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## **Commercial Closure trial – 18 Month Progress Report Performance Testing of Altec P0, P1 and P2 closure**

**Dear Mr Dean Banister**

### **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.

### **18 MONTH TESTING**

After 18 months storage, random samples of each closure were tested for free sulfur dioxide, total sulfur dioxide and the optical density at 420nm. 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 were 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 10 judges was recruited, comprising AWRI staff with extensive experience in wine sensory evaluation, of whom all had participated on the sensory panel at the six or twelve 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, and included one of the reference 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. 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 12 months were used, but with one additional aroma descriptor, namely floral.

Table 1. Sensory attributes scored.

<b>Attribute</b>	<b>Definition</b>
<b>AROMA</b>	
Estery	definition: estery, bubblegum, tropical fruit
Floral	definition: perfumed, musk
Citrus	definition: lemon, lime, orange
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 <sub>2</sub> S/Cabbagey	definition: rotten egg, cabbage, sewerage
Other	
<b>PALATE</b>	
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 six 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 Institute's purpose built sensory facility in isolated, temperature controlled, ventilated tasting booths daylight-type fluorescent lights, with temperature control between 22-24°C. Data was acquired using Fizz 2.00e software (Biosystemes, Couternon, France).

The panellists scored each attribute on a 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 (LSD) value ( $P=0.05$ ), were graphed in the form of a radar (spider) plot.

## RESULTS AND DISCUSSION

### Chemical analysis

The levels of free and total sulfur dioxide in wine are considered to be a 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, 2001) 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 corks, technical corks and synthetic closures. The

changes observed in free and total sulfur dioxide and OD 420 data over 18 months for the reference closures is very comparable to the changes observed in the commercial trail reported at the 20 month after bottling period.

### Sensory Analysis

From the analysis of variance of the sensory data, it was found that there were significant differences among the 14 closures for the aroma attributes: floral, citrus, overall fruit, honey, oxidised, TCA, cork wood, struck flint/rubber and H<sub>2</sub>S/cabbagey; and for the palate attributes: overall fruit, 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 in (Table 3 and 4.)

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 cork wood). ‘Cork wood’ on aroma is not shown on the radar plot due to the values being very close to zero. While there was significant differences for cork wood aroma ratings across the closures, all closures were rated very low and not significantly different from each other

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 six months assessment, the panels used differed slightly in make up, which means that comparing mean values across the two time periods could be potentially misleading. Comparisons among closures at a time point are more meaningful.

### Supplemental Chemical Testing

After the sensory evaluation of wines under the Altec closures, we were asked to confirm the presence or absence of TCA in samples for which a score of less than 1 had been obtained. A score of 1 is the sensory “limit of detection” and unlike instrumental analytical methods, it is not possible to “confirm” the positive result.

For this reason, we have used an instrumental analytical method know as headspace solid phase microextraction gc/ms (HS SPME gc/ms) to confirm the presence/absence of cork taint compounds [especially trichloroanisole (TCA)] in the wine of those samples for which a sensory score above 0 was obtained. The method is fully validated and we have used this in a proficiency test ring to confirm that satisfactory results are being obtained.

Identifying Descriptor	AWRI ID	Sensory Score	TCA (ng/L)
Altec P0 rep #2	CE2249	0.0	nd
Altec P0 rep #4	CE2379	0.1	nd
Altec P1 rep #2	CE2250	0.1	nd
Altec P1 rep #3	CE2366	0.1	nd
Altec P2 rep #3	CE2367	0.3	nd

nd: Indicates that no peaks were observed in the selective ion monitoring mode corresponding to TCA or the fragment ions. The limit of quantitation is  $\leq 1$  ng/L for this instrumental technique. No traces of 2,4- and 2,6-dichloroanisole, 2,3,4,6 tetrachloroanisole (TeCA) or pentachloroanisole were observed. These compounds produce a lesser sensory impact than TCA, but are sometimes present in TCA tainted wine at a lower level.

The results show that there was no evidence for trace levels of TCA.

### References:

Godden P.W., Francis I.L., Field J., Gishen M., Coulter A. D., Valente P., Hoj 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.

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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 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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28 April 2004

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**Attachments:**

**Chemical and Physical Data**

**Table 2. Summary Comparison of Initial and Twelve Month Chemical and Physical Testing of Reference and ALTEC Closures.**

**Figure 1: Change in free Sulfur Dioxide in Wine bottled under Reference and ALTEC Closures.**

**Figure 2: Change in total Sulfur Dioxide in Wine bottled under Reference and ALTEC Closures.**

**Figure 3: Change in OD 420 in Wine bottled under Reference and ALTEC Closures.**

**Sensory Data**

**Table 3: 18 month sensory testing of Reference Closures**

**Table 4: 18 month sensory testing of ALTEC Closures**

**Figure 4: Radar / Spider Plot of Significant Sensory Attributes for wine under Reference and ALTEC Closures**

**Table 2. Summary Comparison of Initial and 18 Month Chemical and Physical Testing on Reference and ALTEC Closures.**

	<b>Initial</b>	<b>18 month</b>	<b>Initial</b>	<b>18 month</b>	<b>Initial</b>	<b>18 month</b>
<b>Auscap-ROTE</b>	Free SO <sub>2</sub> mg/L	Free SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	OD420 a.u.	OD420 a.u.
Mean	38	23	111	92	0.047	0.062
Std deviation	2	1	1	2	0.002	0.002
n	12	12	12	12	12	12
<b>Reference 2</b>	Free SO <sub>2</sub> mg/L	Free SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	OD420 a.u.	OD420 a.u.
Mean	39	19	113	89	0.052	0.071
Std deviation	2	3	2	5	0.004	0.004
n	12	12	12	12	12	12
<b>Reference 3</b>	Free SO <sub>2</sub> mg/L	Free SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	OD420 a.u.	OD420 a.u.
Mean	39	21	112	89	0.052	0.070
Std deviation	2	2	2	3	0.003	0.004
n	12	12	12	12	12	12
<b>Altec P0</b>	Free SO <sub>2</sub> mg/L	Free SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	OD420 a.u.	OD420 a.u.
Mean	38	25	111	97	0.050	0.067
Std deviation	2	1	2	3	0.003	0.003
n	12	12	12	12	12	12
<b>Altec P1</b>	Free SO <sub>2</sub> mg/L	Free SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	OD420 a.u.	OD420 a.u.
Mean	38	25	112	93	0.049	0.066
Std deviation	2	1	2	2	0.002	0.002
n	12	12	12	12	12	12
<b>Altec P2</b>	Free SO <sub>2</sub> mg/L	Free SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	OD420 a.u.	OD420 a.u.
Mean	39	24	113	94	0.050	0.067
Std deviation	2	1	2	2	0.002	0.003
n	12	12	12	12	12	12

### Free SO<sub>2</sub>

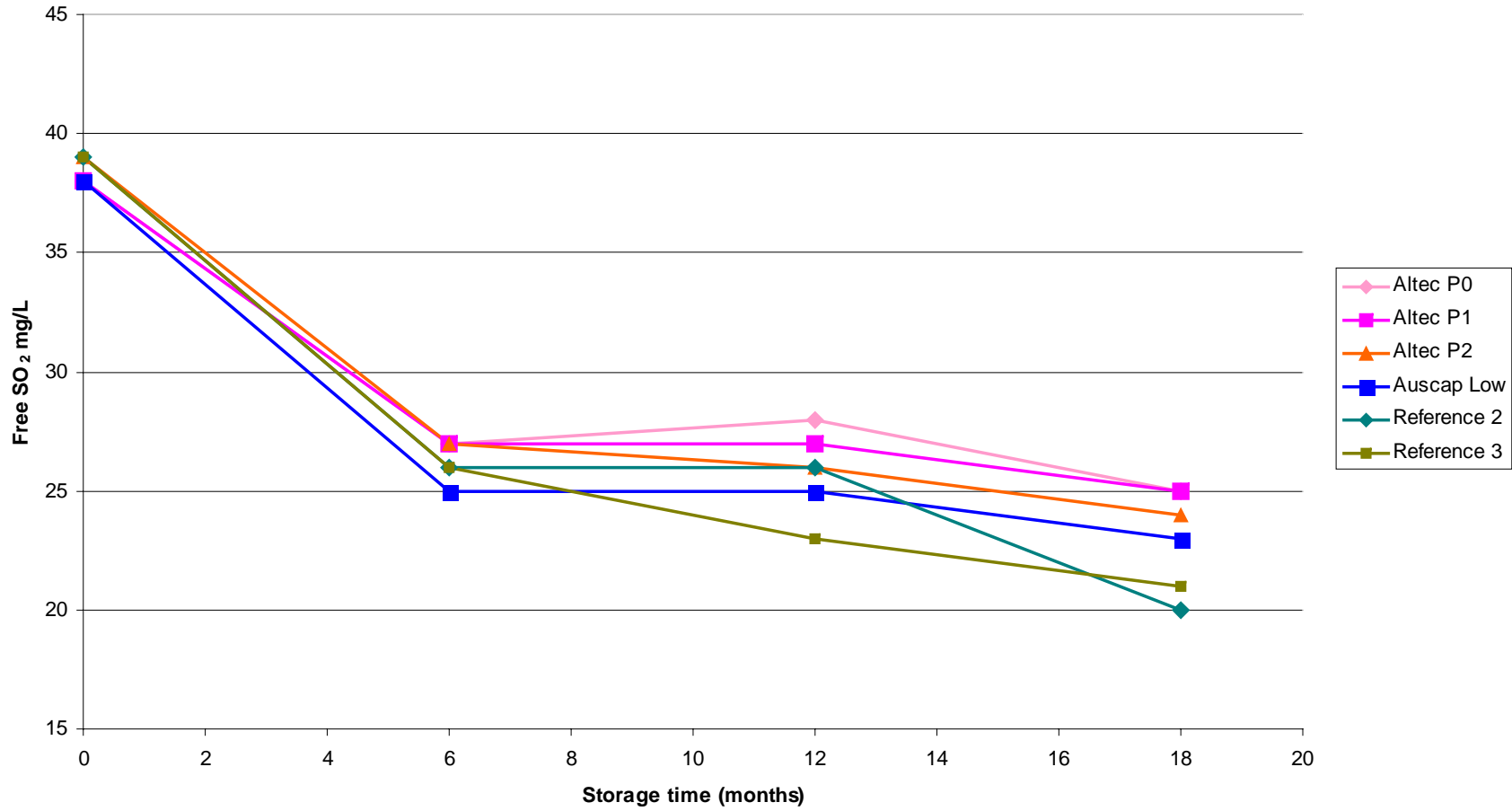
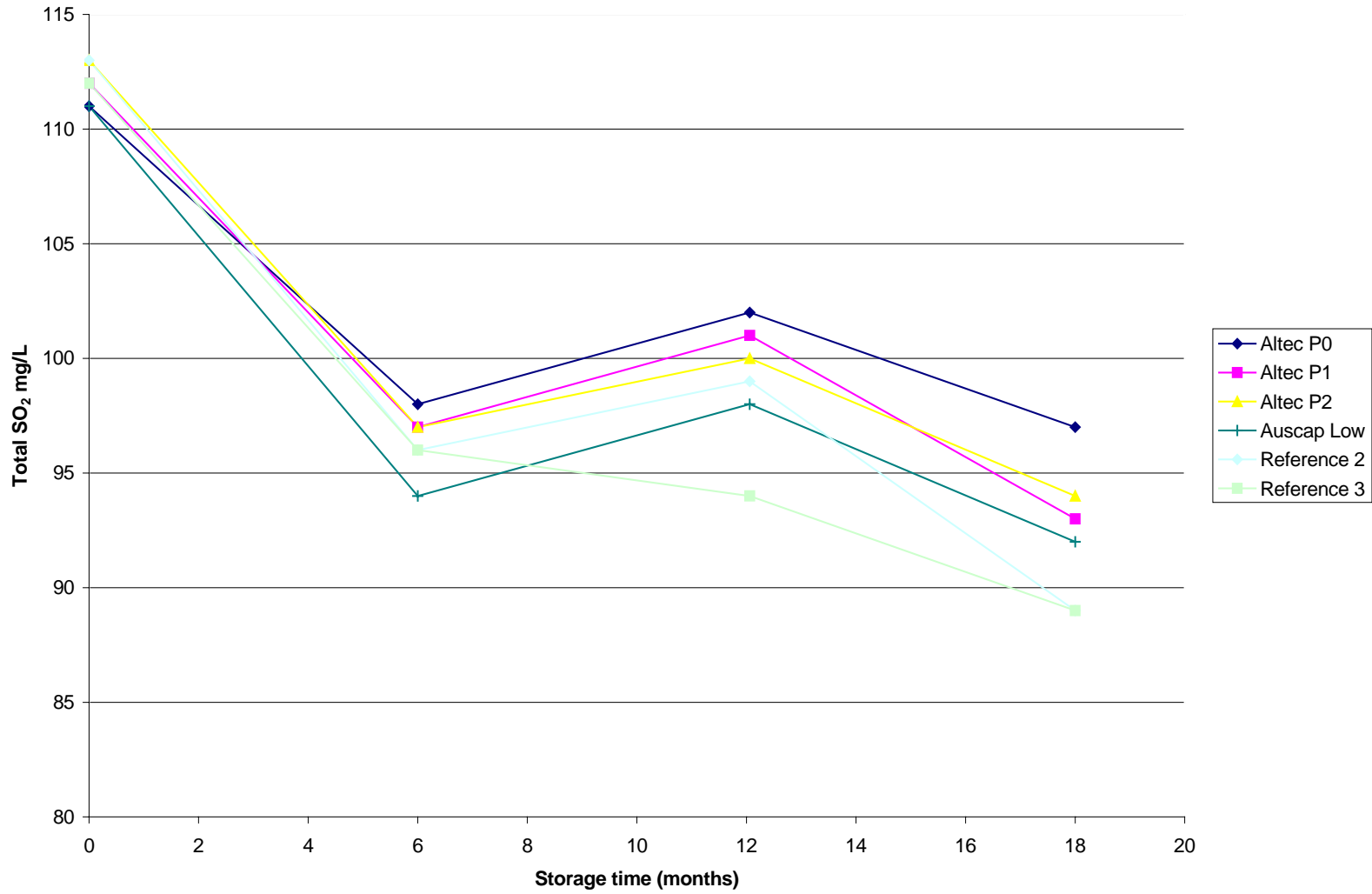


Figure 1: Change in free Sulfur Dioxide in Wine bottled under Reference and ALTEC Closures.

### Total SO<sub>2</sub>



**Figure 2: Change in total Sulfur Dioxide in Wine bottled under Reference and ALTEC Closures.**

Optical Density @ 420 nm

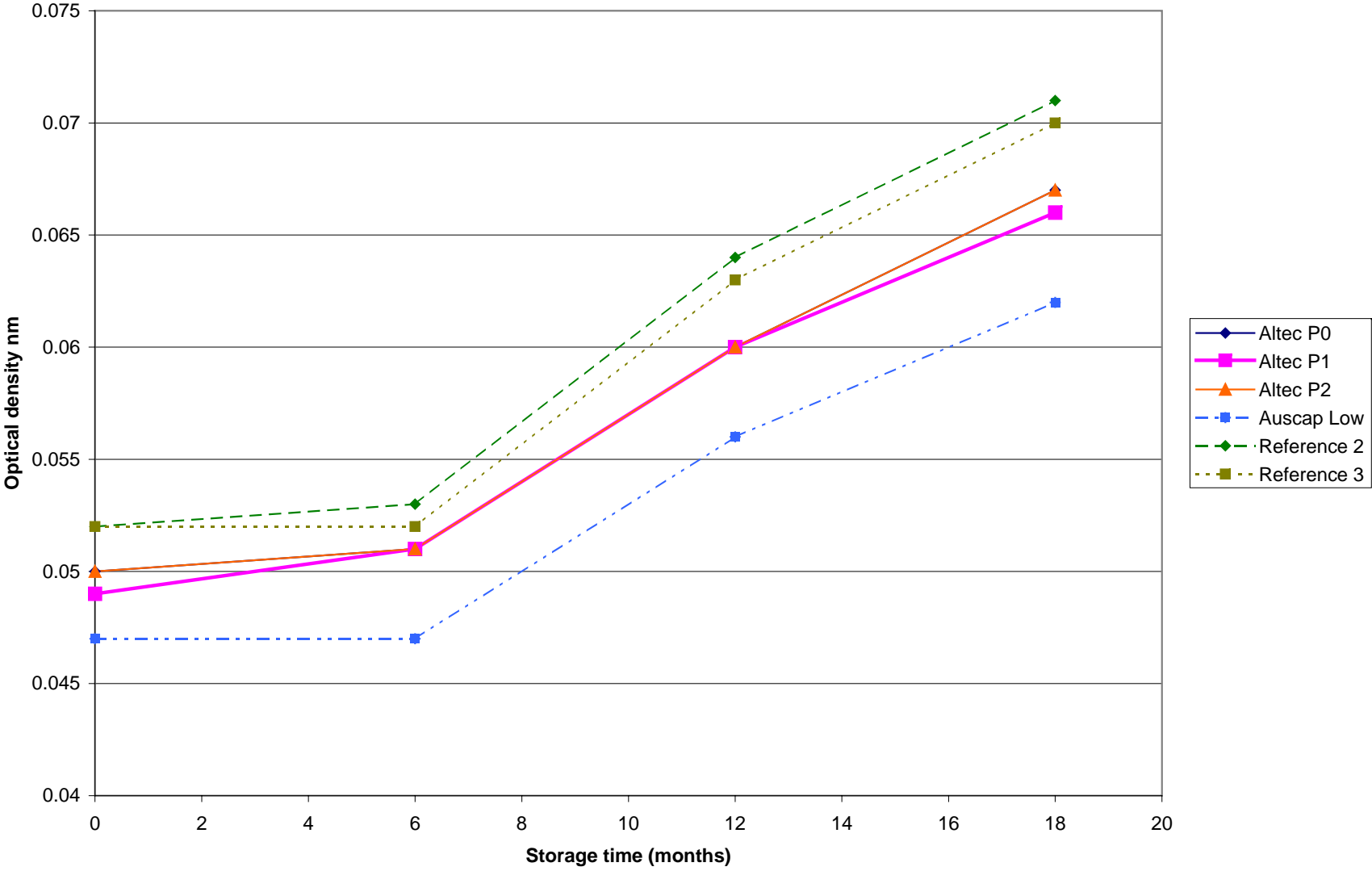


Figure 3: Change in OD 420 in Wine bottled under Reference and ALTEC Closures.

**Table 3: 18 month sensory testing of Reference Closures**

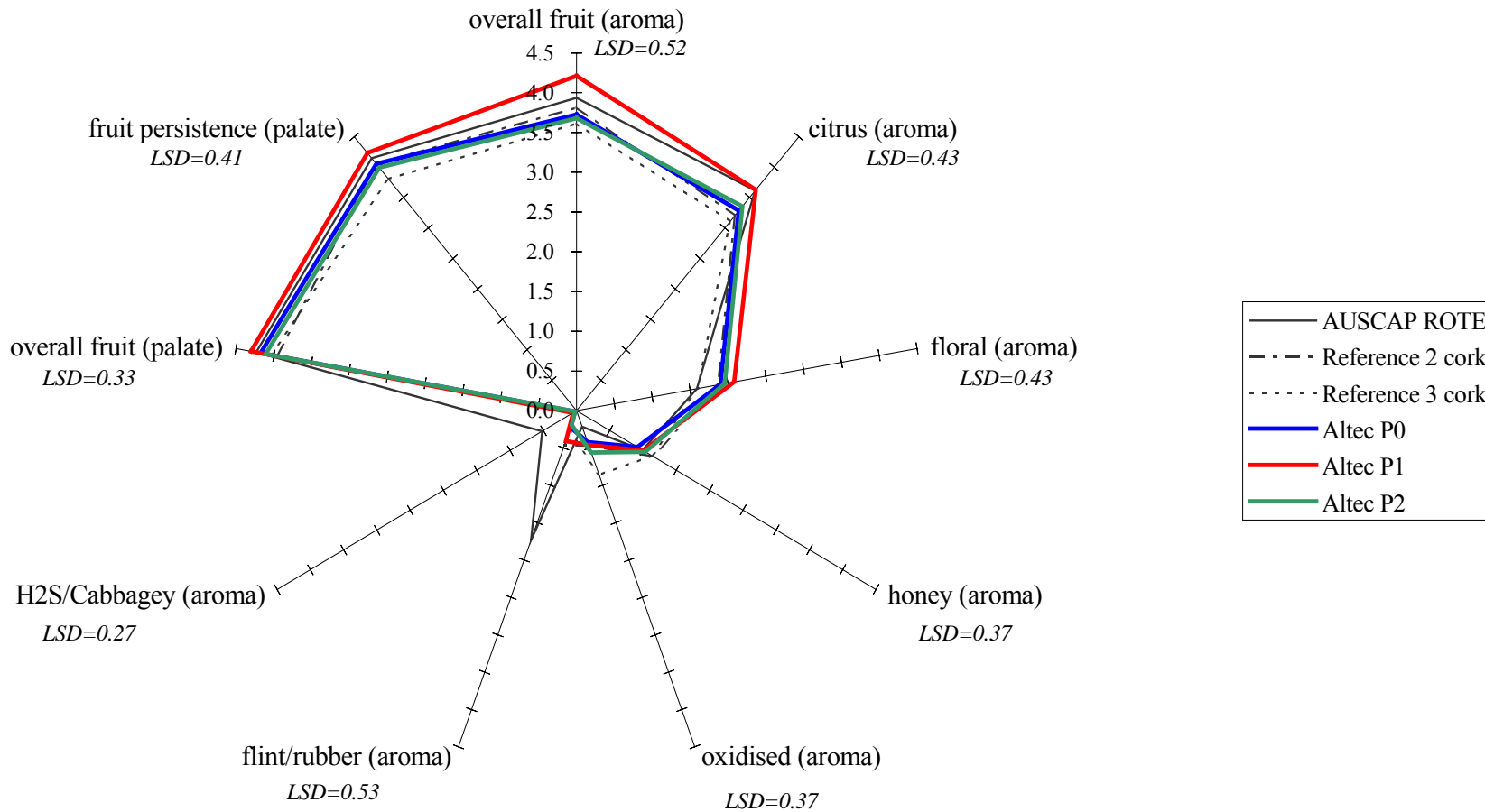
Sensory data for each of the four replicate bottles assessed (mean scores of 10 judges), and mean data for each closure type, averaged across four replicates.

Closure	Replicate	floral (aroma)	citrus (aroma)	overall fruit (aroma)	honey (aroma)	oxidised (aroma)	TCA (aroma)	cork wood (aroma)	flint/rubber (aroma)	H <sub>2</sub> S/ Cabbagey (aroma)	overall fruit flavour (palate)	fruit flavour persistence (palate)	TCA (palate)
<b>AUSCAP-L</b>	1	2.1	4.0	3.9	1.2	0.1	0.0	0.1	1.9	0.5	4.2	4.4	0.0
<b>AUSCAP-L</b>	2	1.6	3.6	4.1	0.8	0.2	0.1	0.0	1.4	0.6	4.2	4.2	0.1
<b>AUSCAP-L</b>	3	1.5	3.7	4.4	1.1	0.2	0.1	0.4	1.7	0.4	4.5	4.0	0.1
<b>AUSCAP-L</b>	4	1.2	3.2	3.4	0.9	0.5	0.0	0.3	2.1	0.6	4.0	3.9	0.0
	<i>Mean</i>	<i>1.6</i>	<i>3.6</i>	<i>3.9</i>	<i>1.0</i>	<i>0.2</i>	<i>0.0</i>	<i>0.2</i>	<i>1.7</i>	<i>0.5</i>	<i>4.2</i>	<i>4.2</i>	<i>0.0</i>
	<i>Std dev.</i>	<i>0.4</i>	<i>0.3</i>	<i>0.4</i>	<i>0.2</i>	<i>0.2</i>	<i>0.1</i>	<i>0.2</i>	<i>0.3</i>	<i>0.1</i>	<i>0.2</i>	<i>0.2</i>	<i>0.1</i>
<b>REF 2</b>	1	1.4	3.3	3.7	0.9	0.4	0.0	0.3	0.4	0.1	3.8	4.0	0.0
<b>REF 2</b>	2	1.9	3.0	3.6	1.2	1.1	0.1	0.2	0.8	0.0	3.7	3.7	0.1
<b>REF 2</b>	3	2.1	3.2	3.8	1.3	0.1	0.1	0.1	0.2	0.0	4.3	4.3	0.1
<b>REF 2</b>	4	2.2	3.4	4.2	1.1	0.4	0.0	0.3	0.5	0.0	4.1	4.3	0.0
	<i>Mean</i>	<i>1.9</i>	<i>3.2</i>	<i>3.8</i>	<i>1.2</i>	<i>0.5</i>	<i>0.0</i>	<i>0.2</i>	<i>0.5</i>	<i>0.0</i>	<i>4.0</i>	<i>4.1</i>	<i>0.0</i>
	<i>Std dev.</i>	<i>0.4</i>	<i>0.2</i>	<i>0.3</i>	<i>0.2</i>	<i>0.4</i>	<i>0.0</i>	<i>0.1</i>	<i>0.2</i>	<i>0.0</i>	<i>0.3</i>	<i>0.3</i>	<i>0.0</i>
<b>REF 3</b>	1	1.9	3.6	4.3	1.3	0.6	0.0	0.0	0.3	0.2	4.5	4.1	0.0
<b>REF 3</b>	2	1.6	3.1	3.6	1.2	0.8	0.1	0.2	0.0	0.0	3.9	3.7	0.1
<b>REF 3</b>	3	1.4	2.8	3.1	1.4	1.2	0.1	0.1	0.5	0.0	4.0	3.9	0.1
<b>REF 3</b>	4	1.5	2.9	3.4	0.7	1.0	0.0	0.2	0.3	0.0	3.8	3.6	0.0
	<i>Mean</i>	<i>1.6</i>	<i>3.1</i>	<i>3.6</i>	<i>1.1</i>	<i>0.9</i>	<i>0.0</i>	<i>0.1</i>	<i>0.3</i>	<i>0.0</i>	<i>4.1</i>	<i>3.8</i>	<i>0.0</i>
	<i>Std dev.</i>	<i>0.2</i>	<i>0.4</i>	<i>0.5</i>	<i>0.3</i>	<i>0.3</i>	<i>0.0</i>	<i>0.1</i>	<i>0.2</i>	<i>0.1</i>	<i>0.3</i>	<i>0.2</i>	<i>0.1</i>

**Table 4: 18 month sensory testing of ALTEC Closures**

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

Closure	Replicate	floral (aroma)	citrus (aroma)	overall fruit (aroma)	honey (aroma)	oxidised (aroma)	TCA (aroma)	cork wood (aroma)	flint/rubber (aroma)	H <sub>2</sub> S/ Cabbagey (aroma)	overall fruit flavour (palate)	fruit flavour persistence (palate)	TCA (palate)
Altec P0	1	1.5	3.3	3.9	0.9	0.5	0.0	0.5	0.3	0.1	4.5	4.4	0.0
Altec P0	2	1.7	3.1	3.3	0.6	0.3	0.1	0.9	0.4	0.0	3.8	3.9	0.0
Altec P0	3	2.4	3.3	3.6	0.8	0.6	0.1	0.0	0.2	0.0	4.0	3.8	0.0
Altec P0	4	2.0	3.5	4.1	1.4	0.5	0.1	0.4	0.0	0.0	4.4	4.2	0.0
	<b>Mean</b>	<b>1.9</b>	<b>3.3</b>	<b>3.7</b>	<b>0.9</b>	<b>0.4</b>	<b>0.1</b>	<b>0.5</b>	<b>0.2</b>	<b>0.0</b>	<b>4.2</b>	<b>4.1</b>	<b>0.0</b>
	<b>Std dev.</b>	<b>0.4</b>	<b>0.1</b>	<b>0.4</b>	<b>0.4</b>	<b>0.1</b>	<b>0.0</b>	<b>0.4</b>	<b>0.2</b>	<b>0.0</b>	<b>0.3</b>	<b>0.3</b>	<b>0.0</b>
Altec P1	1	2.6	3.7	4.3	0.9	0.3	0.0	0.3	0.5	0.2	4.3	4.2	0.1
Altec P1	2	2.2	3.7	4.3	0.8	0.2	0.1	0.2	0.6	0.0	4.4	4.5	0.0
Altec P1	3	1.7	3.8	4.3	1.1	0.7	0.1	0.1	0.3	0.1	4.3	4.4	0.0
Altec P1	4	1.9	3.4	3.9	1.2	0.8	0.0	0.4	0.3	0.0	4.3	3.9	0.0
	<b>Mean</b>	<b>2.1</b>	<b>3.6</b>	<b>4.2</b>	<b>1.0</b>	<b>0.5</b>	<b>0.0</b>	<b>0.2</b>	<b>0.4</b>	<b>0.1</b>	<b>4.3</b>	<b>4.3</b>	<b>0.0</b>
	<b>Std dev.</b>	<b>0.4</b>	<b>0.2</b>	<b>0.2</b>	<b>0.2</b>	<b>0.3</b>	<b>0.0</b>	<b>0.1</b>	<b>0.1</b>	<b>0.1</b>	<b>0.0</b>	<b>0.3</b>	<b>0.0</b>
Altec P2	1	1.6	3.1	3.8	1.1	0.8	0.0	0.5	0.3	0.1	4.0	3.9	0.0
Altec P2	2	2.0	3.1	2.9	1.1	0.8	0.1	0.5	0.0	0.0	4.1	4.0	0.0
Altec P2	3	2.0	3.8	4.2	1.1	0.3	0.3	0.4	0.5	0.0	4.1	4.0	0.0
Altec P2	4	2.3	3.5	3.8	0.9	0.3	0.0	0.1	0.0	0.0	4.3	4.0	0.0
	<b>Mean</b>	<b>2.0</b>	<b>3.4</b>	<b>3.7</b>	<b>1.0</b>	<b>0.6</b>	<b>0.1</b>	<b>0.4</b>	<b>0.2</b>	<b>0.0</b>	<b>4.1</b>	<b>4.0</b>	<b>0.0</b>
	<b>Std dev.</b>	<b>0.3</b>	<b>0.3</b>	<b>0.5</b>	<b>0.1</b>	<b>0.3</b>	<b>0.1</b>	<b>0.2</b>	<b>0.2</b>	<b>0.0</b>	<b>0.2</b>	<b>0.1</b>	<b>0.0</b>



**Figure 4: Radar / Spider Plot of Significant Sensory Attributes vs Closure Material.** Mean values of aroma and palate attributes rated for the three reference closures and the ALTEC closures. Only those attributes that were statistically significant from the ANOVA, adjusted for TCA scores, are shown, excluding cork wood (aroma). LSD: least significant difference. Each value is the mean score from four replicates of each closure presented to 10 judges.