

TOWARDS OFFERING WINE TO THE CONSUMER IN OPTIMAL CONDITION - THE WINE, THE CLOSURES AND OTHER PACKAGING VARIABLES.

A review of AWRI research examining the changes that occur in wine after bottling. PART 2.

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Factors related to the formation of *reductive* character in bottled wine

It is probable that in the future the majority of wine producers will be using closures with lower and more consistent oxygen permeation than the closures that they currently use. The greatest potential risk of such a scenario is the danger of wines producing 'reductive' characters after bottling.

It must be clearly re-stated that screwcaps, or any other closure for that matter, do not cause *reductive* character in bottled wine, and also that the vast majority of wines sealed with screwcaps do not exhibit *reductive* characters. Additionally, many wines sealed with closures other than screwcaps also exhibit *reductive* characters. The AWRI periodically stages *Advanced Wine Assessment Courses*, which are intensive four day courses aimed at preparing experienced tasters to act as wine show judges. The most recent course was held in September 2004, and a slightly higher percentage of wines sealed with cork were considered by participants to exhibit *reductive* character, compared to wine sealed with screwcaps, although that difference was not statistically significant. Clearly, developing an understanding of the causes of such characters in wine sealed with all closures is desirable.

The propensity of *reductive* characters to develop is a function of the composition of the wine at bottling, but the mechanisms are complex and have yet to be fully elucidated. Additionally, whilst the compounds responsible for *reductive* aroma are chiefly assumed to be those containing chemically reduced forms of sulfur, particularly the thiols (a large group of compounds containing chemically reduced forms of sulfur, including mercaptans), it should also be noted that there are probably a great many such compounds in wine that are currently unidentified, and their aromas and aroma thresholds unknown.

As indicated above, in September 2002 the AWRI bottled a second closure trial, using a very similar wine, bottling procedures, and the same bottled-wine storage as was used for the initial trial discussed above. This trial was instigated on a commercial-in-confidence basis, in order to accommodate the many commercial entities that had approached the AWRI to have products tested in a similar fashion to that of the original trial. The results up to 18 months post bottling for the screwcap and reference 2 and 3 corks were published in the August 2004 issue of the AWRI *Technical Review* (Godden et al. 2004). As it had been speculated that the formation of *reductive* character might be related to a combination of the SO₂ concentration, the filling height, and the dissolved oxygen concentration at bottling, the bottling of this trial was used to investigate two of these factors, namely SO₂ concentration and the ullage space at bottling.

The wine (2002 Clare Valley Semillon) was bottled under screwcaps (Auscaps with a tin liner), utilising two filling heights and two SO₂ concentrations. Details of the four treatments are provided in Table 3.

Table 3. Experimental design—trial of the effect of filling height and SO₂ concentration on the formation of reductive characters in bottled wine

'Low' filling height (48 mm ullage, Free SO ₂ 38 mg/L)	'Low' filling height + SO ₂ (47 mm ullage, Free SO ₂ 54 mg/L)
'High' filling height (30 mm ullage, Free SO ₂ 39 mg/L)	'High' filling height + SO ₂ (29 mm ullage, Free SO ₂ 59 mg/L)

As demonstrated in Figure 6, neither the filling height nor Free SO₂ concentration at bottling were found to have influenced the intensity of *reductive (struck flint)* character in the wine sealed with the four screwcap treatments in this trial, 24 months after bottling.

However, it is apparent from Figure 7 that there was some correlation between the free SO₂ concentration and the intensity of *reductive* character, when bottles sealed with all of the closures in the trial with the standard SO₂ concentration were considered. These other closures mainly consist of technical corks, and natural cork closures, some of which have had proprietary treatments applied which might lower their oxygen permeability. For the wine used in this trial, the concentration of free SO₂ at which the intensity of *struck flint* character began to increase markedly was approximately 23 mg/L. A similar relationship was seen in wine from the original closure trial, with the SO₂ concentration at which the intensity of struck flint character increased exponentially being approximately 12 mg/L. This SO₂ concentration was the same at 63 months post bottling (Figure 8) as was observed with wine from that same trial at 24 months post-bottling (data not shown).

Although a positive correlation was found to exist between SO₂ concentration and the intensity of *reductive* character in individual bottles of the wines used for both trials, the relationship is considered to be coincidental and not causal. For an explanation, it is logical to look for a common variable that might lead to both the consumption of SO₂ and the loss of compounds that we describe as 'reductive'. That variable is considered to be the rate of oxygen ingress allowed by the closure.

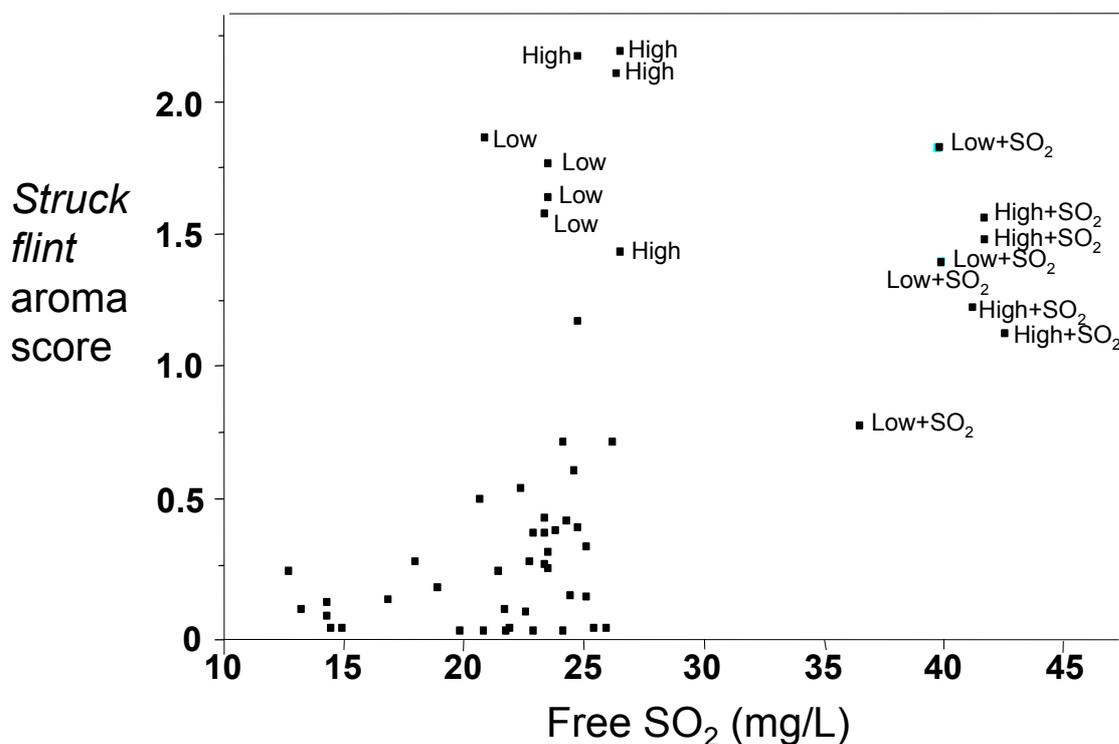


Figure 6. AWRI 'commercial closure trial': Relationship between Free SO₂ concentration (individual bottles) and mean scores for struck flint (scale zero to 9) during sensory evaluation conducted 24 months post bottling.

The lower the rate of oxygen ingress, all other things being equal, the lower will be the rate of SO₂ loss. Additionally, the lower the rate of oxygen ingress, all other things being equal, the lower will be the rate of oxidation of thiols, which might allow the concentrations of the thiols to rise above the sensory threshold *if* the wine has the propensity for this to occur. Thus, a positive correlation between the variables of SO₂ concentration and intensity of *reductive* character should be expected.

This hypothesis is supported by a trial where a Chardonnay wine was sealed under cork, screwcaps, and a portion of the wine was sealed in glass ampoules in the absence of oxygen, which were then stored in an anaerobic environment. Four years post-filling, wine sealed with the screwcaps and ampoules received the same ratings for *oxidised*, but wine from the ampoules was rated significantly higher for the attribute *flint/rubber* during sensory assessment. Whilst this trial was not highly replicated, it does support the presence or absence of oxygen at the time when thiols are being formed as an important factor in determining the degree of development of *reductive* character in bottled wine.

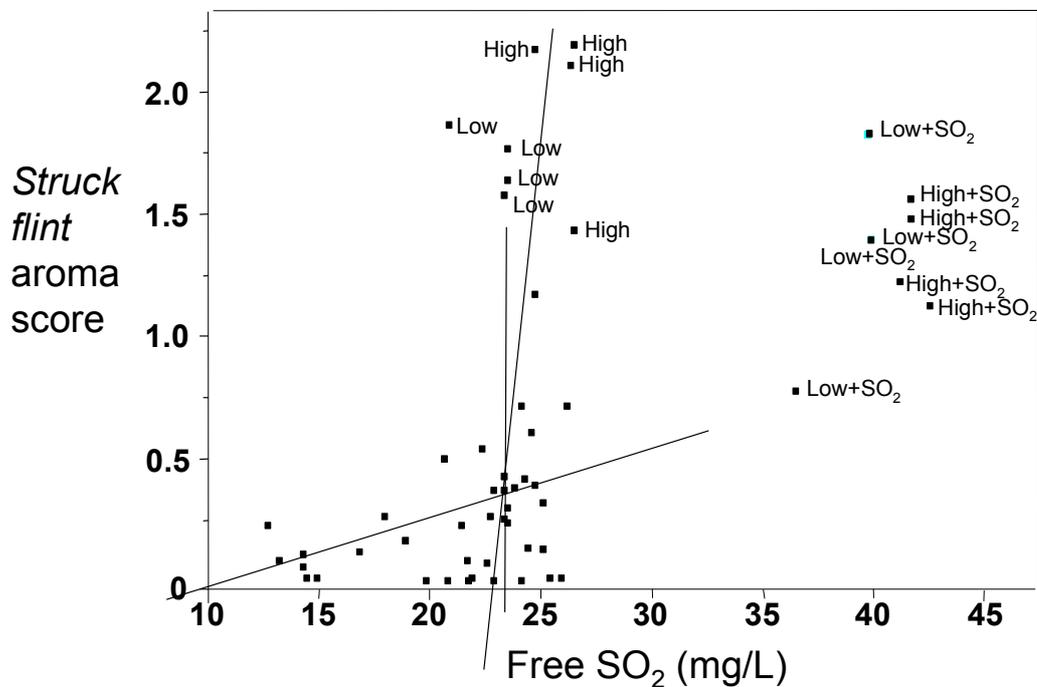


Figure 7. AWRI 'commercial closure trial': Relationship between Free SO₂ concentration (individual bottles) and mean scores for struck flint (scale zero to 9) during sensory evaluation, conducted 24 months post bottling.

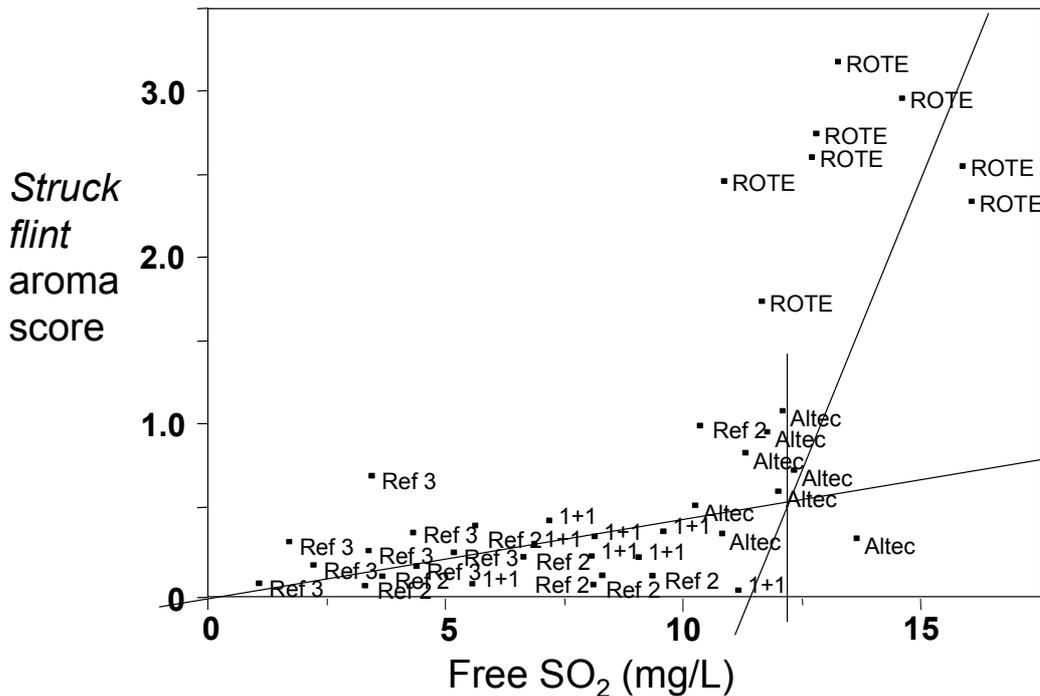


Figure 8. AWRI 'closure trial': Relationship between Free SO₂ concentration (individual bottles) and mean scores for struck flint (scale zero to 9) during sensory evaluation conducted 63 months post bottling (ROTE = roll-on tamper evident screwcap, Ref 2 = Reference 2 cork, Ref 3 = reference 3 cork, 1+1 = One plus One).

With regard to the filling height, no difference was found in the development of *reductive* character post-bottling at the two filling heights utilised in this trial. Additionally, on the last two AWRI *Advanced Wine Assessment Courses* the filling heights (adjusted to a wine temperature of 20°C) have been measured in all bottles sealed with screwcaps. No relationship between filling height and the participants' rating of the incidence or intensity of *reductive* character has been identified.

How might these observations be explained? It is probable that that all of the oxygen present in the headspace and dissolved in the wine at bottling is consumed by chemical reactions in the wine within days or weeks of bottling. Conversely, thiols might be formed over months or years. In the original closure trial, no *reductive* character was evident until 18 months post bottling. Thiols are readily oxidised, and consequently, if they are formed when either the oxygen introduced at bottling or oxygen permeating through the closure is available, then their concentration might not increase to a point above the sensory threshold. However, if thiols are formed after all of the oxygen introduced at bottling has been consumed, and in an environment where zero or only a small amount of oxygen is permeating through the closure, then their concentration might increase to a point above the sensory threshold. For wines with a propensity for this to happen, increased oxygen permeation through the closure, whether it is a screwcap or any other closure, would be advantageous. However, it should be reiterated that the formation of *reductive* character in these circumstances is not a fault of the closure, but rather a problem with the wine. It should also be noted that while such increased oxygen permeation might solve the reduction problem, it could also lead to other unwanted effects on wine development that would negate some of the reasons for using screwcaps or other low oxygen permeation closures in the first place. Additionally, the maximum ullage spaces in screwcapped bottles used for the 2004 *Advanced Wine Assessment Course* were noted to be substantially higher than in bottles used for the 2003 course. It is possible that some wine producers have made a decision to increase ullage space at bottling in order to lessen the risk of later 'reduction'. However, the authors feel that such a strategy is unlikely to be effective, and might only lead to premature wine development which, again, would negate some of the reasons for using screwcaps.

If the hypothesis is correct, one conclusion that can be drawn from these trials is that some oxygen does enter screwcapped wine, as was demonstrated by oxygen permeation (Mocon) testing of a small number of bottles from the original closure trial. It should be noted that this testing was conducted approximately three years after bottling, and that only a small number of samples were tested. The data should not, therefore, be considered as absolutely representative, but is considered useful in the context of a discussion of the development of *reductive* characters in bottled wine.

Table 4. Oxygen permeation (mL O₂ per day) of screwcap, Altec and Reference 2 cork closures from the AWRI closure trial, tested approximately 36 months post bottling

	Mean	Range
Screwcap (n=6)	0.0005	0.0002 - 0.0008
Altec (n=6)	0.0010	0.0007 - 0.0013
Reference 2 cork (n=12)	0.0179	0.0001 - 0.1227

The screwcaps allowed ingress of a mean of 0.0005 mL of oxygen per day, with a range from 0.0002 to 0.0008. The Altec closure, which had retained a similar concentration of SO₂ to the screwcap and also received the next highest ratings for *reductive* characters during sensory evaluation (notwithstanding the TCA taint in those samples), had the next lowest oxygen permeation, with a mean of 0.001 mL of oxygen per day and a range of 0.0007 to 0.0013. The reference 2 corks had a mean permeation of 0.0179 mL of oxygen per day with a range from 0.0001 to 0.1227, i.e. a 1227-fold range.

As with all closures, the amount of oxygen that enters screwcaps is measurable, and the rate of oxygen permeation of all closures is apparently linked to the formation and intensity of *reductive* characters in bottle. However, rather than simply increasing the rate of oxygen permeation of low permeation closures, what are the best strategies for avoiding the formation of *reductive* characters?

The most obvious way in which to avoid post-bottling 'reduction' is to minimise the production of thiols and their precursors, such as thio esters, during winemaking. In most wines, the majority of sulfides and thiol precursors are likely to be formed during fermentation, and the maximum concentration of these compounds is likely to be present at the end of fermentation. Thus, improved fermentation management is likely to be beneficial in minimising the propensity of a wine to later become *reductive*. More careful fermentation management, including optimising yeast culture preparation, avoiding temperature shock and ensuring an adequate supply of nutrients, including oxygen, should all be part of this strategy. A corollary of this is that wines that have suffered fermentation problems are more likely to become *reductive* if bottled with low oxygen permeation closures.

Secondly, conducting the bulk of any copper fining while wine is still on yeast lees is likely to minimise the concentration of residual copper in wine, because yeast cells have strong affinity to adsorb copper. Fining at this stage is also likely to remove the maximum concentration of thiols and thiol precursors (on the assumption that copper does react with thiol precursors), as this is the point at which the maximum concentration of these compounds is likely to be present. Viable yeast lees also have the ability to re-metabolise compounds containing chemically reduced forms of sulphur, and thus delaying the addition of SO₂ post fermentation *might* be useful in allowing this to occur.

A theory postulated to the authors by some Australian winemakers is that there is a finite 'sulfide (thiol and thiol precursor) pool' at the end of fermentation, and a relatively large copper addition at this stage will potentially remove a large proportion of this 'pool'. Over time, because the compounds responsible for *reductive* aroma are probably in complex equilibria, it is possible that the concentration of aroma active compounds will again increase to a concentration above the sensory threshold. Consequently, additional small copper additions might be necessary during wine maturation to again lower the concentration of these compounds to below the sensory threshold. By the time the wine is bottled, the aim is to ensure that the concentration of these compounds has been lowered to a point where further equilibrium shifts will not cause the concentration to again rise above the sensory threshold after bottling. Conversely, copper fining close to bottling is not considered ideal, especially if only temporary removal of *reductive* characters is achieved, and if repeated fining increases the

copper concentration in the wine, thereby increasing the risk of later copper instability. It should also be noted that with Sauvignon Blanc, Chardonnay, and other varieties, compounds containing chemically reduced forms of sulfur are important in varietal expression. Greater care with the timing and magnitude of copper additions should, therefore, be exercised when working with these varieties, and fining trials should be conducted.

Does wine require oxygen to age or develop?

A discussion of the need of some wines for oxygen to prevent the concentration of thiols rising above the sensory threshold leads to the commonly asked question of whether wine requires oxygen to *age* or *develop*. More recently, the question *does wine 'age' under screwcaps?* has also become common. In the authors' experience, what many of these the questioners are really asking is *does wine 'develop' in the same manner under screwcaps as it does under cork?*

With regard to the first question, *does wine require oxygen to age or develop?* the answer is probably no, as demonstrated by the Chardonnay wine sealed in glass ampoules and stored in an anaerobic environment, which displayed sensory characteristics typical of a wine of this type at four years of age. This supports the findings of Jean Ribéreau-Gayon in studies conducted as early as the 1930s, and reported by Ribéreau-Gayon et al. (1976). However, while this question of still of scientific interest, its relevance for commercial situations is diminished when it is understood that the entry of controlled but different amounts of oxygen will change the way in which wine develops in bottle, ie, 'different wines' can be created.

In answer to the second question, *does wine age or develop differently under screwcap?* In the authors' experience the inference from the questioner is often that the manner in which wines age under cork is optimal, and is the benchmark against which development under other closures should be judged. The answer to the question is that understanding the mechanisms of wine development post-bottling should enable wine producers using low oxygen permeation closures to replicate the manner in which wines develop under the best performing corks, consistently for every bottle. It is clear that wines can develop very differently under screwcaps when compared to other closures, but in most cases this is a positive thing, and not a negative. In the AWRI closure trials, and apparently in trials being conducted by many wine producers in many countries, wine development under screwcap is rapidly becoming the benchmark against which the performance of other closures needs to be judged.

When the original closure trial Semillon wine was bottled under fourteen different closures, fourteen different wines began to be created from that point onwards. This phenomenon has been observed with all of the wines used in the AWRI trials discussed in this paper, with the greatest difference being inferred on the Chardonnay wine sealed under screwcaps, cork and glass ampoules. Within two years of filling it was difficult for some of the tasters to believe that it could ever have been the same wine. Importantly, the wines used in the various trials have not only developed at different rates under different closures, but also in different ways. Early in the original closure trial it was clear that the wine under various closures was never going to reach the same 'end point'. That is to say that if it were possible, one could never have picked a single bottle sealed with each closure at different points in time, and compared them and found them to taste the same. Understanding and controlling the factors that lead to the wine under some closures developing in a manner that was preferred to the development under other closures, is the obvious direction in which closure and bottling technology will develop.

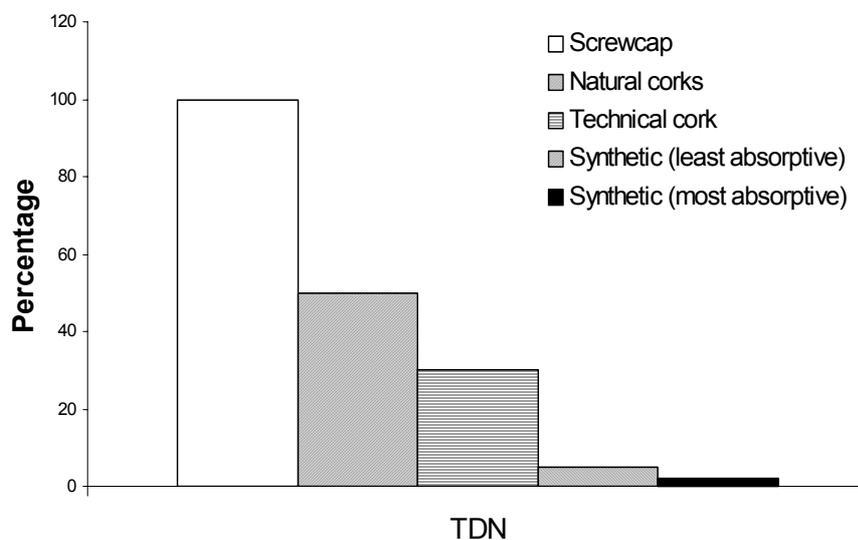
Flavour and aroma scalping

The variation seen in a wine sealed with different closures, or between bottles sealed with the same type of closure, is potentially caused by more than the degree of oxygen permeation. A separate AWRI trial on 'flavour scalping' demonstrates that different closures have the ability to remove certain compounds or groups of chemically related compounds, to a greater or lesser extent (Capone et. al. 2003, Institute publication #744). In this trial, the Semillon wine and a selection of closures from the original closure trial were used, and additional flavour compounds were added to the wine before

bottling. Synthetic corks were found, in general terms, to ‘scalp’ particular compounds to a greater extent than natural corks, which themselves scalped some compounds by up to 50%. However, screwcaps scalped none of the compounds studied. Thus, the way in which wine develops in bottle, and therefore tastes when the bottle is opened, might be profoundly affected by the degree of scalping that has taken place.

However, scalping might not in all cases be a negative, and in future it might be seen as a further tool available to wine producers to modify wine development in bottle in a controlled and reproducible manner. Figure 9 demonstrates the degree of scalping of the compound trimethyldihydronaphthalene (TDN) attributable to various closures. TDN is the compound primarily responsible for a character often described as kerosene-like in aged white wines, particularly those made from the varieties Riesling and Semillon. At certain concentrations, TDN is sometimes considered a positive attribute in aged white wines, but might be considered a negative attribute at high concentrations.

Figure 9. TDN remaining (%) after two years storage in a horizontal position



As demonstrated in Figure 9, all of the closures, except the screwcap, scalped TDN by between approximately 50% (natural cork) and approximately 98% (the most adsorptive synthetic cork). Thus, it is possible that wines sealed with screwcaps might develop an undesirably high concentration of TDN after time in bottle. However, if controlled amounts of polymers similar to those in the most adsorptive synthetic closure could be incorporated into the linings of screwcaps, in order to scalp, as selectively as possible, compounds such as TDN, then wine producers would potentially be able to control the development of this character in their aged wines. Thus, what may be termed ‘designer’ closures specifically manufactured to ensure the optimal development of particular types of wine, might one day be available.

Conclusion

Recognition that one starts to create different wines from the moment a wine is sealed under different closures is one of the most important outcomes from the various closure trials conducted at the AWRI, because the implications of this proposition open up all sorts of exciting possibilities for the future of closing the wine bottle.

In future, the closure and many other bottling variables may be seen to be part of the winemaking process, because the modification in wine aroma and flavour that can be attributed to these variables can be profound, and can apparently be of greater magnitude than those derived from many vineyard or winemaking variables. A full understanding of the mechanisms of wine modification induced by

closures and other variables will allow wine producers to manage such modification to their, and to their consumers' advantage.

The first step, which is already close to commercial reality with screwcaps and some technical corks, is the availability of branded closures with a choice of oxygen permeabilities, and it is apparent that the screwcaps currently offered by different suppliers vary in their oxygen permeability. As the realisation grows of the possible changes in bottled wine that can be induced by allowing low and controlled rates of oxygen permeation, the application of this technology is likely to be as rapid as the uptake of alternative closures themselves.

However, the type of closure used and the oxygen permeability are just two variables that are likely to have an important effect on wine development in bottle. As further variables are examined the science and technology of wine bottling will inevitably become more complex, leading to ever-tighter specifications for closures, bottling procedures, and possibly bottles. This situation might present some wine producers with greater challenges than they face when using traditional closures, and these producers should take a cautious approach to the adoption of new technology, and conduct their own trials.

The use of screwcaps in Australia, New Zealand, and in other parts of the world, and the research that has supported their uptake, has opened a window to the understanding of changes that occur in wine after it is bottled. Elucidation of important variables has begun, and is likely to accelerate. Wine producers are already defining the bottling conditions for different wines, in order for those wines to be in optimal condition when presented to the consumer. The potential market advantage to be gained by producers understanding and successfully applying such technology, cannot be overstated.

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