

Update on the AWRI trial of the technical performance of various types of wine bottle closure

Author's note: This study was not designed to be a survey of closures available in the market place, and not all of the closures available at the time the trial commenced were included. Readers should also 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 represent the performance of each closure when used to seal only one wine, under the conditions defined. Care should, therefore, be exercised in relating the results to other wine types, or to wines bottled and stored under different conditions. It should also be noted that some closures may be suitable for use in products with a short shelf life and may perform well over a particular time span, but less well over a longer term. Winemakers, therefore, need to assess the anticipated shelf life of the wine to be bottled when making decisions on closure use.

Analysis of the concentration of sulfur dioxide at 36 months post bottling

6 The first results from the AWRI Closure Trial were published in the *Australian Journal of Grape and Wine Research (AJGWR)* (Volume 7, number 2) (Godden et al. 2001a)*. That publication provides results up to 20 months post bottling, and copies can be obtained by contacting Ms Val Rechner at asvo@waite.adelaide.edu.au or by telephone on (08) 8303 6607. An update outlining the sulfur dioxide (SO₂) concentration in wine sealed with each of the closures at 21 and 24 months post bottling was provided in *Technical Review 133* (Godden 2001)*, and has also been published in the *Proceedings of the 26th World Congress and 81st General Assembly of the Office International de la Vigne et du Vin*, (Godden et al. 2001b)*. In addition, an update outlining the SO₂ concentration in wine sealed with each of the closures at 30 months post bottling was provided in *Technical Review 137* (Godden 2002)*. Copies of the OIV Proceedings and the *Technical Review* articles are available from the Institute's library.

* The Institute wishes to make it clear that any reference to 'Tage' closures in these publications, and also in this publication, are references to closures that were supplied by Esvin Wine Resources, Auckland, New Zealand in May 1999, and not to closures supplied by Novembal. The Institute has been informed that the closures being examined were made by a Californian manufacturer, and not by Novembal, and Novembal has informed the Institute that these closures are not the same as Tage closures now being made by Novembal. The Institute takes no position on Esvin's right, or that of the Californian manufacturer, to make or supply closures under the name "Tage".

The primary aim of the trial is to assess a range of closure types for a broad range of performance variables, in order to develop protocols for the rapid and reliable evaluation of new or existing closures. A Semillon wine was bottled under controlled conditions in May 1999, at an ISO 9002 certified commercial contract bottling facility, using 14 different closures. The trial protocol was published in the May 1999 issue of *Australian Grapegrower and Winemaker* (Godden 1999), and sufficient wine was bottled to enable the testing of many variables for many of the closures for up to ten years. Results will be published periodically, and testing will continue for as long as it is considered that useful data is being generated. Readers are, therefore, asked to keep in mind that the results reported here, and in the other publications listed above, collectively represent the performance of the closures under examination only up to 36 months post bottling. It is possible that, over time, the relative performance of various closures may change and that closures which have apparently performed well to this point may be found to perform less well in future testing.

The concentration of free and total SO₂ in wine sealed with each of the closures at 36 months post bottling is provided in Table 1 and the results obtained at 30 months are included for comparative purposes.

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Table 1. Mean SO₂ concentration following 30 and 36 months of storage in an inverted position

		30 months	36 months
		inverted storage (n = 8) ^a	inverted storage (n = 20) ^b
Aegis	Free	6 (1) ^c	6 (2)
	Total	59 (4)	58 (6)
Altec	Free	16 (1)	17 (2)
	Total	81 (1)	83 (3)
Auscork	Free	8 (2)	6 (2)
	Total	64 (5)	63 (5)
ECORC	Free	5 (1)	4 (2)
	Total	55 (3)	47 (5)
Integra	Free	6 (2)	5 (2)
	Total	60 (4)	59 (6)
Nomacorc	Free	6 (1)	6 (2)
	Total	63 (2)	62 (4)
NuKorc	Free	9 (1)	9 (3)
	Total	68 (3)	67 (3)
One plus One	Free	14 (2)	12 (2)
	Total	76 (2)	75 (4)
Reference 2, 44mm cork	Free	12 (4)	9 (4)
	Total	73 (7)	67 (9)
Reference 3, 38mm cork	Free	9 (3)	8 (3)
	Total	66 (6)	67 (9)
ROTE	Free	17 (3)	17 (4)
	Total	81 (5)	84 (5)
Supremecorq	Free	7 (1)	6 (2)
	Total	62 (3)	58 (5)
*Californian "Tage"	Free	3 (1)	4 (1)
	Total	47 (6)	47 (5)

^an = 12 for Ref 2, Ref 3 and One plus One, n = 4 for ECORC and Californian "Tage", ^bn = 28 for ROTE, n = 14 for ECORC and Californian "Tage", ^cfigures in parentheses are standard deviations

The data presented in Table 1 can be regarded as an extension of Table 14 of Godden et al. (2001a)* and Table 1 of Godden (2001), and Godden (2002)*. Issues relating to individual bottling runs, and to variations in the concentration of dissolved oxygen in the wine at bottling which may have affected the results reported in Table 1, are discussed in detail in Godden et al. (2001a)*. While these factors are not considered to have affected the significance of the results in any substantive way for any of the closures, readers are encouraged to make themselves aware of these issues when interpreting these results or making decisions on closure use.

The results reported in Table 1 confirm trends that were evident in earlier testing. Wine sealed with the ROTE closure has retained SO_2 to a greater extent than all other closures, but the concentration was not significantly different to the Altec closure. The trend for the technical cork closures (Altec and One plus One) to retain more SO_2 than the synthetic closures has continued.

Full evaluation of the data obtained from sensory analysis conducted at 36 months has yet to be completed and will be reported elsewhere. However, previous analysis has indicated that browning and deterioration of this wine's sensorial attributes accelerates when the concentration of free SO_2 falls below approximately 10mg/L. As such, SO_2 concentration is considered a key indicator of closure performance and it should be noted that the concentration of free SO_2 has now fallen below this apparently critical level with all but three of the closures being examined.

Whilst SO_2 concentration is considered a key performance indicator, tainting of the wine has been found in previous testing to be an important factor with some of the closures that have tended to retain a higher concentration of SO_2 . All bottles of wine sealed with the Altec closure that have been sensorily assessed at all testing intervals have been rated as being tainted with TCA. Gas chromatography-mass spectrometry analysis has been performed on approximately 30% of these bottles, and the concentration of TCA present has typically been found to be in the range of 1 to 2 ng/L. However, data from this trial clearly demonstrates that even at a concentration as low as 1 ng/L, the presence of TCA suppressed the ratings for overall aroma intensity, and of positive fruit-derived characters in wine, during sensory evaluation. In addition, a greater proportion of bottles sealed with the other three cork-based closures, which have been opened for sensory evaluation after 24 and 36 months of storage, have been found to be tainted with TCA compared to those assessed during the first 18 months of the trial, and reported in Godden et al (2001a)*.

In addition to the issue of TCA taint with the cork-based closures, a low intensity reduced or rubber-like aroma has developed, and persists, in wine sealed with the screw cap closure.

Factors relating to the extraction of closures from bottles, their removal from a corkscrew and their manual reinsertion into the bottleneck, may be important with some of the closures being examined and readers are, therefore, encouraged to take these factors into consideration when making decisions on closure use. These variables were discussed in detail in Godden et al (2001a)*, and readers are encouraged to consult this document for

further details. Perusal of the data relating to these variables generated at 36 months indicates that there has been little if any notable change in these measurements since the 18-month time point, for any of the closures being examined.

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Closures - results from the AWRI trial three years post bottling

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Authors note: This study was not designed to be a survey of closures available in the market place, and not all of the closures available at the time the trial commenced were included. In addition, the currently available stocks of some of the closures examined might differ from those available when the trial was implemented, and the results reported represent the performance of each closure when used to seal only one wine, under the conditions defined. It should also be noted that some closures may be suitable for use in products with a short shelf life, and may perform well over a particular time span, but less well over a longer term. Care should be exercised in relating the results to other wine types, or to wines bottled and stored under different conditions.

Abstract

A Semillon wine was bottled using 14 different closures: a screw-cap type, four cork-based closures, and nine closures manufactured from synthetic polymer material. Closure performance was evaluated using a number of measures over 36 months of storage. Wine under the screw cap closure retained the greatest concentration of sulfur dioxide and had the slowest rate of browning. For the other closure types, the sulfur dioxide loss was highest in the group of synthetic closures, intermediate in the conventional corks, and least evident in the technical cork closures. The loss of sulfur dioxide was in general highly correlated with an increase in wine browning (OD_{420}). Closures which tended to result in the best retention of free sulfur dioxide had wine sensory scores for 'citrus' and other fruity aroma attributes that were generally high, whilst scores for the attributes 'oxidised' were relatively low. The opposite situation was observed for wine under closures that retained lower concentrations of free SO_2 . TCA taint was evident for the cork-based closures, at concentrations ranging from 1 to 201 ng/L. Plastic-type aromas were not evident with most synthetic closures. Wine sealed with the screw cap closure exhibited a low-level reductive character with storage, described by the sensory panel as *rubber-like* or *struck-flint*.

Abbreviations and definitions

ROTE - roll-on tamper-evident, 'screw-cap' or 'Stelvin[®]'- like closure; **technical cork closure** - a closure made from cork together with synthetic material, **extraction force** - the maximum force required to remove a closure from the bottle; **total extraction energy** - the total energy required to remove a closure from the bottle, **headspace pressure** - the pressure in the vacuity (headspace) between the closure and the wine, **OD_{420}** - the optical density of the wine at 420 nm (yellow or brown colour in the wine)

Introduction

It can be argued that closing the bottle in a manner that presents a reliable and consistent product to consumers, remains one of the wine industry's greatest technical challenges worldwide. Unpredictable problems due to the performance of the closure may only become apparent once the wine is bottled, and may be outside the winemaker's control. Closures manufactured from cork bark have been used for sealing wine bottles for centuries. In recent decades technical corks (cork-based closures that also contain a synthetic component), and synthetic corks manufactured from polymer materials, have become more widely available.

Screw cap closures were evaluated by the Australian wine industry during the 1970's. Eric *et al.* (1976) found that one of the three screw-cap closures that they evaluated performed well in comparison to the batches of natural cork included in their trial, and considered the result justified the use of the screw cap as a commercial closure for table wines. Rankine *et al.* (1980) considered that their study confirmed the 'superiority' of "Stelvin 358" and "323" closures compared to the batches of natural cork that they examined. However, use of screw cap closures for premium quality wine met with considerable resistance in the marketplace, and although the use of screw cap closures continued, they were mainly used for products that tended to be at low price-points.

More recently, there has been resurgence in the use of screw cap closures for premium quality wines in both Australia and New Zealand, as well as other parts of the world, notably the USA. The authors estimate that approximately 30 million screw cap closures will be used on premium, super premium and icon wines in Australia and New Zealand during 2002.

With a choice of closure types now available, wine producers wish to reliably evaluate and compare the performance of currently available closures, or closures which may become available in the future, and this factor, coupled with the problems discussed above, have provided the impetus for the current study. This paper presents some results obtained from a large comparative trial of various wine bottle closures being conducted at The Australian Wine Research Institute. The first results from the trial were published in the *Australian Journal of Grape and Wine Research* (Godden *et al.* 2001a)^{2,3} and provided results up to 20 months post bottling. Readers are encouraged to consult that document when interpreting the results presented here and if making decisions on closure use, particularly for details of the trial design and implementation, and wine storage conditions.

² The Institute wishes to make it clear that any reference to 'Tage' closures in these publications, and also in the present publication, are references to closures that were supplied by Esvin Wine Resources, Auckland, New Zealand in May 1999, and not to closures supplied by Novembal. The Institute has been informed that the closures being examined were made by a Californian manufacturer, and not by Novembal, and Novembal has informed the Institute that these closures are not the same as Tage closures now being made by Novembal. The Institute takes no position on Esvin's right, or that of the Californian manufacturer, to make or supply closures under the name "Tage".

³ Copies of this publication can be obtained by contacting Ms Val Rechner at asvo@waite.adelaide.edu.au or by telephone on +61 (0) 8 8303 6607.

Following the publication of the first disclosure of the data, results updates outlining the SO₂ concentration in wine sealed with each of the closures at 21, 24, 30 and 36 months post bottling have been published in the Australian Wine Research Institute *Technical Review* (Godden 2001, 2002a and 2002b)². Readers are asked to keep in mind that the results reported in the present paper, and in the previous reports, collectively represent the performance of the closures under examination only up to 36 months post bottling. It is possible that, over time, the relative performance of various closures may change, and that closures which have apparently performed well to this point may be found to perform less well in future testing.

The primary aim of the trial was not solely to compare the performance of a selection of currently available closures - although this has been a positive side benefit of the experiment- but to provide reliable data against which the performance of other closures can be compared, both now and in the future.

The broad objectives of the trial were to:

- collect high quality data on many variables of closure performance, for a range of closure types;
- if possible, use this data to predict the future performance of closures for a range of variables; and
- use this information to develop testing protocols by which new or existing closures can be rapidly and reliably evaluated.

Trial design and bottling procedures

The study commenced in May 1999, when a dry, unwooded Semillon wine was bottled under controlled conditions at an ISO 9002-certified commercial bottling facility, using 14 different closures. The wine chosen for the trial had a low pH, a good balance of free and total SO₂, and fine aromatic properties that would help to emphasise any sensory differences that were later found attributable to the closures. At the time of bottling the wine did not contain any detectable TCA, the compound most commonly associated with cork-type taint in wine, or any compounds that were known to be responsible for premature oxidation of wine. The wine was also considered to be of sufficient quality to age for up to ten years, which is the possible maximum length of the trial.

The closures included in the trial are listed in Table 1. Details of the suppliers of each closure, and the extent to which various closures manufacturers and suppliers participated in the trial are provided in Godden *et al.* (2001)².

Table 1. Closure types being examined, and their source

Closure name	Type of closure	Closure name	Type of closure
Aegis	Synthetic, moulded	NuKorc	Synthetic, extruded
Altec	Technical cork	One plus One 'Twintop'	Technical cork
Auscork	Synthetic, moulded	Reference 2, 44mm cork	Natural cork
Betacorque	Synthetic, moulded	Reference 3, 38mm cork	Natural cork
ECORC	Synthetic, extruded	ROTE "roll-on tamper-evident"	Screw cap
Integra	Synthetic, moulded	Supremecorq	Synthetic, moulded
Nomacorc	Synthetic, extruded	Californian "Tage" ²	Synthetic, moulded

Each of the batches of natural cork was selected as a random sample taken from stocks held by two large Australian wine producers. Both batches of cork had been obtained by the wineries from prominent Australian cork supply companies, which in turn had sourced the cork, as far as can be ascertained, from leading suppliers in Portugal. Both batches of cork had been hydrogen peroxide bleached, and any coating material or printing had been applied in Australia. Four large Australian wine companies each independently graded randomly selected 100 unit samples of both batches of natural cork, using their own assessment procedures. All four companies assessed each batch as being representative of the grades described by the respective suppliers.

The ROTE closures were supplied by the contract bottler of the trial, and of the remaining closures, all except Supremecorq were supplied directly by the manufacturers or agents of each closure, by invitation. Supreme Corq Inc. declined to participate in the trial, and accordingly did not provide a sample of its closures. As a consequence, the Supremecorq closures were taken as a random sample of stocks held by a major Australian wine producer. ECORC supplied closures, but subsequently indicated that it wished to withdraw from the trial.

It was intended that each of the cylindrical closures would be used for duplicate runs of 300 bottles each, and that a single run of 800 bottles would be performed with the screw cap closure. The bottling order was reversed for the second bottling runs, in order to counteract any variables of the bottling process (such as the concentration of dissolved oxygen in the wine or wine temperature), that may have affected the subsequent development of the wine after bottling.

The experimental protocol was motivated by the need to generate data of practical relevance, and all aspects of the design were matched as closely as possible to current Australian wine industry practice. In addition, extensive consultation with closure suppliers ensured that the bottling procedures were optimal for each of the closures used.

Randomly selected bottles sealed with each of the closures were tested periodically, and where possible an equal number of bottles from each bottling run were used for each testing event. Major testing was conducted during the 48 hours following bottling, and after three, six, 12, 18, 21, 24, 30 and 36 months of storage.

The tests conducted on the wine fall into three main categories:

- Physical measurements relating to the removal of closures from bottles and from a corkscrew, and their manual reinsertion into the bottleneck.
- Chemical analyses of wine sealed with each of the closures.
- Sensory analysis of wine sealed with each of the closures.

Only results relating to the key chemical variables of sulfur dioxide (SO₂) concentration, browning of the wine (OD₄₂₀) and results of sensory evaluation, will be presented here. However, details of the performance of various closures for the physical measurements may be important for wine producers when making decisions on closure use, and readers are asked to consult Godden *et al.* (2001)² for details.

Chemical Measurements

Sulfur dioxide concentration

Changes in the concentration of SO₂ over time are of great interest when assessing the development of bottled wine. The mean concentration of free SO₂ in wine sealed with each of the closures at 21, 24, 30 and 36 month following bottling, is presented in Table 1.

At all testing intervals up to 36 months of storage, wine sealed with the ROTE closure contained the highest concentration of free SO₂, and the two technical cork closures (Altec and One plus One) contained the second and third highest concentrations of SO₂, respectively. The SO₂ concentrations in wine sealed with the ROTE and Altec closures was not significantly different. Wine sealed with the technical cork closures tended to retain SO₂ better than wine sealed with the synthetic closures and natural cork. Natural cork, especially Reference 2 up to 30 months post bottling, tended to retain SO₂ better than wine sealed with the synthetic closures being examined. Previous research studies (Eric *et al.* 1976, Rankine *et al.* 1980) also found that ROTE closures preserved the concentration of SO₂ in a range of bottled wines, to a greater extent than natural cork closures.

Godden *et al.* (2001)² found an apparently critical concentration of 10 mg/L of free SO₂ in the Semillon wine being examined, below which the rate of deterioration of the wine in terms of the magnitude of rating of *oxidised* aroma accelerated. Up to the 24 month time point, wine sealed with several of the synthetic closures had retained free SO₂ concentrations above, or close to, this level. However, by 30 months none of the synthetic closures had retained SO₂ at this concentration and the ratings of *oxidised* aroma for wine sealed with these closures had increased substantially (see below). It is of interest that all of the synthetic closures that are most commonly used commercially in Australia have behaved very similarly in terms of SO₂ retention at all testing intervals up to 36 months. It should be also noted that while many of the apparent differences between the SO₂ concentrations in wine sealed with various closures are small, they might still be considered to have oenological or commercial importance in terms of browning of the wine, and ratings for oxidised aroma at various time points.

Table 1. Mean SO₂ concentration for each of the closures^a following 21, 24, 30 and 36 months of storage in an inverted position

		21 months (n = 8) ^b	24 months (n = 20) ^c	30 months (n = 8) ^d	36 months (n = 20) ^e
Aegis	Free	7 (3) ^f	8 (2)	6 (1)	6 (2)
	Total	69 (10)	67 (3)	59 (4)	58 (6)
Altec	Free	17 (3)	18 (2)	16 (1)	17 (2)
	Total	83 (3)	83 (2)	81 (1)	83 (3)
Auscork	Free	9 (4)	10 (2)	8 (2)	6 (2)
	Total	71 (7)	69 (4)	64 (5)	63 (5)
ECORC	Free	4 (2)	6 (1)	5 (1)	4 (2)
	Total	58 (5)	58 (6)	55 (3)	47 (5)
Integra	Free	8 (3)	9 (2)	6 (2)	5 (2)
	Total	68 (4)	67 (3)	60 (4)	59 (6)
Nomacorc	Free	8 (2)	9 (2)	6 (1)	6 (2)
	Total	68 (3)	68 (3)	63 (2)	62 (4)
NuKorc	Free	10 (2)	11 (1)	9 (1)	9 (3)
	Total	74 (4)	72 (1)	68 (3)	67 (3)
One plus One	Free	13 (2)	14 (2)	14 (2)	12 (2)
	Total	76 (3)	77 (3)	76 (2)	75 (4)
Reference 2, 44mm cork	Free	12 (4)	13 (4)	12 (4)	9 (4)
	Total	74 (7)	75 (8)	73 (7)	67 (9)
Reference 3, 38mm cork	Free	9 (3)	11 (2)	9 (3)	8 (3)
	Total	70 (5)	71 (3)	66 (6)	67 (9)
ROTE	Free	19 (3)	19 (3)	17 (3)	17 (4)
	Total	87 (4)	85 (5)	81 (5)	84 (5)
Supremecorcq	Free	8 (3)	9 (2)	7 (1)	6 (2)
	Total	68 (4)	66 (4)	62 (3)	58 (5)
Californian "Tage" ²	Free	3 (2)	5 (1)	3 (1)	4 (1)
	Total	57 (6)	54 (4)	47 (6)	47 (5)

^a Betacorcque SO₂ values were not determined at these time points. ^bn = 16 for the ROTE closure, ^cn = 36 for the ROTE closure, and 14 for the Californian "Tage"² and ECORC closures, ^dn = 12 for Ref 2, Ref 3 and One plus One, n = 4 for ECORC and Californian "Tage"², ^en = 28 for ROTE, n = 14 for ECORC and Californian "Tage"², ^ffigures in parentheses are standard deviations

Browning measures (OD₄₂₀)

Measurement of the optical density of wine at 420 nm (OD₄₂₀) indicates the level of brown colour in a wine. As the development of brown colour is generally considered to be a negative quality factor, this measurement has been considered useful in the determination of the extent to which wine is preserved when sealed with different closures. The OD₄₂₀ was measured in the same intact 12 bottles sealed with each of the closure types at approximately three months after bottling and then at approximately 10-weekly intervals, using a novel method recently developed at the Institute which can reliably measure brown colour in wine through the bottle. This has provided a unique opportunity to follow the development of browning in the same bottles over time, and the resulting data are presented in Figure 1.

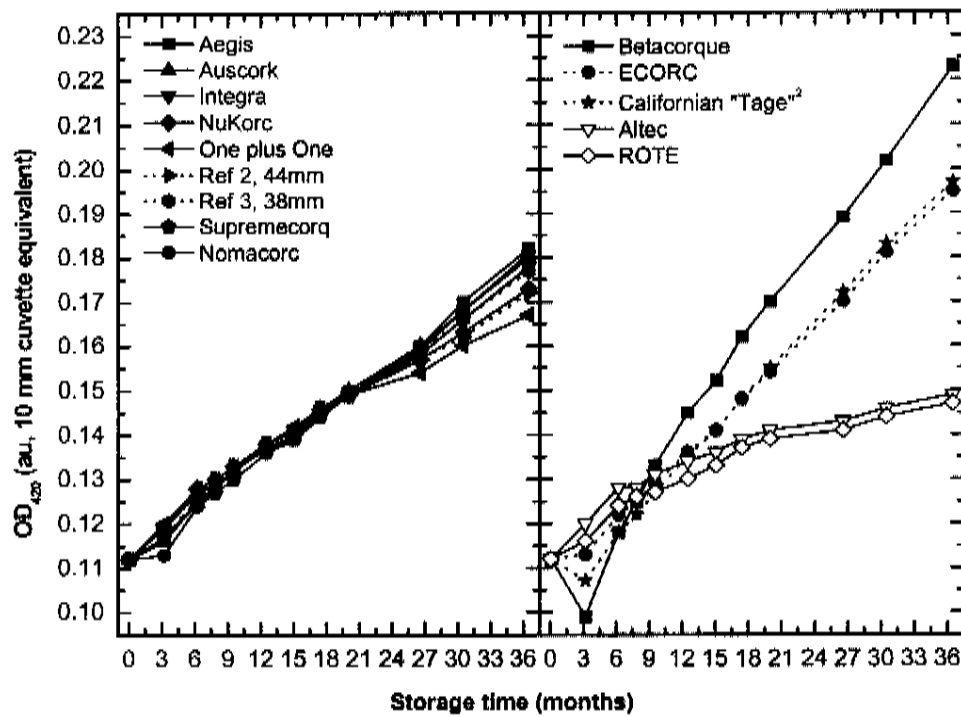


Figure 1. Changes in browning of wine in bottles sealed with each closure type (mean, $n = 12$) which had been stored inverted, as measured by optical density at 420 nm (OD_{420})

The data presented in Figure 1 indicate that trends of either higher or lower rates of brown colour development are apparent in data from early in the trial, even though those trends were not necessarily obvious or statistically significant at the stage when the analyses were performed. It is also apparent that, once established, any trends become more pronounced over time (except for the trend of apparent early reduction in brown colour which was evident with two closures).

The relationship between the OD_{420} measurements and SO_2 concentrations recorded at six, 12 and 18 months was investigated, in order to establish if loss of SO_2 from the wine could be used to predict the future browning of the wine. Strong relationships were found to exist, both with and without the inclusion of data for Betacorque, which displayed the notably lowest SO_2 concentration and highest browning of all of the closures from the earliest measurement. While generally there was a relationship between the degree of browning and SO_2 concentration each time testing was performed, the strongest relationships were found to exist between the SO_2 concentration recorded at six months, and the degree of browning after 12, and particularly 18 months of storage. Thus the concentration of SO_2 was an excellent predictor of brown colour in the wine sealed with each of the closures a full year before the browning actually developed.

Sensory evaluation

A high degree of care and effort has been invested in the way the sensory properties of the wines from the trial were assessed. In order to ensure that objective data was obtained, wines were independently assessed by ten experienced judges in isolated tasting booths under colour masking lights, using blind coded glasses and with each of the tasters assessing the wines in a different, random order. Eight replicate bottles of each closure have been assessed at each time point since the 12 month period, to allow evaluation of bottle to bottle variation among the closures. The assessors were not asked to give a quality judgement, but simply to rate the intensity of appropriate terms which were decided prior to the formal assessments during training and practice sessions. Judge performance criteria such as degree of reproducibility, discrimination and agreement with the other judges, indicated that as a group the judges performed well over the course of the study.

Figure 2 provides a representation of sensory data obtained at the 36 month period, showing a principal component analysis 'map' of the average sensory data for each of the replicate bottles assessed.

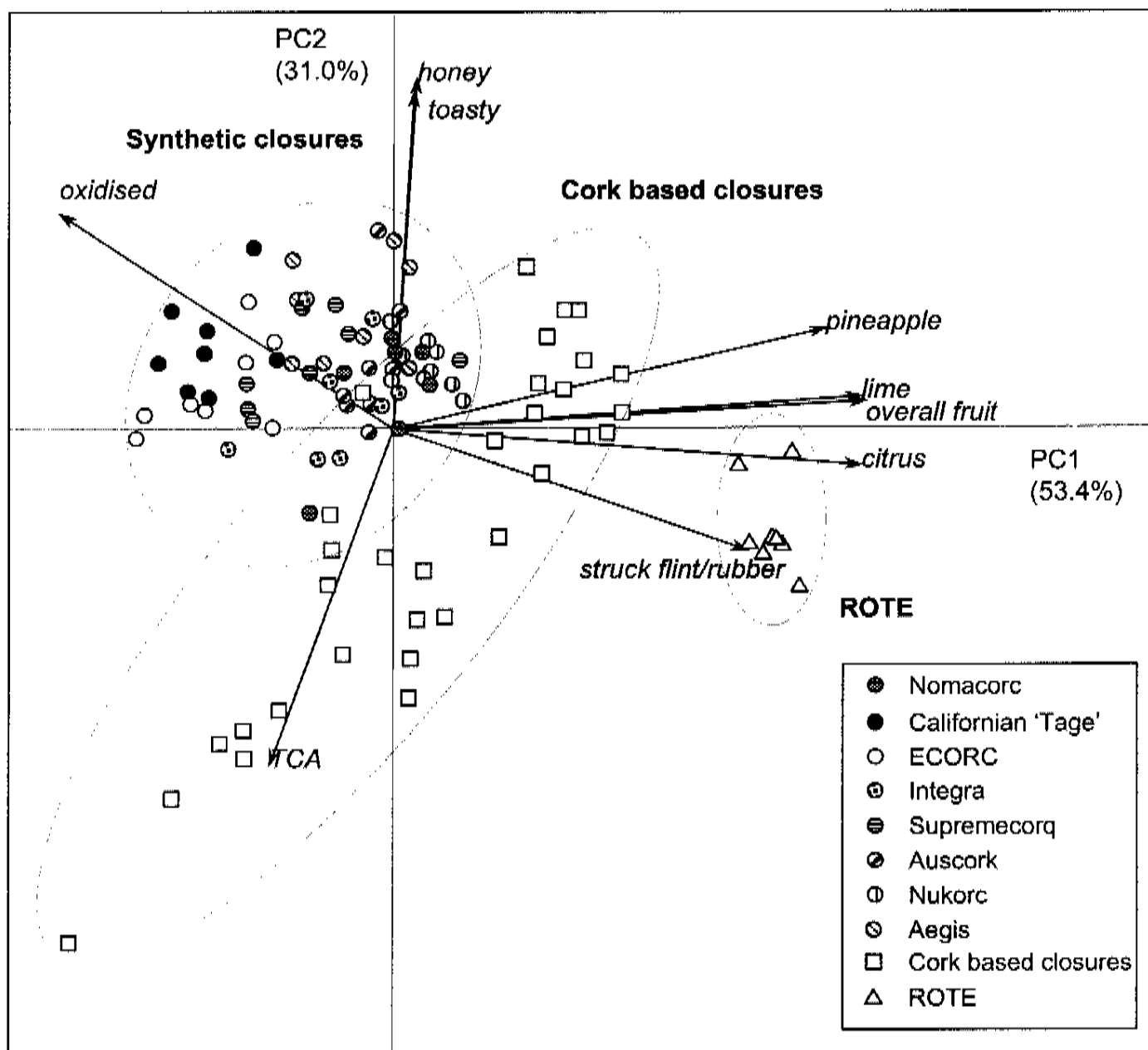
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Figure 2. Principal component biplot for mean aroma ratings (n=10 judges) of each of the replicate bottles of the 13 closures assessed after 36 months post-bottling.

The figure shows how the closures were grouped, with those samples in close proximity being rated similarly, while samples which were dissimilar being distant from each other. The attribute vector arrows provide information regarding how the samples differed. This representation shows that the eight ROTE wines assessed were tightly clustered, being rated highest of all the samples in 'citrus', 'pineapple', 'lime' and 'overall fruit' attributes, and also the highest in a 'struck flint/rubber' attribute. In contrast, the synthetic closures were grouped to the left of the diagram, being rated considerably higher in 'oxidised' aroma than the ROTE samples, and less intense in the fruit type attributes. Within the group of synthetic closures there are other groupings evident, with the ECORC and Californian "Tage"² closure samples, situated to the far left of the figure, being among the highest individual samples in the rating of 'oxidised' aroma.

The cork based closures are plainly less tightly grouped, with some examples being situated close to the ROTE samples, having relatively intense fruit aroma attribute scores, together with relatively high honey and toasty aroma scores, while others are situated to the lower half of the diagram, being rated relatively high in 'TCA' aroma. In addition, a single cork-based closure sample was situated within the synthetic closure group, being higher in 'oxidised' aroma than other cork based samples. No pre-screening of the wines for 'TCA' aroma was done for this study, and it is strongly evident that the aroma of the wines bottled with cork-based closures can be substantially affected when this character is present. It was instructive to count the number of closures which had a 'TCA' score greater than 1.5, in view of the fact that that the average 'TCA' score for the non cork-based closures was 0.1 on the 0-9 scale used to rate the aroma intensity (with the range for the non cork-based closures found

Figure 2. Principal component biplot for mean aroma ratings (n=10 judges) of each of the replicate bottles of the 13 closures assessed after 36 months post-bottling.



to be from 0 to 0.9). Each of the four cork-based closures investigated in this study were affected by 'TCA' aroma by this criteria, with from two of the eight samples affected (the reference 3 cork) to all eight samples affected (the Altec closure). Six of the cork-based closure samples, which did not include any of the Altec closures, had an average TCA score greater than 4.0.

Perceived impact of the trial results

The publication of the first results of this study (Godden *et al.* 2001a) happened by chance to be at a time when announcements were made by some wine producers, particularly those from the Clare Valley region of South Australia, and members of the New Zealand Screwcap Wine Seal Initiative, that screw cap closures would be used for a portion of their production of certain wines. While it can be argued that the results of the trial support this move to use screw caps, the authors caution that only one white wine has been examined in the trial, and extrapolation of the results to other wine types, possibly bottled and stored under different conditions to those of the trial, is unsafe. While the results support the use of screw caps for the white wine in question, the results also demonstrate that some of the synthetic closures examined were also suitable up to two years of storage.

Cork has been virtually the only closure used to seal wine bottles for several hundred years, and a change to other closure types would require a fundamental shift in attitude by producers, wine trade and consumers alike. However, it appears that such an attitudinal shift has begun. It is considered that the major effect of the current trial has been to provide wine producers with independent and objective data on closure performance. The study is therefore considered by the authors to have provided wine producers with information on which they can base their own decisions on closures use, and has provided greater surety of outcome to those who wish to use alternatives to natural cork. The authors also consider that the current trial, by providing objective data on the performance of natural cork relative to other closures, will aid the cork industry to identify, and to potentially solve the problems, which were evident with the cork-based products examined. Independent analysis commissioned by the Australian Grape and Wine Research and Development Corporation (GWRDC) concluded that, due in part to these factors, the current trial could bring forward the availability of completely 'reliable' wine closures by approximately five years. If this were to be the case, it would result in a major economic benefit for the wine industry, and the wine-drinking public, worldwide.

CONCLUSION

The performance of a range of different wine bottle closures has been comprehensively assessed, principally in terms of their effects on the chemical composition and sensory properties of the unwooded Semillon wine. Sulfur dioxide concentration in wine sealed with each of the closures has been found a critical measure in this investigation. Loss of SO₂, presumably due to the degree of oxygen ingress allowed by the closure, has been found to correlate well with the development of brown colour in the wine, and also the rating of *oxidised* character during sensory evaluation, at all testing intervals up to 36 months of storage. Many of the closures

could be considered to have adequately preserved the wine during the first 12 months, and several up to 18 months. By 24 months of storage, most bottles sealed with most of the synthetic closures were considered to be showing adversely high *oxidised* character during sensory evaluation. However, it should be noted that several of the closures examined are marketed for use in wines with a shelf life of two years or less. After 36 months of storage, only wine sealed with the screw cap closure, most of the bottles sealed the cork-based closures, and a relatively low number of individual bottles sealed with various synthetic closures, could be considered not to exhibit an unacceptable level of oxidation. However, many bottles sealed with the cork-based closures opened after 36 months of storage were found to be tainted with TCA to some degree, as discussed below.

Wine sealed with the ROTE closure continues to retain the greatest concentration of SO₂ at 36 months, but the concentration was not significantly different to that in wine sealed with the Altec closure. Wine sealed with the ROTE closure was also found to have retained greater overall freshness and positive fruit-derived sensory characters than wine sealed with any other closure, and also demonstrated the lowest level of brown colour development. However, wine sealed with this closure continued to display a low-level reductive, *rubbery* or *struck flint* character during sensory evaluation. This aroma is likely to be related to chemical changes of the wine volatile compounds over time under the relatively anaerobic conditions, rather than as a result of absorption of extraneous compounds from the closure during storage. All bottles sealed with the Altec closure were again found to be tainted with TCA, albeit at a relatively low concentration. However, data from this trial clearly demonstrates that even at concentrations as low as 1 ng/L, the presence of TCA suppressed the ratings for overall aroma intensity, and of positive fruit-derived characters in wine, during sensory evaluation. In addition, a greater proportion of bottles sealed with the cork-based closures which have been opened for sensory evaluation after 24 and 36-months of storage, have been found to be tainted with TCA, in a range from 1 to 201 ng/L, compared to those assessed during the first eighteen months of the trial. However, the relatively low sample numbers assessed during sensory evaluation makes comment on the overall incidence of TCA taint in the batches of these closures that are being assessed, unsafe.

Considering each of the closure types used, no one closure tested could be considered entirely suitable by all the criteria assessed. Factors such as the anticipated storage time and temperature conditions, are likely to be important considerations for wine producers when making decisions on closure use, as well as the relative importance of avoidance of TCA taint or tolerance of a rubber-like aroma compared to retention of freshness

The concentrations of free SO₂ recorded in wine sealed with each of the closures at 6 months, were also found to be excellent predictors of free SO₂ concentration and browning of the wine after 24 months, and to a lesser extent the rating of *oxidised* aroma in wine sealed with the different closures at 24 months. Thus, it is possible that well conducted and suitably replicated measurements of free SO₂ performed as early as 6 months following bottling, could be used by wine producers to predict the future performance of various closures over a shelf-life of up to two years.

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