BIO Web of Conferences **15**, 02013 (2019) https://doi.org/10.1051/bioconf/20191502013
*42nd World Congress of Vine and Wine*

# Sulphite dioxide reduction in wine: Management and control of oxygen added during bottling

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**Abstract.** Sulphur dioxide ($SO_2$) antiseptic and antioxidant role allows it to preserve the wine from oxygen's negative effects. However, its use is increasingly challenged by the concerns of consumers and producers who want to limit the chemical inputs in wines. During winemaking, many stages can lead to a transfer of oxygen to the wine. Bottling is crucial. In order to limit oxygen addition to the wine, various inerting devices have been developed by manufacturers. The first part of this work aims to understand the influence of bottle inerting sequence, rate work and pressure of inert gas, on the amount of oxygen in the bottle before filling. The results indicate that the level of oxygen brought to the wine depends on the settings implying to adapt them specifically to each bottling setup. Once inerted, the bottles are filled and corked. The influence of the filling nozzle and of the inerting devices on the oxygen addition was studied. The amount of oxygen brought to the wine during bottling was significantly reduced by the use of inerting devices. The influence of the filling nozzle and the setting conditions used was also highlighted. Thus, good management of oxygen addition requires the mastery of the bottling chain.

## 1. Introduction

The amount of oxygen transferred to wine during winemaking strongly affects its development. For red wines, the addition of oxygen can improve the wine's organoleptic quality, particularly by softening the tannins and reducing astringency. For white wines, however, its effect is often negative, as oxygen can affect colour evolution and degrade the wine's sensory qualities [1,2]. As such, winemakers need to control the oxygen level to which wine is exposed, throughout the whole of the production process. Studies show that certain winemaking stages such as pumping, clarification, tartaric stabilization, and conditioning – are critical in terms of oxygen supply. [3,4].

Total package oxygen (TPO) at bottling is a measure of the amount of oxygen transferred to the wine during this stage. It is the sum of the dissolved oxygen (DO) in the wine and the gaseous oxygen in the headspace (HS). TPO can also be considered post-bottling, by taking into account the oxygen transfer rate (OTR) of the selected closure.

Manufacturers now offer technical solutions by which to minimize oxygen intake at various bottling-chain stages. Filling and capping, both of which take place during bottling, are two critical steps in which oxygen transfer is a concern. The most effective inerting process appears to be the inerting of empty bottles, during which it is possible to remove 60–90% of the oxygen contained in the air in an empty bottle [5–7]. The principle is based on the replacement of the air inside the bottle with an inert gas (e.g. gaseous nitrogen insufflation, vacuum, or liquid nitrogen vaporization) [8]. Various techniques have also been developed to manage the HS oxygen. These techniques depend on the type of closure used (e.g. inert gas blowing prior to capping vacuum, Snow-drop$^{TM}$).

The current study looks to evaluate the effects on the TPO of inerting-device settings within a bottling chain, under real bottling conditions.

## 2. Materials and methods

### 2.1. Bottling chain

The bottles used in the tests were Bordeaux-type BVS 75 cL from UNIVERRE PRO UVA SA (Sierre, Switzerland) and BVS $28 \times 44$ mm; SCAP capsules equipped with a SARANEX SU38 EPEBP seal were used for capping.

The trials took place in 2017 at the Agroscope experimental cellar in Changins. We used a Galaxy 2000 monobloc conditioning line designed and assembled by COSTRAL (Riquewihr, France). This packaging line has a small to medium capacity, and an adjustable cycle model that incorporates an empty bottle rinser/inerter, an 11-nozzle filler, and a capper.

Preparation of the empty bottles consists of a rinsing step and an inerting step, carried out by the insufflation of 99.5%-pure nitrogen directly into the bottle, head-down, by means of a cannula. The nitrogen gas came from a nitrogen generator ($N_2$FLO, GENGAZ SRL; Wasquehal, France). The gas injection pressure could be adjusted between 1 and 2.5 bars.

For the filling stage, two nozzle types were tested: 'gravity depression' (GD) nozzles (with liquid-levelling through slight depression) and 'simple gravity' (GS) nozzles (with levelling without re-aspiration).

This line was complemented by additional equipment from the Elvamac brand, to inert the capsules and limit the HS oxygen level. The inerting parameters of the capsule were consistent for all trials.

### 2.2. Gas analysis

We used a NomaSense $O_2$ Prime oxygen analyser from NOMACORC S.A. (Thimister-Clermont, Belgium). To monitor oxygen during bottling, 11 bottles were each equipped with two PSt3 PRESENS GmbH (Regensburg, Germany) pellets: one pellet was immersed in wine to measure the DO, and one was placed in the HS to measure the amount of HS oxygen.

### 2.3. Statistical analysis

The results were statistically analysed with XLSTAT software (version 2016.02.28635; Addinsoft, Paris, France). Differences among variants were determined by analysis of variance ($p < 0.05$).

## 3. Results and discussion

### 3.1. Impact of bottle preparation on TPO

Let us look at the inerting sequence for bottles, prior to filling. The bottles are simultaneously capped immediately at the exit of the rinser/inerter sequence. Four repetitions of three bottles each were performed for each configuration ($n = 12$). The oxygen measurements were undertaken once the 12 bottles were inerted and capped. Between each test, the bottles were ventilated with compressed air to expel the nitrogen.

#### 3.1.1. Impact of inerting sequence

In this subsection, we focused on measuring the effects of the settings of the inerting automaton on the oxygen level at the end of bottle preparation, and on controlling the variability of this level in line with the work rate.

We tested two settings (i.e. settings 1 and 2). Table 1 details the inerting sequences and work rates according to the settings used. The use of setting 2 makes it possible to reduce the duration of nitrogen injection, which leads to costs saving.

Figure 2 shows the effects of the settings and the work rate on the amount of oxygen in the bottle, when using a nitrogen injection pressure of 2 bar. The presented results are the mean of 12 measurements with a 95% confidence interval. Asterisks indicate a significant difference between the two settings for a given rate. For a given setting, different letters indicate a significant difference between rates.

For setting 1, the oxygen levels measured in the bottles ranged from 1.43% to 4.13%. This variability highlights the net impact of the work rate (i.e. significant differences among the three rates). At 2,000 bottles/h, the sequence is much faster than that of 1,300 bottles/h (Table 1). The duration of nitrogen insufflation, combined with low injection pressure, does not allow one to drain all the

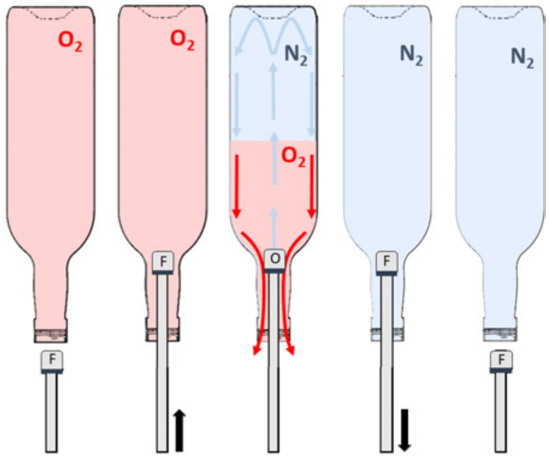


**Figure 1.** Description of the inerting device.

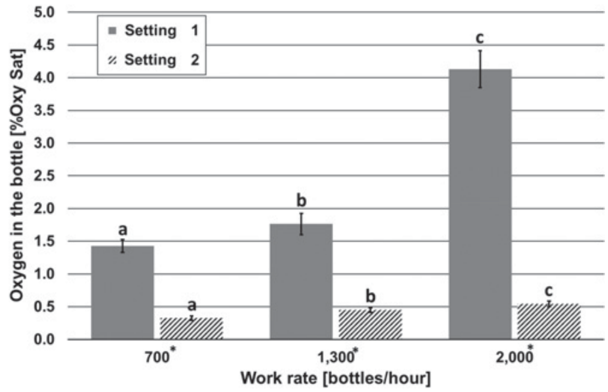


**Figure 2.** Oxygen levels measured in the bottle according to the work rate and the settings of the inerting automaton.

oxygen within the bottle. At 700 bottles/h, the sequence is longer and thus allows for a better inerting; however, the injection continues even after the cannula is out of the bottle, and this wastes inert gas and consequently increases production costs.

The use of setting 2 allowed us to significantly lower the oxygen content measured, for each of the three rates tested. The oxygen levels ranged from 0.33% to 0.54%, which are very low and close to the minimum that can be achieved with nitrogen produced by the generator. Indeed, the technical specifications indicate purity in the range of 99.5–99.8%. Nevertheless, the gaseous oxygen contents measured were statistically different among the three work rates.

Setting 2 seems to be better suited to the bottling conditions used in the Agroscope experimental cellar, as it limits both the oxygen supply to the wine and the variability according to the bottling rate. We thus determined that the use of suitable settings is critical to ensuring the effectiveness of the inerting device.

#### 3.1.2. Impact of gas injection pressure

Besides the duration of the inerting sequence, we studied also the effect of the injection pressure of the inerted gas and the work rate, using setting 2 for the inerting automaton (Fig. 3). The presented results are the mean value of 12 measurements with a 95% confidence interval.

**Table 1.** Inerting sequence characteristics according to the settings used.

| *Work rate* | *700 bottles/h* | | *1,300 bottles/h* | | *2,000 bottles/h* | |
|---|---|---|---|---|---|---|
| **Settings** | **1** | **2** | **1** | **2** | **1** | **2** |
| **Rise/fall sequence duration** | 4s12 ± 0s02 | 3s33 ± 0s06 | 3s55 ± 0s02 | 2s66 ± 0s02 | 2s92 ± 0s05 | 2s38 ± 0s02 |
| **Gas injection sequence duration** | 4s95 ± 0s04 | 1s92 ± 0s02 | 2s44 ± 0s01 | 1s59 ± 0s03 | 1s99 ± 0s03 | 1s48 ± 0s02 |

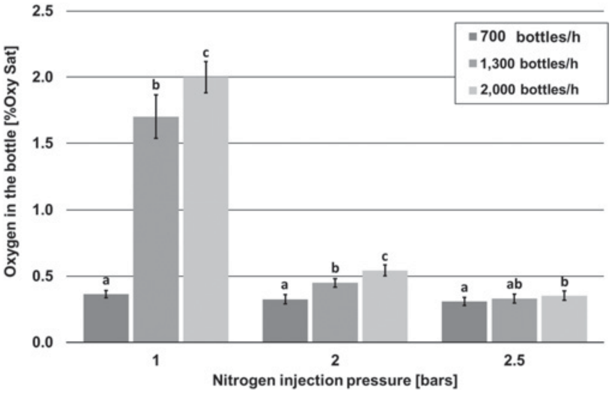


**Figure 3.** Oxygen levels measured in the bottle according to the work rate and the injection pressure of nitrogen gaz.

For a given pressure, different letters indicate a significant difference between the rates.

With an injection pressure of 1 bar, the oxygen values measured in the wine were found to vary widely, depending on the work rate. For a rate of 700 bottles/h, the oxygen level was 0.36 ± 0.03%; this allows to say that setting 2 and an injection pressure of 1 bar is sufficient to obtain good inerting.

For the 1,300 and 2,000 bottles/h rates, the in-bottle oxygen levels were much higher (i.e. 1.70 ± 0.17% and 2.00 ± 0.12%, respectively). These higher oxygen inputs indicate that this level of insufflation pressure is not enough to ventilate all the oxygen from the bottle.

The results obtained for a pressure of 2 bar with setting 2 were presented previously (see subsection 3.1.1). The oxygen levels obtained ranged from 0.33% to 0.54%. The effect of the work rate is substantial, but still lower than that of an injection pressure of 1 bar.

In the case of an injection pressure of 2.5 bars, the oxygen level measured in the inert bottles ranged from 0.31% to 0.35%. At this pressure, the difference between the levels measured at the three rates is almost zero Again, given the nitrogen purity produced by the generator, it is not possible to obtain lower values.

The increase in pressure makes it possible to improve inerting performance with empty bottles, in line with the number of bottles and the oxygen-level variability. Nevertheless, with strong insufflation pressure, the nitrogen consumption is substantial. Clearly, the economic aspects of nitrogen production or the purchase of bottled nitrogen need to be taken into account, and it is

curcial to strike a balance between inerting efficiency and the related cost.

The use of setting 2 significantly improves the inerting of empty bottles. Nitrogen insufflation allows one to ventilate the oxygen within the bottle and achieve very low levels – levels approaching the limit obtained with generator-of produced nitrogen. Low variability persists among the different work rates, and this variability can be neglected with an increase in insufflation pressure.

These tests and the results obtained are relevant in the context of using the Galaxy monobloc by Agroscope – that is to say, at a work rate of approximately 1,300 bottles/h and with Bordeaux-type 75-cL bottles. Indeed, the air–nitrogen mixing dynamics during insufflation will vary with bottle profile and volume. Consequently, the robustness of the settings and the inerting process must relate to the technical and economical objectives of the winemaker, who needs to strike a balance among inerting efficiency, working conditions, and nitrogen consumption.

## 3.2. Impact of bottling and capping on the TPO

Once inerted, empty bottles are filled and then capped. The oxygen inputs must be controlled at these stages. At the filling – plugging stage, several critical points can be identified – for example, the integrity of the nozzles, the filling rate, the type of closure, and the inerting device for the HS.

As mentioned, these trials were carried out in real conditions, during the bottling of "Chasselas Domaine de Changins 2016" at the experimental cellar of Agroscope, Changins.

### *3.2.1. Control of filling nozzle integrity*

The first part of this experiment consisted of checking the integrity and homogeneity of the 11 filling nozzles in terms of oxygen supply. The mean TPO was measured for each spout. Figure 4 shows the mean values of six measurements taken for each spout. The error bars represent a 95% confidence interval. Means that do not share common letters are significantly different according to the Tukey clustering test, at the 95%

In the case of the GD nozzles (Fig. 4A), we found significant variations in TPO as a function of the nozzle number, with values ranging from 3.2 to 4.3 mg/L. Statistical analysis of the differences among the nozzles, allowed us to distinguish three groups: spout 7 caused oxygen enrichment significantly greater than that seen with spouts 2 and 3; spout 8 caused significantly greater wine fortification than spout 2, and the other spouts introduced similar oxygen quantities into the wine.

Separate analyses of the DO and HS oxygen measurements (data not shown) made it possible to confirm that variations observed among the nozzles are due solely to DO differences.

For GS nozzles (Fig. 4B), we found no significant differences among the spouts: the amount of oxygen introduced into the wine during filling was homogeneous, irrespective of the nozzle (i.e. approximately 3 mg/L).

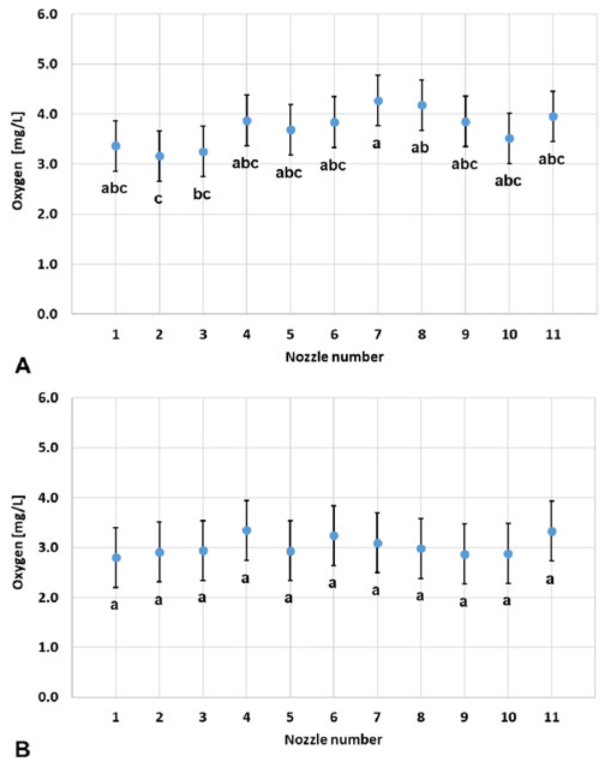


**Figure 4.** TPO measured for the eleven nozzles of the bottling chain and for each type of nozzle.

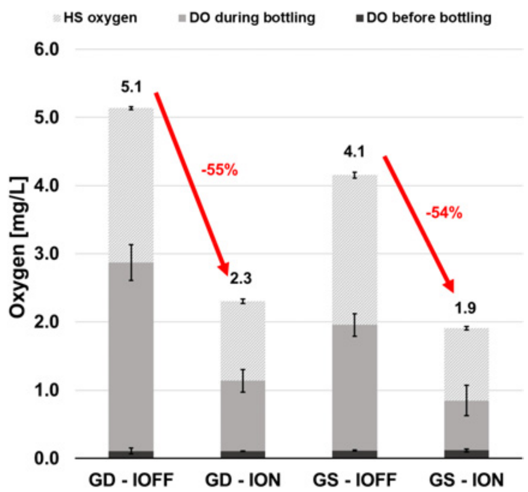


**Figure 5.** Effect of using inerting on the TPO.

### 3.2.2. Impact of inerting device

To confirm the efficiency of the inerting devices, bottling tests were carried out with (ION) and without (IOFF) these devices. Figure 5 shows the mean values of 3 measurements for dissolved oxygen before bottling and 33 measurements for dissolved oxygen during bottling and headspace. The error bars represent a 95% confidence interval.

Irrespective of nozzle type tested, we found that the use of inerting devices reduces TPO (Fig. 5). The pre-bottling DO content in the wine was low (i.e. around 0.1 mg/L under each of the four conditions). In the non-inerted variants, we found TPO values of 4–5 mg/L. In the absence of an inerting device, wine conditioning led to high oxygen enrichment.

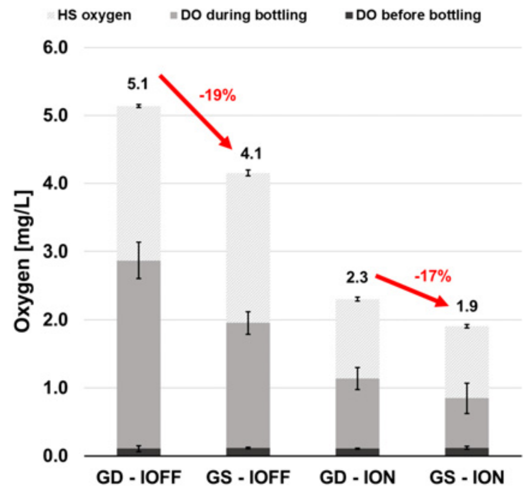


**Figure 6.** Effect on nozzle type on TPO.

When the pre-filling inerting of the empty bottles and the inerting of the HS and the capsule took place, the TPO decreased by more than 50%. The measured values were around 2 mg/L. The DO during the setup decreased by about 60%, while the HS oxygen was reduced by 50%.

During our test configuration, we were unable to distinguish the effects of each inerting station; however, it seems quite obvious that the inerting of empty bottles prior to filling reduces oxygen enrichment of the wine during filling. Similarly, the amount of oxygen trapped in the HS is reduced upon using the Elvaprotect inerting device. To separately check the effect of each inerting station, we would need to undertake additional, individual tests while using the devices.

### 3.2.3. Impact of nozzle type

To determine the performance of the bottling equipment, we tested two nozzle types for the filling step. Figure 6 shows the mean values of 1 measurement for dissolved oxygen before bottling and 11 measurements for dissolved oxygen during bottling and headspace. The error bars represent a 95% confidence interval.

For non-inerted variants, the TPO was found to have decreased, from 5.1 mg/L (with GD nozzles) to 4.1 mg/L (with GS nozzles); this represents an almost 20% decrease. When the inerting devices were activated, the system was clearly more efficient, as explained above. In terms of TPO, the gain obtained with the GS nozzles appeared to be minimal (i.e. $< 0.4$ mg/L), but still represented an approximately 17% decrease. In comparison to the GD nozzles, GS nozzles have a significant impact on the TPO. More specifically, the effect of the spout type is mainly measured by a decrease in DO, in the order of 30%; this is the case, regardless of whether inerting took place. The HS oxygen was only moderately impacted.

### 3.2.4. Impact of work rate

Finally, to complete this study, we assessed the effect of the bottling rate on the level of oxygen introduced into the wine. These tests were carried out by using GS nozzles and various inerting devices. Figure 7 shows the results.

Under these conditions and a 1,000 bottles/h rate, we found a TPO of 1.9 mg/L. At 2,000 bottles/h, the TPO

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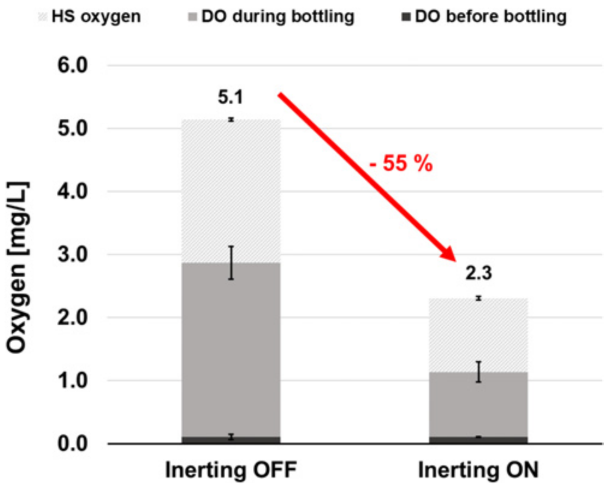


**Figure 7.** Effect of the work rate on TPO using GS nozzle.

reached 0.9 mg/L – a 51% decrease. Imposing a work-rate increase clearly makes it possible to significantly lower the TPO. As Fig. 7 shows, this decrease relates to a decrease not only in HS oxygen (−60%) but also in DO during bottling (−40%).

These test results indicate, once again, the importance of adjusting the settings of the inerting devices installed in bottling equipment to one's working conditions, if one wishes to control the amount of oxygen introduced into the wine.

## 4. Conclusion

In the current study, we found that oxygen-transfer management requires control of the packaging chain. Such controls demand the execution of a set of good practices, from the preparation of the wine to corking. Good practices become even more relevant when the winemaker wishes to limit sulphite dioxide content.

At the draw, simple gravity (GS) nozzles induce an oxygen uptake that is 15–20% lower than that observed under the same conditions but with gravity depression nozzles and with reduced variability among the nozzles.

Regarding bottling, particular attention must be paid to the settings of the various inerting devices, if one wishes to minimize oxygen transfer to the wine.

By combining the use of GS nozzles, a comfortable working pace, and inerting devices, one can produce remarkable results. Indeed, in the current study, we were able to reduce the total oxygen content of the packaging to below 1 mg/L.

From a practical viewpoint, the use of GS nozzles seems perfectly suited for bottling Swiss white wines, which tend to have a higher carbon dioxide content than wines from other regions. Visually, outgassing seems less important during filling, and the virtual absence of foam makes levelling easier and more predictable.

Finally, the robustness of the work settings and the inerting process must align with the technical and economical objectives of the winemaker, who is tasked with striking a balance among inerting efficiency, working conditions, and nitrogen-gas consumption.

We would like to thank Costral for their technical support and the provision of equipment. Vinventions and Elvamac companies for technical support. Thank you to all our Agroscope colleagues who participated in this study, including Philippe Seixas, Emilie Gerber and Matthieu Nussbaum.

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