CONTROL OF THE OXYGEN SUPPLY DURING THE CONDITIONING. PART 2 : BOTTLING AND BAG-IN-BOX

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Conditioning

Operatory conditions:
Before starting the bottling, it is necessary to bring the dissolved oxygen level of the wine to be bottled to a maximal level of 0.5 mg/L. However at this point in time, the oxygen dissolution speed of the wine is close to its maximum (according to Fick's law, confirmed by Vidal et al (16)). It is therefore necessary to use all the means possible mentioned throughout this article in order to prevent see limit all contact with the air.

Circuit (from the tank of wine to be bottled to the entry of the distribution line):
The compactness and the absence of contact with air are positive factors to obtain good results in the middle of the cycle. The volume of the circuit must be reduced to a minimum and must be inerted so as to limit the enrichment at the beginning and the end of the conditioning cycle.
The supply due to the final filtration on cartridges or plates is often negligible and is easily controlled by inerting and by an initial passage of wine through the circuit.

Inertion equipment for bottles:
Certain bottling line producers integrate inertion equipment in their circuit. The bottle arrives under the beak of the inertion equipment, and a vacuum pump allows for the removal of up to 90% of the air in the bottle before the injection of a neutral gas at a light pressure (20 KPa).
The inertion of bottles before filling was observed on two conditioning chains (17). Thanks to the inertion, the average supply observed in both cases was of 0.4mg/L for a initial dissolved oxygen levels of 0,80 and 0,23 mg/L, in this case corresponding to a decrease in the dissolved oxygen supply by 63%. The gas used for the inertion was in the first case alimentary nitrogen and in the second case a mix of 20% CO_2 and 80% N_2. Zingarelli and Gerbi obtained a decrease in the dissolved oxygen supply of 89% (36). Vidal et al. (16) obtained on the same equipment, the lowest supply (≈ 0,6 mg/L) with a mix of 20% CO_2 and 80% N_2. In order to respect the partial pressures between the wine and the gas, a sensible choice would be the use of a mix containing 50% CO_2 for dissolved CO_2 levels in the wine of higher than 700-800mg/L (8).

Wine Bottler:
The are also monoblocks with the inertion of the annular distributor of the bottling line with the returning of the inertion gas introduced by the inertion equipment towards the annular distributor as the bottling proceeds, this therefore can protect the liquid at the moments where the exchanges with the atmosphere can be at their highest since the wine flows in thin layers over the walls of the bottle.

Stopper and encapsulator :
For stoppers, levels of trapped oxygen in the headspace lower than 10% v/v were obtained on lines equipped with inertion equipment before the filling and with stoppering under vacuum (>70KPa) and with a headspace of <6mL (19). These results have since been confirmed (37).
Certain bottling centers are also equipped with a tunnel placed after the wine bottler up until the stopper equipment where the neck is swept with neutral gas. Certain producers also propose stoppers equipped with a neutral gas sweeping system right before the placing under vacuum necessary during the stoppering (36).

For bottles with screwcaps, according to Vidal et al. (19), the snow-drop after filling (3.1 %v/v) is more efficient than the association snow-drop (injection of liquid neutral gas) before filling-sweeping of the headspace with CO_2 (average on 2 lots: 13,5% v/v). Furthermore, the decrease in the oxygen level is low with this last inertion method in comparison to levels measured without inertion (14,8% v/v). The snow drop after filling can be improved and refined by playing with the mass of injected liquid nitrogen and the time between the injection of the neutral gas and the sealing of the ring in order to guarantee that the internal pressure never exceeds 300kPa in absolute pressure (whatever be the stocking temperature) over which pressure the risk of impermeability loss is high.

More recently, an encapsulator equipped with an inertion system using CO_2 to inert the neck and the interior of the screwcap before its application was controlled. The analyses of the trapped gaseous space in the bottle demonstrate clearly the superiority of the inertion of also the interior of the screwcap in comparison to the inertion of only the neck at a CO_2 flow of 40 to 250 L/h, since in the first case the average observed was of 6 %v/v whereas in the second case, it was observed, independent of the CO_2 flow, a level always higher than 15% v/v with a internal overpressure of 3 to 8,5 KPa. Of these measures the INRA confirms and validates the measures completed in Australia by O'Brien and Gibson (38) which report that the filling of the neck and the interior of the screwcap allows for the obtaining of an oxygen level in the headspace of 1,1% v/v, instead of 12,2% v/v with only the inertion of the neck or 20,7% v/v with no inertion.
If no protection measures are taken, the stoppering by screwcap presents a double inconvenience in comparison to a traditional stopper since it has a greater headspace and there is the absence of vacuum use which result in a higher oxygen level. The inerting of the empty bottles and the inertion of the interior of the screwcap or the snowdrop technique before stoppering are techniques which are efficient at decreasing this impact. Finally, one must keep in mind that nitrogen generates a higher internal pressure, since its solubility is 59 times less than that of CO_2.

Closures :
Technicians now have the possibility to adapt the oxygen permeability of the stoppering equipment according to the oxidation potential of the wine, thanks to, notably the knowledge of the Oxygen Transfer Rate (OTR) which one can define as the quantity of oxygen passing across the closure over a certain time and under particular conditions of the temperature, the diameter of the neck of the bottles used, the difference of the partial pressures between the two extremities of the closure and of the atmosphere used (**table 4**). The experimental conditions must be examined attentively before being able to compare OTR values (42), notably the temperature, humidity, nature of the atmospheric gas, the pressure difference between the exterior and the interior of the whole. It is also important to keep in mind, that these permeabilities will result higher than in reality since they are obtained using experimental conditions gas/ closure/ gas (Mocon) or with reductive solutions such as Carmen indigo (26) or BPAA (43) different to the conditions found in a wine bottle.

The homogeneity of the OTR which characterizes each type of closure (26, 37, 44) should also be taken into account, since it contributes to the diminution of sporadic oxidation post-bottling. The availability of the OTR and its standard deviation for each lot could be valuable information for conditioners. In fact, Crochiere demonstrated, that even on an industrial product (Saranex capsules) reputed to be more homogeneous that natural cork, only one lot of 16 tested had the stated permeability (37).

Authors	**Peck J. 2005** (39)	**Godden et al. 2005** (40)	**Lopes et al. 2005** (26)	**Nomacorc 2005** (41)
Method	Mocon Oxtran / air	Mocon Oxtran / air	Carmen indigo	Mocon Oxtran / 100% O_2
Screwcap	0,003-0,039* <0,004-0,017**	0,009-0,034*		
Natural cork	0,30-1,29* 0,49-0,54**	0,004-5,26*	0,03-0,30**	
Technique		0,03-0,056*	0,03-0,045*	
Classic Nomacorc			0,22-0,41**	0,23-0,28**
Injected-formed	0,43-1,29* 0,49-0,54**		0,43-0,69**	0,30-0,86**

Table 4 : comparison of the OTR values found in literature

All the values are expressed in mg/month/closure. The 2 values given correspond either to the minimum or maximum indicated by the authors, or the average decreased or increased by the standard deviation, when the values are known**. Data provided by Nomacorc.*

Bottles:
The conditioner can play with the empty space height of the different bottle formats for bottles to be corked or screwcapped, as well as the length of the cork for bottles to be corked, in order to reduce the height of the headspace and also its volume. It must however respect the filling height pre-established by the bottle maker in order to limit risks, since all impermeable systems have a pressure limit level, notably during the transport and stocking phases during which the bottles can be sporadically submitted to temperatures above 50°C. In fact, it is important to remember that all liquids exposed to elevated temperatures dilate. This dilation compresses the gases present in the headspace and therefore create an increase in the relative pressure in the packaging, which can continue towards infinity when the liquid touches the closure. Therefore, for a headspace where the volume represents 2% of the liquid volume, above 45°C, the internal pressure in the packaging increases very quickly and can rapidly pass 400kPa in absolute pressure. In addition, it is important for the internal pressure in the recipient to not pass an absolute pressure 300KPa, if we want to avoid the faults of leaky bottles (whichever be the closure) and/or those of the discharge of the stopper. The conditioners must remember that for a small headspace, as shown in **table 5,** that the temperatures to not pass above will automatically be low if one wants to avoid leaky bottles (source LNE)

Filling level	Stopper lenght	Leak risk starting from*
63 mm	38 mm	49 °C
	44 mm	43 °C
	49 mm	40 °C
	53 mm	36 °C
55 mm	38 mm	41 °C
	44 mm	34 °C
	49 mm	29 °C
	53 mm	21 °C

Table 5: Influence of the filling level and the stopper length on the risk of leaky bottles

- *For a filling temperature of 20 °C.*

Furthermore, in the framework of a number of stopper trials, the Inter Rhone controlled the dimensions of the necks of 570 bourgogne shaped bottles of the same lot at 3, 15, 30 and 45mm of depth. Around 10% of these bottles were not conforming. This study showed that heterogeneity of the bottle necks should be considered, since they can be responsible for problems during the stoppering, a partial loss of impermeability and leaky bottles.

Stocking position of bottles :
Lopes et al. (45), Skouroumounis et al. (46) describe experiments on the stocking of bottles over several years, which show that the entry of oxygen is slightly lower in bottles laying horizontally when compared to vertical storage. However, according to Mas et al. (47), the wine would clearly be preserved better in bottles conserved horizontally.

Factors responsible for sporadic oxidation post-bottling:
The phenomenon of "undefined oxidation" or "sporadic oxidation post-bottling" is more and more attributable to a series of factors inherent to the operations and bottling materials which lead to increased oxygen levels in a small proportion of bottles rather than the single characteristics of the stoppers (48). As shown in certain studies executed notably in France and Australia, these premature undefined oxidation problems appear generally 6 months after the bottling, since they are due to supplemental levels of total oxygen in the order of 1-3mg per bottle acquired during the bottling.
Among the possible origins, it is possible to cite: the beginning and the end of the bottle filling, the chaotic flow during filling, the stops in the bottling line, the dysfunction of the systems to place the bottles under vacuum during the filling, the stoppering and leveling of the liquid, the disrespect of maximum heights, the performance irregularities of the filling and stoppering equipment, the heterogeneity of the bottle necks and closures, the presence of exogenous auto-oxidants (including ellagin tannins arising from cork stoppers (49) and ascorbic acid) and insufficient levels of free SO_2.

Table 6: Practical advice to reduce oxygen levels in bottles.

Operation or material	Advice	Objective
Initial wine tank to be bottled	Higher than the conditioning chain	Avoid cavitations of the centrifuge pump or the short-circuiting of the feeding pump and the buffering tank
	Close to the bottling area	Reduce the length of the circuit in order to decrease the volume
	Inert the tank before filling (3 times the volume of the wine according to Allen (50)) and maintain with neutral gas	Reduce the amount of gaseous oxygen in contact with the wine in order to limit its dissolution
	Bring the dissolved oxygen levels to less than 0.5mg/L, by gas injection during the filling or by bubbling with stirring rod	Reduce the final dissolved oxygen levels in the bottle
	Choose a tank whose volume is equal to or slightly greater than the volume to be conditioned	Limit the emptying of the tank and the consumption of the inert gas
Tubing	The shortest possible and in stainless steel impermeable to oxygen. Limit use of supple tubing	Reduce the length of the circuit to reduce the volume. Avoid the uptake of air at the junction levels during the pump aspiration.
	Adapt the diameter to the flow required by the bottling tempo: see calculation of the liquid flowing speed (14).	Limit turbulence phenomena and half empty tubes.
	Purges situated on high points of the canalization	Permit for a better evacuation of air by inertion and tubing filled with wine.
	Limit as much as possible corners and tubes suspended to the ceiling	Obtain a better inertion and filling of canalizations

	Control the residual oxygen levels after inertion at the purge points, one time per installation	To determine the necessary inertion time to obtain a threshold of 2% v/v of gaseous oxygen in the circuit (15).
Start of bottling	Inert the circuit	Limit the increase of oxygen supply at the start.
	First pass wine through the circuit (filler included), without return to the source wine tank	
Inertion	Regulate the vacuum to –90 KPa	Eliminate 90% of the oxygen contained in the bottle before the injection of neutral gas
	Injection of neutral gas between 1 and 5g/ bottle according to the tempo, nature of gas and authors (50, 51)	Obtenir une bouteille pleine de gaz neutre juste avant l'emplissage. Obtain a bottle full of neutral gas right before filling.
	Adjust the inertion gas quality according to the CO_2 levels of the wine (30).	Favourize the partial pressure equilibrium between the gaseous phase and the wine and therefore reduce the oxygen dissolution during the filling.
Filler	Regulate the vacuum to –8 KPa.	Limit the depression caused by the liquid leveling in the bottle.
Stopper	Sweep with CO_2, since it is more soluble than N_2 in the wine	Limit the risks of overpressure and reduce the oxygen level in the air chamber of the bottle.
	Regulate the vacuum to –80 to 90 KPa	
Screwcap closer	Injection of liquid nitrogen after the filling or injection of CO_2 in the interior of the screwcap.	Notably decrease the oxygen level in the headspace.
End of filling	Shoot with neutral gas	Limit the oxygen supply increase at the end of the chain
Closures	OTR choice	Adjust the OTR according to the quality and conservation time of the wine
	Verify the OTR (σ OTR) homogeneity.	Limit sporadic oxidation post-bottling
Bottles	Verify the neck dimensions	Limiter problèmes au bouchage, diminution de l'étanchéité à l'oxygène atmosphérique et bouteilles couleuses. Limit problems during the stoppering, decrease the permeability to atmospheric oxygen and leaky bottles
	Choose bottle formats with minimal empty space heights	Limit the headspace volume in a compatible manner with the risks of overpressure due to a high elevation of temperature during the transport or conservation.

Some specifications for bag in box conditioning:
Wines which are conditioned in bag in box are more sensitive to oxidation than wines in bottles (17), which lead to shorter recommended conservation times (52). The main films (EVOH and PETmet) as well at the taps (53, 54) have a high permeability to oxygen, which increase with the humidity level and temperature. For example, EVOH sees its oxygen permeability multiply by 4 and up to 15 when the relative humidity is higher than 80% (55). When the temperature passes from 15 to 30℃, the PETmet film sees its oxygen permeability more than double and that of the Vitop tap passes from 0,25 to 0,65 mL/day (56).
Dufrêne et al. (57) define that the box and the paper quality have a great importance on the « flex-crack » phenomena and on the wear of the films which are responsible for an increase in the gas permeability. Therefore it is advisable to use a non abrasive interior paper to limit the wear, to adjust the interior dimensions of the box and the pocket, to verify the dimensions and ensure the absence of points which are susceptible to cause wear or cuts in the films.

Beyond the advice already cited for the preparation of the wine, the circuit and the use of neutral gases, the flow of the pump which pushes the wine towards the final filters must be calculated considering the average flow of the line and not on the instant flow at the head of the filling. This permits for to limiting of stops during the production, and avoid that the by-pass, mandatory for a volumetric pump, be solicited too often, causing a prejudicial stirring of the wine (57).
After filtration, the wine must be sent to a buffer tank under natural gas pressure. The pressure on the wine at the exit of this tank will be consistent and regulated by taking into account the eventual variations in the wine level, therefore limiting the turbulences and the contact with the gaseous oxygen trapped in the circuit (16).

The major French market suppliers equipped themselves with Form-Seal-Fill units which complete the sealing of the films and fixing of the tap, limiting the manipulations and therefore the associated risks of increased permeability, and a rotating filler fills the pockets hence formed thanks to 4 or 6 heads at a time, permitting for a less turbulent regime.
Concerning the gaseous space, fillers equipped with an adjustable table permit for the obtaining of an enclosed bubble of gas which is as small as possible in the pocket. This bubble is necessary in order to avoid the overflow of the wine during the filling of the pocket. The length of the generator of the gaseous space must be of a maximum of 5cm for BIB of 3 or 5 L (57). The INRA completed measurements of the generators in BIB of 3 and 5L which correspond to respective gaseous space volumes of 60 and 71mL. If we compare the volumetric ratio of headspace/wine per container, a ratio of 2% for 3L and 1.4% for 5L are obtained, in comparison to 0.8% for a bottle of 75cl stoppered with an empty space of 55mm and 1.9% for a screwcap bottle with an empty space of 45mm. These ratios closer to those of the screwcap bottle rather than stoppered prove that one most look to act on the gaseous oxygen level.
Samplings within the gaseous space in the few seconds which follow the attachment of the tap permitted for the observation of a certain efficiency of nitrogen inertion at the base of the filling head and under the tap before the attachment to the film. The inertion permitted for the reduction of the oxygen levels by 22% and to obtain values of 11 to 12% v/v (BIB 5L, INRA internal data). However to our knowledge, the present measures do not allow for the attaining of values lower than 6% v/v.

Conclusion

The integration of measures to calculate dissolved and gaseous oxygen in the conditioning areas and of wine treatment procedures incorporating reasoned protection against oxygen will contribute to the homogeneity of the conditioned lots and to a lengthening of the conservation times of wines all while limiting the addition of sulfites.
The rationalization of operatory conditions, the taking into account by material producers of oxygen protection notably thanks to inerting-filling-stoppering monoblocks and the judicious use of inertion gases throughout the conditioning chain from the reception of the wine up to the filling and closing have allowed for certain installations to obtain levels of trapped oxygen lower than milligrams.
The closure by screwcaps or by stoppers becomes a primordial element for the control of the oxygen supply post conditioning thanks to the taking into account of the OTR and of its reproducibility from one production lot to another.
However, these precautions only constitute an initial response to the problems linked to oxidation phenomena. The conservation temperature always comes up as a primordial element in the evolution of conditioned wines (5, 18). But only a superior comprehension of the reaction mechanisms in place is susceptible to bring reasoned solutions in accordance to the different oenological situations.

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Keywords: dissolved and gaseous oxygen, inertion, SO_2, packaging, bottle, bag-in-box.

Synthesis:

The adaptation of methods for measuring dissolved oxygen in any type of container (thanks to the creation of gas tight circuits) and the continuous control of more than 14.000hl of still wine of all kinds during their conditioning (reception, treatment, bottling or bag in box packaging) have made it possible to characterize oxygen additions during the various operations which the wine undergoes and to propose solutions in order to reduce them, and to limit the use of antioxidants (SO2, ascorbic acid) while preserving the aromas as well as possible and by lengthening the shelf life of conditioned wines.

In general, the final total pick-up depends mostly on the relationship between the volume of wine to be treated and the volume of the circuit.

The most important enrichments occur particularly during the operations of cold tartaric stabilisation and during the bottling, especially if no precautions are taken to protect the wine from the oxygen in the ambient air.

As for the oxygen trapped during conditioning in the headspace, the tests carried out show that the quantity of oxygen trapped in the headspace right after stoppering varies from 0.38 to 3.58 mg per bottle, to which it is necessary to add the dissolved oxygen in the wine whose measured values vary from 0.5 to 6 mg/L.

The rationalization of the operating conditions, the judicious use of neutral gases especially at the beginning and the end of operations, as well as the inertion of bottles before filling and right before closing make it now possible to control and significantly decrease the oxygen pick up during all the operations related to conditioning. The objective is to go below one milligram of total oxygen trapped in bottle or bag in box has already been reached in certain installations.

Finally, the conditioners will continue to have more possibilities to put in practice a qualitative evolution of the desired wine and this also for the closures, which have had significant progress in their homogeneity and their oxygen permeability levels (Oxygen Transfert Rate).