

Results obtained from the testing of the ‘reference closures’ included in the AWRI’s ‘commercial closure trial’

The AWRI ‘closure trial’, which began in May 1999, is a large comparative study of the technical performance of various wine bottle closures. In the 1999 trial a Semillon wine was bottled using fourteen different closures, and changes in the chemical and sensory properties of the wine sealed with each of the closures has been followed over time. Various publications have resulted from this work, most notably AWRI publication #666, and the most recent results relating to testing that was conducted four years after bottling were published in the AWRI 2003 Annual Report (pages 41–43), which is accessible via the AWRI’s web site (www.awri.com.au).

24 Following the first publication of results from this trial in July 2001, the AWRI was approached by a number of companies who were seeking to have the performance of their own closures evaluated in a similar manner. The AWRI’s Industry Services team and Analytical Service facilitated this by instigating a second trial that was conducted in a similar manner to the original trial. An article in the February 2002 issue of *Technical Review* (Godden 2002) invited parties that were interested in participating in the trial to contact the AWRI. The article also provided some information relating to the manner in which the trial would be implemented.

The second trial was instigated on a commercial-in-confidence basis, such that the AWRI is not able to disclose the names of the trial participants, nor discuss the performance of particular closures, unless authorised to do so in writing by the individual participants. To date, only the Sabate company has provided the AWRI with such authorisation. The costs of planning, bottling and conducting the trial were shared between the participants, including the cost of including three ‘reference closures’ (ROTE - roll-on tamper-evident ‘screw cap’, reference 2, 44 mm, and reference 3, 38 mm natural wine cork), against which the performance of other closures might be compared. It was considered important to include some ‘reference closures’ in order that wine industry personnel could form their own opinions on the relative performance of various closures, in situations where trial participants publish data from the trial relating to their own closures. The AWRI is aware that some trial participants have either published, or have presented to wine industry personnel, data relating to the performance of their own closures in this trial. Thus, the primary purpose of this article is to present data obtained from the testing of wine sealed with these ‘reference closures’.

It should be noted that the AWRI's participation in evaluating the closures included in this trial should not be taken to imply that the AWRI endorses these products. In addition, readers should note that the currently available stocks of some of the closures examined might differ from those available when the trial was implemented, and that the results reported here, or supplied by trial participants, represent the performance of each closure when used to seal only one wine under the conditions defined. Care should, therefore, be exercised in relating these results to other wine types, or wines bottled under different conditions. It should also be noted that the results to date only represent the performance of these closures up to 18 months post bottling, and that performance to this time point might not be indicative of future performance. The AWRI strongly advises that wine producers should not rely solely on the results of this, or any other AWRI trial, when making decisions on closure use, and that they should conduct their own evaluations of new closures prior to commercial adoption.

The trial wine was bottled on a contractual basis at *Vinpac's* ISO 9002-certified bottling facility at Angaston, South Australia, on 24 September 2002. Because the bottling line that was used for the 1999 trial was no longer available at the Angaston facility, the most similar available bottling line was used. The nature and degree of quality control applied to the bottling was similar, or exceeded that used for the 1999 bottling, and *Vinpac's* routine preventative maintenance was performed on all equipment prior to the commencement of bottling.

A Semillon wine from the 2002 vintage was used, and this wine was made from fruit grown in the same vineyard as the wine used for the 1999 trial. As far as was practical, the production methods used for the wine were also similar, including the yeast strain used. Notwithstanding that steps were taken to ensure that many aspects of the current trial were as similar as possible to those of the 1999 trial, care should be taken when interpreting and comparing the results obtained from the two trials.

A sample of the wine, taken from the tank at the winery approximately one week prior to bottling, was analysed by the AWRI's Analytical Service, and the resulting data are presented in Table 1. The composition of the wine is considered similar to that of other un-oaked Australian Semillon wines that have been analysed at the AWRI, and in many respects is also similar to the composition of the wine used for the 1999 trial (AWRI publication #666, Table 5). As with all of the testing conducted as part of this trial, most of the analytical methods used were approved methods as defined by the laboratory's National Association of Testing Authorities (NATA) accreditation. The laboratory's quality assurance measures, including standards, blanks, duplicates and control samples, were used where appropriate,

and bottles of wine sealed with each of the closures were tested in rotation. For analyses where it was possible to test multiple samples simultaneously, wine from only one bottle sealed with each of the closures was analysed at a time. Data obtained from analyses conducted on samples sealed with each of the closures during bottling, by staff of the *Vinpac Quality Control Laboratory*, are also presented in Table 1.

The screw cap closures used were 30 mm x 60 mm *Auscaps* with tin / polyvinylidenechloride (PVDC) liners, and were taken from stocks held by the contract bottler.

The batches of cork were obtained as random samples of stocks held by two large wine companies. In each case the cork had been subjected to the standard pre-use quality controls applied by each of those companies, and had been accepted as being both representative of the grades described, and suitable for use in commercial bottling. No further pre-use testing was performed by the AWRI on these or any of the other closures. All of the closures were used in accordance with specifications supplied by the relative manufacturers or suppliers, and all participants (except the companies from which the 'reference closures' were obtained) had a representative present during the bottling runs conducted with their closure(s).

The bottle used for the cylindrical closures was a 750 mL 'flint'-coloured *claret* bottle, manufactured by *ACI Glass Packaging*, Hawthorn, Victoria, (manufacturer's code 5372). For the ROTE closures, a 750 mL *Saverglass* 'flint'-coloured *classic bordelaise* bottle with a BVS (screw thread) finish was used.

Following bottling, the bottles were placed into twelve-bottle cartons which were stored upright for several days before being inverted. The wine was stored in the same facility at The University of Adelaide's *Hickinbotham Roseworthy Wine Science Laboratory*, as continues to be used for wine from the 1999 trial. This facility is maintained at a temperature of approximately 17°C with a relative humidity of approximately 55%. During bottling each bottle was numbered. During the 48 hours following bottling, and then at 6, 12 and 18 months, these numbers were used to randomly select bottles of wine sealed with each of the closures for testing. Sensory analysis was conducted at the 6, 12 and 18 month time points.

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Table 1: Wine composition prior to and during bottling

Analyte	Result recorded one week pre-bottling	Mean result record during bottling
Specific gravity	0.9922	
pH	2.98	
Alcohol %v/v	11.6	
Titrateable acidity g/L	7.0	
Free sulfur dioxide mg/L ^a	24	39 (2) ^b (n=24)
Total sulfur dioxide mg/L ^a	92	111 (2) (n=24)
Glucose+fructose g/L	0.5	
Volatile acidity as acetic acid g/L	0.08	
Succinic acid g/L	0.4	
Malic acid g/L	2.5	
Lactic acid g/L	0.2	
Acetic acid g/L	Not detected	
Tartaric acid g/L	3.2	
Citric acid g/L	0.2	
Dissolved carbon dioxide g/L ^c	1.58	Pre sparging: 0.84 (0.01) (n=75) Post sparging: 0.57 (0.08) (n=40) Pre + post sparging combined: 0.75 (0.14) (n=115)
Dissolved oxygen g/L ^c		Pre sparging 0.46 (0.09) (n=75) Post sparging: 0.46 (0.07) (n=40) Pre + post sparging combined: 0.46 (0.08) (n=115)
Laccase activity	Not detected	
Pink colour	Not detected	
Pinking susceptibility	7	
Precursor content	78	
Ascorbic acid ^d	Not detected	
2,4,6-trichloroanisole	Not detected	
2,3,4,6-tetrachloroanisole	Not detected	
Pentachloroanisole	Not detected	
2,6-dichloroanisole	Not detected	
2,4-dichloroanisole	Not detected	
Copper mg/L	0.08	
Iron mg/L	0.7	
Potassium mg/L	457	
Sodium mg/L	85	
Calcium mg/L	81	
Heat stability	Pass	
Cold stability	Pass	
Atrazine, Azinphos-methyl, Benalaxyl, Captan, Carbaryl, Chlorothalonil, Chlorpyrifos, Chlorpyrifos-methyl, Diazinon, Dicofof (both the isomers and their metabolites), Dimethoate, Ethion, Fenarimol, Fenitrothion, Fenthion, Flusilazole, Iprodione, Maldison, Metalaxyl, Methidathion, Myclobutanil, Oxadixyl, Parathion methyl, Penconazole, Procymidone, Propiconazole, Simazine, Triadimefon, Triadimenol and Vinclozolin	Not detected	

^a an addition of approximately 20mg/L SO₂ was made to the wine between the two sets of analysis, ^b figures in parenthesis are standard deviations, ^c following the first bottling runs (which were conducted using the screw cap closures) the wine was sparged in order to reduce the concentration of dissolved CO₂, ^d approximately 20mg/L of ascorbic acid was added to the wine between when the sample was taken for analysis, and when the wine was delivered to the bottling facility.

During the planning of the trial, it was agreed with all participants that a minimum amount of testing would be conducted on wine sealed with each of the closures at all time points. Participants may commission additional testing of wine sealed with their closures at any time outside the specified trial time points. Some participants have taken this option, and therefore may present industry personnel with additional data relating to compositional variables not discussed in this article, or data relating to testing conducted at time points other than six, 12 or 18 months.

Chemical analysis

The results of chemical analyses conducted on wine sealed with each of the 'reference closures' for various compositional variables since the establishment of the trial, is presented in Table 2.

Table 2. Mean results of the chemical analysis of wine sealed with the ROTE, reference 2 and reference 3 cork closures, for various compositional variables at various time points

	pH	Free SO ₂ mg/L	Total SO ₂ mg/L	Ascorbic acid mg/L	OD ₄₂₀ a.u. ^a	Volatile acidity g/L
ROTE						
48-hours post bottling (n=12)	3.01 (0.03) ^b	38 (2)	111 (1)	14 (1)	0.047 (0.002)	0.08 (0.00)
6 months (n=12)		25 (1) ^c	94 (1) ^c		0.047 (0.004)	
12 months (n=12)		25 (2)	98 (2)		0.056 (0.002)	
18 months (n=12)		23 (1)	92 (2)		0.062 (0.002)	
Reference 2 cork						
48-hours post bottling (n=12)	3.01 (0.03)	39 (2)	113 (2)	15 (1)	0.052 (0.004)	0.08 (0.00)
6 months (n=12)		26 (2)	96 (2)		0.052 (0.006)	
12 months (n=12)		26 (1)	99 (2)		0.064 (0.003)	
18 months (n=12)		19 (3)	89 (5)		0.071 (0.004)	
Reference 3 cork						
48-hour post bottling (n=12)	3.01 (0.03)	39 (2)	112 (2)	14 (1)	0.052 (0.003)	0.08 (0.00)
6 months (n=12)		26 (2)	96 (2)		0.086 (0.116)	
12 months (n=12)		23 (2)	94 (4)		0.063 (0.003)	
18 months (n=12)		21 (2)	89 (3)		0.070 (0.004)	

^a au = absorbance units, ^b figures in parenthesis are standard deviations, ^c n = 11 for free and total SO₂

Sensory evaluation

The sensory analysis for this trial was performed using a panel of 10 judges, comprising AWRI staff with extensive experience in wine sensory evaluation. All but two of the judges had participated on the sensory panel for the 1999 closure trial.

At each testing period a discussion session was held, with the tasters assessing six of the wines from the current study. The selection of these wines was based on a preliminary evaluation and included the ‘reference closures’, and other samples considered to display the largest sensory differences. A list of the terms that was agreed upon by the panelists and used for ratings conducted at either the six, 12 and 18 month time points, is provided in Table 3.

Table 3. Sensory attributes rated at 6, 12 or 18 months

Attribute	Definition or composition of reference standard ^a
Estery ^{c,d}	
Floral ^d	
Citrus ^{c,d}	
Overall fruit aroma ^{b,c,d}	definition: citrus, pineapple
Honey (aroma) ^{b,c,d}	definition: honey
Toasty ^{b,c,d}	
Oxidised (aroma) ^{b,c,d}	definition: bruised apple, aldehyde
Glue/Plastic/Solvent ^{b,c,d}	1 drop plastic bonding glue dissolved in acetone (0.1% v/v)
TCA ^{b,c,d}	2,4,6-trichloroanisole (TCA) 5ng/L
Cork wood ^{b,c,d}	The aroma of fresh cork
Struck Flint/rubber (aroma) ^{b,c,d}	definition: rubber and freshly struck match/flint
H ₂ S/Cabbagey (aroma) ^{b,c,d}	definition: rotten egg, cabbage, sewerage
Reduced (palate) ^{b,c,d}	definition: rubbery, cabbagey
Overall Fruit flavour ^{b,c,d}	
Overall Fruit Persistence (palate) ^{b,c,d}	duration of perceived fruit flavour

^ain 100mL neutral white wine, ^b = rated at 6 months, ^c = rated at 12 months, ^d = rated at 18 months

Practice attribute-rating sessions were carried out in isolated tasting booths prior to the formal assessments. Examples of some of the four-year old Semillon wine under different closures from the previous closure trial were presented during training, to assist panelists in their recognition of attributes such as rubber/struck flint, plastic/glue-like, and oxidised.

For the formal assessment, samples were assessed over four sessions in blind tasting conditions using standardised procedures, and one sample of wine sealed with each of the closures in the trial was assessed at each session. The samples were presented to tasters under sodium lighting, 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 panelists scored defined attributes on a scale of 0–9; where 1 corresponds to ‘just detectable’, 5 to ‘moderate intensity’ and 9 to ‘very strong intensity’. Tasters were also given the opportunity to rate any other attributes evident in any sample.

The results of sensory analysis conducted on bottles of wine sealed with each of the ‘reference closures’ at six, 12 and 18 months following bottling, are presented in Table 4. **It is important to note that all attributes were rated on a scale of zero to nine.**

Table 4. Mean results (rated on a scale of zero to nine) of the sensory analysis of wine sealed with the ROTE, reference 2 and reference 3 cork closures, at various time points post bottling

	Estery (aroma)	Citrus (aroma)	Overall fruit (aroma)	Oxidised (aroma)	Honey (aroma)	TCA (aroma)	Stuck flint/ rubber (aroma)	H ₂ S/ cabbagey (aroma)	Overall fruit flavour (palate)	Fruit flavour persistence (palate)
ROTE										
6 months										
(n=4)	nr ^a	nr	5.0 (0.3) ^b	nr	0.5 (0.1)	0.0 (0.0)	0.5 (0.2)	nr	5.0 (0.2)	4.4 (0.4)
12 months										
(n=4)	3.6 (0.2)	4.3 (0.3)	4.9 (0.4)	nr	nr	0.0 (0.0)	1.4 (0.2)	0.5 (0.2)	4.7 (0.1)	4.7 (0.2)
18 months										
(n=4)	1.6 (0.4)	3.6 (0.3)	3.9 (0.4)	0.2 (0.2)	1.0 (0.2)	0.0 (0.1)	1.7 (0.3)	0.5 (0.1)	4.2 (0.2)	4.2 (0.2)
Ref. 2 cork										
6 months										
(n=4)	nr	nr	5.3 (0.5)	nr	0.7 (0.2)	0.0 (0.0)	0.2 (0.3)	nr	5.0 (0.3)	4.6 (0.3)
12 months										
(n=4)	3.6 (0.4)	4.2 (0.4)	4.8 (0.2)	nr	nr	0.0 (0.1)	0.4 (0.2)	0.1 (0.1)	4.7 (0.1)	4.6 (0.3)
18 months										
(n=4)	1.9 (0.4)	3.2 (0.2)	3.8 (0.3)	0.5 (0.4)	1.2 (0.2)	0.0 (0.0)	0.5 (0.2)	0.0 (0.0)	4.0 (0.3)	4.1 (0.3)
Ref. 3 cork										
6 months										
(n=4)	nr	nr	5.1 (0.8)	nr	0.9 (0.1)	0.5 (0.8)	0.2 (0.1)	nr	5.0 (0.7)	4.4 (0.7)
12 months										
(n=4)	3.4 (0.4)	4.2 (0.3)	4.8 (0.0)	nr	nr	0.1 (0.1)	0.3 (0.3)	0.1 (0.1)	4.4 (0.4)	4.3 (0.2)
18 months										
(n=4)	1.6 (0.2)	3.1 (0.4)	3.6 (0.5)	0.9 (0.3)	1.1 (0.3)	0.0 (0.0)	0.3 (0.2)	0.0 (0.1)	4.1 (0.3)	3.8 (0.2)

^a nr = not rated, ^b figures in parenthesis are standard deviations

The results of sensory ratings at 18 months for the attributes *citrus aroma*, *honey aroma*, *oxidised aroma*, and *stuck flint/rubber aroma*, conducted on bottles of wine sealed with all of the closures being examined in the trial, are presented in Figure 1.

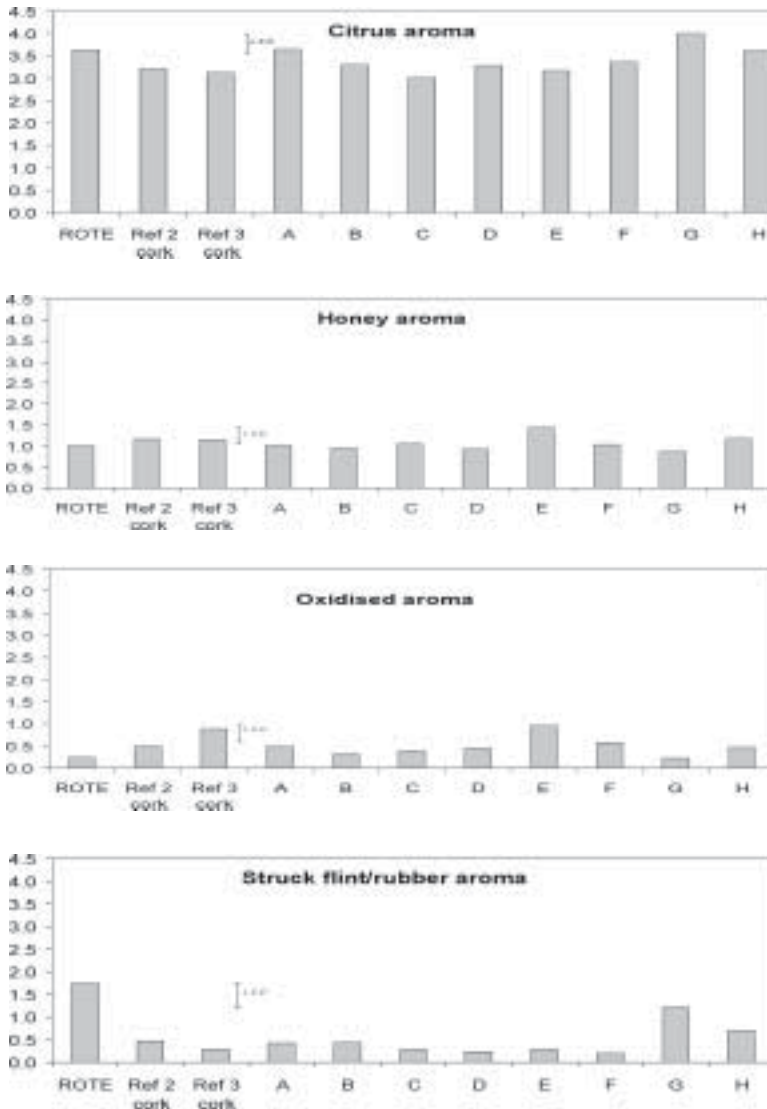


Figure 1. Mean ratings (on a scale of zero to 9) at 18 months post bottling for the attributes *citrus aroma*, *honey aroma*, *oxidised aroma* and *struck flint/rubber aroma*, for all closures

When interpreting the data presented in Table 4 and Figure 1, it is important to note that all sensory ratings were made on a scale of zero to nine. While many of the differences in ratings between closures are small on the zero to nine scale, in some cases these differences are statistically significant, and therefore their potential commercial or oenological importance should not be discounted.

While the AWRI is unable to reveal the identity of the majority of the closures being examined in this trial, a number of cork based closures to which novel treatments aimed at reducing TCA contamination of bottled wine have been applied, are included. The overall incidence of TCA taint identified in wine sealed with those closures, and with the reference cork closures, is considered to be very low up to eighteen months post bottling compared to that identified in the AWRI's previous closure trial which was bottled in May 1999 (Godden et al. 2001), or that discussed in another article published in this issue of *Technical Review* (Cowey and Godden *Technical Review* 151). It is also apparent from the data presented in Figure 1, that as a group, the closures in the current trial appear to be performing well after 18 months with regard to the retention of citrus aroma, and also maintain comparatively low ratings for the attributes *oxidised aroma* and *struck flint/rubber aroma*, when compared to many of the closures examined by Godden et al. 2001. As such, these results might be considered encouraging with regard to the possible future commercial availability of a range of closure types, which offer wine producers increasing predictability of outcome.

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References

Cowey, G.; Godden, P. Compositional data derived from AWRI *Advanced Wine Assessment Courses*: concentrations of *Dekkera/Brettanomyces*-derived compounds and of isovaleric (3-methylbutyric) acid; the incidence of 2,4,6-trichloroanisole (TCA) taint in bottled wine, and; the ullage spaces in screw capped bottles, in a selection of wines from around the world. *Technical Review* (151): 8–18; 2004.

Godden P.W.; Francis I.L.; Field J.; Gishen M.; Coulter A. D.; Valente P.; Hoj P.B.; Robinson E. Wine bottle closures: physical characteristics and effect on composition and sensory properties of a Semillon wine. 1. Performance up to 20 months post-bottling. *Aust. J. Grape Wine Res.* 7(2): 64–105; 2001 (AWRI publication #666).

Godden, P. Proposed commercial closure trial to be conducted by the AWRI Analytical Service. *Technical Review* (136): 1–2; 2002.

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