

# WINES & VINES

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## Oxygen Status at Bottling

### Identifying winery sources for oxidation in Ohio wines

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Oxygen is an important element that can alter the chemical and sensory properties of a wine through oxidation. All wineries need to understand both the impact that oxygen can have on a wine and the critical points in the winemaking process where excess oxygen poses a serious concern or threat. This article, part one in a series, describes oxygen and its effects while looking at the sources of oxygen exposure for wine during the vinification process, bottling and, finally, at the influence of the type of closure on the dissolved oxygen in wine.

The second part of this series will look at a study done by the The Ohio State University's Ohio Agricultural Research and Development Center to determine the dissolved oxygen status

in commercial wines at bottling from 14 Ohio wineries. A total of 12 bottles of wine from the 2014 vintage were collected from each participating winery during the middle of the bottling process. Using a NomaSense oxygen analyzer, dissolved oxygen (DO) and headspace oxygen (HSO) were measured at several storage times ending at 126 days after bottling. In addition, the free and total sulfur dioxide and pH were analyzed in triplicate and compared with the DO and total package oxygen (TPO) levels over time. As will be seen in the results of the study at the end of part two, significant improvements need to be made in reducing wine oxygen levels at the bottling tank and throughout the bottling process for the participating wineries.

### **Sources of DO: vinification**

The amount of oxygen that a wine acquires during winemaking has an impact on its sensory quality. Wines exposed to high levels of oxygen often lose fruitiness and freshness, and develop a brown color. These responses are unwanted in most wines, particularly white wines. Since red wines contain high levels of polyphenols (color and astringency), some beneficial chemical reactions may develop with small amounts of dissolved oxygen.

The main substrates for wine oxidation are phenolic compounds. These molecules are connected with two mechanisms of wine oxidation: enzymatic and nonenzymatic (chemical). Enzymatic oxidation occurs very fast and early in the winemaking operation. During crushing and pressing, polyphenolic oxidase activity leads to brown coloration. Also, the resulting wine may have less varietal character.

Winemakers often prevent enzymatic oxidation by using inert gases such as nitrogen and carbon dioxide, adding sulfur dioxide and settling the juice prior to alcoholic fermentation. Studies have indicated that enzyme activity decreased some 90% with the addition of 50 ppm SO<sub>2</sub>.

Since enzyme activity is inhibited, the most important oxidation mechanism is nonenzymatic, chemical oxidation in wines. Along with ascorbic and tartaric acids, phenolic molecules are substrates for this unwanted oxidation process, a slow reaction. Through a series of several pathways and reactions, chemical oxidation leads to the formation of several compounds including quinones, acetaldehyde and hydrogen peroxide ( $H_2O_2$ ), a strong oxidizing agent. Often, the aroma and flavor of an oxidized wine are described as being caramel, sherry-like, bruised banana and rancid, to name a few descriptors.

To control dissolved oxygen (DO), winemakers frequently minimize oxygen pickup during vinification and prevent its oxidizing effect by adding appropriate amounts of sulfur dioxide. For the latter, attention is given to specific winemaking operations that include racking, pumping, filtration, tartrate stabilization and bottling. Studies indicate that bottle filling is a main source of oxygen exposure, with DO levels from 0.2 ppm to 7.0 ppm, and may be responsible for a 70% increase. Other steps potentially contributing oxygen are processes such as racking, which can have an uptake of as much as 5 ppm oxygen, and filtration, with an uptake of as much as 7 ppm.

Besides the above unit operations, two other aspects also influence oxygen enrichment: wine temperature and volume. Temperature has an important effect on the  $O_2$  level in wines. It is well known that decreasing wine temperature accelerates oxygen uptake. To illustrate, the solubility of  $O_2$  in water at  $50^\circ$  F is 15.4 ppm, while at a higher temperature of  $68^\circ$  F,  $O_2$  solubility is 9.1 ppm. This relationship is decisive in tartrate stabilization, where low temperatures approach  $32^\circ$  F and DO levels may reach 2.7 ppm or higher in wine.

### **Sources of DO: bottling**

For wine volumes during bottling, oxygen up take is less when large volumes of wine are used in comparison to smaller volumes. Studies have found that the beginning and end of a

bottling run add higher DO levels. These increases are caused by the filtration system, dead volume, oxygen content near the wine surface and turbulence in the final bottles.

The guideline for maximum dissolved oxygen in the bottle has been established at 1.0 ppm. To achieve this level, winemakers must employ several measures to lessen the exposure of wines to air—the source of most oxygen exposure. First, all cooperage must be filled to capacity or covered with inert gas such as nitrogen, carbon dioxide or argon, and hoses must be as short as possible with tight coupling links.

The holding tank for the wine prior to bottling needs special attention, such as establishing a desirable wine temperature, using inert gas in wine transfer, avoiding turbulence and stirring without inert gas. Wine bottling is particularly important in controlling oxygen enrichment. Wine turbulence should be kept at a minimum and filling levels maintained at certain guidelines. Also, bottles need to be flushed with nitrogen (usually two to three bottle volumes) and a vacuum provided during filling. In conclusion, wines before bottling should contain less than 0.5 ppm DO.

The DO level in bottled wine is extremely important. However, headspace oxygen (HSO) that is contained in the volume from the top of the wine to the bottom of the closure also is critical to the DO level. The HSO is an additional source and moves gradually into the wine. The amount of oxygen in the headspace volume depends on the closure type: cylindrical, such as cork or a synthetic, or screwcap. A screwcap has the greatest HS (14 ml), while corks vary between 3 ml and 7 ml. Also, the recommended HS height is 15 mm, which avoids cylindrical ejection and leakage after HS gas compression at corking.

Excessive pressures often are caused by low-temperature wines, small HS volumes, high storage temperatures and bottling without HS sparging and vacuum. Studies have found that HS oxygen may increase the DO as much as 60%. Also,

the DO in wine bottles with screwcaps generally experience a DO increase three times greater than cylindrical closures. Another aspect that increases DO in bottled wines concerns the passing of internal gas (air) within the cylindrical closure pores to the HS at the sealing process. However, the amount of O<sub>2</sub> escaping to the bottled wine depends on the internal pressure, storage time and bottle orientation during bottle aging.

### **Sources of DO: closure type**

The third source that affects DO level relates to the closure type used in bottling. The oxygen-transfer rate (OTR) varies widely among closures. Because of this variation, types of closures influence the chemical and sensory properties of bottled wines. For example, research findings have indicated that screwcaps maintain higher levels of free SO<sub>2</sub> and result in less browning compared to natural, synthetic and technical corks in certain white varietal wines. However, screwcaps may offer reduced characters at extended storage periods.

In another instance, synthetic closures may bring about oxidized flavor during prolonged storage. Also, performance studies of natural corks have shown variability in OTR, which may lead to sensory differences in wine. Certainly, this response depends on composition and wine precursors.

Although wines may be vinified within the guideline levels for DO (<1.0 ppm) and HSO (<2.0 ppm), this does not excuse winemakers from adding SO<sub>2</sub>. This chemical is unique because it offers both antioxidant and antimicrobial properties. In wine, SO<sub>2</sub> prevents unwelcomed browning and oxidation processes. These responses are due to chemical reactions that are much slower than enzymatic oxidation.

The use of SO<sub>2</sub> and its interaction with O<sub>2</sub> is complex, involving several pathways and reactions. The ability of SO<sub>2</sub> to react with wine oxidants and prevent spoilage makes it indispensable in producing high-quality wines. Sulfur dioxide reacts with many

wine compounds such as acetaldehyde, pyruvic acid, glutaric acid, glucose and phenols, just to name a few.

One main oxidant in wines is  $\text{H}_2\text{O}_2$  (hydrogen peroxide), which is produced from phenolic compounds. This powerful oxidant reacts with  $\text{SO}_2$ , eliminating its effect in wine, and prevents oxidation. Keep in mind that  $\text{SO}_2$  in wines consists of two forms: free and bound. The former is the most active and is responsible for its antioxidant and antimicrobial properties. Also, the effectiveness of  $\text{SO}_2$  is influenced by such factors as pH, temperature, oxygen, phenolics and  $\text{SO}_2$  levels.

It is often reported that white wine should maintain a free sulfur dioxide content to achieve 0.8 ppm molecular  $\text{SO}_2$  based on wine pH with red wines being maintained at 0.6 ppm molecular relative to wine pH prior to bottling. Some general recommendations for free  $\text{SO}_2$  levels are: dry white wine (30-40 ppm) and dry red wine (20-30 ppm). It is important to remember that 4 ppm  $\text{SO}_2$  reacts with 1 ppm  $\text{O}_2$ , and the free  $\text{SO}_2$  should not fall below 10-12 ppm during the life of the wine.

### **Introducing part two: the study**

The second part of this article will describe in greater detail the scope of the study taking into account the size of the wineries, the type of filling and closure operations as well as the level of dissolved oxygen in the holding tank and during bottling. The amount of oxygen in the headspace of bottles is discussed as well as “total package oxygen” (the sum of dissolved oxygen and headspace oxygen), and the results of analyses for sulfur dioxide and pH levels will be reviewed. Finally, the results and implications for the Ohio wine industry will be examined.

If this study represents a majority of the Ohio wine industry and beyond, then it emphasizes the extremely important issue of maintaining appropriate dissolved and headspace oxygen levels in our wines at bottling.

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