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

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Abstract

An important outcome of the AWRI's research on wine closures is the recognition that when a wine is bottled under different closures, different wines begin to be created from that point onwards. Other workers have apparently expanded this concept to other bottling variables such as the filling height, the concentration of Free SO_2 at bottling, and the mixture of gases in the headspace of bottles post filling. The ability to link such variables to wine development post bottling creates the possibility of reliably predicting, and therefore optimising, wine development in bottle. This would enable wine producers to consistently offer wines to the market in optimal condition. As understanding of these factors increases, ever-tighter specifications for both closures and bottles may be set in order to minimise variation in wine development between bottles. The gap between reliable closure performance and wine producers' expectations of the manner in which closures need to perform, is smallest with manufactured closures. In particular, screwcaps have the proven ability to deliver the desired homogeneity. Technical corks, some synthetic closures, and novel closure technologies such as membrane barriers to oxygen and TCA for natural or synthetic corks, might all be able to deliver the tighter specifications demanded by wine producers, and the development of such technologies will inevitably continue. Understanding of the impact of the closure on wine development has been elucidated by the AWRI's various closure trials. The original trial commenced in May 1999 with the bottling of a Semillon wine under 14 different closures. Results of testing conducted at 60 and 63 months post bottling for five closures (roll-on tamper evident 'ROTE' or 'screwcap' closures, Altec and One + One technical corks, and 'reference 2' and 'reference 3' natural wine corks) showed that the ROTE and the Altec closures continued to retain a significantly higher concentration of SO_2 in the wine compared to the One + One and reference 2 cork closures, which in turn retained a significantly higher concentration of SO_2 in the wine compared to the reference 3 cork closures. Higher SO_2 concentration continued to show a strong negative correlation with optical density at 420 nanometers for all closures. Likewise, during sensory evaluation, ratings for *overall fruit aroma* and *citrus* aroma were strongly positively correlated with SO_2 concentration, and were negatively correlated with ratings for *oxidised*. Ratings for *struck flint* and *rubber* aroma were also, to a lesser degree, positively correlated with SO_2 concentration. In a second trial, a Semillon wine was bottled under screw caps with both a 'high' and a relatively 'low' concentration of SO_2, and at two filling heights. Sensory evaluation of the wine

conducted two years post bottling showed no relationship between increased SO_2 concentration and ratings for *H_2S/cabbagey* aroma, and an inverse relationship between increased SO_2 concentration and ratings for *struck flint*/*rubber* aroma. However, it is considered possible that the elevated concentration of free SO_2 in the 'high SO_2' treatments (42 mg/L for the 'high fill height', and 39 mg/L for the 'low fill height' treatments respectively) might have interfered with the assessors' ability to evaluate the wines. Nevertheless, these data, and those from the first trial, suggest no causal relationship between increasing SO_2 concentrations and increasing ratings for 'reductive' characters during sensory evaluation. Additionally, in this second trial the varied ullage at the time of filling had no influence on ratings for *flint/rubber* and *H_2S/cabbagey* during sensory evaluation conducted two years post bottling, at either SO_2 concentration. In a third trial a Chardonnay wine was bottled with screwcaps, and a portion of the wine was also sealed in glass ampoules in the absence of oxygen. Four years post-filling, the wine sealed by both methods received the same rating for the attribute *oxidised* during sensory evaluation, but the wine sealed in ampoules was rated significantly higher for the attribute *reduced*. Wine under both treatments had retained a similar concentration of SO_2.

The currently available stocks of some of the closures examined, and, therefore, their performance, might differ from those available when these trials commenced. Similarly, the closures have been used to seal only the wine types defined under the conditions described, and care should, therefore, be exercised when relating the results reported here to other wine types, or to wines stored in less than industry best practice storage conditions. Full details of the wine, the bottling procedures and storage conditions are contained in the Australian Journal of Grape and Wine Research *7 (2), 62-105.*

Any reference to 'Tage' closures in this presentation or in the publications referred to, are references to closures manufactured by APM in the USA, and not by Novembal in Europe. The closures were obtained from the Australasian agent, Esvin Wine Resources. The AWRI takes no position on the rights of APM or Esvin to manufacture or sell closures under the name 'Tage'.

Introduction

A key aspect of the success of the Australian wine industry, and of other non-traditional wine producing countries, has been a focus on the consumer and on delivering wine to them in optimal condition. Those employed in the Australian wine industry are working to ensure that this success continues. The recent and continuing shifts in the demographics of wine production and consumption around the world are resulting in increased market pressures for producers in many countries, but the future of those producers is to a large extent in their own hands. To paraphrase Alan Kay, the *inventor* of personal computing, *the best way for them to predict their future is to invent it.*

The Australian wine industry set itself a 25-year strategy to invent its own future, and that strategy document *Strategy 2025* (Winemakers Federation of Australia 1996) states that Australia will become the world's most influential producer of branded wine within that 25-year period. The industry is confident of achieving that objective. Those wine production regions that are facing a crisis of falling sales also require a strategic approach to address their problems, and a cornerstone of any such strategy *must* be to deliver wines to the market in the best possible condition. Packaging wine in a haphazard and unreliable manner would doom any such strategy to failure.

For those wine producing countries that are facing increased market pressures, the need to adopt modern bottling and packaging technologies is the most compelling and yet in many cases there is little apparent sign of it happening. Indeed, laws in some countries impede the adoption of such technology. For instance, some laws effectively rule-out the adoption of screwcap closures by denying wines bottled in such a manner the 'appellation' status to which they would otherwise have been entitled. Additionally, unlike in Australia, various regulations compel many producers to bottle and label wine at the site where it was made, in order to qualify for appellation or quality statements on their labels. This has the effect of condemning many otherwise sound wines to being poorly prepared for bottling, and to being packaged using antiquated equipment by people whose primary expertise is not wine bottling. In contrast, in Australia and New Zealand contract bottling by experts is common, and producers utilise the best equipment available which is operated by world-experts in the field. It is considered likely that this is contributing to the objective decisions that are being made by a growing number of wine consumers around the world, to purchase these bright, fresh, well-prepared and well-packaged wines. Meanwhile, in Australia, New Zealand and elsewhere, knowledge of how to optimise wine development post bottling is growing, and is being widely and rapidly adopted.

The subject of wine development post-bottling is complex, and a large number of variables are involved which are interlinked in complex relationships. A complete understanding of the manner in which wine develops in bottle might be the 'Holy Grail' of wine research for many, as it is a key aspect of delivering wines to consumers in optimal condition. Gradual elucidation of some of the important factors indicates that in future it might be possible to reliably predict, and thereby optimise, wine development in bottle, and for the first time this might allow wine producers to be confident that they are offering their consumers wines in the best possible condition. As understanding of this subject grows, what might in future be achievable

when closing the wine bottle is profound and exciting, and the potential market advantage to be gained by those who understand and apply such technology, cannot be overstated.

This paper, therefore, attempts to link together the research projects of several teams at The Australian Wine Research Institute (AWRI), which are providing insights related to wine development post-bottling. This work goes beyond looking at the performance of different closures. While the closure is perhaps the most obvious variable that might influence wine development in bottle, it is only one factor. However, many of the other variables discussed are based on the premise of using closures that have lower and more consistent oxygen permeation than do traditional closures. The authors believe that in future most wine producers will be using closures that have lower and more consistent oxygen permeation than the closures that they currently use, and all producers should, therefore, already be defining the specifications of closure performance that they require, and be demanding closures that deliver to those specifications. The sooner that all producers the world-over are demanding the same thing, the sooner that a range of closures that perform to those specifications will become available.

The AWRI closure trial

All producers strive to optimise the quality of their grapes in the vineyard, and to maintain and enhance that quality through winemaking. In spite of this, gross quality loss as a result of packaging routinely occurs and is apparently accepted by many in the world's wine industry. The starting point of the AWRI's work on wine packaging was to avoid this quality loss, by:

- helping to facilitate greater choice and greater reliability of closures and of other packaging materials;
- developing an understanding of the mechanisms by which quality is lost; and
- developing strategies to avoid that quality loss.

The stated objective of the original closure trial in 1998 was simply, *'To facilitate greater choice and greater reliability of closures'*.

The AWRI maintains a very positive relationship with stakeholders in the closure debate and senior technical personnel from the cork industry presented an excellent technical workshop at the *12th Australian Wine Industry Technical Conference*, which was staged in Melbourne in July 2004. While encouraging information was presented at that workshop, particularly relating to technical corks, the fact remains that the variability and tainting of wine that too often derives from traditional closures is inconsistent with the objective of confidently presenting wines to consumers in optimal condition. Much of our current understanding of wine development in bottle has only been gained by using screwcaps, and it is clear that the gap between our desired specifications of closure performance, and what closures are currently able to deliver, is smaller with screwcaps than with other closures.

While on a purely objective basis it can be stated that screwcaps are currently the closure which most closely offers the possibility of offering wine to our consumers in optimal condition, it is inevitable that in future other closures that perform to the same specifications will become available. Technical corks are developing rapidly, and the results of trials conducted at the AWRI with prototypes of some novel technologies that are now commercially available, such as membranes for corks or synthetic corks that apparently lower oxygen permeation and act as a barrier to TCA, are very promising. For many applications, synthetic cork is already a well entrenched and accepted closure, as evidenced by the volume currently used, and the development of synthetic cork technology is sure to continue.

The promotion of the AWRI's trial results, particularly the success of screwcaps, remains part of the AWRI's strategy to achieve its stated objective of facilitating greater reliability and choice of closures, and provides all those involved in producing closures with a reference point.

Perhaps the most important outcome of the closure research at the AWRI is the postulate that one begins a process of creating different wines from the moment a wine is sealed with different closures. Accepting this proposition creates many exciting possibilities for the future of closing the wine bottle. More recently, other workers from Australian and New Zealand wine production and contract bottling companies have apparently expanded this concept of 'creating different wines' to other bottling variables. The *1st International Screwcap Closure Symposium* was held in New Zealand in November 2004, and papers and tastings were presented that supported this concept. It is apparent for instance, that bottling a wine under different commercially available screwcaps, each with slightly different oxygen permeation rates, might also result in 'different wines' being created. Further, tastings at the symposium indicate that in a Sauvignon Blanc wine, changing bottling variables such as the filling height by 5 mm increments or free SO_2 concentration by 5 mg/L increments, both created 'different wines' after time in bottle. The differences between the 'wines' were obvious to the first author, and were considered to be strongly related to the treatments that had been applied.

Figure 1 presents a graphical representation of the changes to the aroma profile of the Semillon wine used in the original AWRI closure trial 36 months after bottling, which can be attributed to the closures. This type of sensory evaluation is now being routinely conducted in the Australian wine industry by researchers, closure suppliers, wine producers and contract bottling companies, and demonstrates that the 'differences between wines' can be objectively quantified. To understand fully the significance of this concept, one also needs to appreciate that the 'differences' can be profound, and when tasting wines from the AWRI trials it can be difficult to come to terms with the fact that the 'wines' were once the same wine. In many cases it is apparent that the differences are of greater magnitude than those that might be attributable to many vineyard and winemaking variables.

These concepts have important implications. An understanding of the factors that determine the way in which wine develops in bottle will allow producers to manage them. Consequently, for the first time it might be possible to predict and influence the manner in which wine develops in bottle in a reproducible manner and the winemaker's role will thus continue after wine is bottled. This will enable wine

producers to present wines to consumers that display the purest possible expression of their 'terroir', which in many situations is not the case at present.

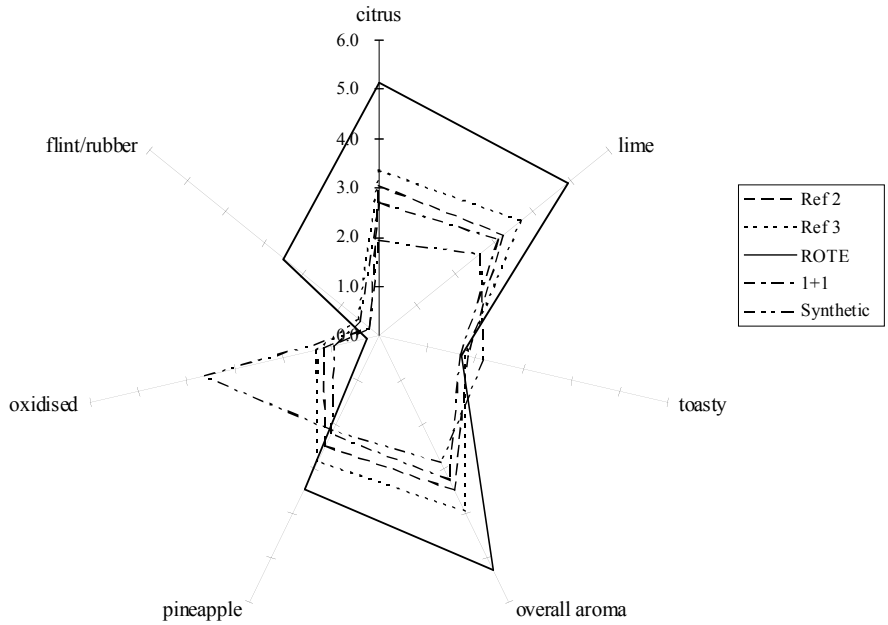

Figure 1. 'Spider plot' of descriptive sensory analysis of the AWRI closure trial Semillon wine conducted 36 months post bottling for selected attributes, rated on a scale of zero to nine.

Wine producers and contract bottlers in Australia and New Zealand are already defining the optimal bottling conditions for various wines, based on assessing the 'optimal' shelf life or cellaring potential of each wine. It is clear that those conditions, including the screwcap used, might be quite different for different wines and even for wines made from the same grape variety. For instance, it might be that for Riesling *x* with an optimal shelf life of 18 months, those conditions will be different than for Riesling *y* with an optimal cellaring potential of five years, which in turn might be different to Riesling *z* with an optimal cellaring potential of ten years. Once a full understanding of wine development in bottle is achieved, producers will have the ability to offer their products to consumers in optimal or 'perfect' condition. This will be a powerful technology that has the potential to assist producers to increase their market penetration and market share.

It is to Australia's advantage, therefore, that so many wine producers in other parts of the world are apparently reluctant to also adopt alternative closure technologies and that regulations in some countries effectively impede the adoption of such technology. However, it must be considered likely that with time, other countries will also seize upon the potential power of this technology and also make the shift to more reliable

closures. Indeed, it will be necessary for them to do so if producers in those countries are to be confident that they are presenting their wines to consumers in the condition that their winemakers intended, and also be in a position to defend their definitions of 'terroir'. Clearly, therefore, in the belief that producers the world over share a collective goal, the authors believe that the goal will be achieved more quickly if all producers adopt the technology together. The critical mass supplied by the traditional wine producing countries would ensure that closure and bottle manufacturers would be able to improve continually their products in response to the ever tighter specifications that are likely to be set.

Results of the initial AWRI closure trial up to 63 months post bottling

The original AWRI 'closure trial' commenced in May 1999, when a dry white Semillon wine was bottled under 14 different closures. The composition of the wine pre and post bottling is presented in Table 1.

Table 1. Wine composition at the time of bottling

Compositional variable	Value
Measures made immediately before bottling[a]	
Tartaric acid	3.8 g/L
Citric acid	0.1 g/L
L - malic acid	1.2 g/L
Lactic acid	0.1 g/L
Acetic acid	0.5 g/L
Glucose plus fructose	0.3 g/L
Laccase activity	Not detected
Pink colour	Not detected
Pinking susceptibility	4 au[b] x 10^3
Pinking precursor content	58 au x 10^3
Specific gravity	0.9929
Turbidity	0.17 NTU [c]
2,4,6–trichloroanisole, 2,3,4,6-tetrachloroansole, pentachloroanisole, 2,6-dichloranisole, 2,4-dichloranisole	None detected
Measures made after bottling[d]	
pH	3.1
Alcoholic strength	11.1 % v/v
Titratable acidity (at pH 8.2)	6.2 g/L as tartaric acid
Volatile acidity	0.58 g/L as acetic acid
Free SO_2	30 mg/L
Total SO_2	95 mg/L
OD_{420}	0.112 au[b]
Dissolved carbon dioxide	0.5 g/L
Ascorbic acid	42 mg/L

Adapted from Godden et al. 2001
[a]analyses carried out on a tank sample
[b]absorbance units
[c]nephelometer turbidity units
[d]analyses made on bottled wine within 48 hours of bottling (mean, n=14 closures x 12 bottle replicates)

Fourteen different closures were included in the trial, and details of the closures and their manufacturers/suppliers are provided in Table 2. It should be noted that the trial commenced before *Sabate Altec* and *Amorim Twintop* closures treated by the *Diam/ Diamond* or *ROSA* processes respectively, became available.

Table 2. The closures studied and their source

Closure name	Type of closure	Source
Aegis	Synthetic, moulded	Southcorp Packaging, Melbourne, Victoria
Altec (Not Diam/ Diamond)	Technical cork	Sabate USA, San Francisco, USA
Auscork	Synthetic, moulded	J. B. Macmahon Pty Ltd, Forestville, South Australia
Betacorque	Synthetic, moulded	Betacorque Limited, Blackwater, United Kingdom
ECORC	Synthetic, extruded	ECORC A.S., Oslo, Norway
Integra	Synthetic, moulded	Anthony Smith Australasia Pty Ltd, Regency Park, South Australia
Nomacorc	Synthetic, extruded	Newpak Australia Pty Ltd, Wingfield, South Australia
NuKorc	Synthetic, extruded	NuKorc Pty Ltd, Wingfield, South Australia
One + One 'Twintop' (Not ROSA)	Technical cork	Amorim Cork Australia Pty Ltd, Dandenong South, Victoria
Reference 2, 44 mm cork	Natural cork	Random sample of stock held by a major Australian wine producer
Reference 3, 38 mm cork	Natural cork	Random sample of stock held by a major Australian wine producer
ROTE (Screwcap) Auscap with aluminium liner	Screwcap	Auscap, Braybrook, Victoria
Supremecorq	Synthetic, moulded	Random sample of stock held by a major Australian wine producer
Californian 'Tage'	Synthetic, moulded	Esvin Wine Resources, Auckland, New Zealand

Both of the batches of natural cork were selected as random samples taken from stocks held by two large Australian wine companies. Each batch had been obtained by the wineries from leading Australian cork supply companies, which in turn had sourced the cork, as far as could be ascertained, from leading suppliers in Portugal. The corks had been hydrogen peroxide bleached, and any coating material or printing had been applied in Australia. Four large Australian wine companies independently graded each batch using their own assessment procedures. All four companies assessed each batch as being representative of the grades described by the respective cork suppliers.

The contract bottler of the trial supplied the screwcap closures, and of the remaining closures, all except Supremecorq were supplied directly by the manufacturers or agents of each closure, by invitation. Supreme Corq Inc. declined to participate in the trial, and accordingly did not provide a sample of its closures. As a consequence, the Supremecorq closures were taken as a random sample of stocks held by a major Australian wine producer. ECORC supplied closures, but subsequently indicated that it wished to withdraw from the trial.

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All of the closures were used according to the specifications recommended by the suppliers of the closures. These were obtained from the published specifications where available, or from special instructions supplied by the manufacturers or suppliers of particular closures. Representatives of all but two of the closure suppliers, and of the cork industry, were present at the bottling.

The first results were published in the *Australian Journal of Grape and Wine Research* in July 2001 (Godden et al. 2001, Institute publication No. 666). Subsequent publications have updated the results as the trial has progressed, and are listed at the end of this paper.

Perhaps as an indication of the interest in, and importance of issues related to wine closures, results of the trial have been widely reported. Media monitoring conducted during the 18 months following the first publication of results revealed at least 220 articles in the print and electronic media, published in nine countries and in six languages, which directly referred to the trial. A search of the Internet using the Google search engine performed in December 2002 revealed hundreds of references to the trial.

Much of the media interest focussed on the performance of the screwcaps. It was no surprise that the screwcap closures performed so well in the trial, as the efficacy of these closures had been demonstrated in work conducted at the AWRI in the 1970s (Eric et al. 1976, (Institute publication #139), Rankine et al. 1980). Perhaps this focus on screwcaps was in part due to the coincidence of timing of the release of the results complementing the promotion of screwcaps by some producers of Riesling wines in the Clare Valley of South Australia and also the fledgling *Screwcap Initiative* in New Zealand, on which the press had already run stories. The authors consider that the results helped to add objectivity to the media debate, and an apparent change in the nature of closures reporting occurred post July 2001, with greater objectivity and science, and less subjectivity and emotion.

The trial continues, with wine sealed with five of the original fourteen closures (Altec, One plus One, Reference 2 and Reference 3 cork and screwcap) being examined from 2003 onwards.

Figure 2 plots the loss of free sulfur dioxide (SO_2) in wine sealed with each of the closures up to 63 months post-bottling. The screwcap and Altec closures continue to retain significantly higher concentrations of SO_2 than the reference 2 and One + One closures, which in turn retain significantly higher concentrations of SO_2 than the reference 3 corks. Two of the synthetic closures, ECORC and Californian Tage, performed similarly, and retained less free SO_2 than the other commercially available synthetic closures. The remaining synthetic closures all performed very similarly. It should be noted that the trial commenced six years ago, and it is considered probable that, in general, the synthetic corks would retain greater SO_2 today than did the versions available when the trial commenced.

With minor exceptions, early trends in SO_2 data have become more pronounced over time. This strongly implies that the 1 and 2 mg/L differences in SO_2 concentrations between wine sealed with different closures recorded at 6 months post-bottling were real differences, which were of later oenological and possibly commercial importance.

Thus the six months post-bottling SO_2 data proved to be strongly predictive of data recorded 24 months post-bottling for the variables SO_2 concentration (R^2 = 0.89), optical density at 420 nm (OD_{420}) (R^2 = 0.90) and, to a lesser degree, ratings for *oxidised* during sensory evaluation (R^2 = 0.75), in wine sealed with each of the closures (means of 12 bottles, Betacorque excluded).

Throughout the trial, these three variables have strongly correlated for all of the closures. Figure 3 presents data for OD_{420} recorded from the same 12 intact bottles of wine sealed with each of the closures, over time. These data have been obtained using a modified spectrophotometer and novel analytical method developed at the AWRI (Skouroumounis et al. 2003, Institute publication number No. 731).

Wine sealed with the screwcap, being the closure that had retained the highest free SO_2 concentration, maintained substantially lower OD_{420} than wine sealed with the One + One and reference 2 corks. Similarly, ECORC and Californian Tage, two closures that had similarly low SO_2 retention up to 36 months post-bottling, had almost identical ratings for OD_{420}.

The ability to quantify wine development in unopened bottles is a powerful technology to aid our understanding of the subject. Not only is this method being used as a research tool, commercial companies have also used it to sort batches of wine that demonstrate sporadic or random colour development between bottles. Additionally, further work at the AWRI in the field of NIR spectroscopy has also demonstrated the possibility of estimating many other wine components in unopened bottles, possibly including SO_2 concentration.

As with earlier testing, various sensory attributes continue to correlate strongly at 63 months with both SO_2 concentration and OD_{420} values, with higher ratings for *overall fruit* and *citrus* aroma correlating with higher SO_2 concentrations and lower OD_{420} values, and lower SO_2 concentrations and higher OD_{420} values correlating strongly with *oxidised* aroma.

Figure 4 demonstrates that the relationship between ratings for *citrus* (and for *overall fruit*, data not shown) and SO_2 concentrations, continues to be linear at 63 months post-bottling, except for the individual bottles that contained the highest concentrations of TCA, and for the screwcap bottle that was considered to exhibit the strongest *reduced* character. It is perhaps noteworthy that these relationships appear to have remained near-linear even in the range of what might be considered sub-optimal free SO_2 concentrations of less than 5 mg/L; perhaps again indicating that small differences in SO_2 concentration might be of oenological importance.

Notwithstanding greater SO_2 retention, lower colour development and superior sensory ratings, wine sealed with the screwcaps has been rated higher for the characters *rubber* and *struck flint* from 18 months post-bottling through to the testing conducted 63 months post-bottling. The potential for wines with a propensity for 'reduction' to develop such characters if sealed with low oxygen permeation closures is an important issue, and is discussed in greater detail below. This is primarily a winemaking issue, and 'reduction' is not *caused* by the closure. While in some situations, all other things being equal, increased oxygen permeability of screwcaps or of other low permeation closures might diminish or avoid the development of

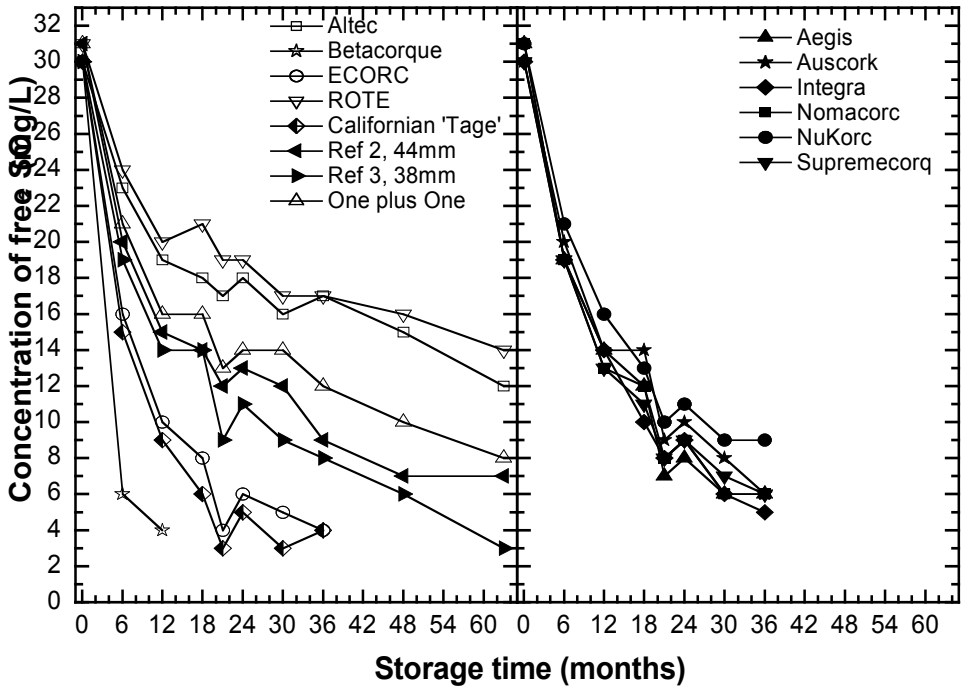

Figure 2. Mean concentration (n=12) of free SO_2 in wine sealed with each of the closures over time for bottles stored inverted.

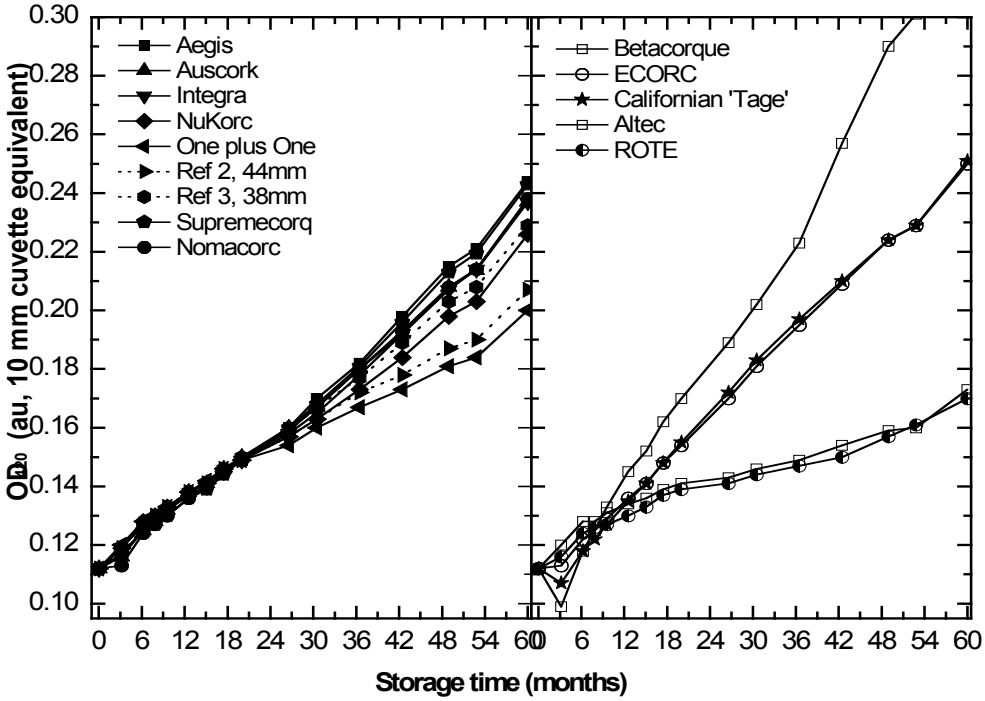

Figure 3. Mean (n=12) optical density at 420 nm in wine sealed with each of the closures over time for bottles stored inverted.

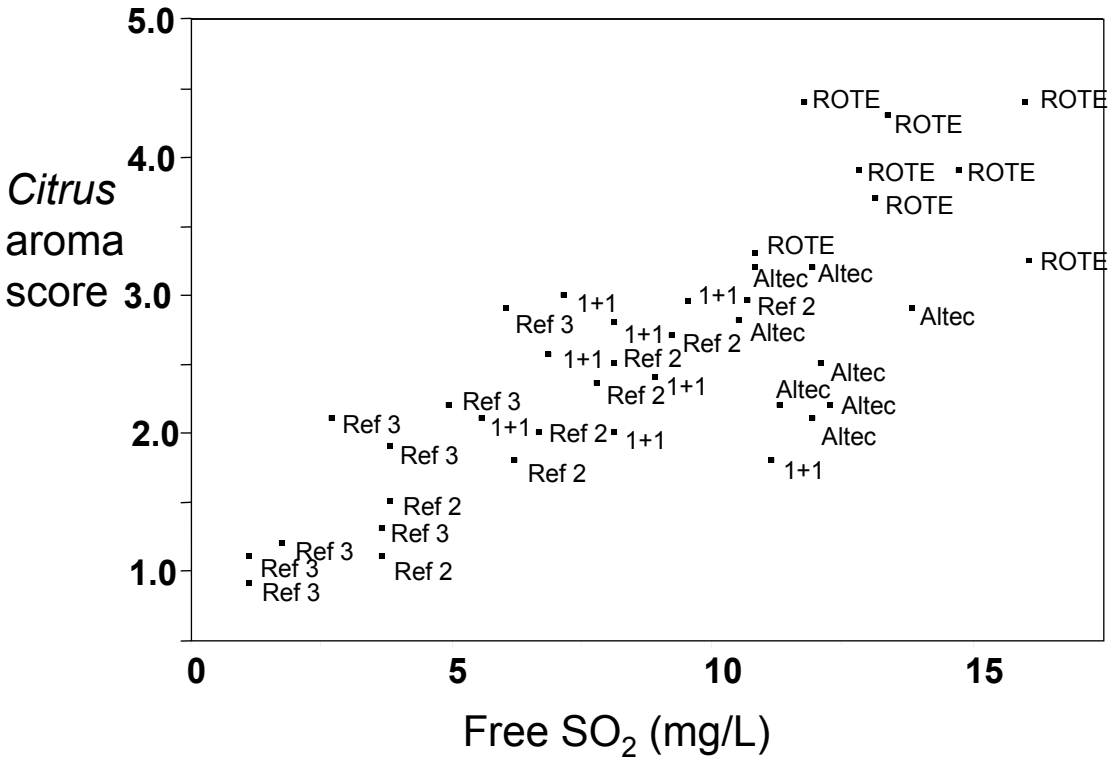

Figure 4. Relationship between Free SO_2 concentration (individual bottles) and mean scores for *citrus* (scale of zero to 9) during sensory evaluation conducted 63 months post bottling (ROTE = roll-on tamper evident screwcap, Ref 2 = Reference 2 cork, Ref 3 = reference 3 cork, 1+1 = One plus One).

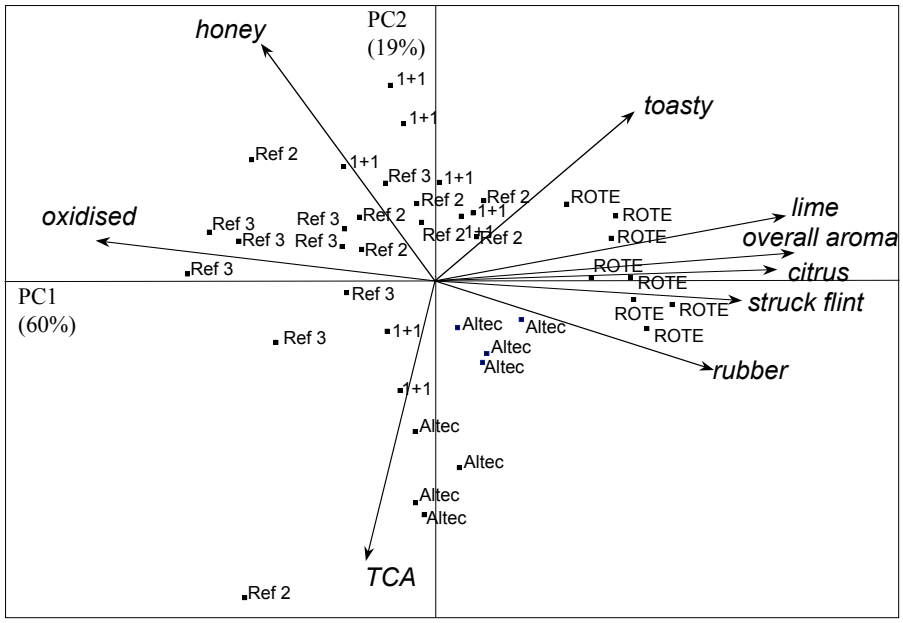

Figure 5. Biplot of principal components 1 and 2 for scores of sensory descriptive analysis of individual bottles, assessed at 63 months post bottling (ROTE = roll-on tamper evident screwcap, Ref 2 = Reference 2 cork, Ref 3 = reference 3 cork, 1+1 = One plus One).

reductive characters post-bottling, the authors do not consider that this hypothesis should be used as an argument for changing the permeability of low permeation closures. Rather, producers contemplating the adoption of low permeation closures should first consider if the winemaking techniques that they adopt lead to wines that are prone to 'reduction', and whether they are prepared to modify their winemaking to avoid this potential problem.

Figure 5 presents data from sensory analysis conducted at 63 months post-bottling, for eight individual bottles of each of the five closures tested. Perhaps the most notable factor is the relatively tight grouping of the screwcap bottles compared to bottles sealed with the cork and, to a lesser extent, the technical cork closures. Additionally, the difference between the screwcap and other closures for the intensity of fruit characters appears to be increasing over time, compared with similar analysis conducted earlier in the trial (data not shown). It is also apparent, for instance, that despite very similar SO_2 concentrations wine sealed with the reference 2 and One + One closures are somewhat different in their *honey* and *toasty* characters, further illustrating the concept of creating 'different wines'.

Despite the Altec closure continuing to retain a high SO_2 concentration and low OD_{420}, virtually every bottle tested in the trial has been affected by TCA taint in a range of concentrations between approximately 1 and 1.5 ng/L. These concentrations were found to suppress the ratings of positive fruit attributes in the Semillon wine by approximately 40%, suggesting that concentrations of TCA as low as 1 ng/L have the potential to negatively impact on the consumer's enjoyment of wine. However, it should be noted that in a similar trial bottled in September 2002, no TCA has been detected in wine sealed with prototype Altec *Diamond*/*Diam* closures in the first two years following bottling (data not shown).

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 SO_2 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 SO_2 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 SO_2 concentrations. Details of the four treatments are provided in Table 3.

Table 3. Experimental design–trial of the effect of filling height and SO_2 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 SO_2 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 SO_2 concentration and the intensity of *reductive* character, when bottles sealed with all of the closures in the trial with the standard SO_2 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 SO_2 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 SO_2 concentration at which the intensity of struck flint character increased exponentially being approximately 12 mg/L. This SO_2 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 SO_2 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 SO_2 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.

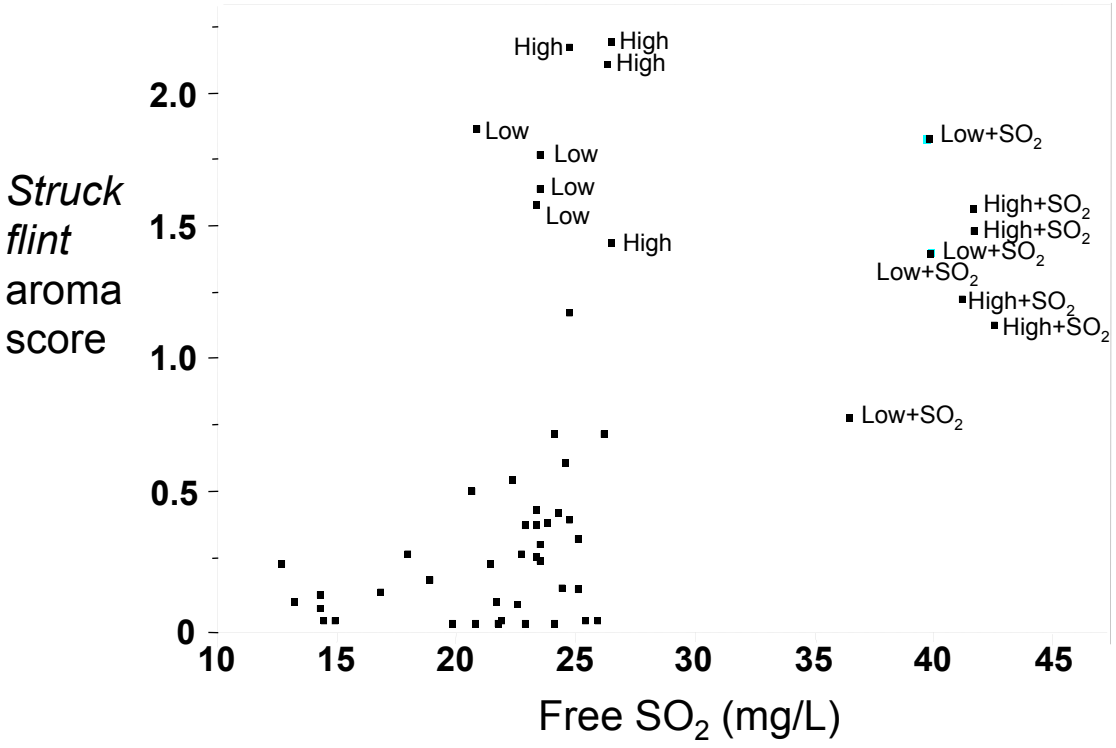

Figure 6. AWRI 'commercial closure trial': Relationship between Free SO_2 concentration (individual bottles) and mean scores for *struck flint* (scale zero to 9) during sensory evaluation conducted 24 months post bottling.

The lower the rate of oxygen ingress, all other things being equal, the lower will be the rate of SO_2 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 SO_2 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.

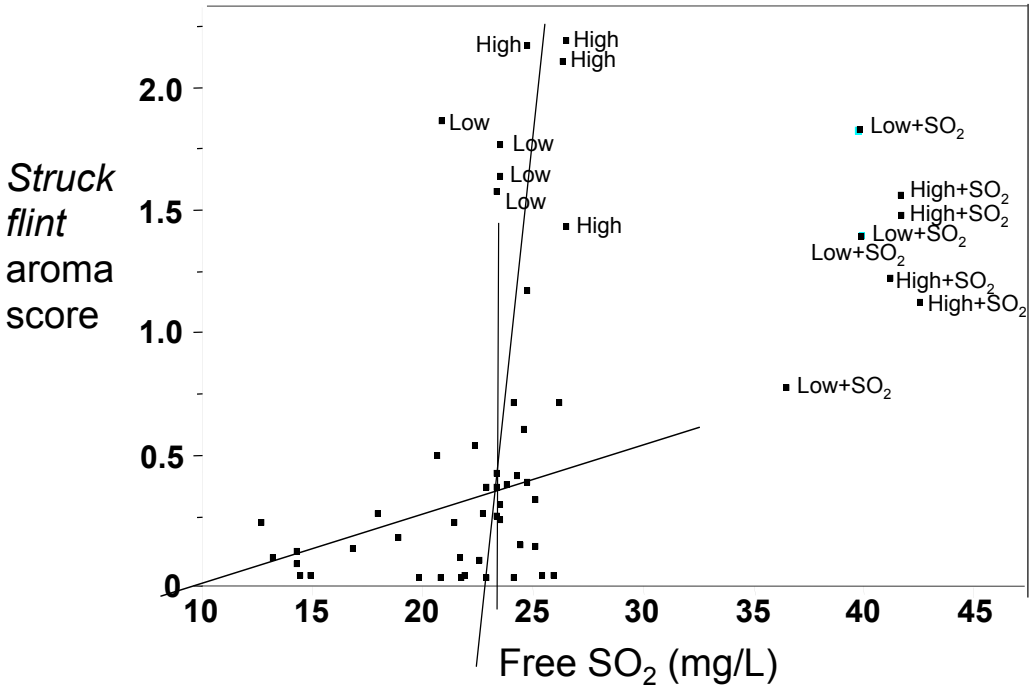

Figure 7. AWRI 'commercial closure trial': Relationship between Free SO_2 concentration (individual bottles) and mean scores for *struck flint* (scale zero to 9) during sensory evaluation, conducted 24 months post bottling.

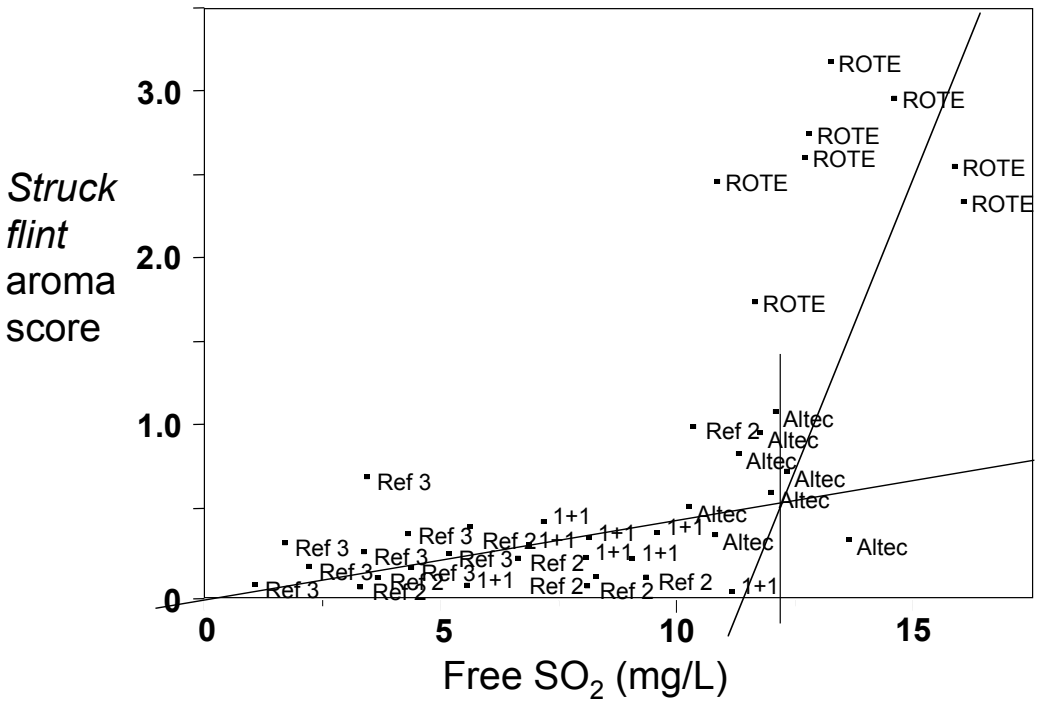

Figure 8. AWRI 'closure trial': Relationship between Free SO_2 concentration (individual bottles) and mean scores for *struck flint* (scale zero to 9) during sensory evaluation conducted 63 months post bottling (ROTE = roll-on tamper evident screwcap, Ref 2 = Reference 2 cork, Ref 3 = reference 3 cork, 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 O_2 per day) of screwcap, Altec and Reference 2 cork closures from the AWRI closure trail, 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 SO_2 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 SO_2 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 14 different closures, 14 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 No. 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.

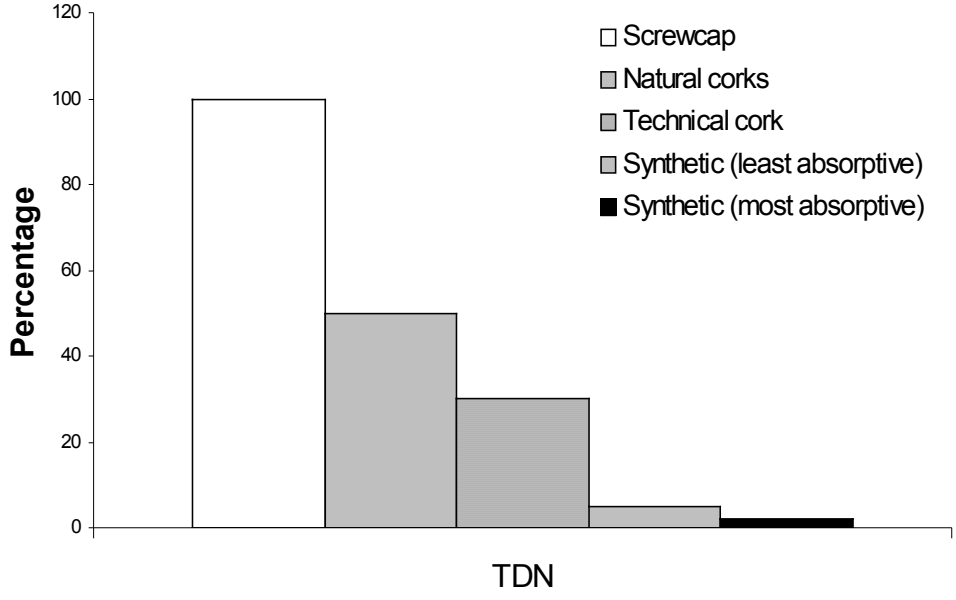

Figure 9. TDN remaining (%) after two years storage in a horizontal position

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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