The impact of closure type and storage conditions on the composition, colour and flavour properties of a Riesling and a wooded Chardonnay wine during five years' storage

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Abstract

This paper presents the results from an investigation to assess the development of a Riesling and a wooded Chardonnay wine over five years following the imposition of several treatments at bottling. The wines were bottled under a screw cap closure, two different natural corks, a synthetic closure and in a glass ampoule. In addition, the effect of storage orientation was investigated. The bottled wines were stored under controlled temperature and humidity conditions. Various analyses were carried out on replicate bottles from each treatment, including sulfur dioxide and ascorbic acid concentration, sensory analysis of appearance and aroma attributes, and spectral measures. The largest treatment effect resided with the nature of the closure. Wines sealed with the synthetic closure were relatively oxidised in aroma, brown in colour, and low in sulfur dioxide compared to wines held under the other closures. A *struck flint/rubber* (*reduced*) aroma was discernible in the wines sealed under the screw caps or in glass ampoules. Wines sealed under natural bark corks in this study showed negligible *reduced* characters. The bottle orientation during storage under the conditions of this study had little effect on the composition and sensory properties of the wines examined.

Keywords: *browning, white wine, aroma, taste, cork, screw cap, ROTE, spoilage, development, ageing, sulfur dioxide, CIELAB, oxygen ingress, oxygen permeation, oxygen diffusion*

Introduction

The flavours of wines change as they age from those that are fruit-derived and fermentation-derived to *developed* characters. These *developed* characters often relate to the grape variety/ies from which the wine was made and continue to evolve over time. They are largely the result of various acid-catalysed reactions occurring in the wine during ageing. Oxidative spoilage of bottled wine differs from ageing by being primarily the result of reactions promoted by oxygen and it results in colour, aroma and taste changes different from those produced during ageing. As an example, oxidised Riesling wine is commonly described as having lost its *floral* and *citrus* notes, having acquired *nutty*, *sherry-like* and *cooked vegetable* notes, and becoming *brown* in colour, whereas aged Riesling has also lost the *floral* and *fresh citrus* notes but has acquired typical bottle age characters such as *toastiness* and *cooked citrus*, and developed a *golden* colour (Iland and Gago 1995). It is therefore the goal of wine producers to ensure that the conditions of wine bottle maturation and storage favour wine development rather than wine oxidation.

The primary compounds involved in wine oxidation are oxygen (the initiator of the process), phenolic compounds (the main oxidisable substrates that are presumably the precursor to the pigments formed during browning) and metal ions such as Fe^{+++}, Cu^{++} and Mn^{++} (catalysts). Other factors such as temperature, pH and light also have an effect (Silva Ferreira et al. 2002, Macias et al. 2001). Two factors likely to be mainly responsible for the levels of dissolved oxygen in bottled wines, and potentially under the control of the producer, are the oxygen levels in the headspace at bottling and the rate of ingress of oxygen into the bottle through the closures and/or the closure/bottle interface (Waters et al. 1996, Caloghiris et al. 1997, Waters and Williams 1997, Godden et al. 2001). The choice of wine closure type is therefore likely to have a considerable impact on the extent of wine oxidation.

There have been several reports of studies evaluating the impact of different closures and storage conditions on wine development (Chatonnet et al. 2000, Godden et al. 2001, Mas et al. 2002) with the most extensive to date being that of Godden et al. (2001). In contrast to Godden et al. (2001), Chatonnet et al. (2000) reported that wines with natural bark corks retained more of the antioxidant, SO_2, than wines sealed with the 'technical cork' closures (natural cork with a synthetic component). From both compositional and sensory data, Mas et al. (2002) concluded that wine was better preserved when bottles were stored horizontally, rather than upright, and that the most suitable closures for wine development were natural corks. They concluded that the use of synthetic closures and the screw-caps allowed oxidation of the wines. Godden et al. (2001) evaluated fourteen different closure types, and, in contrast to Mas et al. (2002), demonstrated that the screw cap closure in that study was the best performer in terms of minimising wine oxidation. These wines, however, developed in a different way to those under the other closures and also acquired a low level of a *struck flint/rubber* aroma. Such aromas have been described in other wines sealed under screw-cap closures (Francis et al. 2003).

As part of ongoing research on wine oxidation, we initiated a study to determine the effect of closure type and storage conditions on wine oxidation and development. The impact of other winemaking inputs such as ascorbic acid addition at bottling were also assessed and are reported separately (Skouroumounis et al. 2005).

In the study of the impact of closures and storage conditions reported here, the focus is on the compositional and sensory differences between wines sealed under different closures, and the physical performance of those closures. This study was undertaken on a Riesling and a wooded Chardonnay wine; two wine styles of economic importance to the Australian wine industry. These wines also differed in their content and composition of phenolics, the compounds likely to be involved in the formation of pigments contributing to brown colour, and of flavour compounds and their precursors, likely to be involved in the development or masking of oxidised and aged flavour.

Materials and methods

Skouroumounis et al. (2005) provides full details of wines, analysis and treatment of the data. Additional information specific to this closure study is as follows. The closures used included a roll-on-tamper-evident (ROTE) screw-cap closure (Auscap, Braybrook, Victoria), two reference 2 natural bark corks (both 44 × 24 mm, Cork 1 and Cork 2) and a synthetic closure produced by the moulding method (36.3 × 21.4 mm, Synthetic). The ROTE and Synthetic closures were taken from stocks held by Penfolds Wines and Corks 1 and 2 were selected from a range representing commercial stocks supplied by the agents or manufacturers of the corks.

Bottling was performed in mid August 1999 at Penfolds Nuriootpa packaging line in the following order: Riesling without ascorbic acid addition with Cork 1, with Cork 2, with Synthetic, with ROTE; Riesling with ascorbic acid addition with ROTE, with Synthetic, with Cork 2, with Cork 1; Chardonnay without ascorbic acid addition with Cork 1, with Cork 2, with Synthetic, with ROTE; Chardonnay with ascorbic acid addition with ROTE, with Synthetic, with Cork 2, with Cork 1.

Wine was pumped from a pallet tanker to the buffer tank under CO_2 cover. Pre-run checks of fill height, wine temperature, closure insertion depth, torque required to remove ROTE and headspace pressure were made and adjustment made as necessary before bottling proceeded. Eighty bottles were run through the system, then the filler was emptied and remaining wine placed back into the pallet tanker. For four replicates of each treatment, the wine temperature measured after bottling ranged from 14.7°C to 15.8°C. Fill heights measured for the cylindrical closures were between 65 and 71 mm, insertion depths for the Cork 1 and Cork 2 were from 0 to 2.5 mm and for the synthetic were from 1.5 to 3 mm, fill volume ranged from 746 mL to 757 mL and headspace pressure under the cylindrical closures ranged from –5 kPa to –20 kPa. The closures were replaced in the hopper and then the process repeated with the next closure. For ROTE, the corker was removed and ROTE applicator assembled. Fill heights measured for the ROTE were between 49 and 54 mm, torque ranged from 8 to 12 N and fill volume from 752 mL to 757 mL. The bottles were held upright for at least 12 hours. The following day, the wines were transported to the Hickinbotham Roseworthy Wine Science Laboratory (HRWSL) and placed in their final storage position.

Replicate bottles of the treatments were stored at the HRWSL at constant temperature and humidity for a period of five years. The storage temperature averaged 15.8°C, with maximum deviation over a day being not more than 1°C. Humidity averaged 65.1% over this period. Full details are as described in Godden et al. (2001).

A subset of the wines was stored in an office at the AWRI, where no continuous temperature control or any humidity control was operative.

The extent of wine travel along the 'long' exterior face of the cylindrical closures was estimated by inspection of the closures in the bottle at various time periods during storage.

Measurements of absorbance at 420 nm (A_{420}) and from 300 to 800 nm of wines were undertaken in the bottle using the method of Skouroumounis et al. (2003). Wines were also analysed spectrally to obtain the CIELAB parameters L*, a*, b* at various times after bottling, under the fluorescent illuminant F2 to be comparable to the lighting conditions of the visual assessment.

Directional paired comparison testing (Meilgaard et al. 1999) was carried out at various times with a panel of 40 to 60 assessors assessing one to eight pairs per session for visual *brownness/darkness*. On one occasion, after 3 weeks with a panel of only 14 assessors, two sets per session were assessed for several aroma and palate attributes. The balanced reference method of duo-trio difference testing (Meilgaard et al. 1999) was used at six months post-bottling, with the full panel of assessors asked to evaluate the samples by aroma only.

For descriptive analysis at two-and-a-half years post-bottling, a consensus descriptive analysis training method was applied. The visual attributes *yellow, orange, brown, green* and *overall colour intensity* were selected by the panel, and all but the last were defined with the assistance of printed 4 × 10 cm strips of graduated shades of these colours and were used to rate the wines.

For aroma descriptive analysis the method as detailed previously (Godden et al. 2001) was closely followed, with some variations as detailed in Skouroumounis et al. (2005). The assessors rated aroma attributes for each of the wine types in a randomised complete block design with ten replicate assessments over ten sessions. A closely similar procedure was followed for the 48 and 60 month post-bottling sensory study.

Genstat version 6.1.0.200 (Lawes Agricultural Trust, Rothamsted Experimental Station, UK) was used, together with JMP 5.0 (SAS Institute, Cary, NC), for data analysis, unless specified otherwise. The details of the statistical analysis are given in Skouroumounis et al. (2005).

Results

Wine travel differences among the cylindrical closures

Wine travel along the exterior of the long axis of the cork closures was determined visually from the changed colour of wet cork; no colour change is evident with Synthetic closures, hence the extent of wine travel could not be assessed by this method for these closures. The data from bottles of the different wine types (Chardonnay or Riesling, either with or without ascorbic acid addition at bottling) but same closure type and storage orientation were pooled (Figure 1) as they were not significantly different (data not shown). Wine travel occurred only to a relatively small extent along Cork 2 and was unaffected by storage orientation. There was extensive travel along Cork 1, particularly when stored in the inverted position, although wine travel was still evident along the exterior of the closure in the upright position, i.e., even when not in contact with the bulk of the wine in the bottle.

Colour differences among the wines measured instrumentally

1. Absorbance at 420 nm

Changes in absorbance at 420 nm (A_{420}) over five years for the Chardonnay wine to which ascorbic acid was added at bottling and sealed under either Cork 2, Synthetic or

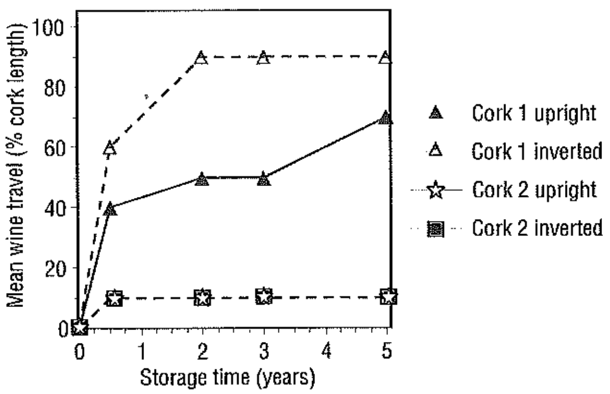

Figure 1. The effect of closure type and storage position on wine travel on the exterior of the cork towards the end of the bottle.
Wine travel, expressed as % of total cork length, for the two cork types and their two storage positions was the mean of pooled data from bottles of Chardonnay and Riesling wine with and without ascorbic acid addition (n = 136 to 144 bottles for storage times of 0.5 and two years, n = 46 to 49 bottles for three years storage time, n = 69 to 84 at five years storage).

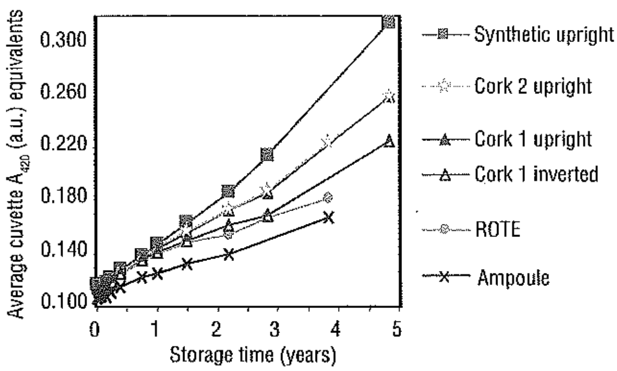

Figure 2. The effect of closure type and storage position on A_{420} of stored wine.
Mean A_{420} values over five years storage for the Chardonnay wine to which ascorbic acid was added at bottling sealed with either of the closure types and stored either upright or inverted (n = 10 to 36 bottles for all treatments except for Ampoules, n = 4, duplicate analyses were performed on each replicate bottle). The data for Cork 2 and Synthetic sealed wines with ascorbic acid and stored inverted are not shown because they were very similar or identical to their upright equivalents. Standard deviations were less than 5% for most data points and less than 10% for all.

under ROTE during upright storage or Cork 1 during both upright and inverted storage are shown in Figure 2. The change in A_{420} over 46 months for this same wine sealed in glass ampoules is also shown.

The trends exhibited by these data are representative of those for Riesling wine, except for those wines sealed in the ampoules and for both wines to which no ascorbic acid was added at bottling. The mean values of A_{420} for wines after three years storage are given in Appendix Table 1. The A_{420} values of the Chardonnay sealed with Cork 1 were lower when bottles were stored in the inverted position. Storage orientation did not, however, affect the A_{420} values of the other closure types (Appendix Table 1). For both bottled wines, excluding the ampoules, there was no observable difference in A_{420} between closure types up to 5 months after bottling, but from then on, the rate of increase of A_{420} was Synthetic > Cork > ROTE. For the Chardonnay wine, the A_{420} values for wines in ampoules were consistently lower than those for wines sealed with other closure types, at all time points assessed. For the Riesling wine, the data for wines in ampoules was similar to that of wines in ROTE (data not shown).

A restricted maximum likelihood (REML) analysis on the A_{420} values was undertaken 34 months after bottling (Appendix Table 2) and confirmed that both closure type and storage orientation influenced the A_{420} values.

Up to six bottles of each of the treatments of the Riesling wine and of a subset of the treatments of the Chardonnay wine were also stored upright immediately following bottling in an office where the temperature and humidity was not controlled. Under these conditions, the rate of increase in A_{420} was greater than for the same wines stored under controlled conditions (see data between 9 and 34 months for Riesling wine in Figure 3).

Nine months after bottling, as described in Skouroumounis et al. (2005), a portion of the Chardonnay wines

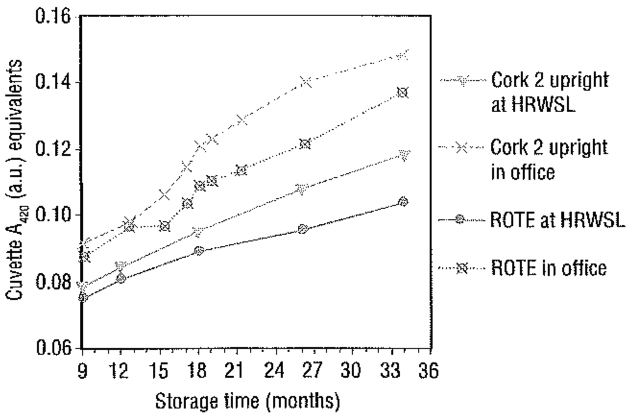

Figure 3. The effect of storage conditions on A_{420} of stored wine. Mean A_{420} values for the Riesling wine to which ascorbic acid was added at bottling sealed with either Cork 2 or ROTE and stored upright in either HRWSL under controlled temperature and humidity or in an office without control for 34 months after bottling (n = 30 bottles for HWRSL and 5 bottles for office, duplicate analyses were performed on each replicate bottle, standard deviations were less than 5% for all data points).

sealed with synthetic closure was transferred and then stored for 15 months in an anaerobic hood. The storage temperature was 22°C throughout the storage period, warmer than in the constant temperature and humidity storage room used for the main storage trial, and similar to the office. During this time period when some wines were stored in the anaerobic hood, the rate of increase in A_{420} of these wines in the anaerobic hood was much greater than for the same wines stored under all closure types in the HRWSL, and confirmed that temperature alone can affect the rate of increase of A_{420} in wine (Figure 4).

2. CIELAB

Wine colour was also measured instrumentally three years after bottling using the CIELAB coordinates, L* (a measure of intensity, the higher the value the lighter the colour), a* (positive values relate to *redness*) and b* (positive values relate to *yellowness*) (see data for wines sealed with natural bark corks and synthetics in Figure 5). For both wine types, the CIELAB coordinates discriminated among the treatments. Closure type affected the lightness (L*) of the wines; the lightest wines (highest L*) were those sealed under ROTE or cork, the darkest (lower L*) were those sealed with the synthetic closure. The value of the parameter ΔE^*_{ab}, a measure of differences in colour between samples of different closures, was also calculated (data not shown). For both wines, the ΔE^*_{ab} values between either Cork 1 or Cork 2 and the Synthetic were greater than 1, indicating that these samples would be perceived different to each other in colour by eye. After five years storage, the ΔE^*_{ab} values for pairs differing in closure type were all greater than 1.

Colour differences in wine determined visually

A visual assessment of colour of wines sealed under Cork 1, Cork 2 or Synthetic closure was also undertaken two-and-a-half years after bottling. In order to obtain an overview of the differences among the samples assessed by the panel, the data is shown in Figure 6 in a principal component analysis form. The mean scores for the colour

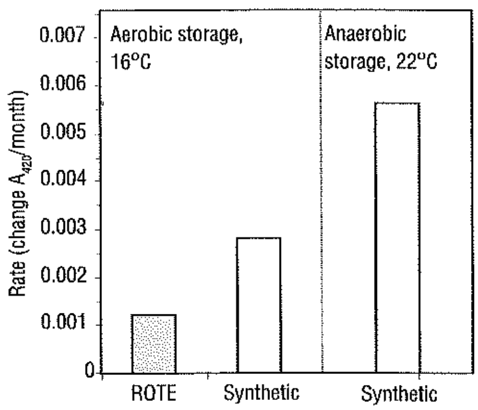

Figure 4. The effect of closure type and storage conditions on rate of change in A_{420}.
Chardonnay wines held upright and to which ascorbic acid was added at bottling were stored either aerobically in HRWSL at 16°C or in an anaerobic hood at 22°C, for a 15 month time period between 9 and 24 months post bottling. Rates were calculated from the difference in A_{420} values (cuvette equivalents) from the two time points divided by the number of months elapsed between the measurements.

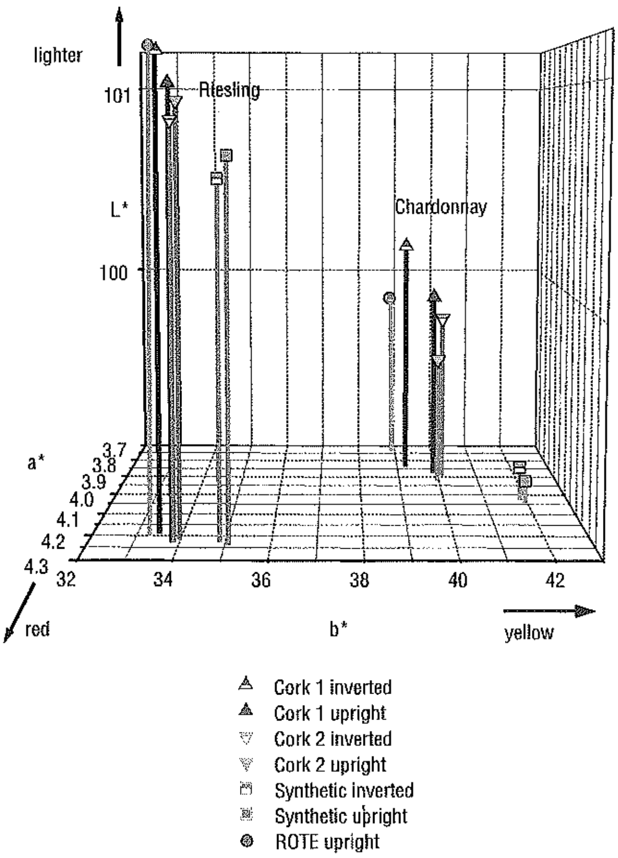

Figure 5. The effect of closure type and storage position on CIELAB coordinates, L*, a*, b* after three years storage at the fluorescent illuminant F2.
Values are means of ten replicates per treatment of the wines to which ascorbic acid was added at bottling. The data for the Riesling wines is shown on the left and that for the Chardonnay wines on the right of the figure. Some L* values are greater then 100% due to zeroing the instrument against air rather than water.

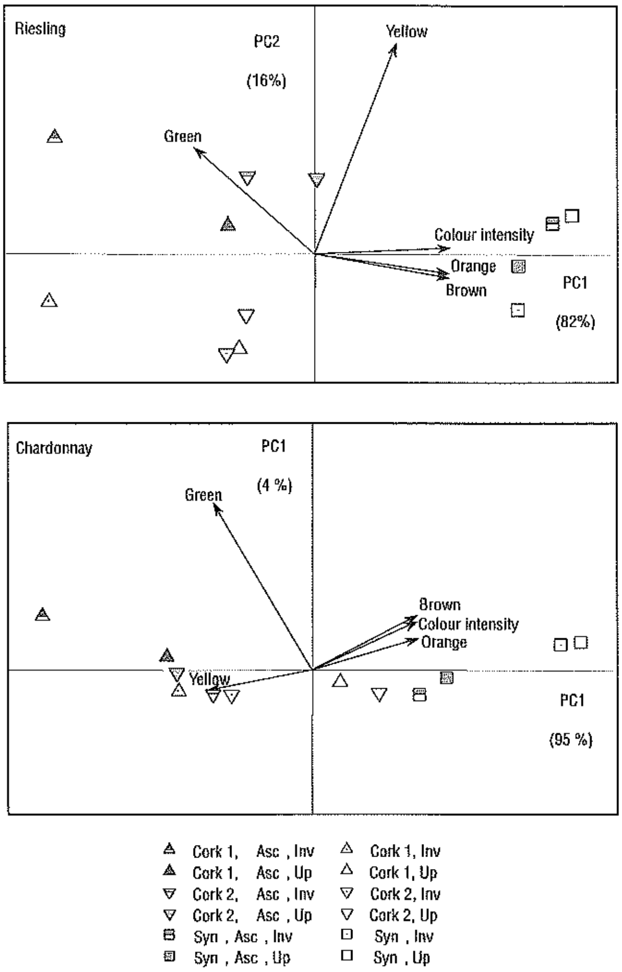

Figure 6. The effect of the treatments on visual colour after three years storage.
Biplot of principal components 1 and 2 for sample mean scores of visual descriptive analysis data for the Riesling and the Chardonnay wine, assessed after two and a half years storage. Vectors for visual colour attributes and the means for the ten replicate bottles are shown.

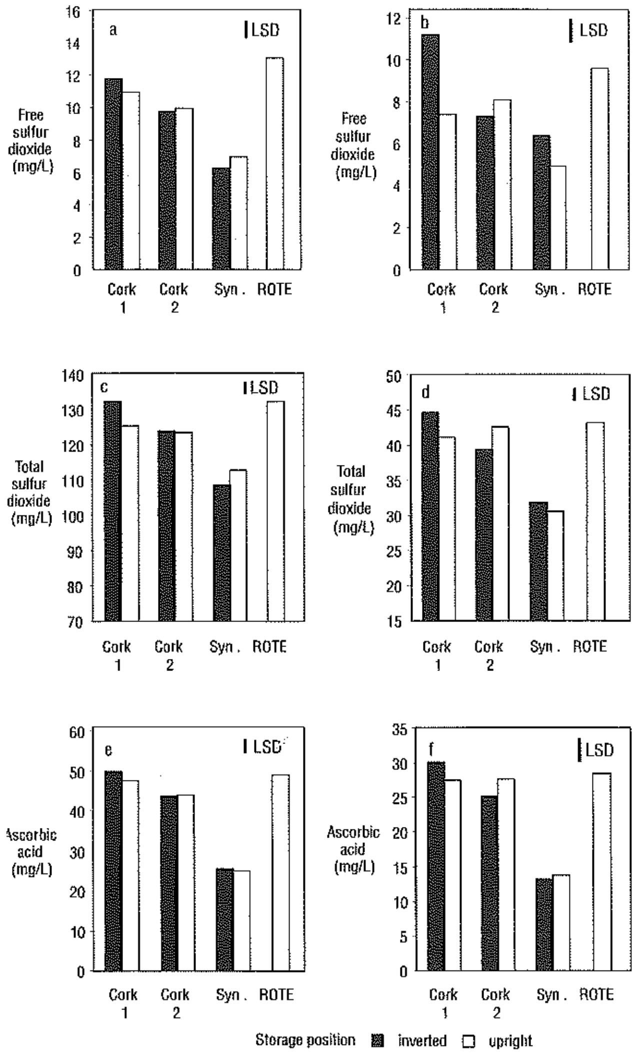

Figure 7. The effect of closure type and storage orientation on the concentration of free and total SO_2, and ascorbic acid after three years storage.
Bar charts represent means of 10 replicates per treatment (after three years storage) of concentration of (a, b) free SO_2, (c, d) total SO_2 and (e, f) ascorbic acid of Riesling and Chardonnay wines, to which ascorbic acid was added at bottling. Least significant differences (LSD) at the 5% level are as indicated.

attributes and the ANOVA summary are given in Appendix Tables 3 and 4, respectively.

For both varieties a similar pattern was found, with the largest effect that differentiated the wines being the closure type. For both the Riesling and Chardonnay sample sets, the Synthetic closed wines were clearly rated highest for *brown* and *orange* colour and highest in *overall colour intensity*, compared to the samples bottled under other closures. From the mean data and the ANOVA, it is clear that while the closure effect was the most important as to the perceived colour for both the Riesling and Chardonnay wine (Cork 1 similar to Cork 2, and Synthetic highest in *brown, orange* and *overall colour intensity*), storage upright could modulate the effects in the cork sealed wines and also that the presence or absence of ascorbic acid was of importance (see Skouroumounis et al. 2005). For wine sealed with Cork 1 or Cork 2, upright storage generally resulted in wines with higher scores for *brown, orange* and *overall colour intensity*.

Differences in the concentrations of SO_2 and ascorbic acid

The concentrations of SO_2 and ascorbic acid in the wines were determined three years after bottling (Appendix Table 1). A summary of the REML statistical analysis of the effect of the treatments on the levels of free and total SO_2 and ascorbic acid in the wines is given in the Appendix Table 5 and the mean data for the wines with ascorbic acid addition at bottling is presented in Figure 7. For a discussion of the impact of ascorbic acid on the concentrations of SO_2 see Skouroumounis et al. (2005).

The closure type was the treatment with the most obvious influence on the concentrations of SO_2 and ascorbic acid. Both wines sealed under the Synthetic closure had consistently lower levels of free and total SO_2 and ascorbic acid. Storage orientation appeared to have little impact on the levels of ascorbic acid in either wine. For the levels of total SO_2, both wines were affected by the orientation with varying degrees depending on closure type.

Differences in aroma and flavour among the wines

As described previously (Skouroumounis et al. 2005), the concentrations of two aroma compounds, 1,1,6-trimethyl-1,2-dihydronaphthalene (TDN, *aged Riesling, farm-feed, kerosene*) and β-damascenone (*fruity, cooked apple*) were determined three years after bottling (Appendix Table 9). Wines sealed with ROTE had the highest levels of TDN and wines sealed under the synthetic closure had the lowest. Within each closure type, similar levels or slightly less TDN was present in wines to which ascorbic acid had been added at bottling (as described in Skouroumounis et al. 2005).

Also after three years storage, two aroma descriptive analysis studies were carried out as described in Skouroumounis et al. (2005). The mean data for the attributes are given in Appendix Tables 6 and 7. Those individual samples with *TCA* mean scores above 1.0, a criterion that in the past (Godden et al. 2001) has been found to indicate a degree of TCA aroma that could be considered unacceptable, were removed from the data set before further analysis. Out of the 80 bottles sealed with Cork 1 and 80 with Cork 2, 4 and 10 samples were removed respectively, a cork taint incidence of 5 and 13%, respectively, three years after bottling. None of the wines sealed with the synthetic closure, under ROTE or in glass ampoules had *TCA* scores above 1.0.

In order to obtain an overview of the differences among the samples assessed by the panel for the two varieties, the data is shown in Figure 8 in a principal component analysis form. For both varieties a similar pattern was found, with the largest effect that differentiated the wines being the closure type. For both the Riesling and Chardonnay sample sets, the ROTE closed wines were generally rated highest in the fruity attributes, compared to the samples bottled under other closures. Both the Chardonnay and Riesling ROTE closed wines were also rated as relatively high in a *reduced struck flint/rubber* aroma. The Synthetic closed wines were rated as generally low in the fruity attributes in both sample sets, and high in *oxidised, wet wool, toasty* and *plastic* aroma. The Cork closed samples were situated on the right of the figures in both cases, indicating relatively high perceived fruit intensity. While the closure type was the most obvious and largest effect on the sensory properties of the samples, there was a further notable effect of the ascorbic acid addition at bottling generally resulting in wines with less oxidised and/or more fresh fruity aromas (see Skouroumounis et al. (2005) for full discussion of this effect).

To further explore the effects of imposed treatments on wine sensory properties, a further series of ANOVAs were run for the data set, excluding the ROTE closure, to assess the effect of storage orientation, closure type and ascorbic acid addition on the sensory properties of the wines. The results of the ANOVA for the Riesling and the Chardonnay data sets are summarised in the Appendix Table 8, with scores for selected mean aroma attributes shown in Figure 9.

For the *fresh citrus* and *cooked citrus* attributes of the Riesling wine, the ANOVA suggested that closure type had the main influence on the score. It is clear in Figure 9 that Riesling wines sealed under ROTE were highest in these attributes; Cork 1 was similar to Cork 2, and wines under the Synthetic closure were lowest. Storage orien-

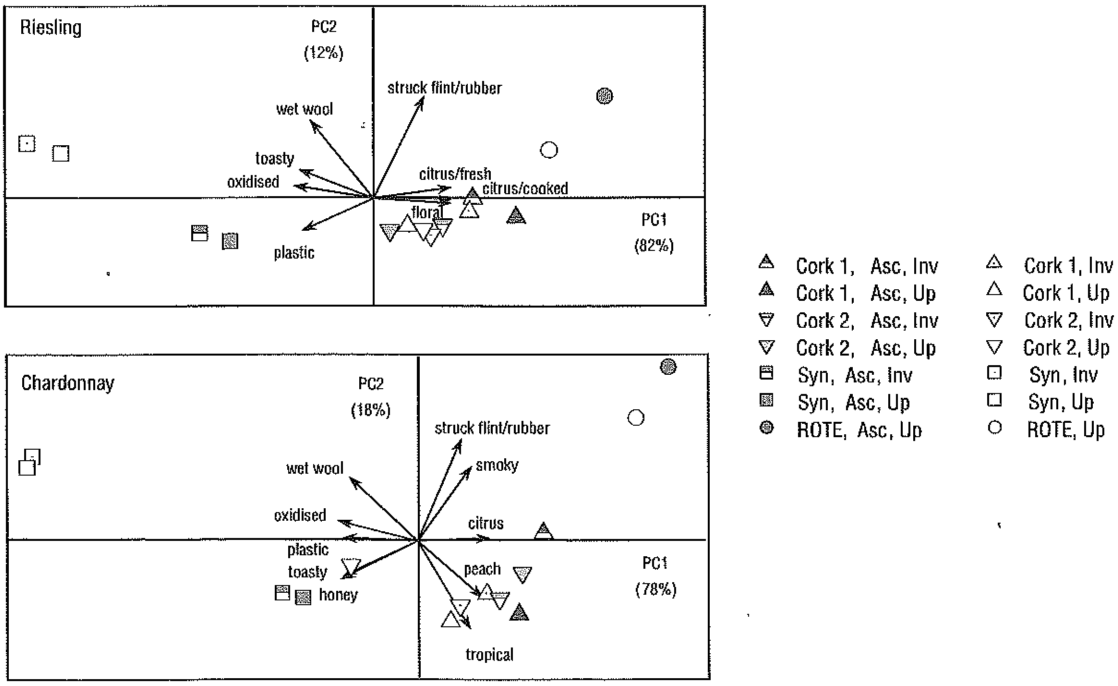

Figure 8. The effect of the treatments on aroma after three years storage.
Biplot of principal components 1 and 2 for sample mean scores of sensory descriptive analysis data for the Riesling and the Chardonnay wine, assessed after 36 months storage. Vectors for sensory attributes and the means for the ten replicate bottles of the 14 sample types are shown calculated from the scores of 11 panellists. Individual samples with TCA mean scores above 1.0 were not used to generate this biplot.

tation had little effect. Similarly, the *citrus* scores of the Chardonnay wines were little affected by storage orientation and wines sealed under ROTE were highest in *citrus*, Cork 1 was similar to Cork 2, and wines under the Synthetic were lowest in this attribute. The ANOVA suggested that the *honey* scores in the Chardonnay wines were affected by both closure type and storage orientation. In general *honey* was highest in the wines under Synthetic and lowest in wines under ROTE (Figure 9).

For the *oxidised* score, the ANOVA suggested, for the Riesling, that sample storage orientation had no influence, and the effect of ascorbic acid addition depended on the closure type (Appendix Table 8). For Chardonnay, storage orientation, ascorbic acid addition and closure type all influenced the *oxidised* score. For a discussion on the impact of ascorbic acid on this aroma attribute see Skouroumounis et al. (2005). It is clear from Figure 9 that the closure effect was the most important contributor to the perceived *oxidised* aroma for both wines (ROTE lowest, Cork 1 similar to Cork 2, and Synthetic highest). In general, bottle orientation during storage had little further impact on the *oxidised* score.

As noted above, three years after bottling, both the Chardonnay and Riesling ROTE closed wines were rated as relatively high in a *reduced, struck flint/rubber* aroma compared to wines under the other closures (Figure 9).

To investigate the *struck flint/rubber* aroma attribute further, two replicates of the upright stored ROTE and Cork 2 closed Riesling and Chardonnay wines, together with two replicates of the wines sealed in glass ampoules, were subjected to aroma descriptive analysis after four years storage. The mean data for the attributes are given in Appendix Tables 13 and 14. The ANOVAs carried out from the data for each of the attributes rated for the two sample sets indicated that for the Chardonnay set only the attributes *oxidised, honey, smoky* and *struck flint/rubber* varied significantly across the closures (Appendix Table 15). For the Riesling set the attributes *citrus-fresh, floral, honey, struck flint/rubber* and *oxidised* were rated significantly different across the closures, while ascorbic acid was a significant effect for *citrus-fresh, floral* and *oxidised* (Appendix Table 15).

The wine sealed anaerobically in the ampoules was clearly rated higher in the *struck flint/rubber* attribute compared to the ROTE closed wines, which in turn were rated higher than Cork sealed wines (Figure 10). For the *oxidised* attribute, ampoule and ROTE sealed wines were rated similarly low compared to Cork 2 sealed wines (Figure 10).

Discussion

The work reported here was undertaken to examine the effect on white wine oxidation and development of a number of different factors that wine producers can influence. These include the choice of closure type, storage conditions such as bottle orientation, storage temperature and time, and the choice of addition of ascorbic acid at bottling. In this paper the emphasis is on the impact of closure type and storage conditions. For a discussion of the impact of ascorbic acid see Skouroumounis et al. (2005).

The choice of closure for sealing wine intended for bottle maturation before consumption is likely to have a

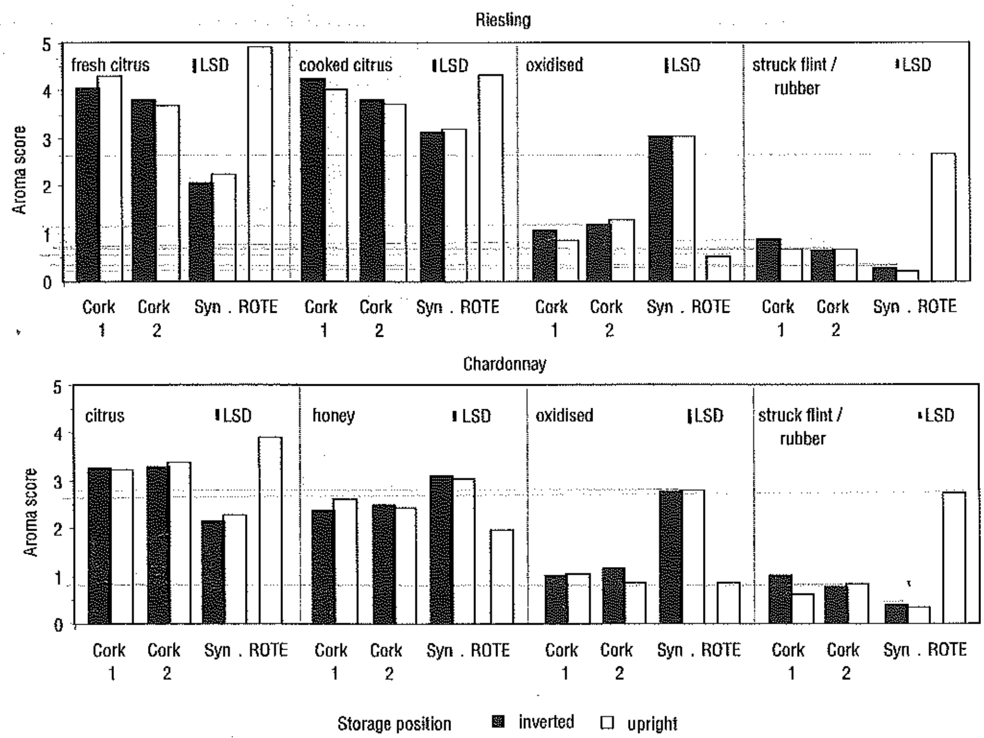

Figure 9. The effect of closure type and storage position on selected aroma scores after three years storage.
Values are means of seven to ten replicates per treatment of the Riesling and Chardonnay wines to which ascorbic acid was added at bottling. Individual samples with TCA mean scores above 1.0 were not used to generate this data. Least significant differences (LSD) at the 5% level are as indicated.

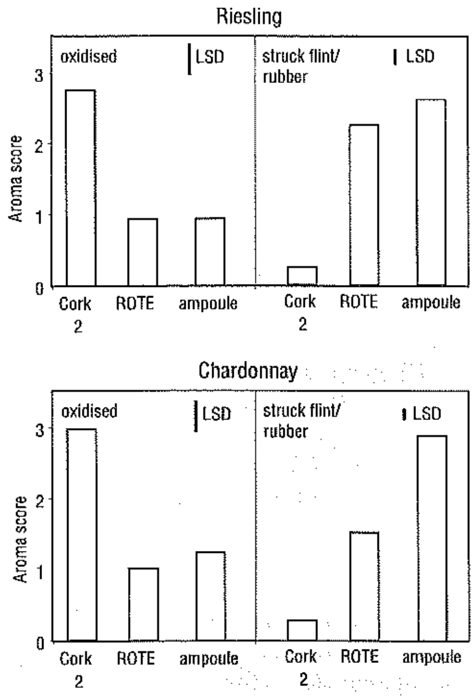

Figure 10. The effect of closure type on the *struck flint/rubber* and *oxidised* aroma score after four years.
Values are means of two replicates per closure of bottles stored upright of wines to which ascorbic acid was added at bottling, assessed by ten panellists. Least significant differences (LSD) at the 5% level are as indicated.

major impact on the degree of oxidation of the wine, as observed in other studies (Chatonnet et al. 2000, Godden et al. 2001, Mas et al. 2002). By all measures of wine oxidation used in this study (for both wines), storage under ROTE resulted in the least oxidised wines, a performance matched only by that of the ampoules. Use of the synthetic closure resulted in wines with the greatest oxidation in the set at three and four years storage, as observed in other studies (Godden et al. 2001). It is likely that these results are related to the ability of the closures to exclude oxygen (Caloghiris et al. 1997, Casey 1994, Godden et al. 2001, Keenan et al. 1999). The anaerobic study described in Skouroumounis et al. (2005) also demonstrated the major role played by oxygen in wine colour development.

Accompanying the lowest scores for oxidation in wines sealed under ROTE or in ampoules were the highest scores for a *struck flint/rubber* aroma attribute (Figure 9), as observed by others (Francis et al. 2003, Godden et al. 2001). The character appears to be related to either little or no oxygen ingress through the closure/package. It seems unlikely to be due to taints arising from, or aroma compounds produced from precursors derived from the screw cap or its wad because it also occurred in wines sealed in glass ampoules (Figure 10). Development of this character may depend on the presence of grape or fermentation derived precursors in wine at bottling. Conceivably, its development after bottling could be prevented by treatments before bottling, such as addition of copper sulfate. These observations should be followed up by studies with closures that exclude oxygen and ullages of known oxygen content.

The formation of TDN, a grape-derived aroma compound responsible for bottle-aged Riesling character, is reported to be accelerated by oxidation (Simpson 1979) and was present in oxidised white wine from Portugal (Silva Ferreira et al. 2003). The factor with the greatest impact on TDN levels in this study was closure type; the wines sealed under ROTE had the highest concentration, and the wine under the Synthetic closure has the lowest concentration of TDN (Appendix Table 9). However, this is most likely due to absorption of TDN by the Synthetic and natural closures (Capone et al. 2003) rather than due to oxidation. The data in this present study illustrates that overall closure performance relates to its barrier properties to oxygen, the absence of taints and to its ability to absorb specific flavour compounds from wine.

In some cases, inverted storage of the Cork closed wines resulted in the wine with less oxidised characters than upright storage, although the effect of storage orientation was relatively minor (Figure 9). It has long been asserted that natural bark corks need to remain moist to prevent shrinkage of the closure in the neck of the bottle. It is also possible that wine present in the cork reduces oxygen ingress into the bulk of the bottled wine by both reduced solubility in moist cork compared to dry cork and by reaction of wine components trapped in the cork with oxygen. The two corks examined here differed substantially in their estimated wetness (Figure 1), but appeared to perform similarly overall. Our estimates of cork wetness in this study were based on visual assessment of wine travel. This aspect of closures could be related to surface treatments applied to corks as well as to cork moisture. It was of interest that there was observable wine travel up the long axis of Cork 1 even when the bottles were stored upright, presumably due to surface migration from the meniscus up the sides of the bottle to the cork.

Storage time has an important role in the development of a bottled wine. In this study measurements of A_{420} on at least 30 replicate bottles for each treatment have been taken throughout their storage period (Figure 2). As described by Skouroumounis et al. (2005), provided one is not comparing wine without added ascorbic acide to wine with ascorbic acid addition, these values are a measure of the extent of wine browning and show that colour continues to change, even after contact of the wine with oxygen ceases, such as in an ampoule or in an anaerobic environment. Furthermore, our data from experiments in which wines were exposed to either one discrete 'dose' of oxygen or several 'doses' over one month (Skouroumounis et al. 2005) suggest that oxygenation events before or at bottling may not generate detectable changes in wine colour until some months later.

The storage temperature and its control is likely another most important factor for colour development. For the closures used in this study, storage at ambient, uncontrolled temperature and humidity resulted in a 50 to 100% increase in the rate of change in A_{420} compared to storage under controlled temperature (approx. 16°C) and humidity (approx. 70%, Figure 3). The data for the ROTE closures indicate that temperature can have a direct effect on colour development through accelerating chemical reactions even without significant oxygen ingress. This is also supported by the observation of greater increase in the rate of change in A_{420} for the wines stored anaerobically compared to the comparable wines stored in HRWSL (Figure 4). The importance of temperature and humidity control on bottled wine oxidation should be confirmed in storage and transport facilities used by the wine industry.

In conclusion, the data from this study confirm that wine development is influenced by the choice of closure used to package the wine. This is primarily due to the closure's ability to act as a barrier to oxygen. Oxygen ingress through closures is likely to be related to properties of the closures themselves as well as physical factors relating to the closure/bottle interface. In this study it appeared that too high an oxygen ingress rate, as exhibited by the synthetic closures, led to oxidised flavour and also that too low an oxygen ingress rate, as shown by the screw cap seals and the glass ampoules, led to the development of *reduced, struck flint/rubber* aromas in these wines. Given the range of oxygen transfer rates for different natural corks and within a batch of one particular natural cork type reported by others (Silva et al. 2003, Blair Duncan, unpublished data), natural corks could fit into both the 'too high and 'too low' category. It is likely that the extent to which these *reduced* characters develop in wines depends not only on the oxygen barriers properties of the closure used to seal the wine but also on the overall wine composition, the level of specific precursors to these aromas in the wines and the presence of other aroma compounds that may modulate the perception of these specific aromas.

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Appendix

Appendix Table 1

Mean[1] values of A_{420} (cuvette equivalents), and mean concentrations of free SO_2, total SO_2 and ascorbic acid for the Chardonnay and Riesling wine at three years post-bottling.

Wine	Addition at bottling	Closure	Storage position	A_{420} (a.u.)[2]	Free SO_2 (mg/L)	Total SO_2 (mg/L)	Ascorbic acid (mg/L)
Riesling	Ascorbic acid added	Cork 1	upright	0.116	11	125	47
			inverted	0.106	12	132	50
		Cork 2	upright	0.119	10	123	44
			inverted	0.116	10	124	44
		Synthetic	upright	0.131	7	113	25
			inverted	0.131	6	108	25
		ROTE	upright	0.104	13	132	49
	No ascorbic acid added		Cork 1	upright	0.103	10	122 ne
			inverted	0.093	11	129	ne
		Cork 2	upright	0.102	9	121	ne
			inverted	0.100	10	123	ne
		Synthetic	upright	0.112	6	109	ne
			inverted	0.114	5	105	ne
		ROTE	upright	0.094	11	124	ne
Chardonnay	Ascorbic acid added	Cork 1	upright	0.187	7	41	27
			inverted	0.170	11	45	30
		Cork 2	upright	0.188	8	43	28
			inverted	0.187	7	39	25
		Synthetic	upright	0.214	5	30	14
			inverted	0.213	6	32	13
		ROTE	upright	0.167	9	43	28
	No ascorbic acid added		Cork 1	upright	0.174	6	36 ne
			inverted	0.159	8	40	ne
		Cork 2	upright	0.178	5	33	ne
			inverted	0.166	7	39	ne
		Synthetic	upright	0.203	1	21	ne
			inverted	0.202	2	23	ne
		ROTE	upright	0.152	9	43	ne

[1] Values are means of ten replicates
[2] absorbance units
ne = not examined

Appendix Table 2

Results of the REML analysis for A_{420} (cuvette equivalents) for the Chardonnay and Riesling wine at 34 months post-bottling: degrees of freedom (df) and statistical significance[a].

Wine	Closure	Storage position	Ascorbic acid	Closure × Storage position	Closure × Ascorbic acid	Storage position × Ascorbic acid	Closure × Storage position × Ascorbic acid
Riesling	NA	NA	NA	***	**	ns	ns
Chardonnay	NA	NA	***	*	ns	ns	ns
df	3	1	1	2	3	2	2

[a] NA: not analysed because higher order interaction including the treatment was statistically significant at least at the 5% level
ns: not significant, * $P < 0.05$, ** $P < 0.01$, and *** $P < 0.001$.

Appendix Table 3

Mean[1] scores assessed in the *Flint* bottle for each colour attribute for the three cylindrical closures for the Chardonnay and Riesling wine at two and a half years post-bottling.

Wine	Addition at bottling	Closure	Storage position	*Colour intensity*	*Green*	*Brown*	*Yellow*	*Orange*
Riesling	Ascorbic acid added	Cork 1	upright	3.70	1.07	2.11	4.34	2.49
			inverted	2.93	1.58	1.65	4.34	1.89
		Cork 2	upright	4.25	1.12	2.58	4.50	3.04
			inverted	3.70	1.12	2.17	4.49	2.64
		Synthetic	upright	5.84	0.85	3.72	4.58	4.30
			inverted	5.75	0.84	3.65	4.54	4.13
	No ascorbic acid added	Cork 1	upright	3.84	0.98	2.34	3.98	2.80
			inverted	2.84	1.37	1.77	3.92	2.04
		Cork 2	upright	3.81	1.03	2.41	4.07	2.89
			inverted	3.61	0.99	2.44	3.97	2.73
		Synthetic	upright	5.58	0.86	3.60	4.39	4.08
			inverted	5.37	0.73	3.56	4.32	4.01
Chardonnay	Ascorbic acid added	Cork 1	upright	4.45	0.93	2.87	5.05	3.56
			inverted	3.57	1.28	2.16	5.22	2.64
		Cork 2	upright	4.42	0.87	2.83	5.03	3.56
			inverted	4.61	0.73	2.97	5.00	3.81
		Synthetic	upright	6.60	0.47	4.68	4.41	5.54
			inverted	6.38	0.43	4.52	4.57	5.35
	No ascorbic acid added	Cork 1	upright	5.41	0.61	3.97	4.56	4.49
			inverted	4.09	0.81	2.73	4.91	3.33
		Cork 2	upright	5.68	0.52	4.22	4.45	4.71
			inverted	4.61	0.72	3.06	4.85	3.76
		Synthetic	upright	7.78	0.42	5.97	4.05	6.58
			inverted	7.58	0.43	5.91	4.15	6.43

[1] Values are means of ten bottle replicates

Appendix Table 4

Results of the ANOVAs for each colour attribute for the three cylindrical closures for the Chardonnay and Riesling wine at two-and-a-half years post-bottling: degrees of freedom (df) and statistical significance [a].

Wine	Attribute	Closure	Storage position	Ascorbic acid	Closure × Storage position	Closure × Ascorbic acid	Storage position × Ascorbic acid	Closure × Storage position × Ascorbic acid
Chardonnay	*Intensity*	NA	NA	NA	*	ns	*	ns
	Brown	NA	NA	NA	*	ns	*	ns
	Yellow	***	**	***	ns	ns	ns	ns
	Orange	NA	NA	***	*	ns	ns	ns
	Green	NA	NA	NA	**	**	ns	ns
	df	2	1	1	2	2	1	2
Riesling	*Intensity*	NA	NA	ns	*	ns	ns	ns
	Brown	***	**	ns	ns	ns	ns	ns
	Yellow	NA	ns	NA	ns	*	ns	ns
	Orange	NA	NA	ns	*	ns	ns	ns
	Green	NA	NA	ns	***	ns	ns	ns
	df	2	1	1	2	2	1	2

[a] NA: not analysed because higher order interaction including the treatment was statistically significant at least at the 5% level
ns: not significant, * $P < 0.05$, ** $P < 0.01$, and *** $P < 0.001$.

Appendix Table 5

Results of the REML analysis for concentration of free SO_2, total SO_2 and ascorbic acid for the four closures for the Riesling and Chardonnay wine at 36 months post-bottling: degrees of freedom (df) and statistical significance[a].

Wine	Attribute	Closure	Storage position	Ascorbic acid	Closure × Storage position	Closure × Ascorbic acid	Storage position × Ascorbic acid	Closure × Storage position × Ascorbic acid
Riesling	*Free* SO_2	***	ns	**	ns	ns	ns	ns
	Total SO_2	NA	NA	***	***	ns	ns	ns
	Ascorbic acid	***	ns	–	ns	–	–	–
Chardonnay	*Free* SO_2	***	*	***	ns	ns	ns	ns
	Total SO_2	NA	NA	NA	ns	**	*	ns
	Ascorbic acid	***	ns	–	ns	–	–	–
	df	3	1	1	2	3	1	

[a] –: not tested, NA: not analysed because higher order interaction including the treatment was statistically significant at least at the 5% level
ns: not significant, * $P < 0.05$, ** $P < 0.01$, and *** $P < 0.001$.

Appendix Table 6

Mean[1] scores for each aroma attribute for the Riesling wine at three years post-bottling.

Addition at bottling	Closure	Storage position	*Fresh citrus*	*Cooked citrus*	*Floral*	*Honey*	*Kerosene*	*Toasty*	*Wet wool*	*Plastic*	*Struck flint/rubber*	*Oxidised*
Ascorbic acid added	Cork 1	upright	4.3	4.0	2.4	2.5	1.0	1.5	0.1	0.3	0.6	0.8
		inverted	4.1	4.2	2.1	2.7	1.1	1.9	0.1	0.4	0.9	1.0
	Cork 2	upright	3.7	3.7	1.8	2.4	1.1	1.5	0.2	0.8	0.7	1.3
		inverted	3.8	3.8	2.1	2.7	1.0	1.5	0.2	0.6	0.6	1.2
	Synthetic	upright	2.2	3.2	1.2	2.8	1.0	2.1	0.1	0.8	0.2	3.0
		inverted	2.0	3.1	1.2	2.8	1.0	2.1	0.3	0.9	0.2	3.0
	ROTE	upright	4.9	4.3	2.3	1.9	1.4	1.5	0.3	0.1	2.7	0.5
No ascorbic acid added	Cork 1	upright	3.4	3.8	1.9	2.6	1.0	1.6	0.1	0.4	0.6	1.8
		inverted	4.0	4.0	2.2	2.5	1.0	1.7	0.1	0.4	0.9	1.1
	Cork 2	upright	3.4	3.6	2.0	2.4	1.1	1.5	0.3	0.6	0.5	1.6
		inverted	3.8	3.8	1.9	2.5	1.1	1.5	0.2	0.5	0.3	1.3
	Synthetic	upright	1.4	2.7	0.8	3.0	0.9	2.4	1.2	0.8	0.3	4.4
		inverted	1.5	2.6	0.8	2.8	1.0	2.4	1.4	1.0	0.4	4.5
	ROTE	upright	4.8	4.0	2.4	2.1	1.3	1.5	0.2	0.4	1.9	0.7

[1] Values are means of seven to ten replicates. The mean data were calculated after excluding those individual samples with *TCA* mean scores above 1.0, a criterion that in the past (Godden et al. 2001) has been found to indicate a degree of *TCA* aroma that could be considered unacceptable.

Appendix Table 9

Concentration of TDN and β-damascenone after upright storage of bottled wine for 36 months.

Wine	Addition at bottling	Closure	Concentration of β-damascenone (μg/L)	Concentration of TDN (μg/L)
Riesling	Ascorbic acid added	Cork 1	6.3 (0.3) [1]	22.6 (0.9)
		Cork 2	6.6 (0.9)	24.4 (3.4)
		Synthetic	6.2 (0.4)	7.8 (0.4)
		ROTE	5.2 (0.3)	46.9 (0.2)
	No ascorbic acid added	Cork 1	7.4 (0.7)	24.2 (0.4)
		Cork 2	7.0 (0.7)	30.0 (1.9)
		Synthetic	–	–
		ROTE	6.6 (0.9)	52.8 (2.6)
Chardonnay	Ascorbic acid added	Cork 2	4.0 (0.3)	2.0 (0.0)
		Synthetic	4.2 (0.2)	0.5 (0.0)
		ROTE	4.0 (0.3)	3.0 (0.0)
	No ascorbic acid added	Cork 2	4.8 (0.2)	2.0 (0.0)
		Synthetic	–	–
		ROTE	4.4 (0.6)	3.5 (0.3)

[1] Means of five replicates with standard deviation in parentheses
– = not determined

Appendix Table 10

Mean[1] scores for each aroma attribute for the Cork 1, Cork 2 and Synthetic sealed Riesling wine stored inverted at 60 months post-bottling.

Addition at bottling	Closure	*Citrus*	*Foral*	*Honey*	*Toasty*	*Kerosene*	*Oxidised*	*Plastic*	*TCA*	*Struck flint/ rubber*
Ascorbic acid added	Cork 1	4.0	2.3	2.6	2.2	2.1	1.1	0.0	0.0	0.7
	Cork 2	3.4	1.7	2.2	1.9	2.0	1.8	0.4	0.5	0.4
	Synthetic	1.8	0.9	2.6	1.8	1.3	5.3	0.3	0.0	0.0
No ascorbic acid added	Cork 1	3.8	2.0	2.4	2.1	2.1	1.4	0.3	0.2	0.3
	Cork 2	3.7	2.0	2.7	2.0	2.1	1.7	0.2	0.1	0.1
	Synthetic	1.2	0.5	2.1	1.9	1.1	6.1	0.2	0.0	0.0

[1] Values are means of four replicates.

Appendix Table 11

Mean[1] scores for each aroma attribute for the Cork 1, Cork 2 and Synthetic sealed Chardonnay wine stored inverted at 60 months post-bottling.

Addition at bottling	Closure	*Overall fruit*	*Honey*	*Toasty/ buttery*	*Vanilla*	*Smoky*	*Oxidised*	*Plastic*	*TCA*	*Struck flint/ rubber*
Ascorbic acid added	Cork 1	4.0	2.8	3.0	2.3	2.3	0.9	0.0	0. 0	0.5
	Cork 2	3.0	2.8	2.6	1.9	1.4	2.5	0.1	0.2	0.1
	Synthetic	1.5	2.6	1.9	1.2	1.1	5.3	0.2	0.0	0.0
No ascorbic acid added	Cork 1	3.6	2.8	2.9	2.0	1.6	1.4	0.2	0.1	0.1
	Cork 2	2.4	2.9	2.2	1.6	1.5	3.4	0.1	0.4	0.0
	Synthetic	1.0	2.4	1.6	1.1	1.0	6.9	0.2	0.1	0.1

[1] Values are means of four replicates.

Appendix Table 12

Results of the ANOVAs for each attribute for the Cork 1, Cork 2 and Synthetic sealed Riesling and Chardonnay wine stored inverted at 60 months post-bottling: degrees of freedom (df) and statistical significance[a].

Wine	Attribute	Ascorbic acid	Closure	Closure × Ascorbic acid
Riesling	*Citrus*	ns	***	ns
	Floral	ns	***	ns ($P = 0.058$)
	Honey	NA	NA	*
	Toasty	ns	ns	ns
	Kerosene	ns	***	ns
	Struck flint/rubber	*	***	ns
	Oxidised	ns	***	ns
	Plastic	NA	NA	*
	df	1	2	2
Chardonnay	*Overall fruit*	***	***	ns
	Vanilla/caramel	ns ($P = 0.095$)	***	ns
	Honey	ns	*	ns
	Smoky	ns	***	ns ($P = 0.056$)
	Toasty/buttery	ns ($P = 0.06$)	***	ns
	Plastic	ns	ns	ns
	Struck flint/rubber	NA	NA	*
	Oxidised	***	***	ns ($P = 0.08$)
	df	1	2	2

[a] NA: not analysed because higher order interaction including the treatment was statistically significant at least at the 5% level, ns: not significant, * $P<0.05$, ** $P<0.01$, and *** $P<0.001$.

Appendix Table 13

Mean[1] scores for each aroma attribute for the upright stored Cork 2, ROTE, and glass ampoule sealed Riesling wine at 48 months post-bottling.

Addition at bottling	Closure	*Fresh citrus*	*Cooked citrus*	*Floral*	*Honey*	*Toasty*	*Kerosene*	*Oxidised*	*Plastic*	*TCA*	*Struck flint /rubber*	*Wet wool /wet dog*
Ascorbic acid added	Cork 2	2.09	3.50	2.23	2.86	2.09	2.05	2.73	0.68	0.23	0.23	0.14
	ROTE	2.86	3.05	1.50	2.64	1.86	1.86	0.91	0.55	0.09	2.23	0.14
	Glass ampoule	2.68	2.91	1.64	2.36	1.86	1.64	0.91	0.95	0.05	2.59	0.23
No ascorbic acid added	Cork 2	1.36	2.86	1.32	3.59	2.27	2.09	4.32	0.91	0.00	0.09	0.05
	ROTE	3.00	4.00	2.27	2.45	1.27	2.09	1.36	0.64	0.18	0.91	0.00
	Glass ampoule	2.36	3.05	1.27	2.45	1.73	2.09	1.09	0.36	0.09	2.50	0.32

[1] Values are means of duplicate samples.

Appendix Table 14

Mean[1] scores for each aroma attribute for the upright stored Cork 2, ROTE, and glass ampoule sealed Chardonnay wine stored inverted at 48 months post-bottling.

Addition at bottling	Closure	*Citrus*	*Peach*	*Tropical*	*Honey*	*Toasty /buttery*	*Vanilla /caramel*	*Oxidised*	*Plastic*	*TCA*	*Struck flint /rubber*	*Wet wool /wet dog*
Ascorbic acid added	Cork 2	1.86	2.55	1.91	3.27	2.91	2.86	2.95	0.27	0.23	0.27	0.68
	ROTE	2.14	2.59	2.18	2.55	2.64	2.86	1.00	0.32	0.09	1.50	0.14
	Glass ampoule	2.05	2.45	1.95	2.14	2.50	2.59	1.23	0.55	0.00	2.86	0.45
No ascorbic acid added	Cork 2	1.64	2.55	1.95	2.55	2.23	3.32	2.86	0.77	0.09	0.05	0.32
	ROTE	2.00	2.95	2.68	2.27	2.64	2.95	1.05	0.18	0.14	1.82	0.18
	Glass ampoule	2.27	2.82	2.18	2.18	2.64	2.59	0.91	0.41	0.09	2.50	0.36

[1] Values are means of duplicate samples.

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Appendix Table 15

Results of the ANOVAs for each attribute for the ROTE, Cork 2 and glass ampoule sealed Riesling and Chardonnay wine at 48 months post-bottling: degrees of freedom (df) and statistical significance[a].

Wine	Attribute	Ascorbic acid	Closure	Closure × Ascorbic acid
Riesling	*Citrus-fresh*	*	**	ns
	Citrus-cooked	ns	ns	ns
	Floral	NA	NA	**
	Honey	ns	*	ns
	Toasty	ns	ns	ns
	Kerosene	ns ($P = 0.07$)	ns	ns
	Wet wool / wet dog	ns	ns	ns
	Struck flint/rubber	ns	***	ns
	Oxidised	**	***	ns
	Plastic	ns	ns	ns
	df	1	2	2
Chardonnay	*Citrus*	ns	ns	ns
	Peach	ns	ns	ns
	Tropical	ns	ns ($P = 0.06$)	ns
	Vanilla/caramel	ns	ns	ns
	Honey	ns	*	ns
	Smoky	ns	***	ns
	Toasty/buttery	ns	ns	ns
	Wet wool / wet dog	ns	ns	ns
	Plastic	ns	ns	ns
	Struck flint/rubber	ns	***	ns
	Oxidised	ns	***	ns
	df	1	2	2

[a] NA: not analysed because higher order interaction including the treatment was statistically significant at least at the 5% level, ns: not significant, * $P < 0.05$, ** $P < 0.01$, and *** $P < 0.001$.