



Commercial Closure trial – 30 Month Progress Report Performance Testing of Procork's Membrane Cork Closure

Dear Dr Gregor Christie,

PROTOCOL

The protocol has not changed since the commencement of the commercial closure trial. Where relevant information to this stage of the work is used a brief description is provided.

Cartons containing 12 randomised bottles were selected from the cartons previously stored inverted in the cellar on pallets with approximately 64 cartons to the pallet.

30 MONTH TESTING

After 31 months storage, random samples of each closure were tested for free sulfur dioxide, total sulfur dioxide and the optical density at 420 nm. Further samples were used for sensory evaluation. The results and number of replicates for each test are identified in the tables of results in Tables 2 for Chemical/Physical Data, and Tables 3 and 4 for Sensory Data.

EXPERIMENTAL

Methods of chemical analysis

Optical density was determined by measurement of the absorbance at 420nm on a Varian UV/visible spectrophotometer. Free and total sulfur dioxide was measured using the aspiration method. Most of these methods are approved methods covered by the laboratories NATA accreditation. All analyses were performed by trained staff and were performed in conjunction with quality assurance measures including standards, blanks, duplicates and control samples where appropriate. The quality control measures were required to meet established criteria before acceptance of the analytical data.

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 on the sensory panel at the 24 month assessment of these wines. An initial discussion session was held, with the tasters assessing six of the wines from the current study. These wines were selected based on a preliminary evaluation to identify those samples displaying the largest sensory differences. The tasters assessed the wines in silence, followed by a discussion regarding the sample's characteristics, to confirm the attributes that would be rated in the subsequent formal sessions. A list of the terms that was agreed upon by the panellists is given in Table 1..

Note that the same attributes as rated at 24 months were used with the exception of herbaceous and the aroma term 'citrus' has been split into 'fresh citrus' and 'cooked citrus'.

Table 1. Sensory Attributes Scored.

Attribute	Definition
<i>Aroma</i>	
Estery	definition: estery, bubblegum, tropical fruit
Floral	definition: perfumed, musk
Fresh Citrus	definition: lemon, lime
Cooked Citrus	definition: orange peel, marmalade
Herbaceous	definition: grassy, green bean
Overall fruit	definition: citrus, pineapple
Honey	-
Toasty	definition: aged Semillon aroma, complex buttered, toasty
Oxidised	definition: bruised apple, aldehyde
Glue/Plastic/Solvent	-
TCA	definition: musty, mouldy
Cork wood	definition: woody, corks soaked in wine
Struck flint/rubber	definition: rubber, struck match/flint
H ₂ S/cabbagey	definition: rotten egg, cabbage, sewerage
<i>Palate</i>	
Acidity	
Overall Fruit	
Fruit Flavour Persistence	
Glue/Plastic/Solvent	
TCA	
Cork wood	
Reduced	definition: rubbery, struck match/flint, cabbage

Following the discussion session, one practice rating session with 10 samples was carried out in isolated tasting booths using the same format as for the formal sessions.

For the formal sessions, samples were assessed in blind tasting conditions using standardised procedures. Fifteen wines were assessed at a session, being one example of each closure in the study and one spike or repeat for quality control purposes. Four bottles of each closure type were assessed over four sessions. The samples were presented to tasters in coded, covered XL5 (ISO standard) glasses, in a random order with a constant volume of wine in each glass (25mL). The tasters were instructed to assess each wine for aroma and then palate. The tests were carried out in the AWRI's purpose built sensory facility in isolated, temperature controlled, ventilated tasting booths under sodium colour masking lights, with temperature control between 22-24°C. Data was acquired using Fizz 2.00M software (Biosystemes, Couternon, France). The sensory evaluations were held over two consecutive days with sessions held in both the morning and afternoon of each day.

The panellists 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.

Data analysis was carried out using Genstat 6 (VSN International, UK). Analysis of variance was carried out testing for the effect of closure and bottle replicate nested within closure type, using a mixed model treating judges as a random effect. Due to a highly significant TCA effect a further analysis of variance, adjusting for the variation in TCA scores, ie treating TCA aroma ratings as a co-variate, was carried out. Mean values from this analysis of variance, together with Least Significant Difference value ($P=0.05$), were graphed in the form of a radar plot.

RESULTS AND DISCUSSION

Chemical analysis:

The levels of free and total sulfur dioxide in wine are considered to be critical parameters with respect to the stability of the wine and provides protection against oxidation and therefore accelerated development of the wine. As was evident from the AWRI research closure trial (Godden, 2002) losses of free and total sulfur

dioxide occur with time irrespective of the various closure types under trial. The losses over time in that trial were found to be less with the ROTE type closure than natural corks, technical corks and synthetic closures.

The changes observed in free and total sulfur dioxide during this trial for the reference closures are *comparable*, but not identical to the changes observed in the commercial trial reported at the 30 month post bottling period (Godden *et al.*, 2002).

The Procork closures have a slightly higher mean free and total sulphur dioxide than the Reference ROTE type closures although statistically the values are similar, this has resulted in similar low levels of oxidation, as measured by the increase in OD420 absorbance (Figure 3) for the ROTE type reference closures and the Procork membrane cork closures. This is expected, but cannot be compared with the original trial as the OD420 absorbance data have not been reported.

The Procork closures retained slightly more free and total sulphur dioxide than the Reference 2 and 3 closures resulting in a slightly lower level of oxidation, as measured by the increase in OD420 absorbance (Figure 3) than the reference 2 and 3 closures. Again this is expected, but cannot be compared with the original trial as the OD420 absorbance data have not been reported.

Sensory Analysis

From the analysis of variance of the sensory data, it was found that there were significant differences among the 10 closures for the aroma attributes: estery, floral, citrus, overall fruit, honey, oxidised, TCA and struck flint/rubber; and for the palate attributes: overall fruit, fruit flavour persistence and reduced. The other attributes rated did not differ significantly among the closures.

The data from the attributes that were statistically significant across all closures are presented for each replicate in Table 3 (reference closures) and Table 4 for the Procork membrane cork closure.

Figure 4 shows mean values and least significant differences for each of the attributes that were rated significantly different across the closures, except TCA and reduced. 'Reduced' on the palate is not shown on the figure due to the values being very close to zero. The most significant difference is for flint/rubber aroma, where the reference 2 and 3 closures were rated 0.3 and 0.4 respectively, whereas the ROTE closure was scored at **2.3** and Procork's closure was **0.7**. (A score of 1 is considered just detectable). Honey characters were rated lower in the ROTE (1.3) and Procork (1.5) closures than the Reference 2 (2.0) and 3 (1.5) closures, and oxidised characters were not perceptible in either the ROTE or Procork closures. The fresh citrus character (aroma) and fruit flavour persistence (palate) were higher in wine under the ROTE and Procork closures, than the Reference 2 and 3 closures.

The wine under the Procork membrane cork closure was rated highest amongst the perceived fruit levels, and the differences across the closures are not easily predicted based on the level of sulfur dioxide present. It must be noted that the tasters will undoubtedly be influenced to a degree by the other characters, such as oxidation/honey and reduced characters.

It should be noted that although the methods used for the sensory assessment at this time point were closely similar to those used at the previous assessments, assessors can vary in their responses from study to study, which means that comparing mean values across time periods could be potentially misleading. Comparisons among closures at a time point are more meaningful.

Note: While we have every confidence in these results, factors such as the manufacturing variations between batches have not been evaluated. This trial relates only to one style of wine and clearly winemakers should carefully test the shelf life of their product and the characteristics of their style of wine they wish to use.

References:

Godden P.W., Francis I.L., Field J., Gishen M., Coulter A. D., Valente P., Høj P.B. and Robinson E., Wine bottle closures: physical characteristics and effect on composition and sensory properties of a Semillon wine. Performance up to 20 months post-bottling. *Australian Journal of Grape and Wine Research*, 7, 64-105, 2001.

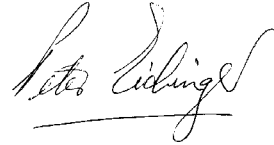
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Godden, P.W., Update on the AWRI trial of the technical performance of various types of wine bottle closure: Analysis of the concentration of sulfur dioxide at 21 and 24 months post bottling. Tech.Rev. 133:1-3; 2001.

Godden, P.W., Update on the AWRI trial of the technical performance of various types of wine bottle closure: Analysis of the concentration of sulfur dioxide at 30 months post bottling. Tech.Rev. 137:7-10; 2002.

Godden, P.W., Update on the AWRI trial of the technical performance of various types of wine bottle closure: Analysis of the concentration of sulfur dioxide at 36 months post bottling. Tech.Rev. 139:6-10; 2002.

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Manager-Analytical Service
5 November 2004

Attachments:

Chemical and Physical Data

Table 2. Summary Comparison of Initial and 30 Month Chemical and Physical Testing of Reference and Procork's Membrane Cork Closures.

Figure 1: Change in free Sulfur Dioxide in Wine bottled under Reference and Procork's Membrane Cork Closures.

Figure 2: Change in total Sulfur Dioxide in Wine bottled under Reference and Procork's Membrane Cork Closures.

Figure 3: Change in OD 420 in Wine bottled under Reference and Procork's Membrane Cork Closures.

Sensory Data

Table 3: 30 month sensory testing of Reference Closures

Table 4: 30 month sensory testing of Procork's Membrane Cork Closures

Figure 4: Radar / Spider Plot of Significant Sensory Attributes vs Procork's Membrane Cork Closure Material

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Table 2. Summary Comparison of Initial and 30 Month Chemical and Physical Testing on Reference and Procork's Membrane Cork Closure.

	Initial	30 month	Initial	30 month	Initial	30 month
ROTE	Free SO ₂ mg/L	Free SO ₂ mg/L	Total SO ₂ mg/L	Total SO ₂ mg/L	OD420 a.u.	OD420 a.u.
Mean	38	21.6	111	91.2	0.047	0.069
Std deviation	2	1.7	1	2.5	0.002	0.002
n	12	12	12	12	12	12
Reference 2	Free SO ₂ mg/L	Free SO ₂ mg/L	Total SO ₂ mg/L	Total SO ₂ mg/L	OD420 a.u.	OD420 a.u.
Mean	39	20.4	113	89.5	0.052	0.080
Std deviation	2	1.9	2	3.1	0.004	0.003
n	12	12	12	12	12	12
Reference 3	Free SO ₂ mg/L	Free SO ₂ mg/L	Total SO ₂ mg/L	Total SO ₂ mg/L	OD420 a.u.	OD420 a.u.
Mean	39	14.2	112	89.5	0.052	0.084
Std deviation	2	3.2	2	3.1	0.003	0.006
n	12	12	12	12	12	12
Procork's Membrane Cork Closure	Free SO ₂ mg/L	Free SO ₂ mg/L	Total SO ₂ mg/L	Total SO ₂ mg/L	OD420 a.u.	OD420 a.u.
Mean	39	23.5	113	94.1	0.051	0.075
Std deviation	2	1.0	3	2.0	0.003	0.002
n	12	12	12	12	12	12

Free Sulfur Dioxide

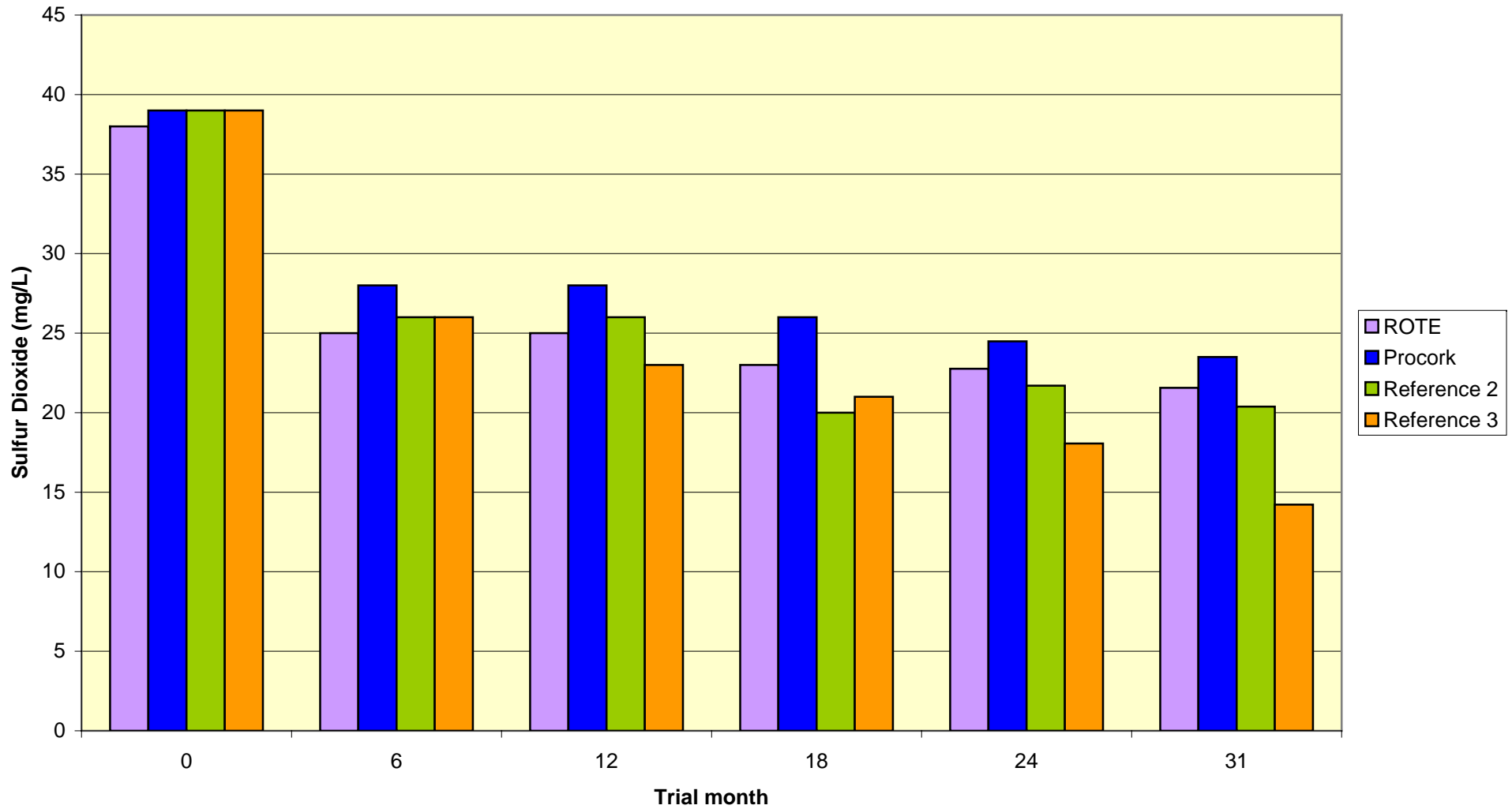


Figure 1: Change in free Sulfur Dioxide in Wine bottled under Reference and Procork’s Membrane Cork Closure.

Total Sulfur Dioxide

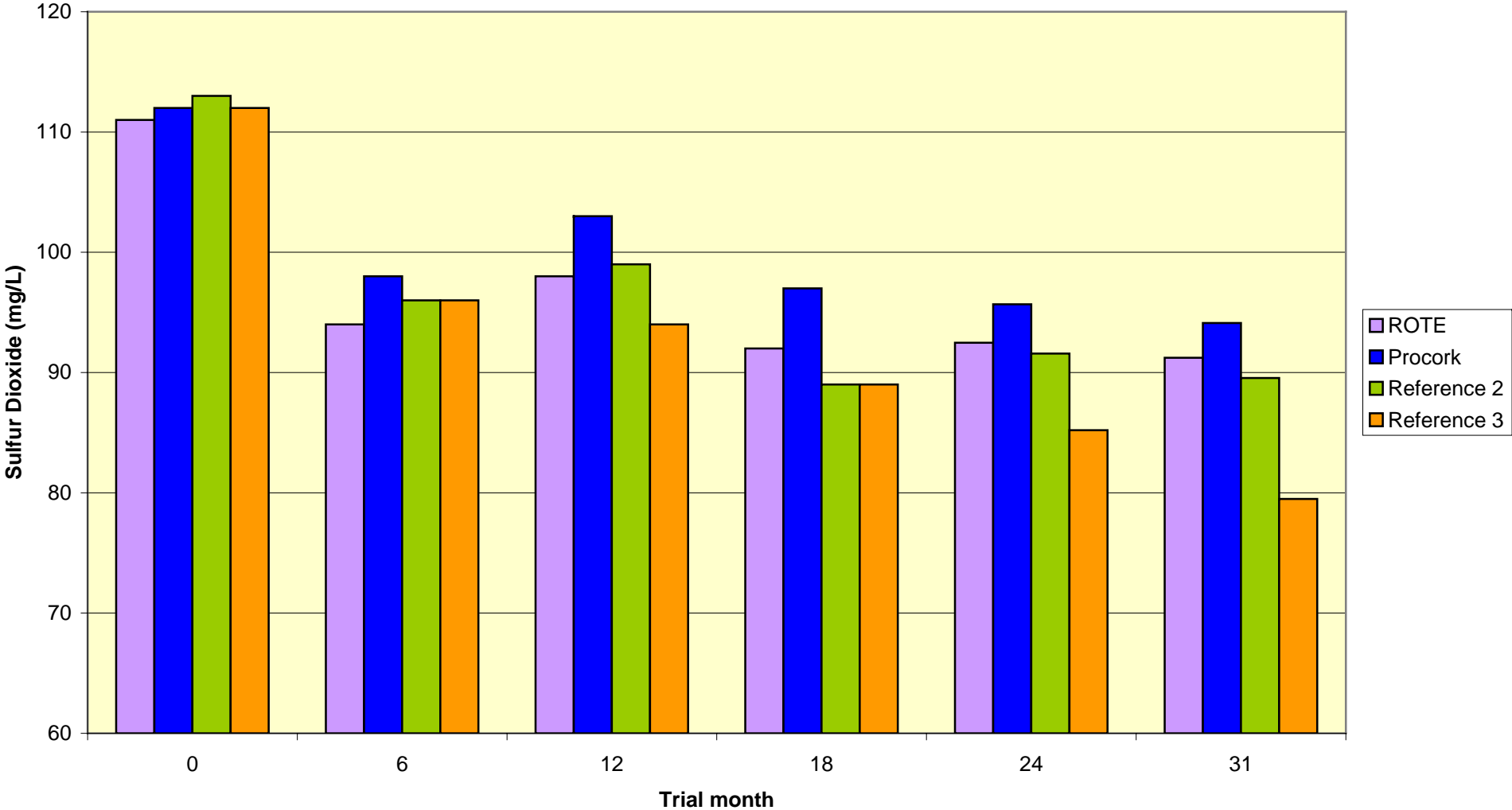


Figure 2: Change in total Sulfur Dioxide in Wine bottled under Reference and Procork’s Membrane Cork Closure.

OD 420

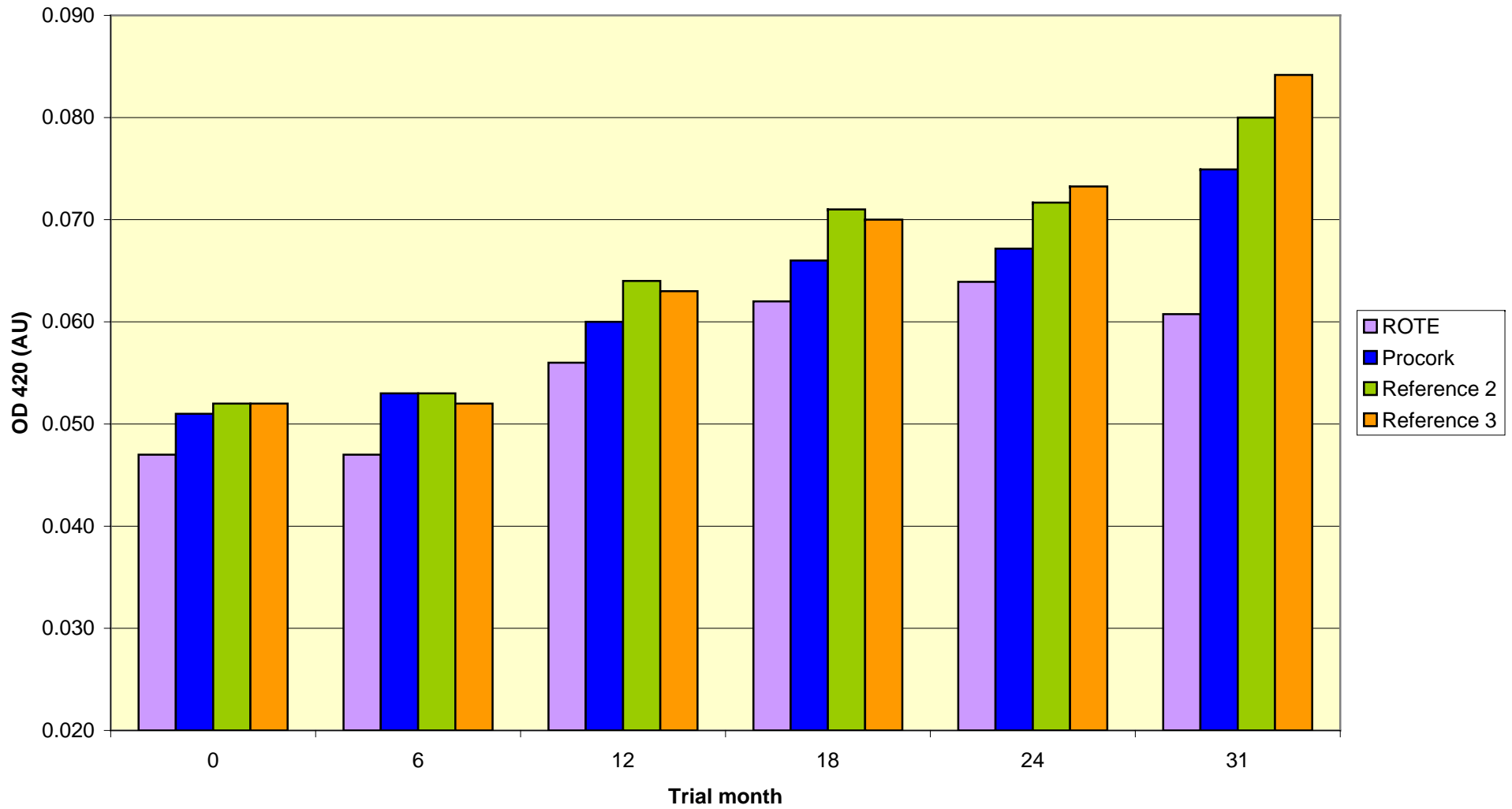


Figure 3: Change in OD 420 in Wine bottled under Reference and Procork's Membrane Cork Closures.

Table 3: 30 month sensory testing of Reference Closures

closure	replicate	estery (aroma)	floral (aroma)	fresh citrus (aroma)	cooked citrus (aroma)	overall fruit (aroma)	honey (aroma)	oxidised (aroma)	TCA (aroma)	struck flint/rubber (aroma)	H ₂ S/ cabbagey (aroma)	acidity (palate)	overall fruit flavour (palate)	fruit flavour persistence (palate)	TCA (palate)
ROTE (Low SO₂)	1	2.4	1.9	3.8	3.3	4.6	1.2	0.0	0.3	2.5	0.3	6.3	5.0	4.3	0.1
	2	2.2	1.2	2.9	3.1	3.9	1.3	0.5	0.0	2.4	0.3	5.9	4.7	4.0	0.0
	3	2.5	2.1	3.4	3.1	4.4	1.4	0.6	0.0	2.0	0.0	6.4	5.3	4.6	0.0
	4	2.0	1.3	3.7	3.5	4.4	1.3	0.4	0.1	2.3	0.3	6.1	5.2	4.9	0.1
	<i>Mean</i>	<i>2.3</i>	<i>1.6</i>	<i>3.4</i>	<i>3.2</i>	<i>4.3</i>	<i>1.3</i>	<i>0.4</i>	<i>0.1</i>	<i>2.3</i>	<i>0.2</i>	<i>6.2</i>	<i>5.0</i>	<i>4.5</i>	<i>0.1</i>
	<i>Std dev.</i>	<i>0.2</i>	<i>0.4</i>	<i>0.4</i>	<i>0.2</i>	<i>0.3</i>	<i>0.1</i>	<i>0.3</i>	<i>0.1</i>	<i>0.2</i>	<i>0.1</i>	<i>0.2</i>	<i>0.3</i>	<i>0.4</i>	<i>0.1</i>
Ref 2 cork	1	2.4	1.4	2.8	3.3	3.7	1.2	0.4	0.3	0.6	0.0	6.0	4.4	4.0	0.1
	2	2.4	1.4	2.7	2.9	3.8	2.2	1.3	0.1	0.1	0.1	6.0	4.1	3.7	0.1
	3	2.4	1.4	2.5	3.6	4.4	2.7	0.9	0.0	0.1	0.0	5.9	4.5	4.2	0.0
	4	1.9	1.6	1.9	3.2	3.4	1.8	1.9	0.6	0.3	0.0	6.0	4.0	3.8	0.4
	<i>Mean</i>	<i>2.3</i>	<i>1.5</i>	<i>2.5</i>	<i>3.3</i>	<i>3.8</i>	<i>2.0</i>	<i>1.1</i>	<i>0.2</i>	<i>0.3</i>	<i>0.0</i>	<i>6.0</i>	<i>4.3</i>	<i>3.9</i>	<i>0.2</i>
	<i>Std dev.</i>	<i>0.2</i>	<i>0.1</i>	<i>0.4</i>	<i>0.3</i>	<i>0.4</i>	<i>0.6</i>	<i>0.6</i>	<i>0.3</i>	<i>0.2</i>	<i>0.0</i>	<i>0.1</i>	<i>0.3</i>	<i>0.2</i>	<i>0.2</i>
Ref 3 cork	1	2.0	1.2	2.9	3.2	3.7	1.8	1.2	0.3	0.5	0.0	5.9	4.6	4.2	0.1
	2	2.3	1.9	2.7	3.8	4.3	2.0	1.2	0.0	0.6	0.3	6.4	4.6	4.4	0.0
	3	1.9	1.4	1.9	2.9	3.1	2.9	2.7	0.0	0.1	0.0	5.9	3.6	3.3	0.0
	4	2.5	1.6	2.2	3.0	3.7	1.9	0.9	0.1	0.3	0.0	6.2	4.3	3.9	0.1
	<i>Mean</i>	<i>2.2</i>	<i>1.5</i>	<i>2.4</i>	<i>3.2</i>	<i>3.7</i>	<i>2.2</i>	<i>1.5</i>	<i>0.1</i>	<i>0.4</i>	<i>0.1</i>	<i>6.1</i>	<i>4.3</i>	<i>3.9</i>	<i>0.1</i>
	<i>Std dev.</i>	<i>0.3</i>	<i>0.3</i>	<i>0.4</i>	<i>0.4</i>	<i>0.5</i>	<i>0.5</i>	<i>0.8</i>	<i>0.1</i>	<i>0.2</i>	<i>0.1</i>	<i>0.2</i>	<i>0.5</i>	<i>0.5</i>	<i>0.1</i>

Sensory data for each of the four replicate bottles assessed (mean scores of 10 judges), and mean data for each closure type, averaged across four replicates.

Table 4: 24 month sensory testing of Procork's Membrane Cork Closures

Sensory data for each of the four replicate bottles assessed (mean scores of 10 judges), and mean data for each closure type, averaged across four replicates.

closure	rep	estery (aroma)	floral (aroma)	fresh citrus (aroma)	cooked citrus (aroma)	overall fruit (aroma)	honey (aroma)	oxidised (aroma)	TCA (aroma)	struck flint/rubber (aroma)	H ₂ S/ cabbagey (aroma)	acidity (palate)	overall fruit flavour (palate)	fruit flavour persistence (palate)	TCA (palate)
Procork	1	2.8	1.9	3.7	3.0	4.7	1.1	0.2	0.3	0.9	0.0	5.9	5.1	4.6	0.1
	2	2.4	1.7	2.9	2.6	3.6	1.3	0.3	0.0	1.2	0.1	5.5	4.9	4.4	0.0
	3	2.2	1.7	3.7	3.4	4.7	1.7	0.6	0.0	0.6	0.0	5.3	5.0	4.7	0.0
	4	2.2	1.6	3.0	3.4	4.2	2.0	0.8	0.3	0.2	0.0	5.9	4.6	4.5	0.3
	<i>Mean</i>	<i>2.4</i>	<i>1.7</i>	<i>3.3</i>	<i>3.1</i>	<i>4.3</i>	<i>1.5</i>	<i>0.5</i>	<i>0.1</i>	<i>0.7</i>	<i>0.0</i>	<i>5.7</i>	<i>4.9</i>	<i>4.6</i>	<i>0.1</i>
	<i>Std dev.</i>	<i>0.3</i>	<i>0.1</i>	<i>0.5</i>	<i>0.4</i>	<i>0.5</i>	<i>0.4</i>	<i>0.3</i>	<i>0.2</i>	<i>0.4</i>	<i>0.1</i>	<i>0.3</i>	<i>0.2</i>	<i>0.1</i>	<i>0.1</i>

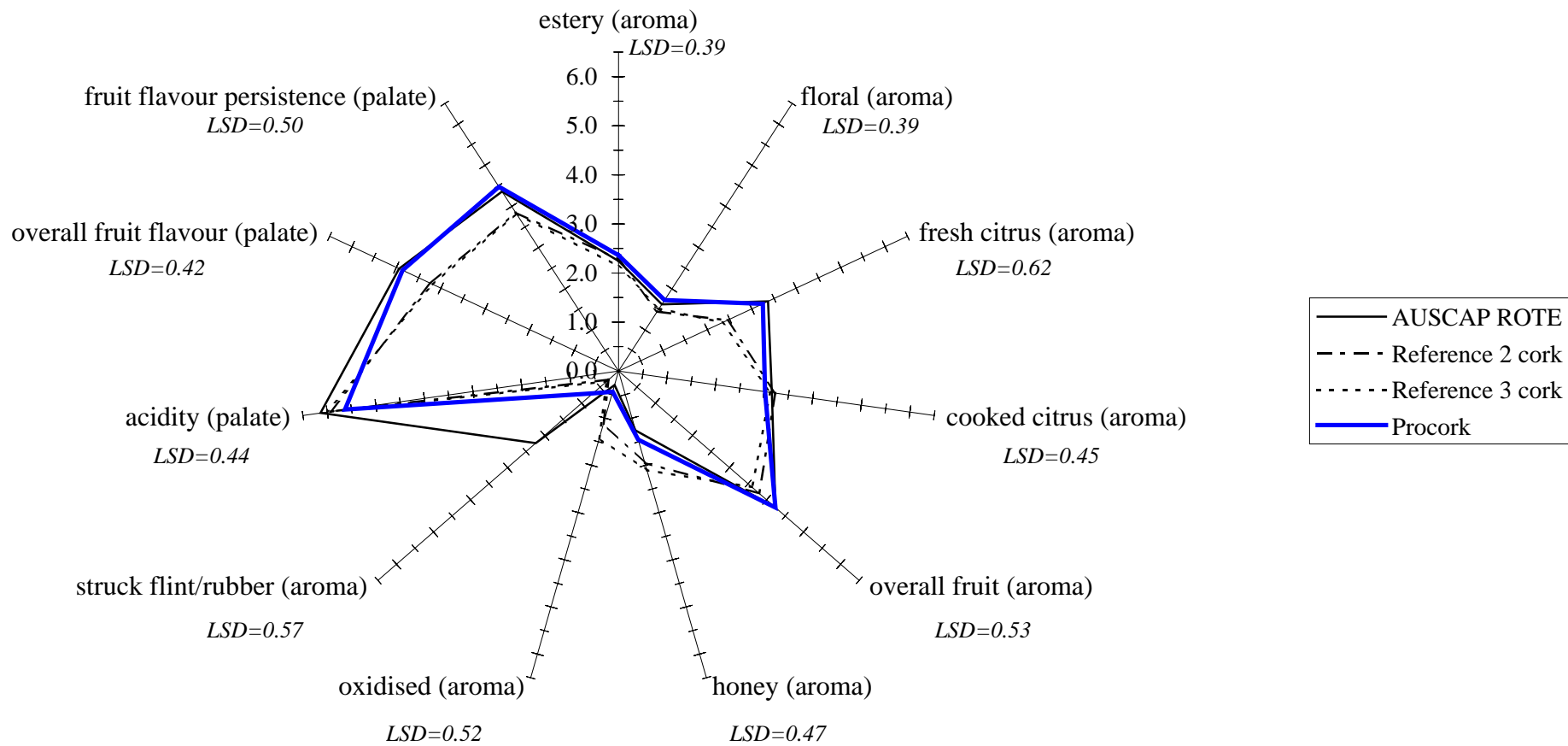


Figure 4: Radar / Spider Plot of Significant Sensory Attributes vs Procork Closure Material.

Only those attributes that were statistically significant from the ANOVA, adjusted for TCA scores, are shown, excluding reduced (palate). LSD: least significant difference. Each value is the mean score from four replicates of each closure presented to 10 judges.