural-5-metil | 18.84 | 2.00 | 0.76 |
| 4-Oxopentanedioic acid | 18.91 | 1.83 | 0.26 |
| Dihydroxycetone | 19.04 | 2.36 | n.d. |
| (or 1,3-Dihydroxypropan-2-one) | 19.89 | 0.67 | 0.55 |
| Pyran-2,6(3H)-dione | 21.40 | 1.05 | 0.07 |
| 2-Acetylfruran (or 2-Furyl methyl ketone) | 21.58 | 9.17 | 15.57 |
| Phenylic alcohol | 21.73 | 0.62 | 0.38 |
| Diethyl butanedioate (or Diethyl succinate) | 21.86 | 0.54 | 0.31 |
| Glycerin acetate (or 1-acetylglycerol) | 22.15 | 7.03 | 1.07 |
| Pyranone | 22.18 | 12.15 | 12.77 |
| Ethyl succinate | 22.43 | 0.59 | 0.01 |
| 5-Hydroxymaltol | 22.85 | 3.61 | 7.72 |
| Succinic acid (or Butanedioic acid) | 23.18 | 1.65 | 0.54 |
| 2,3-Dihydrobenzofuran | 23.56 | 10.16 | 0.09 |
| Hydroxymethylfurfural | 27.07 | 5.27 | 6.00 |

3.4. Analysis of Real Wine Samples for Determining the Aromas

As just reported in the previous section, Figure 2 shows the chromatogram obtained after the direct injection of a real white wine (Fiano) sample, particularly the sample fermented in cherry barrel, whereas the Table 4 reports the determinations performed by means of direct injection and GC-MS/QqQ of Fiano wines fermented in cherry barrel and steel tank.

First, it should be evidenced the high VOCs resolution to be achieved by this methodology. In the literature, many papers dealing the VOCs determination for the wine aroma characterization are present [23,45–54]. Basically, such issue is achieved by means of analytical methods based on extraction (i.e., liquid–liquid extraction, pre-column clean-up, cartridge extraction, headspace procedure, etc.) followed by GC-MS or HPLC-MS analysis whereas for the first time, at authors’ knowledge, this paper describes a direct injection of real wine sample into the GC-MS/QqQ equipment, never tested before. The results obtained are very good; 38 compounds have been detected in wine samples fermented by two different ways, i.e., cherry barrel and steel tank. Table 4 highlights the different levels reached by VOCs during the two different fermentations: as it can be seen, the wine fermented in cherry barrel systematically shows higher VOC levels than the wine fermented in steel tank (except 1-methoxybutan-2-ol, phenethyl alcohol, succinic acid and tyrosol), as well as a greater number of compounds (i.e., acetic acid, diglycerol, dioxirane, 1,3-dioxane-2-methyl-4-methyl, dihydroxycetone). The reason can be reasonably due to the different materials used for fermentation. In fact, the cherry barrel, basically a wooden barrel, allows a greater wine breathing and consequently a greater presence of aromas in the wine. On the other hand, the steel tank is more hermetic and reduces the possibility of aeration of the wine and therefore the VOCs formation that characterize the relative aroma.

4. Conclusions

The qualitative/quantitative determination of secondary aromas is still an interesting task in the wine science even if such issue is well-studied. The literature reports analytical methods based on extraction procedures followed by GC or HPLC separation: this paper shows a direct injection of real white wine samples into the GC-MS/QqQ system. This procedure allows us to analyze the sample without any chemical pretreatment: the results on alcohol solution standards are good compared with liquid–liquid extraction and headspace analysis as well as on the real samples. The procedure has been applied two real Fiano wine samples.
evidencing the VOC profile differences between two different fermentations, i.e., in barrel cherry and steel tank: the wood container allows to get higher VOC levels and greater compound number. The next step will be to test such procedure to other fermented beverages, e.g., beer, marsala, fortified wines, liqueurs.

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