



**Commercial Closure trial – 36 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.

36 MONTH TESTING

After 36 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 420 nm 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 laboratory's 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 nine judges was recruited, comprising AWRI staff with extensive experience in wine sensory evaluation, of which all had participated on the sensory panel at the 31 month assessment of these wines. An initial practice rating session was held, with the tasters assessing a sample of each of the wines from the current study. A list of the terms that was rated by the panellists is given in Table 1, which was the same attributes as rated at 31 months.

Table 1. Sensory attributes scored.

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

For the formal sessions, samples were assessed in blind tasting conditions using standardised procedures. Thirteen wines were assessed at a session, being one example of each closure in the study including the three reference closures. 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 (28 and 29 October 2005) 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 JMP 5.1 (SAS Institute, Cary, NC). 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 covariate, 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 provide 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.

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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 36 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. Both the Reference ROTE type closure and the Procork closure have shown only a slight loss of sulphur dioxide compared to the 24 and 31 month post bottling period.

The Procork closures retained significantly more free and total sulphur dioxide than the Reference 2 and 3 cork closures resulting in a lower level of oxidation, as measured by the increase in OD₄₂₀ absorbance (Figure 3) for 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 13 closures assessed for the aroma attributes estery, floral, fresh citrus, cooked citrus, overall fruit, honey, toasty, oxidised, TCA, struck flint/rubber and H₂S/Cabbagey; and for the palate attributes: overall fruit flavour, fruit flavour persistence and TCA. 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 closure.

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.

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 H₂S/Cabbagey. The aroma attribute 'H₂S/Cabbagey' is not shown on the figure due to the values for all closures being very close to zero, with the maximum mean ratings for any closure being 0.6. The flint/rubber aroma attribute showed a significant difference between the reference closures, the reference 2 and 3 closures were rated **0.4** and **0.1** respectively, whereas the ROTE closure was scored at **2.1**. The Procork closure was **1.3** (a score of 1 is considered just detectable). Honey and toasty characters were rated lower in the ROTE (**1.3/1.4**) and Procork (**1.4/1.3**) closures than the Reference 2 (**1.7/1.7**) and 3 (**2.5/1.3**) closures. The fresh citrus character (aroma) was markedly higher in wine under the ROTE (**3.4**) and Procork (**3.3**) closures, than the Reference 2 (**2.7**) and 3 (**1.8**) closures. The Procork and ROTE closures had similar mean scores in attributes such as estery, floral and cooked citrus.

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.

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.

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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 36 months post bottling. Tech.Rev. 139:6-10; 2002.

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21 November 2005

Attachments:

Chemical and Physical Data

Table 2. Summary Comparison of Initial and 36 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 OD₄₂₀ in Wine bottled under Reference and Procork's Membrane Cork Closures.

Sensory Data

Table 3: 36 month sensory testing of Reference Closures

Table 4: 36 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 36 Month Chemical and Physical Testing on Reference and Procork's Membrane Cork Closure.

	Initial	36 month	Initial	36 month	Initial	36 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	21.7	111	92.7	0.047	0.071
Std deviation	2	2.1	1	2.7	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	17.5	113	86.3	0.052	0.084
Std deviation	2	4.6	2	8.1	0.004	0.005
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	13.5	112	79.8	0.052	0.087
Std deviation	2	4.1	2	7.5	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	22.8	113	94.7	0.051	0.076
Std deviation	2	1.5	3	1.8	0.003	0.003
n	12	12	12	12	12	12

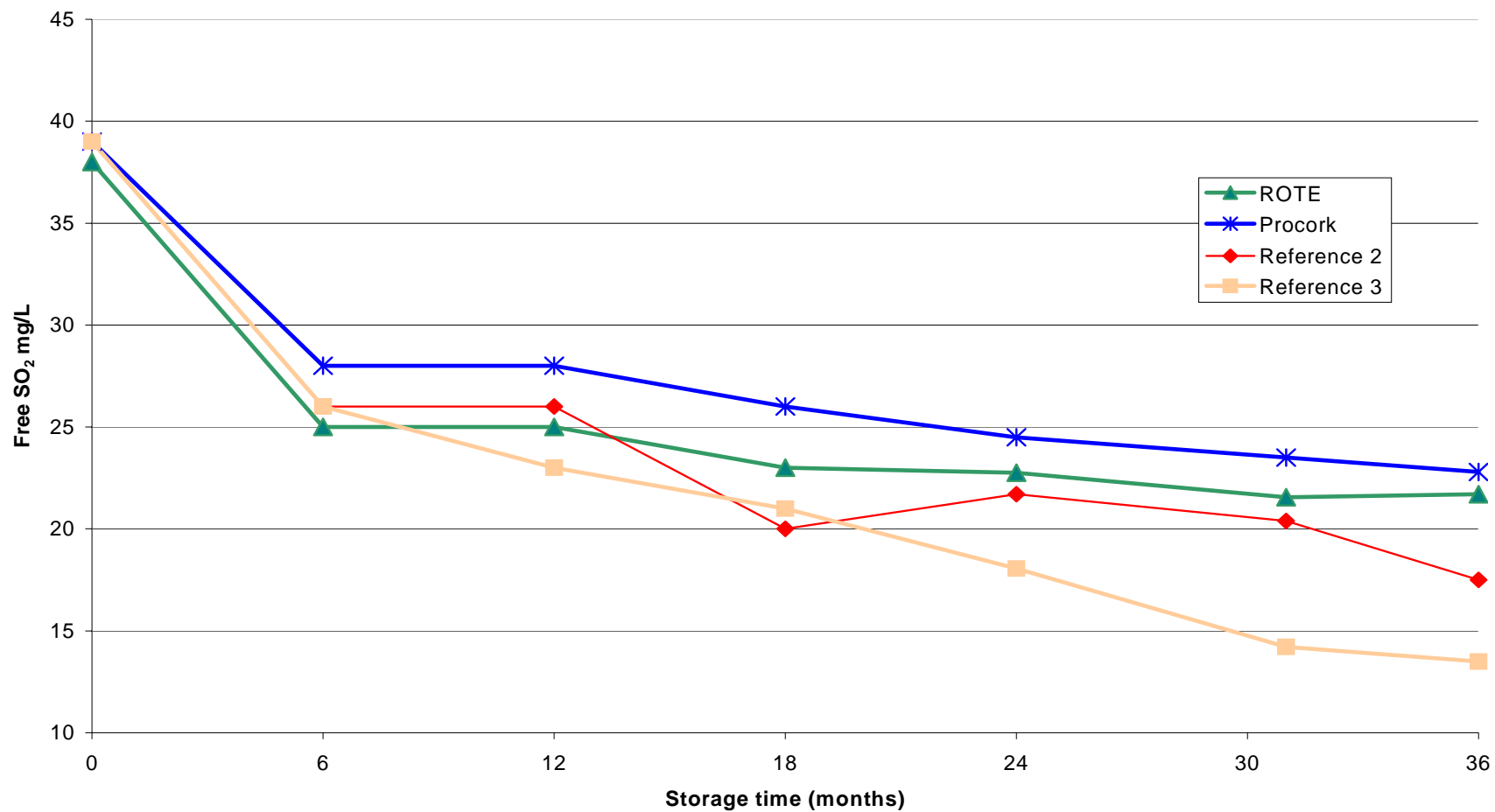


Figure 1: Change in free Sulfur Dioxide in Wine bottled under Reference and Procork’s Membrane Cork Closure.

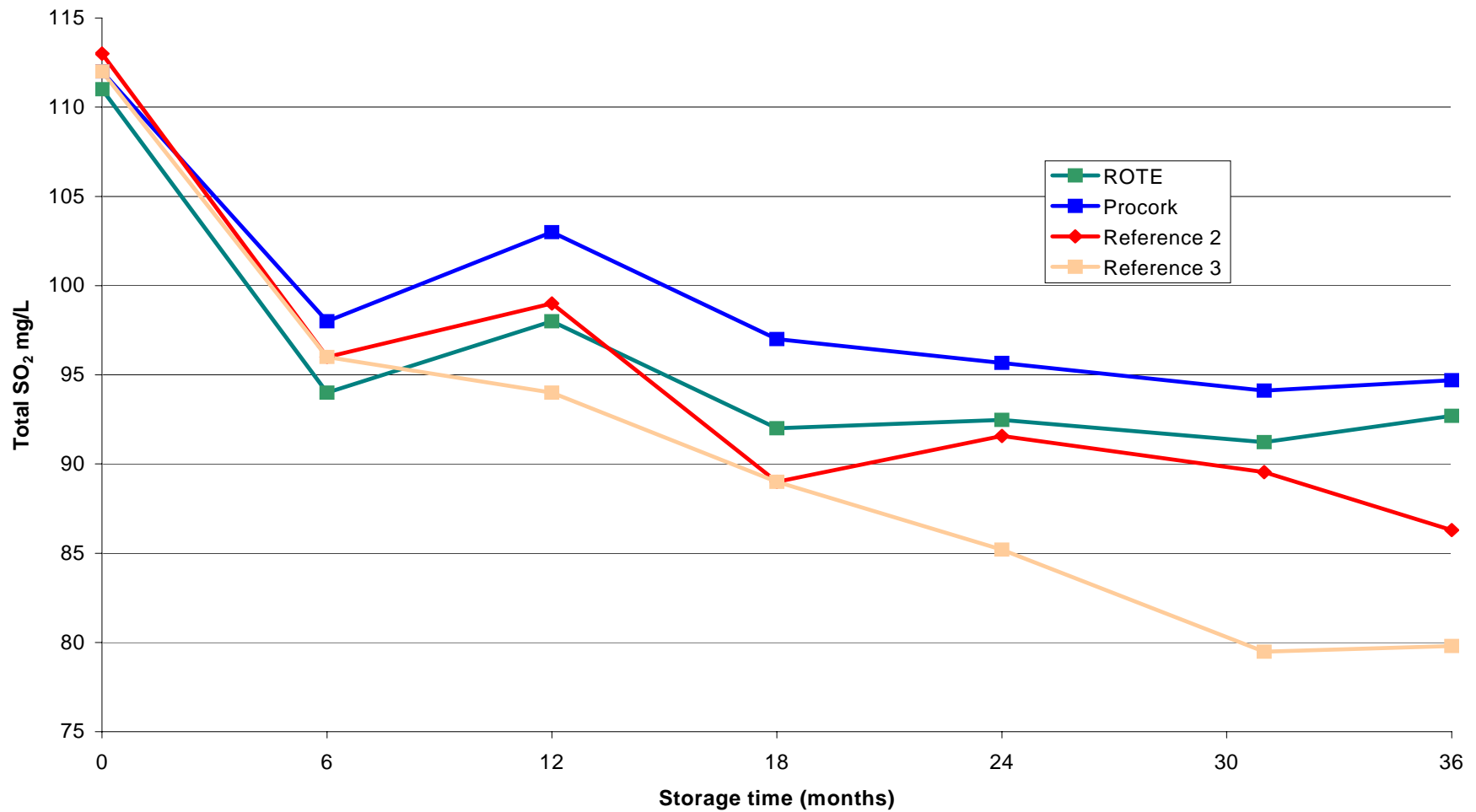


Figure 2: Change in total Sulfur Dioxide in Wine bottled under Reference and Procork’s Membrane Cork Closure.

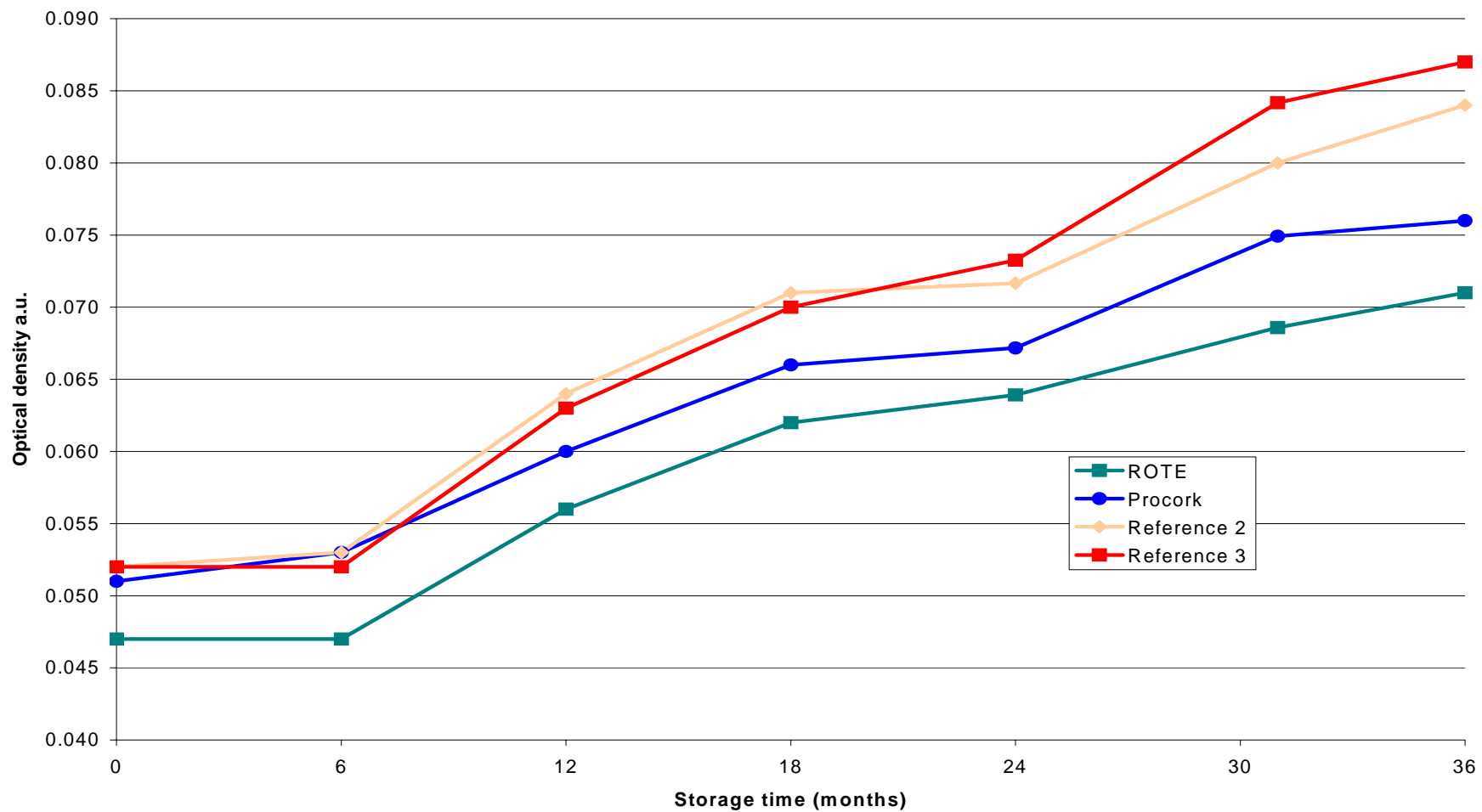


Figure 3: Change in OD₄₂₀ in Wine bottled under Reference and Procork's Membrane Cork Closures.

Table 3: 36 month sensory testing of Reference Closures

closure	replicate	estery (aroma)	floral (aroma)	fresh citrus (aroma)	cooked citrus (aroma)	overall fruit (aroma)	honey (aroma)	toasty (aroma)	oxidised (aroma)	TCA (aroma)	struck flint/rubber (aroma)	H ₂ S/ cabbagey (aroma)	overall fruit flavour (palate)	fruit flavour persistence (palate)	TCA (palate)
ROTE (Low SO₂)	1	2.2	2.0	3.5	3.3	4.5	1.4	1.6	0.3	0.0	2.3	0.0	5.0	4.4	0.0
	2	2.1	1.7	3.3	3.4	4.2	1.2	1.4	0.5	0.0	2.3	0.6	4.4	4.3	0.0
	3	2.0	1.6	3.5	3.6	4.2	1.5	1.6	0.2	0.0	1.6	0.0	4.4	4.2	0.0
	4	2.2	2.0	3.2	3.5	3.9	1.2	0.9	0.2	0.0	2.5	0.6	5.0	4.1	0.0
	<i>Mean</i>	<i>2.1</i>	<i>1.8</i>	<i>3.4</i>	<i>3.5</i>	<i>4.2</i>	<i>1.3</i>	<i>1.4</i>	<i>0.3</i>	<i>0.0</i>	<i>2.1</i>	<i>0.3</i>	<i>4.7</i>	<i>4.3</i>	<i>0.0</i>
	<i>Std dev.</i>	<i>0.1</i>	<i>0.2</i>	<i>0.1</i>	<i>0.1</i>	<i>0.2</i>	<i>0.1</i>	<i>0.3</i>	<i>0.1</i>	<i>0.0</i>	<i>0.4</i>	<i>0.3</i>	<i>0.3</i>	<i>0.1</i>	<i>0.0</i>
Ref 2 cork	1	1.0	1.2	1.1	1.4	1.7	1.1	0.5	0.6	4.5	0.0	0.0	2.8	2.5	0.0
	2	2.3	1.5	3.1	3.0	4.0	1.7	1.7	0.2	0.0	0.6	0.0	4.6	4.5	0.0
	3	1.7	1.5	2.3	3.2	3.5	1.7	1.9	0.7	0.0	0.4	0.0	4.5	4.2	0.0
	4	2.6	2.2	2.6	2.8	4.2	1.8	1.6	0.5	0.0	0.3	0.6	4.4	4.5	0.3
	<i>Mean (exclud. Rep 1)</i>	<i>2.2</i>	<i>1.7</i>	<i>2.7</i>	<i>3.0</i>	<i>3.9</i>	<i>1.7</i>	<i>1.7</i>	<i>0.4</i>	<i>0.0</i>	<i>0.4</i>	<i>0.2</i>	<i>4.5</i>	<i>4.4</i>	<i>0.1</i>
	<i>Std dev. (exclude. Rep 1)</i>	<i>0.4</i>	<i>0.4</i>	<i>0.4</i>	<i>0.2</i>	<i>0.4</i>	<i>0.0</i>	<i>0.2</i>	<i>0.3</i>	<i>0.0</i>	<i>0.1</i>	<i>0.3</i>	<i>0.1</i>	<i>0.2</i>	<i>0.2</i>
Ref 3 cork	1	2.2	1.6	2.2	2.6	3.6	2.2	1.7	1.4	0.0	0.1	0.0	4.2	4.0	0.0
	2	1.2	0.6	1.5	2.2	2.5	2.5	1.1	2.7	0.5	0.0	0.0	3.1	2.8	0.0
	3	1.4	1.4	1.6	2.3	2.7	2.7	1.3	2.7	0.0	0.2	0.0	3.3	2.9	0.0
	4	1.8	1.6	1.8	2.5	3.1	2.7	1.3	2.1	0.0	0.0	0.0	3.3	2.9	0.0
	<i>Mean</i>	<i>1.7</i>	<i>1.3</i>	<i>1.8</i>	<i>2.4</i>	<i>3.0</i>	<i>2.5</i>	<i>1.3</i>	<i>2.2</i>	<i>0.1</i>	<i>0.1</i>	<i>0.0</i>	<i>3.5</i>	<i>3.1</i>	<i>0.0</i>
	<i>Std dev.</i>	<i>0.4</i>	<i>0.4</i>	<i>0.3</i>	<i>0.2</i>	<i>0.5</i>	<i>0.2</i>	<i>0.3</i>	<i>0.6</i>	<i>0.2</i>	<i>0.1</i>	<i>0.0</i>	<i>0.5</i>	<i>0.5</i>	<i>0.0</i>

Sensory data for each of the four replicate bottles assessed (mean scores of 9 judges), and mean data for each closure type, averaged across four replicates.

Table 4: 36 month sensory testing of Procork’s Membrane Cork Closures

Sensory data for each of the four replicate bottles assessed (mean scores of 9 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)	toasty (aroma)	oxidised (aroma)	TCA (aroma)	struck flint/rubber (aroma)	H2S/ Cabbag ey (aroma)	overall fruit flavour (palate)	fruit flavour persistence (palate)	TCA (palate)
PROCORK	1	2.4	1.8	3.7	3.3	4.4	1.4	1.7	0.2	0.0	1.7	0.3	4.7	4.6	0.0
	2	2.2	1.6	3.6	3.0	4.4	1.4	0.9	0.5	0.0	0.8	0.0	4.4	3.9	0.0
	3	2.2	2.0	3.3	3.1	4.1	1.3	1.0	0.1	0.5	0.8	0.0	4.4	4.2	0.2
	4	1.9	1.7	2.6	3.0	3.5	1.6	1.5	0.3	0.0	2.0	0.0	4.1	3.9	0.0
	<i>Mean</i>	<i>2.1</i>	<i>1.8</i>	<i>3.3</i>	<i>3.1</i>	<i>4.1</i>	<i>1.4</i>	<i>1.3</i>	<i>0.3</i>	<i>0.1</i>	<i>1.3</i>	<i>0.1</i>	<i>4.4</i>	<i>4.1</i>	<i>0.0</i>
	<i>Std dev.</i>	<i>0.2</i>	<i>0.2</i>	<i>0.5</i>	<i>0.2</i>	<i>0.4</i>	<i>0.1</i>	<i>0.4</i>	<i>0.2</i>	<i>0.2</i>	<i>0.6</i>	<i>0.1</i>	<i>0.2</i>	<i>0.3</i>	<i>0.1</i>

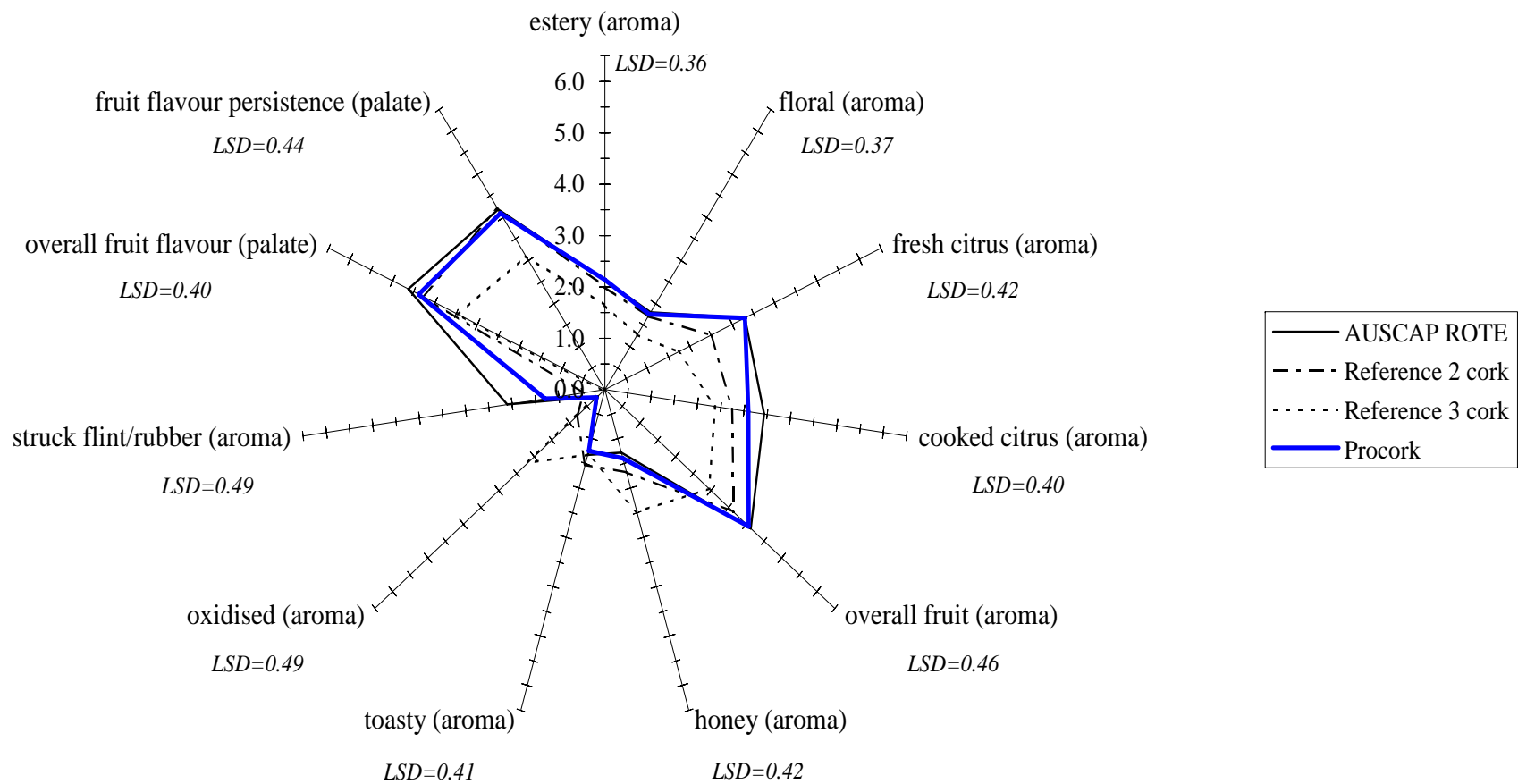


Figure 4: Radar / Spider Plot of Significant Sensory Attributes vs Procork Closure Material.

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.