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Dear Mr Dean Banister,

## Commercial Closure Trial – 36 Month Progress Report Performance Testing of Altec P0, P1 and P2 closure

### PROTOCOL DETAILS

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. These same samples were used for sensory evaluation immediately after the chemical analysis. 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 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 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 were the same attributes as rated at 31 months.

**Table 1. Sensory Attributes Scored.**

<b>Attribute</b>	<b>Definition</b>
<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
<i>Glue/Plastic/Solvent</i>	
TCA	musty, mouldy, cork taint
Cork wood	woody, corks soaked in wine
Struck flint/rubber	rubber, struck match/flint
H <sub>2</sub> S/cabbagey	rotten egg, cabbage, sewerage
<i>Palate</i>	
Acidity	
Overall Fruit	
Fruit Flavour Persistence	
<i>Glue/Plastic/Solvent</i>	
TCA	
Cork wood	
Reduced	rubbery, struck match/flint, cabbage

Following the discussion session, a single practice rating, comprising 10 samples, was carried out in the 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 single 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.

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 Altec P0 and P1 closures were similar in mean free and total sulfur dioxide to the ROTE, while the P2 was slightly lower and was similar to the reference 2 closure. All three Altec closures were significantly higher than the reference 3 closure. The OD<sub>420</sub> measurements for all three Altec closures were very similar. When compared with the reference 2 and 3 closures, the Altec closures showed a lower level of oxidation, but this level was greater than that for the ROTE closure. This trend is expected, but cannot be compared with the original trial as the OD<sub>420</sub> 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 H<sub>2</sub>S/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 in Table 3 (Reference Closures) and Table 4 for the Altec Closures.

Figure 4 shows mean values and least significant differences for each of the attributes that were rated significantly different across the closures, except TCA (aroma and palate) and H<sub>2</sub>S/Cabbagey. The aroma attribute 'H<sub>2</sub>S/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 Altec P0, P1 and P2 closures were **0.1**, **0.2** and **0.0** respectively, indicating that there was no evidence of the reduced characters (flint/rubber) having formed (a score of 1 is considered just detectable).

Amongst the reference closures, oxidised characters measured for the reference 2 and ROTE closures were similar, at **0.4** and **0.3** respectively, but the reference 3 closure was rated as 'most' oxidised at **2.2**. The honey character was similar for the reference 2 and ROTE closure (**1.7** and **1.3** respectively) while the Altec closures were all very similar at **2.0** or **2.1**. Overall fruit aroma and flavour for the Altec closures were all similar to, but slightly lower than, the reference 2 closure. The presence of some aromas (*eg* TCA or oxidised characters) has been observed to affect panellist perception of fruit intensity.

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 assessment at 18 months, 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.

**Note:** While we have every confidence in these results, factors such 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.

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## 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.

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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**Randell Taylor**  
**Acting Manager-Analytical Service**  
23 November 2005

## Attachments:

### Chemical and Physical Data

**Table 2. Summary Comparison of Initial and 36 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<sub>420</sub> in Wine bottled under Reference and ALTEC Closures.**

### Sensory Data

**Table 3: 36 month sensory testing of Reference Closures**

**Table 4: 36 month sensory testing of ALTEC Closures**

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

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**Table 2. Summary Comparison of Initial and 36 Month Chemical and Physical Testing on Reference and ALTEC Closures.**

	<b>Initial</b>	<b>36 month</b>	<b>Initial</b>	<b>36 month</b>	<b>Initial</b>	<b>36 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	OD <sub>420</sub> a.u.	OD <sub>420</sub> a.u.
Mean	38	<b>21.7</b>	111	<b>92.7</b>	0.047	<b>0.071</b>
Std deviation	2	2.1	1	2.7	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	OD <sub>420</sub> a.u.	OD <sub>420</sub> a.u.
Mean	39	<b>17.5</b>	113	<b>86.3</b>	0.052	<b>0.084</b>
Std deviation	2	4.6	2	8.1	0.004	0.005
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	OD <sub>420</sub> a.u.	OD <sub>420</sub> a.u.
Mean	39	<b>13.5</b>	112	<b>79.8</b>	0.052	<b>0.087</b>
Std deviation	2	4.1	2	7.5	0.003	0.006
n	12	12	12	12	12	12
<b>Altec P0</b>	Free SO <sub>2</sub> mg/L	Free SO <sub>2</sub>	Total SO <sub>2</sub> mg/L	Total SO <sub>2</sub> mg/L	OD <sub>420</sub> a.u.	OD <sub>420</sub> a.u.
Mean	38	<b>21.4</b>	111	<b>94.0</b>	0.050	<b>0.077</b>
Std deviation	2	1.4	2	3.0	0.003	0.001
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	OD <sub>420</sub> a.u.	OD <sub>420</sub> a.u.
Mean	38	<b>20.9</b>	112	<b>91.7</b>	0.049	<b>0.077</b>
Std deviation	2	1.6	2	2.5	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	OD <sub>420</sub> a.u.	OD <sub>420</sub> a.u.
Mean	39	<b>18.9</b>	113	<b>88.4</b>	0.050	<b>0.078</b>
Std deviation	2	1.7	2	2.6	0.002	0.002
n	12	12	12	12	12	12

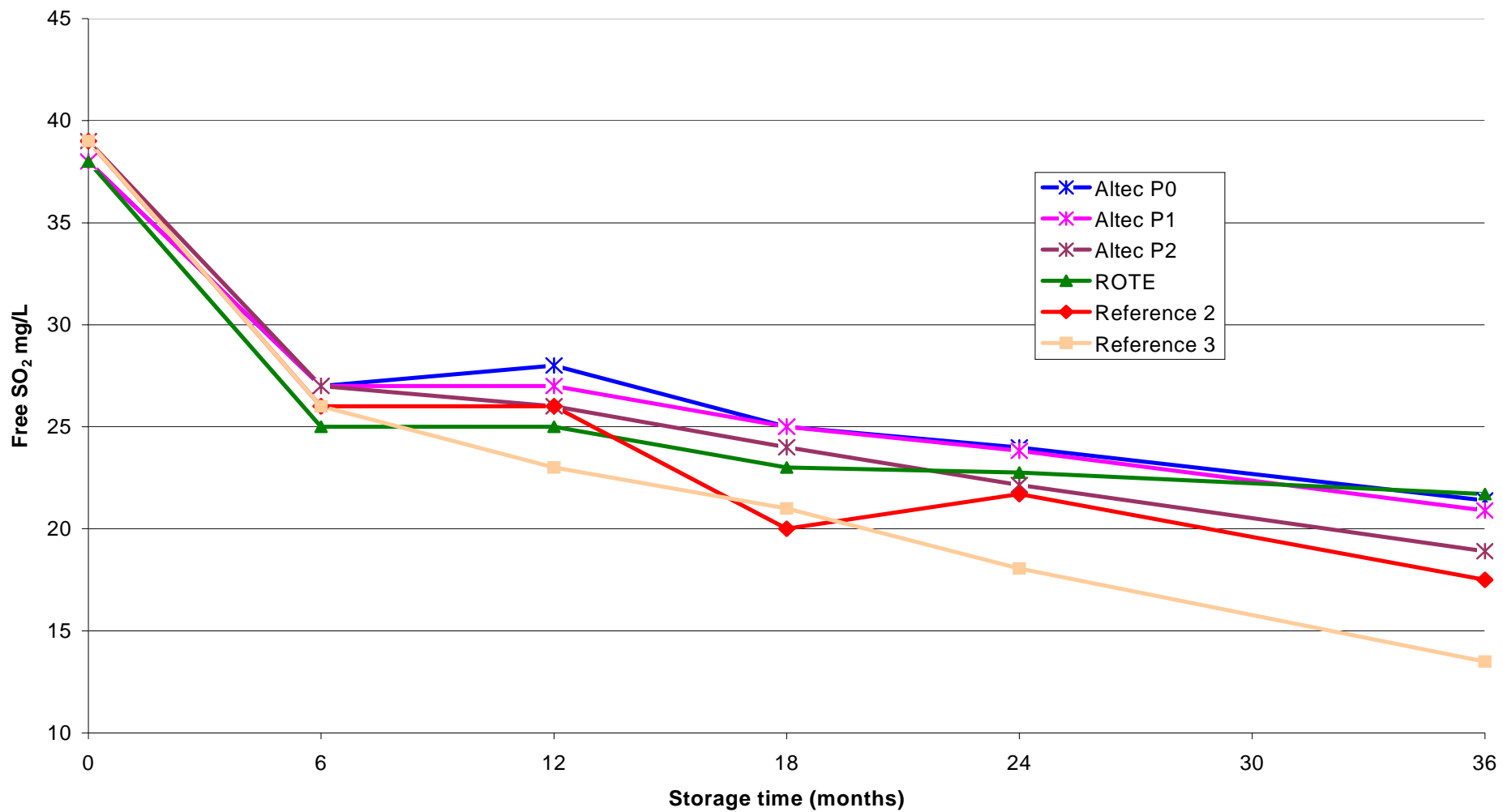
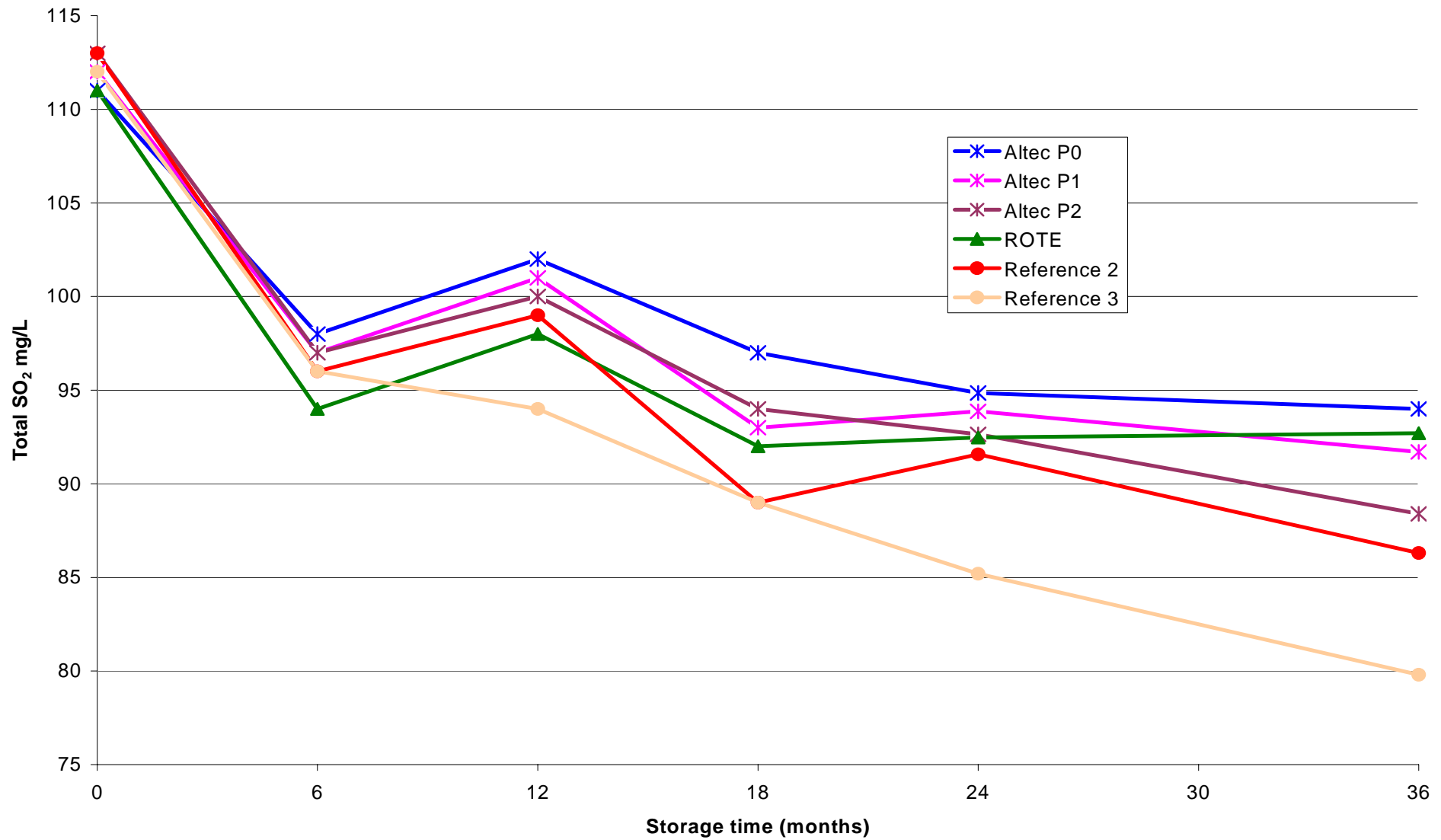


Figure 1: Change in free Sulfur Dioxide in Wine bottled under Reference and ALTEC Closures.



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

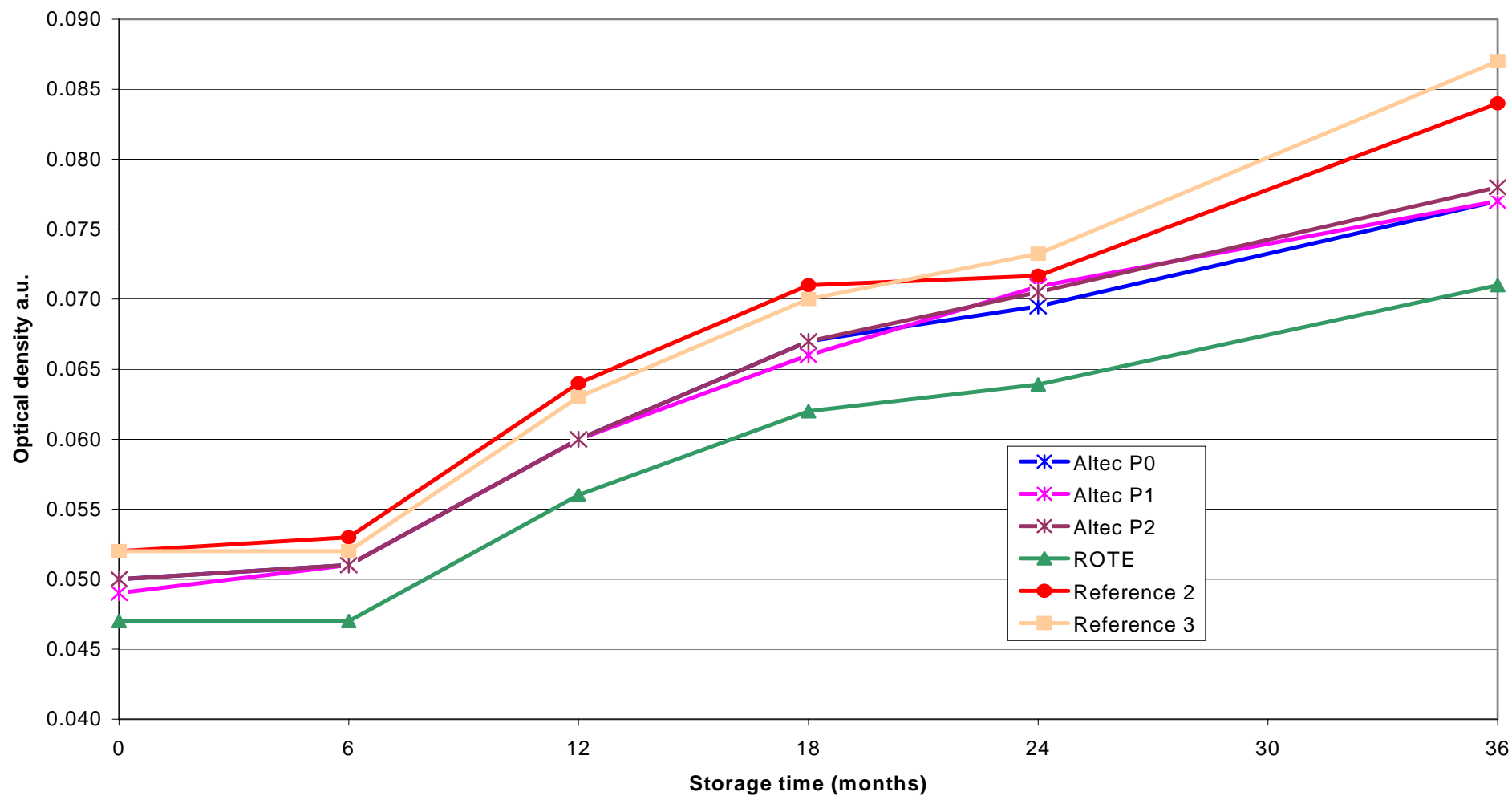


Figure 3: Change in OD<sub>420</sub> in Wine bottled under Reference and ALTEC Closures.

**Table 3: 36 month sensory testing of Reference Closures**

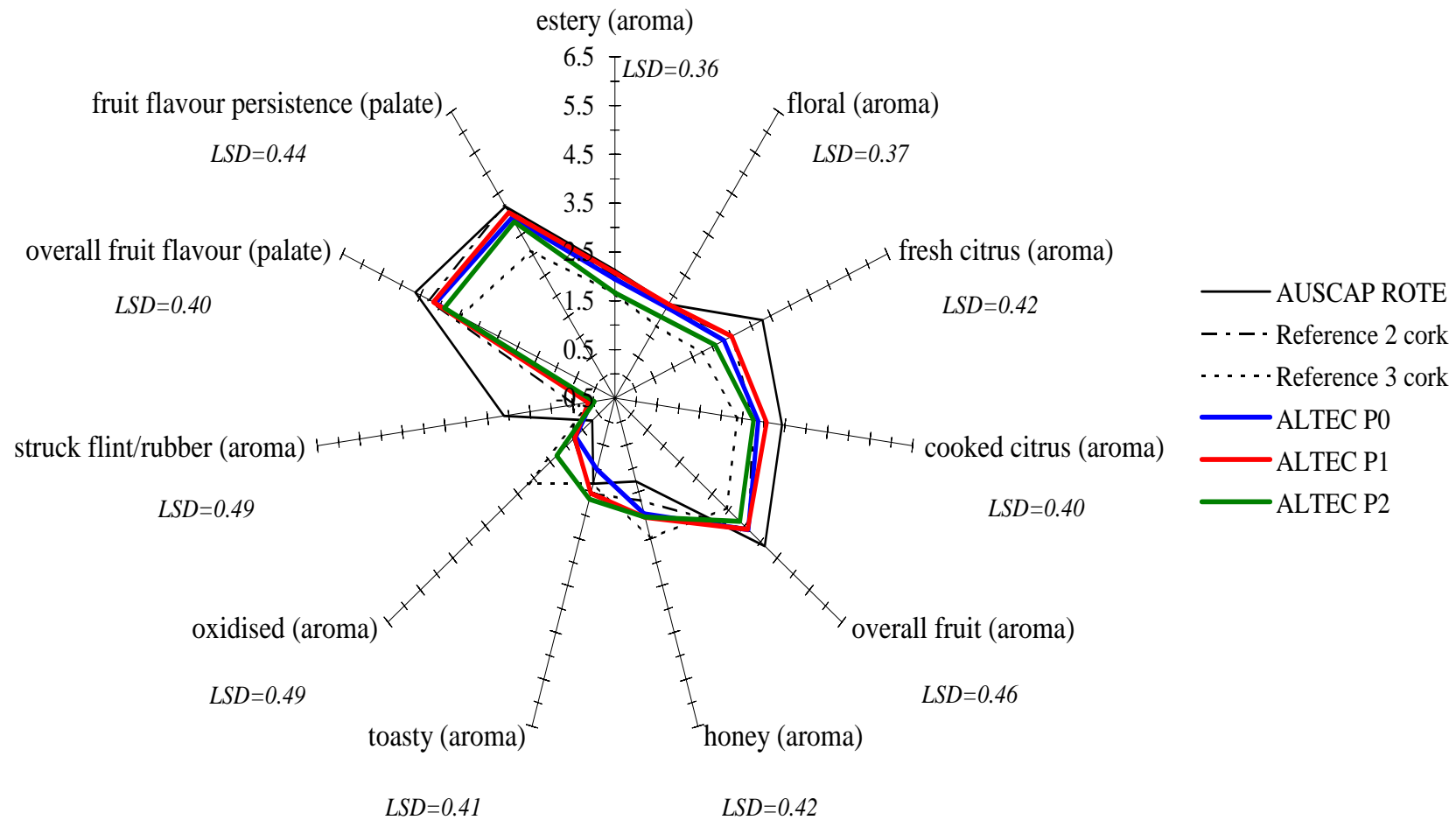
<b>closure</b>	<b>replicate</b>	estery (aroma)	floral (aroma)	fresh citrus (aroma)	cooked citrus (aroma)	overall fruit (aroma)	honey (aroma)	toasty (aroma)	oxidised (aroma)	TCA (aroma)	struck flint/rubber (aroma)	H <sub>2</sub> S/ cabbagey (aroma)	overall fruit flavour (palate)	fruit flavour persistence (palate)	TCA (palate)
<b>ROTE (Low SO<sub>2</sub>)</b>	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>		2.1	1.8	3.4	3.5	4.2	1.3	1.4	0.3	0.0	2.1	0.3	4.7	4.3	0.0
<i>Std dev.</i>		0.1	0.2	0.1	0.1	0.2	0.1	0.3	0.1	0.0	0.4	0.3	0.3	0.1	0.0
<b>Reference 2 cork</b>	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>		2.2	1.7	2.7	3.0	3.9	1.7	1.7	0.4	0.0	0.4	0.2	4.5	4.4	0.1
<i>Std dev. (exclud. Rep 1)</i>		0.4	0.4	0.4	0.2	0.4	0.0	0.2	0.3	0.0	0.1	0.3	0.1	0.2	0.2
<b>Reference 3 cork</b>	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>		1.7	1.3	1.8	2.4	3.0	2.5	1.3	2.2	0.1	0.1	0.0	3.5	3.1	0.0
<i>Std dev.</i>		0.4	0.4	0.3	0.2	0.5	0.2	0.3	0.6	0.2	0.1	0.0	0.5	0.5	0.0

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 ALTEC Closures**

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

<b>closure</b>	<b>replicate</b>	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 <sub>2</sub> S/ cabbagey (aroma)	overall fruit flavour (palate)	fruit flavour persistence (palate)	TCA (palate)
<b>Altec P0</b>	1	1.9	1.8	2.5	2.9	3.6	1.8	1.2	0.5	0.0	0.0	0.0	3.8	3.9	0.0
	2	2.1	1.6	2.1	2.6	3.3	1.8	0.8	1.3	0.0	0.0	0.0	4.0	3.7	0.0
	3	2.0	1.9	2.6	3.3	4.1	2.4	1.2	0.7	0.0	0.3	0.0	4.6	4.2	0.0
	4	1.8	1.7	2.3	2.9	3.7	2.1	1.0	0.7	0.0	0.0	0.0	4.3	4.1	0.0
<i>Mean</i>		<i>2.0</i>	<i>1.7</i>	<i>2.4</i>	<i>2.9</i>	<i>3.7</i>	<i>2.0</i>	<i>1.1</i>	<i>0.8</i>	<i>0.0</i>	<i>0.1</i>	<i>0.0</i>	<i>4.2</i>	<i>4.0</i>	<i>0.0</i>
<i>Std dev</i>		<i>0.1</i>	<i>0.2</i>	<i>0.2</i>	<i>0.3</i>	<i>0.3</i>	<i>0.3</i>	<i>0.2</i>	<i>0.4</i>	<i>0.0</i>	<i>0.2</i>	<i>0.0</i>	<i>0.4</i>	<i>0.2</i>	<i>0.0</i>
<b>Altec P1</b>	1	2.1	1.6	2.4	2.9	3.4	1.7	1.5	0.8	0.0	0.1	0.0	4.0	3.9	0.0
	2	2.0	1.6	2.8	3.3	4.2	2.7	1.9	0.6	0.0	0.1	0.0	4.6	4.4	0.0
	3	2.2	2.0	2.7	2.9	3.7	1.8	1.5	0.6	0.0	0.3	0.0	4.1	4.2	0.0
	4	2.1	2.1	2.3	3.3	3.4	2.1	1.4	1.3	0.0	0.1	0.0	4.2	4.0	0.1
<i>Mean</i>		<i>2.1</i>	<i>1.8</i>	<i>2.6</i>	<i>3.1</i>	<i>3.7</i>	<i>2.1</i>	<i>1.6</i>	<i>0.8</i>	<i>0.0</i>	<i>0.2</i>	<i>0.0</i>	<i>4.2</i>	<i>4.1</i>	<i>0.0</i>
<i>Std dev.</i>		<i>0.1</i>	<i>0.2</i>	<i>0.3</i>	<i>0.2</i>	<i>0.4</i>	<i>0.4</i>	<i>0.2</i>	<i>0.4</i>	<i>0.0</i>	<i>0.1</i>	<i>0.0</i>	<i>0.3</i>	<i>0.2</i>	<i>0.1</i>
<b>Altec P2</b>	1	1.5	1.6	1.8	2.7	3.4	1.9	1.6	1.4	0.1	0.0	0.0	3.8	3.7	0.0
	2	1.9	1.3	2.4	2.9	3.6	1.8	1.2	0.7	0.1	0.0	0.0	4.1	4.1	0.0
	3	1.9	2.0	2.6	2.9	3.9	2.0	2.1	1.0	0.0	0.0	0.0	4.1	4.0	0.0
	4	1.5	1.2	1.7	2.6	2.8	2.6	1.8	2.4	0.0	0.0	0.0	3.7	3.7	0.0
<i>Mean</i>		<i>1.7</i>	<i>1.5</i>	<i>2.1</i>	<i>2.8</i>	<i>3.4</i>	<i>2.1</i>	<i>1.7</i>	<i>1.4</i>	<i>0.1</i>	<i>0.0</i>	<i>0.0</i>	<i>3.9</i>	<i>3.9</i>	<i>0.0</i>
<i>Std dev.</i>		<i>0.2</i>	<i>0.4</i>	<i>0.4</i>	<i>0.2</i>	<i>0.5</i>	<i>0.4</i>	<i>0.4</i>	<i>0.7</i>	<i>0.1</i>	<i>0.0</i>	<i>0.0</i>	<i>0.2</i>	<i>0.2</i>	<i>0.0</i>



**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 9 judges.