AWRI Report

AWRI Commercial Closure Trial – Twenty Four Month Report. Performance testing of the Álvaro Coelho Irmãos S.A. Nanocork closure

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Executive Summary

Shelf life of a wine is influenced by a combination of factors including vinification, packaging methodology, storage, transport conditions and importantly, closure selection.

Engaged by Álvaro Coelho Irmãos S.A. in May 2007, AWRI Commercial Services undertook a 24 month study to evaluate changes in wine chemical and sensory properties related to the performance of the Nanocork closure compared to those occurring in the same wine bottled under three reference closures, namely:

- A Reference Two natural cork;
- A Supremecorq X2 synthetic closure; and
- An Alcan Stelvin screwcap closure.

The Nanocork at the 24 month timepoint was found to produce a fresh, intense, fruity wine with long persistence on the palate. In contrast, the Reference Two and Reference Synthetic closures produced relatively similar wines to each other, characterised by honey, esters and oxidation characters and a higher OD_{420} than other closures entered in the trial. The enhanced oxidation characters exhibited by the Reference Two and Reference Synthetic closures suggest that they are approaching the end of their shelf lives, whereas the fresh fruity characters of the Nanocork suggest it has further to go before reaching its shelf life.

When compared to the Reference Screwcap, the Nanocork displayed elevated fresh fruit intensity in aroma and palate, plus a long palate length. In contrast, the Reference Screwcap displayed moderate levels of reductive characters, in particular struck flint, and had a lower fruit intensity.

Reductive characters were observed under the Reference Screwcap along with elevated levels of free and total SO_2 and the compound methanethiol.

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1.0 Introduction

Wine closure type has been demonstrated by The Australian Wine Research Institute (AWRI) over several studies as playing a significant role in wine style development (Godden *et al.*, 2001; Godden, 2001; Godden 2002; Godden *et al*., 2005).

Given the ongoing nature of closure development, there is necessity to regularly evaluate novel closure technologies or next-generation versions of existing closures.

Presented in this report is a trial conducted by the AWRI tracing the chemical and sensorial development of a 2007 Clare Valley Semillon wine bottled under the Nanocork and three Reference closures (Reference Two natural cork, Reference Synthetic closure and Reference Screwcap) over a 24 month period from May 2007 – May 2009.

2.0 Materials & Methods

2.1 The closures

The participating companies were asked to provide a minimum of 800 closures to the AWRI, from which 300 closures were selected randomly from the hopper during each bottling run at VinPac, Angaston. A holdback sample was taken of the remaining closures in case later testing was required.

The three reference closures used in this study included:

- Reference Screwcap - Standard Stelvin Saran-tin lined screwcap produced by Alcan;
- Reference Synthetic - Supremecorq X2 (length 45 mm); and
- Reference Two natural cork - (length 44 mm) from a QC checked batch sourced from the stock from a major winery.

2.2 The bottles

All bottles were new, and were purchased from O-I Glass, Adelaide, through VinPac International. The bottles used for the cylindrical closures were 'flint'-coloured 'claret' bottles (manufacturer's code 5372). For the Screwcap closures, a similar 'flint'-coloured' bottle with a BVS screw thread was used.

2.3 The wine

The wine chosen was a 2007 Semillon purchased from Constellation Wines Australia's, Leasingham Winery, located in the Clare Valley, South Australia. The winemakers at Leasingham were requested to produce a wine of similar characteristics as previously used in AWRI closure trials (unoaked, around 11 % alcohol, relatively neutral with some fresh citrus, tropical notes, taint and fault free) and to use normal commercial practice in the making of the wine including in the clarification and fining of the wine.

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2.4 The bottling

Bottling operations were performed on 16 May, 2007, at VinPac International Pty Ltd, Angaston, South Australia, a large ISO 17025-certified contract bottling facility. VinPac's routine quality control procedures were performed on all equipment prior to bottling. The wine was filtered through a 0.45µm filter and bottled at a temperature of 17-19^{o}C.

Closures were applied according to the supplier's specifications, or in the absence of specifications, in accordance with good manufacturing practise based on VinPac's experience with the closure type. All participants had the option of exclusively observing the bottling of their closures.

300 bottles of wine were bottled for each closure, with each bottle labelled with consecutive numbers and the closure type. Random bottles taken from the bottling line were tested for dissolved oxygen and dissolved carbon dioxide using an Orbisphere instrument.

2.5 Storage

The 300 bottles (for each closure) were randomised at packaging so a random sample containing 12 bottles could be selected without opening multiple cartons. The labelled bottles were immediately packed into cardboard cartons and sealed. Cartons were stacked on pallets. The cartons remained upright for 24-36 hours after which they were inverted and then transported to the Hickinbotham Roseworthy Wine Science Laboratory cellar adjacent to the AWRI. Here they were stored inverted in the wine storage area on pallets with approximately 64 cartons to the pallet. The temperature in the storage facility was maintained at approximately 17^{o}C and 55 % humidity.

2.6 Pre-bottling analyses

Tank samples of the finished wine were submitted to AWRI Commercial Services for pre-bottling analyses including alcohol, organic acids, free sulfur dioxide (free SO_2), total sulfur dioxide (total SO_2), glucose+fructose, volatile acidity (VA), specific gravity (SG), titratable acidity (TA), pH, pinking, ascorbic acid, dissolved carbon dioxide, laccase activity, optical density at 420 nm (OD 420nm), and metals (copper, iron, potassium, sodium), as well as chloroanisoles and agrochemical residues. The results for these analyses were presented in the initial report.

A preliminary sensory evaluation was also performed by expert AWRI wine tasters to ensure the wine was suitable for the closure study. The wine was described as a pleasant, fairly full bodied and flavourful Semillon with the following sensory attributes: clean with no faults, fairly neutral, with some estery and tropical fruit notes, quite acidic on the palate.

2.7 Post bottling analyses (24 - 48 hours)

Initial testing was performed on 12 randomly selected bottles within 48 hours of bottling by AWRI Commercial Services. The tests involved were alcohol, pH, TA, VA, free SO_2, total SO_2 and OD 420nm.

The results for this time point were discussed in the initial (post bottling) bottling report.

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2.8 Six to twenty four month analyses

Twelve bottles of each closure were removed from storage at 6, 12, 18 and 24 months and analysed for free SO_2, total SO_2, OD_{420}, sensory properties and for physical parameters as shown in Table 1. A suite of low molecular weight sulfur analyses was offered as an option at the 18 and 24 month timepoints. When requested, low molecular weight sulfur analysis was performed on 3 replicate bottles, including the three reference closures.

Table 1. Summary of analyses conducted from 6 – 24 months.

Storage time	Analysis date	Free SO_2, Total SO_2 & OD_{420nm}	Sensory panel analysis	Extraction and reinsertion force	Low molecular weight sulfur compounds
6 months	12 - 16 November, 2007	Yes	Yes	Yes	No
12 months	19 - 23 May, 2008.	Yes	Yes	No	No
18 months	17 - 21 November, 2008	Yes	Yes	No	Yes. 5 - 12 December, 2008
24 months	18 - 22 May, 2009	Yes	Yes	No	Yes. 15 - 19 June, 2009

The results and number of replicates for each test at the 24 month timepoint are presented in Appendices 1 and 2. Data from the 6, 12 and 18 month timepoints was discussed in previous reports.

2.9 Methods of chemical analysis

All analysis was performed by AWRI Commercial Services' NATA accredited (ISO 17025 certified) wine laboratory.

Free and total sulfur dioxide was measured using Flow Injection Analysis (FIA, Lachat). Optical density was determined by measurement of the absorbance at 420 nm on a Varian UV/visible spectrophotometer. Low molecular weight sulfur analysis was conducted via a gas chromatograph fitted with a sulfur detector.

All analyses were performed by trained staff and were performed in conjunction with NATA accredited quality assurance measures including standards, blanks, duplicates and control samples. The quality control measures were required to meet established criteria before acceptance of the analytical data.

2.10 Method of sensory evaluation

A panel of ten judges was recruited, comprising AWRI staff with extensive experience in wine sensory evaluation, of whom all had participated in at least one of the previous testing times for this study. An initial discussion session was held on 18 May, 2009, with the tasters assessing six of the wines from the current study. These wines were selected based on a preliminary evaluation by the project sensory team to identify those samples displaying the largest sensory differences, and included the Reference Screwcap and Reference Synthetic closures. The tasters assessed the wines in silence, followed by a discussion regarding the sample's characteristics, to decide upon the attributes that would be rated in the subsequent formal sessions. The attribute list was slightly modified from that applied at the earlier testing times. A list of the terms that was agreed upon by the panellists to rate in the formal sessions is given in Table 2.

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An initial practice rating session was held, with the tasters assessing a sample of each of the wines from the current study under the same conditions as the formal sessions but with a constant presentation order. Following the practice session, panel performance was assessed and all panellists were considered to be performing adequately.

Table 2. Sensory attributes scored.

Attribute	Definition/synonyms
Aroma	
Estery	Confectionary, banana lolly
Fresh Citrus	Lemon, lime, zest
Cooked Citrus	Cooked lime, brown lime cordial
Tropical fruit	Includes pineapple
Stonefruit	Peach, apricot
Overall fruit	Overall fruit aroma
Honey	Includes caramel, butterscotch
Toasty	Includes nutty
Oxidised	Aldehyde, bruised apple, cardboard, wet dog, lanolin, wet wool
Plastic	Fresh PVC, beachball, glue, play dough
TCA	TCA, musty, mouldy
Struck flint	Struck match, toward burnt rubber
Cabbagey	Rotten egg, cabbage, sewerage
Other (aroma)	Any other term - please record on 'other descriptors' sheet
Palate	
Acidity	Perceived acidity
Overall fruit	Overall fruit flavour on palate
Fruit Flavour Persistence	Fruit flavour persistence
Plastic	PVC, beachball, glue, play dough
Reductive	Struck flint, burnt rubber, hardness on the palate
Oxidised	Aldehyde, bruised apple, cardboard, wet dog, lanolin, wet wool
Other (palate)	Any other term - please record on 'other descriptors' sheet

For the formal sessions, samples were assessed by the judges independently in blind tasting conditions using standardised procedures. Fourteen wines were assessed at a session, being one example of each closure in the study including the three Reference closures. The samples were assessed in two sets of seven, with a forced rest between sets. Four bottles of each closure type were assessed over four sessions over three days, 19 May, 20 May and 22 May. The samples (30 mL) were presented to tasters in 3-digit coded, covered XL5 (ISO standard) glasses, in a random presentation order across judges at 22-24°C. The tasters were instructed to assess each wine for aroma and then palate, and then move to the next sample. The tests were carried out in the AWRI's sensory facility in isolated tasting booths under sodium colour masking lights. Data was acquired using Fizz 2.30B software (Biosystemes, Couternon, France).

The panelists scored each attribute on a structured line scale of 0-9; where 1 corresponded to just detectable, 5 to a moderate intensity and 9 to a very strong intensity. Tasters were also given the opportunity to rate any other attributes evident in any sample.

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Data analysis was carried out using GenStat Release 10.1 (VSN International). Analysis of variance was carried out testing for the effect of closure type, and bottle replicate nested within closure type, followed by a Fisher's Least Significant Difference mean comparison test (P=0.05). This allows an assessment of whether the mean attribute values for each closure, are sufficiently different to be considered statistically significantly different. In other words, if the difference between two mean values is greater than the LSD value, they are significantly different.

2.11 Method of statistical analysis

All data was analysed by standard ANOVA techniques using the statistical package JMP (v5.0.1a, SAS Institute, U.S.A). During analysis, outliers in data sets, i.e. where value > μ ± 95% confidence intervals, were excluded. All Least Significant Difference (LSD) values used for analysis and presented in this report are for pooled values for the whole data set, except where otherwise indicated. Note. LSD is the 95% confidence interval.

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3.0 Results

Twenty four month chemical and sensory analysis results are presented in sections 3.1 to 3.5. Chemical results for free and total SO_2 and OD_{420} are provided in sections 3.1 to 3.3, low molecular weight sulfur analyses in section 3.4 and sensory analyses in section 3.5.

All twenty four month chemical and sensory analysis data is reported in Appendices 1 and 2.

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3.1 Chemical analysis – Free SO_2

Results for free SO_2 at the 24 month timepoint presented below in Figure 1 show the following:

- After starting at a concentration of 36mg/L, free SO_2 concentrations for the three reference closures and the Nanocork over the 24 month storage period were observed to undergo a rapid decrease over the first 6 months, following which free SO_2 results stabilised;
- The Reference Screwcap closure generally sustained a higher level of free SO_2 than the other closures throughout the trial;
- Whilst there is an apparent scatter of data at the zero timepoint, all analyses at this point were within the acceptable tolerances associated with FIA analysis of free SO_2. This is discussed in further detail later in the report;
- Statistically significant differences ($p>0.95$) were observed between the free SO_2 results of the Nanocork and the Reference Two natural cork, with the Nanocork having a higher free SO_2 value;
- Statistically significant differences ($p>0.95$) were observed between the free SO_2 results of the Nanocork and the Reference Screwcap closure, with the Nanocork having a lower free SO_2 value; and
- There were no statistically significant differences ($p>0.95$) in free SO_2 between the Nanocork and the Reference Synthetic closure.

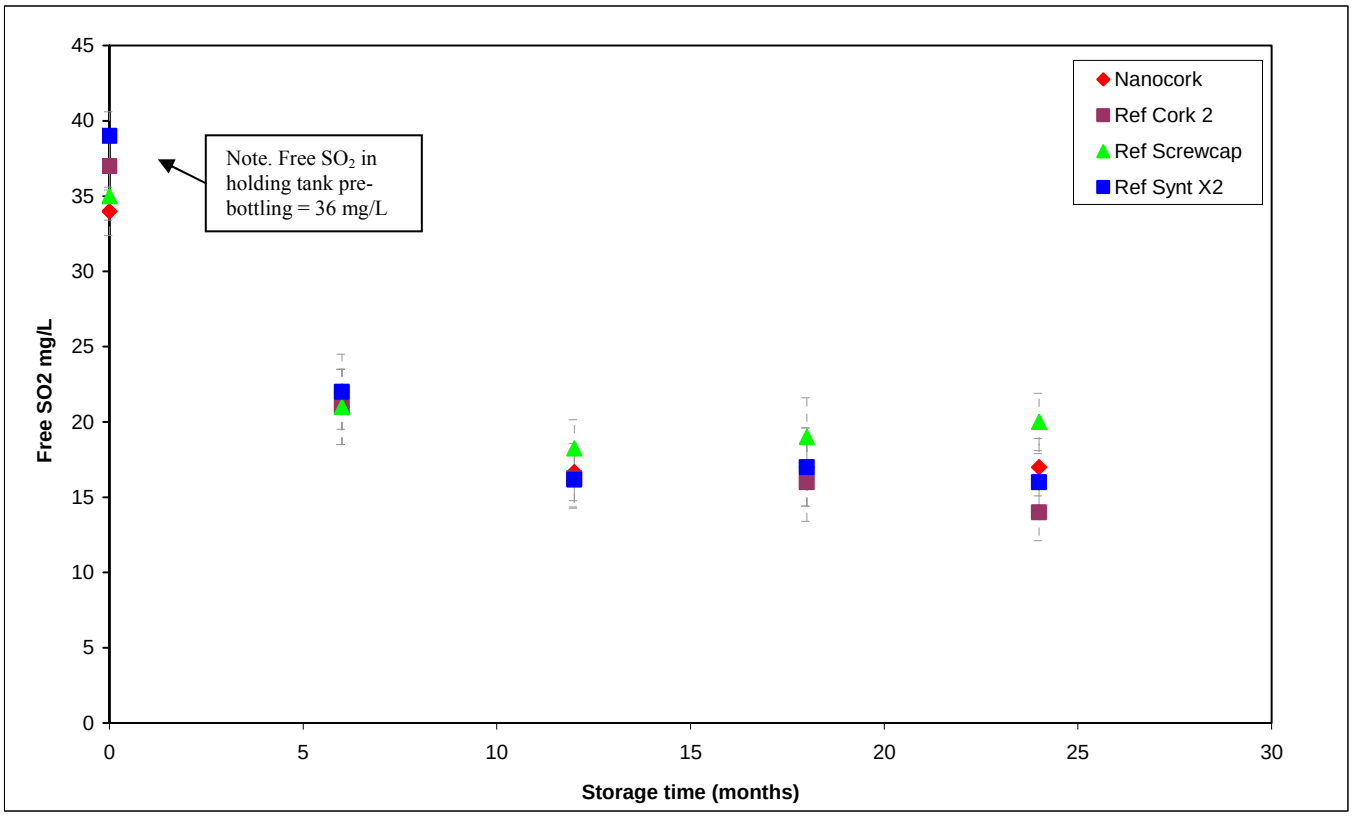

Figure 1. Free SO_2 *vs.* storage time. Error bars denote LSD.

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3.2 Chemical analysis – Total SO_2

Results for total SO_2 at the 24 month timepoint presented below in Figure 2 show the following:

- After starting at a concentration of 121mg/L, total SO_2 concentrations for the three reference closures and the Nanocork over the 24 month storage period were observed to undergo a rapid decrease over the first 6 months, following which total SO_2 values for the Nanocork, Reference Screwcap and Reference Synthetic closures stabilised, whilst the Reference Two natural cork experienced a gradual decline;
- The Reference Screwcap closure generally sustained a higher level of total SO_2 than the other closures throughout the trial;
- Whilst there is an apparent scatter of data at the zero timepoint, all analyses at this point were within the acceptable tolerances associated with FIA analysis of total SO_2. This is discussed in further detail later in the report;
- No statistically significant differences ($p>0.95$) were observed between total SO_2 values of the Nanocork, Reference Screwcap and Reference Synthetic closures; and
- Statistically significant differences ($p>0.95$) were observed between the total SO_2 results of the Nanocork and the Reference Two natural cork, with the Nanocork having a higher total SO_2 value.

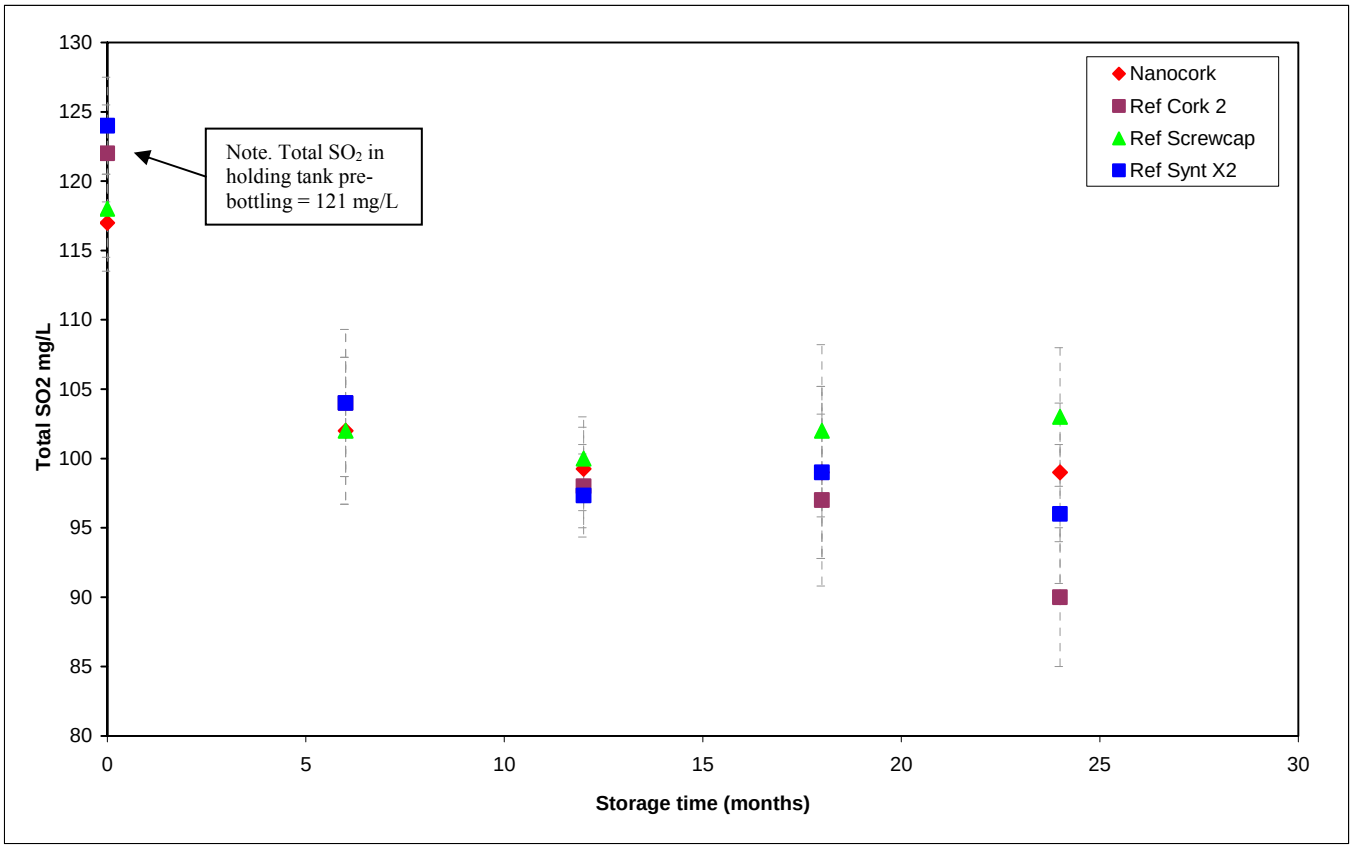

Figure 2. Total SO_2 *vs*. storage time. Error bars denote LSD.

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3.3 Chemical analysis – OD_{420}

The OD_{420} results presented below in Figure 3 show the following:

- The overall trend of the past 24 months for the Nanocork and reference closures was to increase in OD_{420};
- Statistically significant differences ($p>0.95$) were observed between the OD_{420} results of the Nanocork and the Reference Screwcap, with the Nanocork having a higher OD_{420}; and
- Statistically significant differences ($p>0.95$) were observed between the OD_{420} results of the Nanocork Reference Two natural cork and the Reference Synthetic closure, with the Nanocork having a lower OD_{420}.

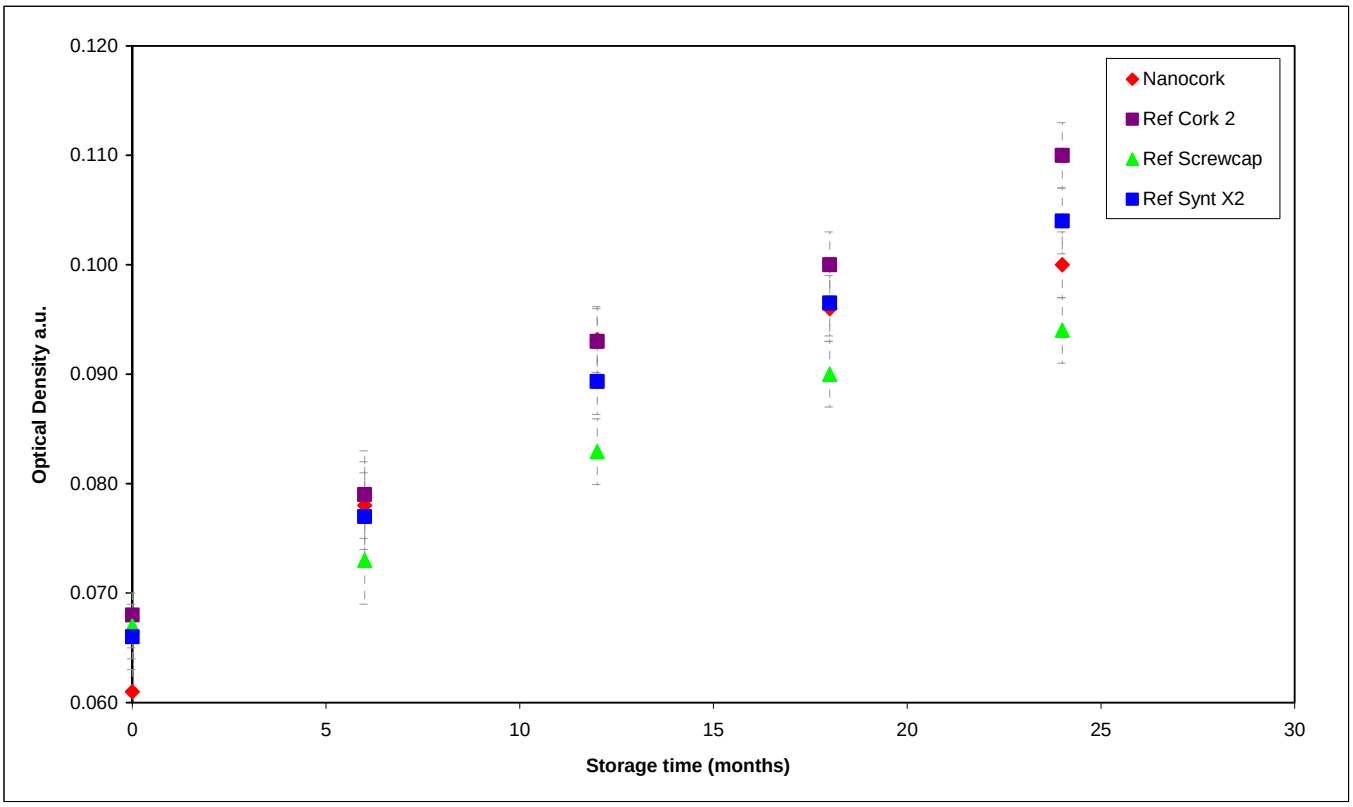

Figure 3. OD 420nm *vs*. storage time. Error bars denote LSD.

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3.4 Chemical analysis – Low molecular weight sulfur compounds

Low molecular weight sulfur compound analyses at the 24 month timepoint presented in Figure 4 show the following:

- Decreased concentrations of carbon disulfide and dimethyl sulfide and an increased concentration of hydrogen sulfide were observed under the Nanocork and reference closures since the 18 month timepoint;

- The concentration of methanethiol in the wine under the Reference Screwcap remained steady at 4µg/L since the 18 month timepoint, approximately double the aroma threshold of 1.5µg/L;

- Statistically significant differences ($p>0.95$) were observed between the hydrogen sulfide results of the Nanocork and the Reference Synthetic closure, with the Nanocork having a lower hydrogen sulfide concentration; and

- Statistically significant differences ($p>0.95$) were observed between the methanethiol results of the Nanocork and the Reference Screwcap closure, with the Nanocork having a lower methanethiol concentration.

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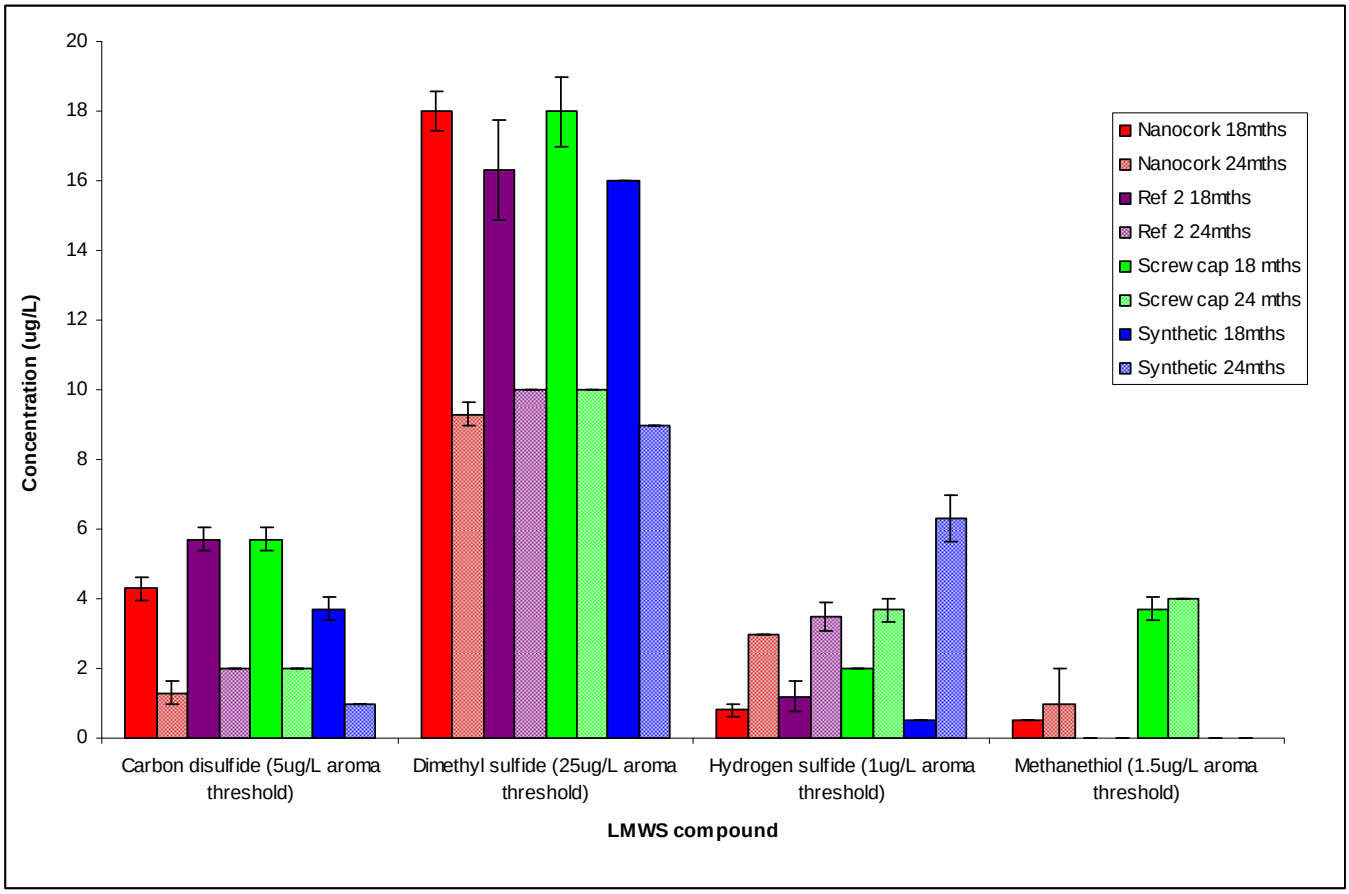

Figure 4. Low molecular weigh sulfur analyses showing compounds detected at 18 and 24 months. Note. The low molecular weight sulfur compound aroma thresholds were developed using highly trained sensory panellists. The aroma threshold of most wine consumers would be greater than these values, usually at least 2 times. Error bars denote Standard Error between the replicates.

3.5 Sensory analysis

From the analysis of variance of the sensory data, it was found that there were significant differences among the closures studied for all attributes. There were no statistically significant differences amongst the replicates.

The data from the attributes that were statistically significant across all four closures tested are presented for each replicate in Appendix 2, and the mean values for the closures are provided in Figure 5. Figure 5 also provides an LSD value.

Significant differences between the closures for sensory attributes scoring ≥1* are summarised below in Table 4.
* Note. Sensory attributes scoring <1 are considered to be minor/less important components of the wine flavour and aroma.

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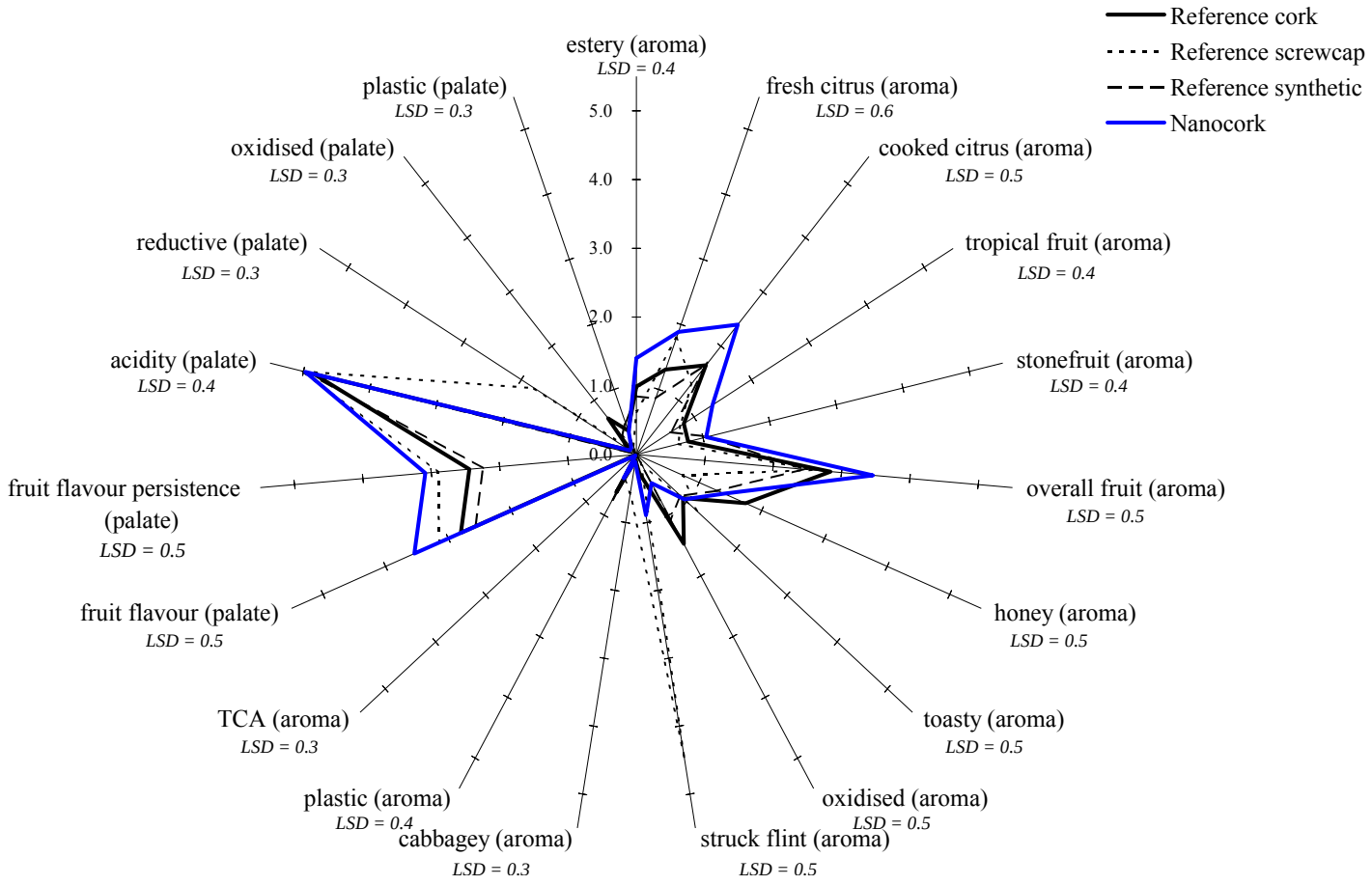

Figure 5: Radar plot showing mean values of aroma and palate attributes rated for the three reference closures and the Nanocork closure. LSD: least significant difference. Each value is the mean score from four replicates of each closure presented to ten judges.

Table 4. Significant differences between the closures for sensory attributes scoring ≥1. ref2 = Reference Two natural cork, sc = Reference Screwcap, syn = Reference Synthetic cork, nano = ACI Nanocork.

Closure	estery (aroma)	fresh citrus (aroma)	cooked citrus (aroma)	tropical fruit (aroma)	Stone fruit (aroma)	overall fruit (aroma)	honey (aroma)	oxidised (aroma)	struck flint (aroma)	fruit flavour (palate)	fruit flavour persist-ence	Re-ductive (palate)
Nanocork	>ref2 >sc >syn	>syn	>ref2 >sc >syn	>ref2 >sc >syn	>sc	>ref2 >sc >syn	>sc	<ref2 <syn	>ref2 <sc >syn	>ref2 >syn	>ref2 >syn	<sc
Reference cork	<nano	-	<nano	<nano	-	<nano	-	>nano	<nano	<nano	<nano	-
Reference screwcap	<nano	-	<nano	<nano	<nano	<nano	<nano	-	>nano	-	-	>nano
Reference synthetic	<nano	<nano	<nano	<nano	-	<nano	-	>nano	<nano	<nano	<nano	-

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4.0 Discussion

Results of this 24 month study revealed several differences between the chemical analysis data and sensory properties of some of the wines. The following issues highlighted by these results are noteworthy of discussion:

- The performance of the Nanocork closure relative to the reference closures;
- The development of reductive characters;
- Apparent observation of closure variability at the zero timepoint; and
- Shelf life potential of wines due to closure type.

4.1 Nanocork versus reference closures

Relative performance of the Nanocork versus the three reference closures is best summarised in the Principal Component Analysis (PCA) biplot below (Figure 6), which describes the relative similarity of closures based upon both their chemical and sensory analysis data.

The descriptors developed for the wines under the different closure technologies are shown as small red dots on the graph. Closures are represented by larger coloured dots. Each large coloured dot represents the average results for a closure technology tasted 24 months after bottling. The direction of the dot from the origin indicates the relevant attributes describing that wine under that closure. The further the dot from the origin – the greater the intensity of the attributes that were recorded.

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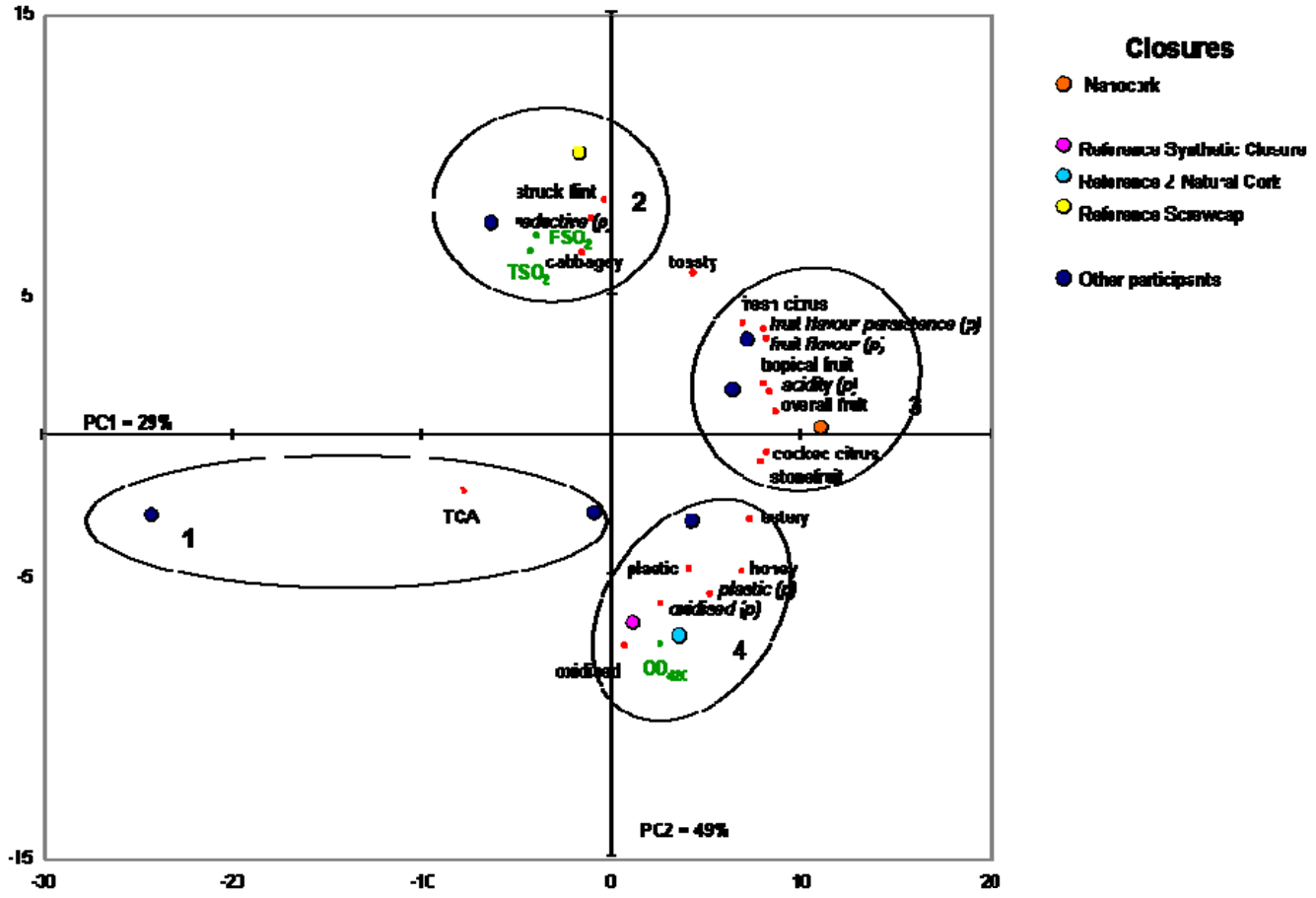

Figure 6. Twenty four month PCA biplot showing similarities between the Nanocork, reference and other trial participant's closures based upon their chemical and sensory attributes. PC1 & PC2 = 79%.

PCA analysis of the wines under the different closures at the 24 month timepoint (Figure 6) yielded four main clusterings, namely:

1. Wines affected by TCA;
2. Wines with a high free and total SO_2, coupled with reductive sensory characteristics;
3. Wines showing intense and persistent fresh fruit on the nose and palate; and
4. Wines showing a higher OD_{420}, coupled with developed sensory characters such as honey and esters, ranging to oxidised characters.

According to the PCA biplot in Figure 6, following 24 months of storage, the wine bottled under the Nanocork fell into Cluster 3, and therefore, displayed intense and persistent fresh fruit on the nose and palate.

In comparison to the wines under the Reference Two natural cork and Reference Synthetic closures which fell into Cluster 4, the wine under the Nanocork was therefore, quite different. Where the wine under the Nanocork displayed intense and persistent fresh fruit on the nose and palate, the wines under the Reference 2 natural cork and Reference Synthetic closures displayed honey, estery and oxidised characters.

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The wine that developed under the Nanocork closure was also completely different to that of the Reference Screwcap, which was found to belong to Cluster 2. Where the wine bottled under the Reference Screwcap displayed struck flint aroma, a reductive palate, and little fruit intensity, the wine bottled under the Nanocork displayed elevated fruit intensity and persistence of fruit flavour.

4.2 The development of reductive characters

The relationship between reductive character development and the presence of low molecular weight sulfur compounds is best summarised in the Principal Component Analysis (PCA) biplot below (Figure 7), which describes the relative similarity of closures based upon both their chemical and sensory analysis data.

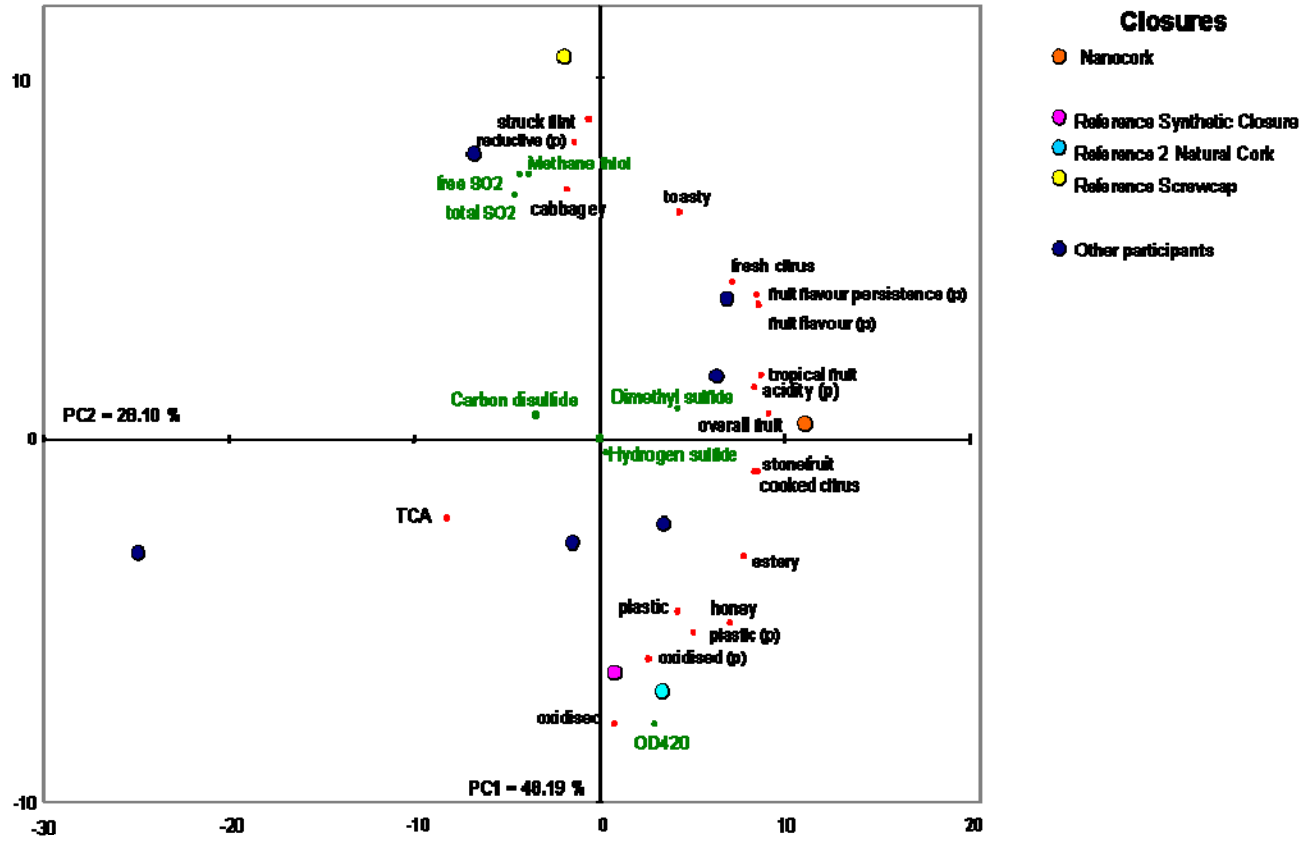

Figure 7. Twenty four month PCA biplot showing relationship between LMWS compounds, SO_2, OD_{420} and sensory properties of the Nanocork, reference and other trial participant's closures. PC1 & PC2 = 76%.

Of the four low molecular weigh sulfur compounds detected in the wines bottled under the Nanocork and reference closures, methanethiol was the only compound that appeared to correlate with the sensory analysis results. This is best demonstrated by the Reference Screwcap in Figure 7, which shows a correlation between elevated levels of struck flint and cabbagey aromas, a reductive palate, high free and total SO_2 concentrations and methanethiol.

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4.3 Apparent observation of closure variability at the Zero timepoint

Throughout the period of this trial, several participants have fielded questions about why the free and total SO_2 data generated at the zero timepoint (24 – 48 hours after bottling) appears to be widely scattered compared to the data at other timepoints. This can be readily explained by looking at how the samples were analysed at the zero timepoint compared to subsequent timepoints.

Samples at the zero timepoint were submitted for SO_2 analysis via FIA in sequential blocks of 12 replicate closures, rather than in a randomised order. For any run of samples on the FIA, a standard wine is analysed to check instrument performance, the acceptable range for which is ±4mg for SO_2 values < 80mg/L and ±5% for SO_2 for values > 80mg/L. During the zero timepoint SO_2 analyses, there was an upwards (but acceptable in terms of method tolerance for the control wine) drift in SO_2 values. Being analysed in sequential blocks, this led to some of the closures recording apparently higher free and total SO_2 levels later in the run when compared to other wines analysed earlier in the run. This was identified following analysis of the data at the zero timepoint, leading to all analyses being conducted in a randomised run order at subsequent timepoints.

Figure 6 below explains this more clearly:

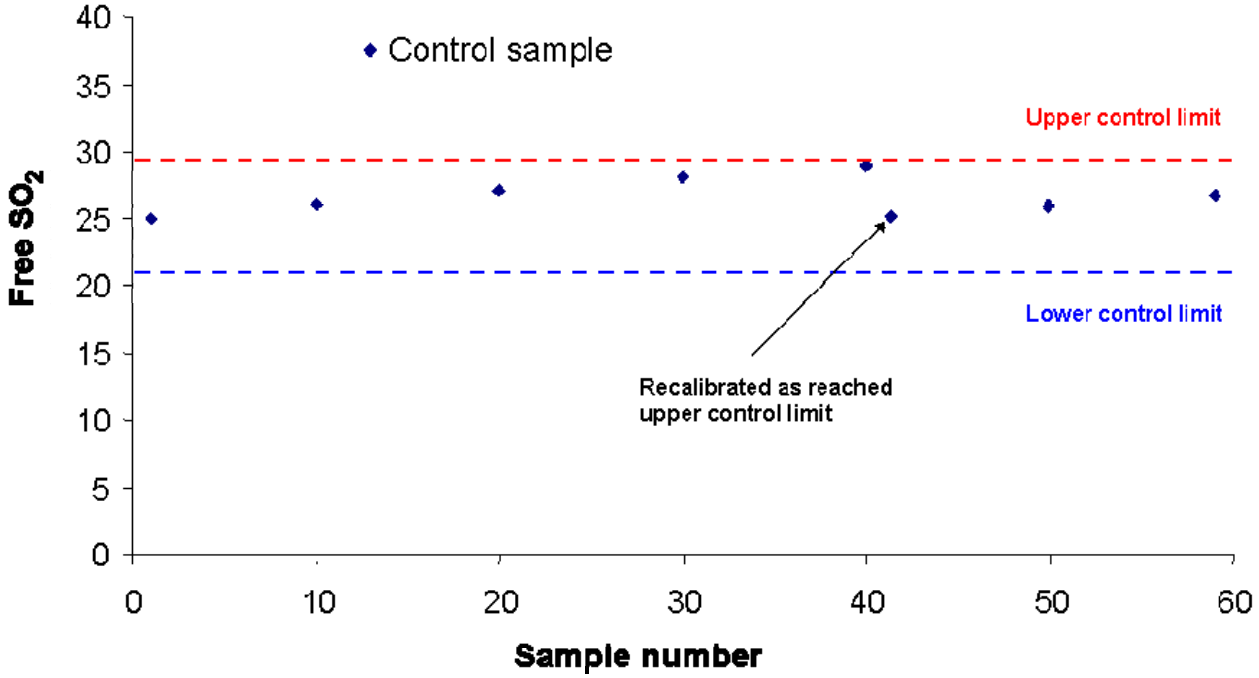

Figure 6. Explanation of apparent drift in SO_2 levels at the zero timepoint,

Figure 6 shows a typical run of SO_2 samples on the FIA, with the control sample analysed every 10 samples. Therefore, according to analytical uncertainty, the control sample is within specification when between 21 and 29 mg/L. During the run in this example, the free SO_2 drifted upwards over 40 samples from 25 mg/L to the upper control limit of 29mg/L. Following this, the FIA was re-calibrated at sample 41, then the run was continued. SO_2 results for the samples analysed earlier in the run were therefore, slightly lower than those analysed towards sample 40, but still within specification of the method.

Zero timepoint free and total SO_2 values measured at the bottling facility were determined to be 36/121, which correlate well with our results.

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4.4 Shelf life potential of wines due to closure type

Shelf life of a wine is influenced by a combination of factors including vinification, packaging methodology, storage, transport conditions and importantly, closure selection.

As a rule of thumb, wines have reached the end of their shelf life and begin showing obvious sign of oxidation when free SO_2 levels fall below 10mg/L (Godden, 2001).

Whilst none of the wines in this study as yet have reached the theoretical 10mg/L free SO_2 threshold, it is apparent through their enhanced ratings in oxidative characters in the sensory data that the Reference 2 natural cork and the Reference Synthetic closure are showing definite signs of nearing their shelf lives. The Nanocork however, is still producing a wine with intense and persistent fresh fruit characters on the nose and palate, suggesting it has further to go before reaching its shelf life.

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5.0 Conclusion

Based on the experimental results of the 24 month time point of the closure trial, it is possible to conclude the following:

- The Nanocork at the 24 month timepoint was found to produce a fresh, intense, fruity wine with long persistence on the palate. In contrast, the Reference Two and Reference Synthetic closures produced relatively similar wines to each other, characterised by honey, esters and oxidation characters and a higher OD_{420} than other closures entered in the trial. The enhanced oxidation characters exhibited by the Reference Two and Reference Synthetic closures suggest that they are approaching the end of their shelf lives, whereas the fresh fruity characters of the Nanocork suggest it has further to go before reaching its shelf life.

- When compared to the Reference Screwcap, the Nanocork displayed fresh fruit, a long palate length and enhanced fruit intensity. In contrast, the Reference Screwcap has moderate levels of reductive characters, in particular struck flint, and a lower fruit intensity; and

- Reductive characters were observed under the Reference Screwcap closure along with elevated levels of free and total SO_2 and the compound methanethiol.

Note:
The results in this trial are subject to the methods and equipment that were used for preparation of samples and their analysis. Different results may be obtained for testing where alternative methods and equipment are used.
The results in this report describe technical performance only and should not be used to suggest endorsement by AWRI of any of the products tested. Where the results of this report are cited as part of any product endorsement, it is recommended that the form and presentation of the data are provided to the AWRI for review first. Where this does not occur, the AWRI reserves the right to request withdrawal of the material and to seek suitable remedy from the author of the relevant material where appropriate.

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Attachments

APPENDIX 1 Twenty four month testing, Chemical data

APPENDIX 2 Twenty four month testing, Sensory evaluation

References

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APPENDIX 1: Twenty four month testing – Chemical data

Table 1.Twenty four month testing. Raw free & total SO_2, OD_{420} results for reference and Nanocork closures.

Closure	Initial	Initial	Initial	24 months	24 months	24 months
Reference Screwcap	Free SO_2	Total SO_2	OD_{420}	Free SO_2	Total SO_2	OD_{420}
(Saran tin/Alcan)						
	mg/L	mg/L	a.u.	mg/L	mg/L	a.u.
Mean	**35**	**118**	**0.067**	**20**	**103**	**0.094**
Std Error	0.21	0.5	0.001	0.23	0.57	0
N	12	12	12	12	11	11
Reference 2	Free SO_2	Total SO_2	OD_{420}	Free SO_2	Total SO_2	OD_{420}
Natural Cork						
	mg/L	mg/L	a.u.	mg/L	mg/L	a.u.
Mean	**37**	**122**	**0.068**	**14**	**90**	**0.110**
Std Error	0.13	0.58	0	0.68	1.7	0.001
N	12	12	12	12	12	12
Reference Synthetic (Supremecorq X2)	Free SO_2	Total SO_2	OD_{420}	Free SO_2	Total SO_2	OD_{420}
	mg/L	mg/L	a.u.	mg/L	mg/L	a.u.
Mean	**39**	**124**	**0.066**	**16**	**96**	**0.104**
Std Error	0.15	0.58	0	0.17	0.79	0
N	12	12	12	12	12	12
ACI Nanocork	Free SO_2	Total SO_2	OD_{420}	Free SO_2	Total SO_2	OD_{420}
	mg/L	mg/L	a.u.	mg/L	mg/L	a.u.
Mean	**34**	**117**	**0.061**	**17**	**99**	**0.098**
Std Error	0.18	0.63	0	0.34	0.84	0
N	12	12	12	12	12	11

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APPENDIX 1: Twenty four month testing – Chemical data

Table 2. Twenty four month testing. Low molecular weight sulfur compound results for reference and Nanocork closures.

	Carbon disulfide (µg/L)	Diethyl disulfide (µg/L)	Diethyl sulfide (µg/L)	Dimethyl disulfide (µg/L)	Dimethyl sulfide (µg/L)	Ethanethiol (µg/L)	Ethyl thioacetate (µg/L)	Hydrogen sulfide (µg/L)	Methanethiol (µg/L)	Methyl thioacetate (µg/L)
Ref Synt X2 43	1	Not Detected	Not Detected	Not Detected	9	Not Detected	Not Detected	7	Not Detected	Not Detected
Ref Synt X2 116	1	Not Detected	Not Detected	Not Detected	9	Not Detected	Not Detected	7	Not Detected	Not Detected
Ref Synt X2 249	1	Not Detected	Not Detected	Not Detected	9	Not Detected	Not Detected	5	Not Detected	Not Detected
Ref Screwcap 150	2	Not Detected	Not Detected	Not Detected	10	Not Detected	Not Detected	4	4	Not Detected
Ref Screwcap 228	2	Not Detected	Not Detected	Not Detected	10	Not Detected	Not Detected	4	4	Not Detected
Ref Screwcap 247	2	Not Detected	Not Detected	Not Detected	10	Not Detected	Not Detected	3	4	Not Detected
Ref Cork 2 194	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
Ref Cork 2 199	2	Not Detected	Not Detected	Not Detected	10	Not Detected	Not Detected	3	Not Detected	Not Detected
Ref Cork 2 289	2	Not Detected	Not Detected	Not Detected	10	Not Detected	Not Detected	4	Not Detected	Not Detected
ACI Nano 83	2	Not Detected	Not Detected	Not Detected	10	Not Detected	Not Detected	3	Not Detected	Not Detected
ACI Nano 183	1	Not Detected	Not Detected	Not Detected	9	Not Detected	Not Detected	3	3	Not Detected
ACI Nano 257	1	Not Detected	Not Detected	Not Detected	9	Not Detected	Not Detected	3	Not Detected	Not Detected

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APPENDIX 2: Twenty four month testing – Sensory evaluation aroma terms

Closure	Rep	*estery*	*fresh citrus*	*cooked citrus*	*tropical fruit*	*Stone fruit*	*overall fruit*	*honey*	*toasty*	*oxidised*	*plastic*	*TCA*	*struck flint*	*cabbage y*
Nanocork	1	*1.5*	*1.7*	*2.7*	*1.1*	*1.0*	*3.4*	*1.4*	*0.5*	*0.1*	*0.3*	*0.1*	*1.3*	*0.1*
	2	*1.5*	*2.9*	*1.9*	*1.7*	*1.3*	*4.0*	*1.5*	*1.4*	*0.0*	*0.4*	*0.0*	*0.7*	*0.1*
	3	*1.5*	*1.9*	*2.1*	*1.4*	*0.9*	*3.3*	*1.5*	*1.4*	*1.1*	*0.3*	*0.0*	*0.8*	*0.1*
	4	*1.2*	*1.1*	*2.9*	*1.1*	*1.1*	*3.2*	*1.3*	*0.6*	*0.7*	*0.6*	*0.0*	*0.8*	*0.1*
Reference cork	1	*1.1*	*1.6*	*1.5*	*1.0*	*0.9*	*3.3*	*2.1*	*1.2*	*1.4*	*0.2*	*0.0*	*0.2*	*0.0*
	2	*0.7*	*1.2*	*2.0*	*0.8*	*0.5*	*2.7*	*1.3*	*0.8*	*1.5*	*0.7*	*0.0*	*0.7*	*0.0*
	3	*0.8*	*1.3*	*1.6*	*0.7*	*1.0*	*2.7*	*2.2*	*0.9*	*1.7*	*0.4*	*0.0*	*0.1*	*0.2*
	4	*1.4*	*1.1*	*1.5*	*0.9*	*0.7*	*2.6*	*1.3*	*0.8*	*1.4*	*1.1*	*0.0*	*0.0*	*0.1*
Reference screwcap	1	*0.6*	*2.1*	*1.3*	*0.7*	*0.7*	*2.8*	*0.8*	*1.4*	*0.0*	*0.6*	*0.0*	*5.0*	*0.4*
	2	*0.7*	*1.4*	*1.3*	*0.6*	*0.4*	*2.2*	*0.9*	*1.5*	*0.0*	*0.0*	*0.0*	*3.8*	*0.7*
	3	*0.7*	*2.3*	*1.2*	*0.8*	*0.7*	*2.9*	*0.3*	*1.0*	*0.6*	*0.5*	*0.0*	*4.5*	*0.2*
	4	*0.5*	*1.4*	*1.4*	*1.0*	*0.8*	*2.5*	*0.9*	*1.0*	*0.0*	*0.5*	*0.0*	*4.6*	*1.0*
Reference synthetic	1	*0.9*	*0.9*	*1.5*	*0.6*	*0.7*	*2.6*	*1.6*	*1.2*	*0.8*	*0.7*	*0.0*	*0.0*	*0.0*
	2	*1.1*	*0.8*	*1.9*	*0.5*	*1.3*	*2.3*	*1.5*	*0.9*	*1.4*	*0.6*	*0.2*	*0.0*	*0.0*
	3	*0.7*	*1.0*	*1.4*	*0.6*	*0.9*	*2.2*	*1.0*	*0.6*	*1.3*	*0.8*	*0.0*	*0.0*	*0.0*
	4	*0.8*	*0.7*	*2.0*	*0.7*	*1.5*	*2.7*	*1.1*	*0.8*	*0.8*	*0.8*	*0.0*	*0.2*	*0.0*

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APPENDIX 2: Twenty four month testing – Sensory evaluation palate terms

Closure	Rep	*acidity*	*overall fruit*	*fruit flavour persistence*	*plastic*	*reductive*	*oxidised*
Nanocork	1	*4.8*	*3.7*	*2.9*	*0.8*	*0.2*	*0.0*
	2	*5.3*	*3.9*	*3.3*	*0.3*	*0.2*	*0.0*
	3	*5.0*	*3.4*	*3.2*	*0.1*	*0.0*	*0.3*
	4	*4.8*	*3.2*	*2.9*	*0.3*	*0.0*	*0.2*
Reference cork	1	*4.6*	*2.8*	*2.7*	*0.4*	*0.4*	*0.1*
	2	*5.1*	*2.9*	*2.4*	*0.6*	*0.1*	*0.7*
	3	*4.7*	*2.7*	*2.5*	*0.1*	*0.0*	*0.9*
	4	*4.9*	*2.8*	*2.2*	*0.4*	*0.0*	*0.9*
Reference screwcap	1	*5.3*	*3.5*	*3.1*	*0.2*	*1.8*	*0.0*
	2	*4.7*	*3.1*	*2.8*	*0.0*	*1.6*	*0.0*
	3	*5.0*	*2.8*	*2.5*	*0.1*	*1.8*	*0.0*
	4	*5.0*	*3.1*	*3.2*	*0.3*	*1.8*	*0.2*
Reference synthetic	1	*5.0*	*2.8*	*2.4*	*0.5*	*0.0*	*0.1*
	2	*4.9*	*2.3*	*2.1*	*0.3*	*0.0*	*0.3*
	3	*4.9*	*2.4*	*2.3*	*0.4*	*0.0*	*0.6*
	4	*5.0*	*2.8*	*2.2*	*0.8*	*0.0*	*0.3*

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