



CLOSURE STUDY REPORT



Introduction

Through this trial, Sabaté wanted to contribute to the closure debate and confirm its commitment to developing better performing closures, working with the trade to provide an ever improving quality of wine to the consumer.

Sabaté proposed that the trial be driven by a panel of respected UK wine trade professionals who would design, participate in and support the trial. Sabaté's transparency, both during the trial process and now through this report, is a contribution to the breaking down of the traditional barriers of secrecy and denial built over the years between the cork industry and the rest of the trade.

Sabaté have hoped to spark further lively and relevant discussion throughout this trial, and are now confident that the results will do just that. This will no doubt broaden the debate and further illustrate Sabaté's commitment to proactive involvement and transparent communication with the wine trade decision makers.

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Background

Sabaté spent ten years of research in developing a composite closure that would be totally innovative, consistent and reliable. The resulting closure was Altec. Launched in 1995, it far exceeded the wine industry's expectations and sales soared. Sabaté carried out quality control checks on random batches of closures leaving the factory, but the technology did not exist at that time to analyse every batch of closures leaving the factory. In 1997, Sabaté received the first set of results from the Australian Wine Research Institute (AWRI) trial and although the Altec closures performed consistently well, all had shown detectable levels of TCA throughout the samples. A period of negative publicity followed and for a recovery to be successful, Sabaté needed a dynamic strategy that would become industry acknowledged. Having recognised the need to constantly improve its Altec closures and thus better serve the wine market's needs, Sabaté proposed the following actions:

1 - Implementing a quality control standard whereby every batch of Altec is analysed using solid phase microextraction (SPME), gas chromatography (GC) and mass spectrometry (MS) for releaseable TCA before leaving the factory. Any batch with an average releaseable TCA of more than 3ppt is rejected and not sold as wine closures.

2 - Commissioning an original and open research trial led by a forum of UK wine trade professionals in order to add scientific data to the closure debate.

THIS TRIAL PRESENTED AN EXCELLENT OPPORTUNITY TO FINALISE AND VALIDATE THE RESULTS OF TWO R&D PROJETS LAUNCHED IN RECENT YEARS :

A - DIAMOND PROCESS : substantial financial investment, since 1996, has permitted an intense research and development programme to create a CO₂ super-critical extraction process that treats the cork flour in order to remove TCA.

B - PERMEABILITY : to determine the effect of different closure permeability levels on wines.



FIGURE 1
ALTEC CLOSURE

Summary

A PANEL OF RESPECTED AND OPINION-FORMING MEMBERS OF THE UK WINE TRADE WAS CONVENED IN ORDER TO MAKE THIS TRIAL AS SCIENTIFICALLY VALID AND INDEPENDENT AS POSSIBLE.

2,640 bottles of wine (50:50 Cabernet Merlot, Chile and VDP Gascogne, France as chosen by the panel) were closed with 6 various closures, including 3 new Altec formulation prototypes with varying permeabilities, 2 original Altec formulation closures (one pre quality control procedure and one post) and a ROTE at the Waverley Group's bottling facility. The panel chose to actually taste 660 bottles across 5 tastings and to have all 2,640 bottles technically analysed at Campden & Chorleywood Food Research Association (CCFRA). CCFRA was selected by the panel because of their development of a new technique to technically analyse for TCA down to 0.2ppt (parts per trillion) which had previously been unattainable - no other trial to date has measured down to this level.

The panel members defined the following criteria for scoring the wines at the tastings: fruit, freshness, finish, overall and finally whether they perceived TCA to be present in the sample.

THE TRIAL'S CHOSEN STATISTICIAN, RUSSELL GERRARD OF CITY UNIVERSITY, LONDON WAS GIVEN BOTH THE TASTER'S AND THE TECHNICAL RESULTS TO ANALYSE. HIS FINDINGS INCLUDE:

> All closures within the trial measured average releasable TCA batch levels below 2ppt and were therefore well within Sabaté's quality control standard of <3ppt.

> The diachronic results from the trial indicate that both prototype 2 and 3 performed better than prototype 1 (whose permeability was the original Altec formulation) according to the fruit, freshness, finish and overall scores. Prototype 3 actually slightly outperformed prototype 2, but internal trials at Sabaté, confirmed by a US panel(*), indicated browning on wines closed with this permeability. The permeability of prototype 2 is now being used by Sabaté in the manufacture of Evolution and Reference closures.

> The data shows that the notion of a taster TCA threshold is not tenable. On the whole, the proportion of bottles identified as containing TCA increases steadily as the actual measured TCA content increases.

> 95% of the tasters involved in the trial had a consistently higher chance of detecting TCA in white wines than in reds.

> Sabaté's three prototypes showed average releasable TCA levels under 0.2ppt (according to the trial's statistician, anything under 0.2ppt is not actually present). These prototypes had been treated using the super-critical CO₂ extraction process, known as the Diamond process.

() Panel of Californian winemakers, commissioned in 2002 by Sabaté in collaboration with ETS laboratories, Santa Helena, California.*

^ Aims

1 > To determine the effect of varying levels of closure permeability on wines

2 > TCA

To assess any correlation between perceived and actual TCA

To test Sabaté's new quality control standard of <3ppt average releasable TCA per batch

To assess the performance of a viable TCA-cleaning process

3 > To contribute valid scientific data to the closure debate.

Firm scientific findings

Increased dialogue between all sectors of the wine trade

The development of better closures with performance benefits

Better understanding of the relationships between perceived and actual TCA

BENEFIT ^

The Panel

TO ENSURE IMPARTIALITY WITHIN THE TRIAL, A PANEL OF RESPECTED AND OPINION-FORMING WINE WRITERS, BUYERS AND TECHNICAL MINDS WAS ESTABLISHED.

THE PANEL WERE ASKED TO DESIGN A TRIAL PROCESS THAT WOULD BE BEYOND SUSPICION OR REPROACH. THE FOLLOWING PEOPLE WERE INSTRUMENTAL IN SETTING UP THE TRIAL PROCEDURES AND PROTOCOLS.

INDEPENDENT

Rosemary George MW	Freelance
Derek Smedley MW	Freelance
Peter McCombie MW	Freelance
Liz Robertson MW	Freelance

PRESS

Charles Metcalfe	Freelance
Robert Joseph	Freelance
Jo Burzynska	Harpers
Nigel Huddleston	OLN
Tim Palmer	The Grocer

ON TRADE

Henry Chapon	Hotel du Vin
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RETAILERS

Howard Winn	J Sainsbury
Helen McGinn	Tesco
Julian Brind MW	Waitrose
Tony Allen	Oddbins

WHOLESALER

Alastair Maling MW	IWS
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If original panel members were unable to attend a tasting, another suitable taster was sent in their place, including Justin Howard-Sneyd and Laura Jewel of Sainsbury's, Lindsay Talas of Tesco, Jo MacDonald of Thresher Group and freelance journalist Andy Knott.

THE CLOSURES

THE FOLLOWING 6 CLOSURES WERE PROPOSED BY SABATÉ AND AGREED BY THE PANEL FOR ANALYSIS IN THE TRIAL:

3 prototypes with differing permeability levels, all of which had been treated with the supercritical CO₂ extraction process and should therefore contain <0.2ppt releasable TCA:

- PROT 1 STANDARD ALTEC FORMULATION**
- PROT 2 "PERMEABILITY +" FORMULATION**
- PROT 3 "PERMEABILITY ++" FORMULATION**

PRE QC

1 pre-quality control standard formulation Altec, untreated. Samples were taken from surplus stock manufactured before the <3ppt quality standard had been introduced around the time of the first AWRI trial.

POST QC

1 post-quality control standard formulation Altec, untreated. Samples chosen at random by Peter McCombie MW at the Sabaté factory in Perpignan after the <3ppt quality standard had been introduced.

ROTE 1

Roll On Tamper Evident closure hermetically sealed with no apparent TCA risk - standard closure used by Waverley/ IWS for library samples.

The Wines

THE FOLLOWING SELECTION OF WINES WAS MADE AND BOTTLED BY THE WAVERLEY GROUP. THE PANEL TASTED THE LISTED WINES AND SCORES WERE ATTRIBUTED ACCORDING TO THREE DISTINGUISHING FEATURES: FRESHNESS, FRUIT AND FINISH, TOGETHER WITH AN OVERALL SCORE.

WHITES

Soave, Italy

Pinot Grigio, Italy

VDP Gascogne, Varietal Colombard, France

REDS

Valpolicella, Italy

Cabernet/ Merlot, Chile

WHICH WINES WERE CHOSEN?

Wine	Freshness	Fruit	Finish	Overall
VDP Gascogne, France	4/6	4/6	4/6	15/20
Cabernet/ Merlot, Chile	5/6	5/6	5/6	16/20

These wines received the highest average scores from the tasters and were considered to be the most vulnerable to potential sensory deviation. These scores were used by the tasters as a benchmark for the trial tastings. At this point, samples of the chosen wines were sent to CCFRA for full analysis.

HOW MANY BOTTLES NEED TO BE TESTED?

Three months of complete data from Sabaté's quality control procedure were used to identify a statistically valid number of wines to be trialled. At the 480 bottle point the decrease in accuracy becomes insignificant, therefore 480 of each closure (50:50 red and white) were chosen to be tested (only 240 bottles of ROTE were tested due to the panel's belief that there would be less variance between bottles).

Of the 480 bottles, all were analysed at CCFRA using solid phase microextraction (SPME), gas chromatography (GC) and mass spectrometry (MS), and 120 of these bottles were actually tasted by the panel.

The tasting

TIMETABLE

The panel chose to taste the wine one month post bottling and every three months thereafter.

The actual dates according to taster availability were as follows:

> TASTING 1	December 2001
> TASTING 2	January 2002
> TASTING 3	May 2002
> TASTING 4	August 2002
> TASTING 5	January 2003

PROCEDURE

One week prior to each tasting, 72 bottles of each cork-based closure and 36 bottles of ROTE were delivered to CCFRA (396 bottles in total) for analysis without being tasted.

24 bottles of each cork-based closure and 12 bottles of ROTE were taken for actual tasting by the panel.

Samples of each bottle opened for tasting were immediately taken and sealed without air. All samples were collected by CCFRA and taken back to the laboratory within 8 hours.

Tasting was carried out 'blind'. Each bottle was unlabelled except for its unique identification number and ROTE bottles were decanted to ensure anonymity .

Tasters scored the wines against the pre-defined criteria, using the initial selection scores as benchmarks.

The panel were asked to complete the following information for each sample tasted:

UNIQUE BOTTLE ID	FRUIT	FRESHNESS	FINISH	OVERALL	TCA Y/N?	COMMENTS
Number used by Cube and CCFRA for bottle ID	Scored from 0-6	Scored from 0-6	Scored from 0-6	Scored from 0-20	Whether or not the taster perceived TCA to be present	If the taster identified another taint in the wine other than TCA

Turn to APPENDIX for results graphs.

The tasters

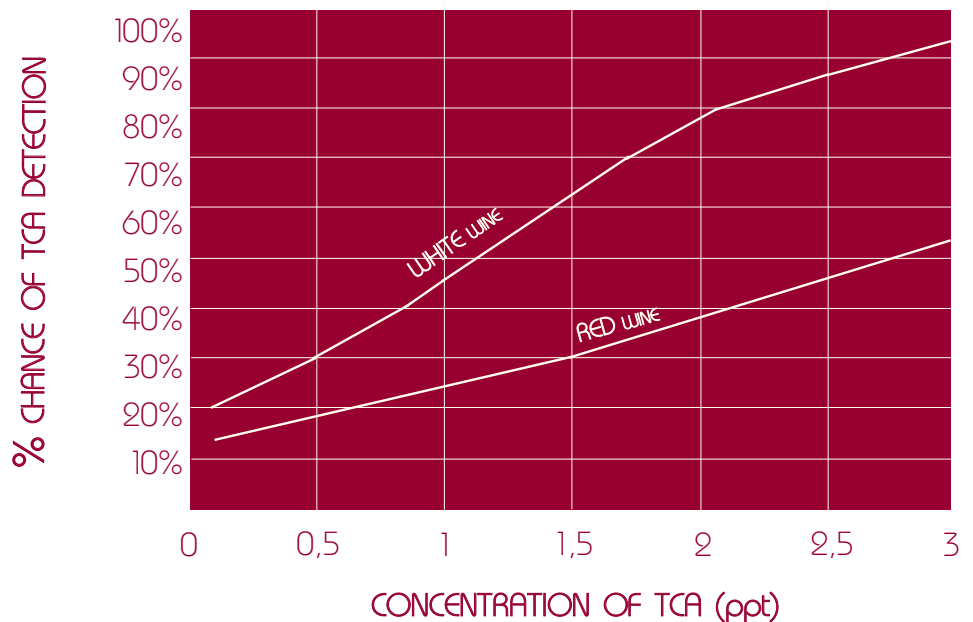
DID PANEL VARIATION AFFECT THE DATA ?

It was suggested that the variation in tasters across the tastings may have affected the results. Statistical analysis of the tasters across the tastings however shows that it is impossible to state there is a difference between the rate of detection of TCA from tasting to tasting due to panel composition. At low levels of TCA, other faults may have been mistakenly identified as TCA due to the dumbing of the fruit and freshness characteristics.

DOES A TCA THRESHOLD EXIST ?

The notion of a threshold above which it is more probable that tasters will detect TCA is not tenable. Russell Gerrard states that, the proportion of bottles identified as containing TCA increases steadily as the measured TCA content increases, rather than jumping from a lower value to a higher as the TCA content passes some cut-off point. This can be seen clearly on the graph below.

FIGURE 2: TCA DETECTION



NB. It should be taken into account when reading this graph that those tasters who correctly detected TCA when it was present tended also to report TCA when technical analysis showed that it was not present, ie. false positives. This graph does not take into account these false positives.

Conclusions

> The difference between groups of tasters does not have a significant effect on the results.

> There is an indication of a difference in performance between the three levels of permeability with regard to sensory descriptors.

The sensory results from the trial indicate that both Prototype 2 (P2) and Prototype 3 (P3) performed better than Prototype 1 (P1 : Original Altec formulation). P3 slightly outperformed P2, but internal trials at Sabaté, confirmed by the US panel, indicated slight oxidation. The permeability of P2 is now being used by Sabaté in the manufacture of Evolution and Reference closures.

> There is a significant correlation between actual and perceived TCA however, as anticipated, the perceived TCA was higher due to the tasters being experts and actively looking for it.

> The perception of TCA in the Prototypes and ROTE is low. There is, however, a very slightly higher perception of TCA in the ROTE than in the Prototypes which is confirmed by actual TCA levels at tastings 1 and 5.

> TCA detection rates differ according to wine type - TCA appears to be more obvious in white wine (but also more often wrongly identified).

> Both Pre- and Post-QC closures succeeded in achieving average TCA levels of below 2 ppt and are therefore well within existing QC threshold of 3 ppt.

> The Prototypes have all proved to show an insignificant level of TCA (<0.2ppt) meaning that the super-critical CO₂ TCA extraction process works and produces closures which are virtually TCA free.

> It seems to be untenable to assume an accurate average TCA threshold for the tasters.

QUESTIONS

THIS TRIAL HAS OPENED UP NEW QUESTIONS IN THE ONGOING SEARCH FOR THE MOST SUITABLE CLOSURES FOR WINE.

- > Do different permeabilities affect perceived TCA, and to what extent ?
- > Why was TCA both perceived and found in the ROTE ?
- > Can tasters accurately account for TCA when assessing wines containing only trace elements of TCA ?
- > Why is actual and perceived TCA higher in white wines than red when all other factors were the same ?

In his article "Grain of Truth" (attached in appendix), Jamie Goode questions how different the TCA perception results would be should a panel of non expert tasters have undertaken the tastings.

Glossary

ALTEC > the result of ten years of extensive laboratory research, Altec closures were introduced to the market in 1995. The closures are composed of fine cork particles (very rich in suberin and physically separated from lignin-rich particles, which have been swept aside, like dust), synthetic microspheres (closed cells made from thermoplastic polymers encapsulating a volatile fluid) and a unique polyurethane binding agent (the formulation of which is free from plasticisers such as phtalates). After expanding, the microspheres occupy the small spaces between the cork particles, thereby making up new closed cells that contribute their own elasticity to that of the cork micro-particles and strengthen the structure of the closure. Altec closures offer significant benefits in terms of elasticity, tightness and dust rate.

DIAMOND PROCESS > this supercritical CO₂ extraction process was developed in 1997 in partnership with the CEA's Supercritical Fluids and Filtration Membranes laboratory and is therefore aimed at selectively extracting any potential organic contaminants from cork by using CO₂ in its supercritical stage (an intermediary fluid state between liquid and gas, with the extraction power of a liquid and the penetration power of a gas). Supercritical CO₂ makes it possible to solubilize most low molecular weight organic compounds, which makes it particularly relevant for 'undesireable' compounds such as chlorophenols, and more importantly, chloroanisoles - in the

case of cork, 2,4,6 trichloroanisole.

EVOLUTION > internal research and development at Sabaté along with the results from this trial has led to the manufacture of two new closures: Evolution and Reference. The closures have the permeability of P2 which performed better than the original Altec formulation of P1. For all batches of Evolution the 2,4,6 releasable TCA control limit has been fixed at 3,0 ppt, considering technical tolerance.

PPT > the concentration of TCA in wine is measured in parts per trillion or nanograms per litre (ng /l).

RATE OF PERMEATION > the rate of flow of a gas through a porous material. The higher the permeability, the higher the rate of ingress of oxygen through or past the cork to the wine.

REFERENCE > (see Evolution). For Reference batches the 2,4,6 releasable TCA control limit has been fixed at 1,5 ppt, considering technical tolerance. Reference closures have the permeability of the P2.

TCA > Chloroanisoles (e.g. 2,4,6 TCA) are formed when chlorophenols are methylated by micro organisms. The transformation into chloroanisole could be a detoxification mechanism for elimination of the chlorine or certain chlorinated compounds, noxious for certain micro organisms.

The panel members
and
other participating
tasters

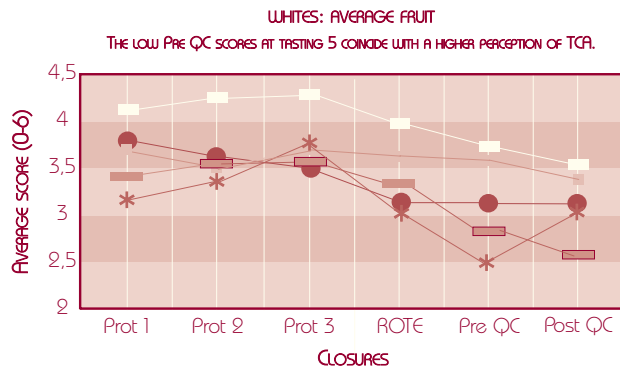
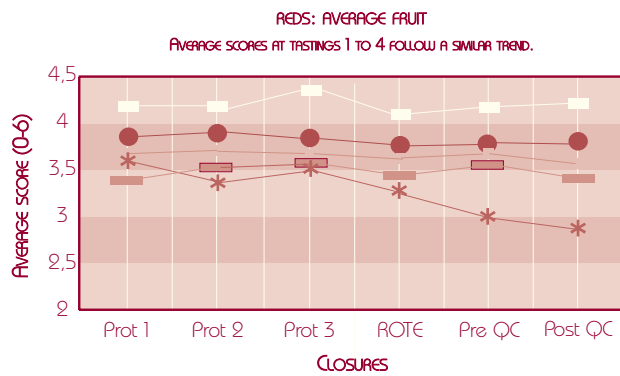
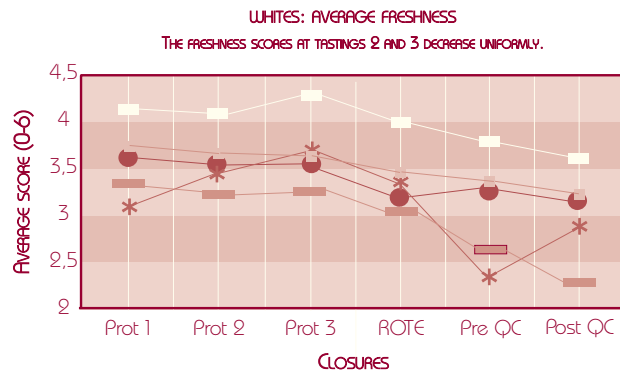
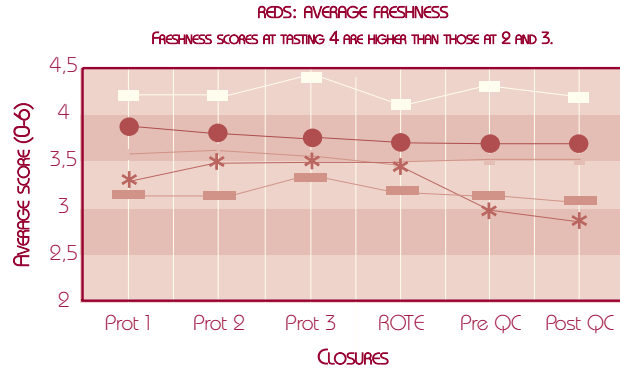
Alastair Maling & Alan Armstrong,
Waverley Group

**Campden & Chorleywood
Food Research Association**

EHD London Ltd

Russell Gerrard,
*Cass Business School,
City of London University*

Appendix



>>> KEY

□ TASTING 1

● TASTING 2

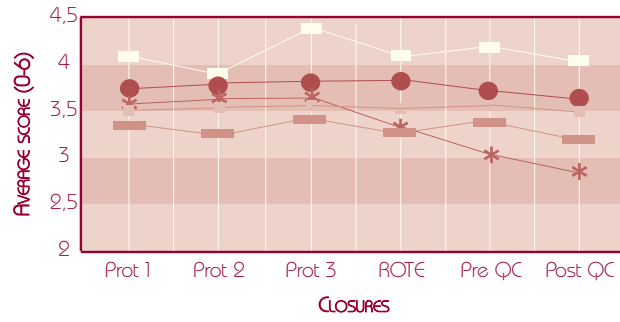
□ TASTING 3

* TASTING 4

■ TASTING 5

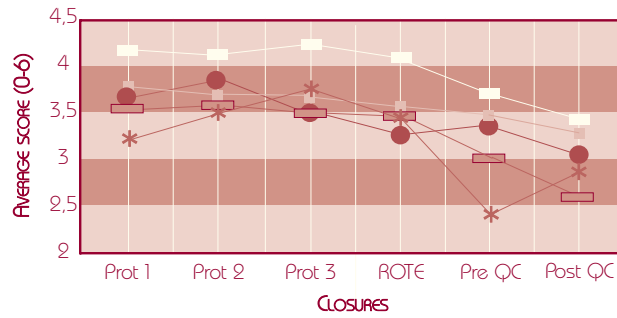
REDS: AVERAGE FINISH

THE REDS WERE GIVEN MORE CONSISTENT AVERAGE FINISH SCORES THAN THE WHITES.



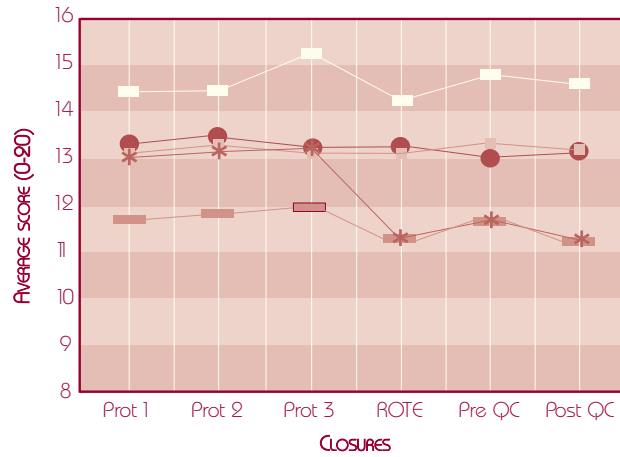
WHITES: AVERAGE FINISH

PROT 3 WAS GIVEN THE HIGHEST AVERAGE SCORES ACROSS THE TASTINGS.



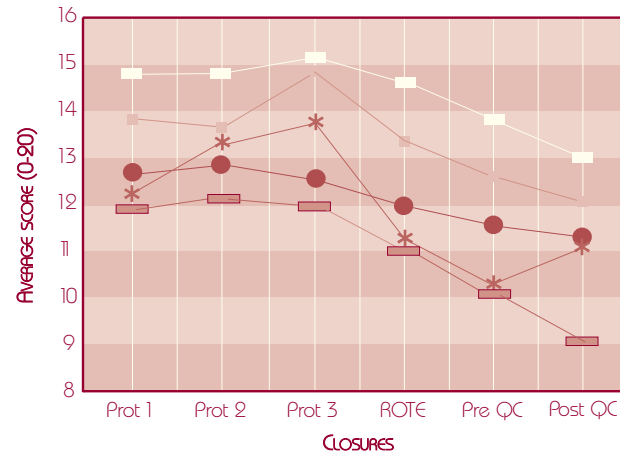
REDS: AVERAGE OVERALL

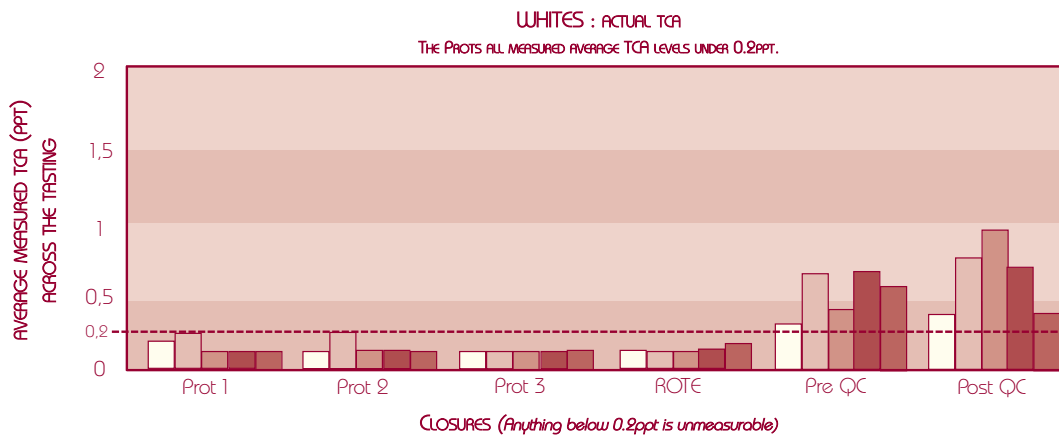
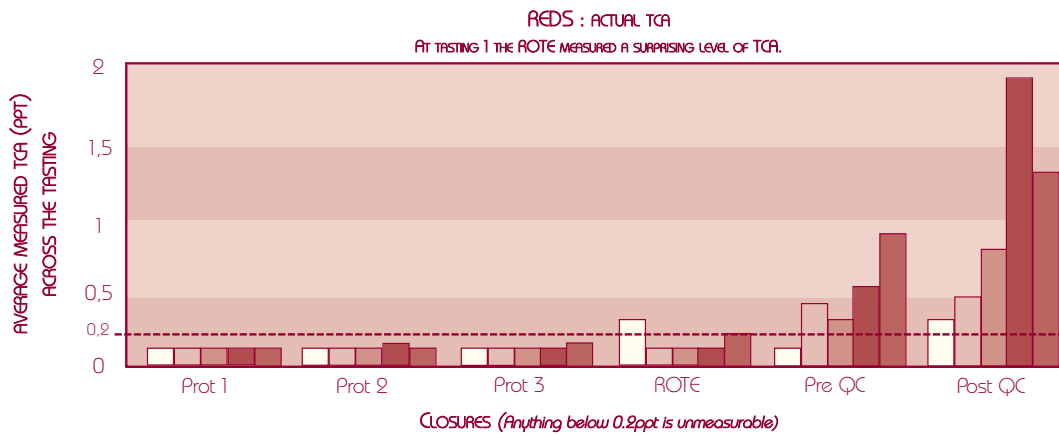
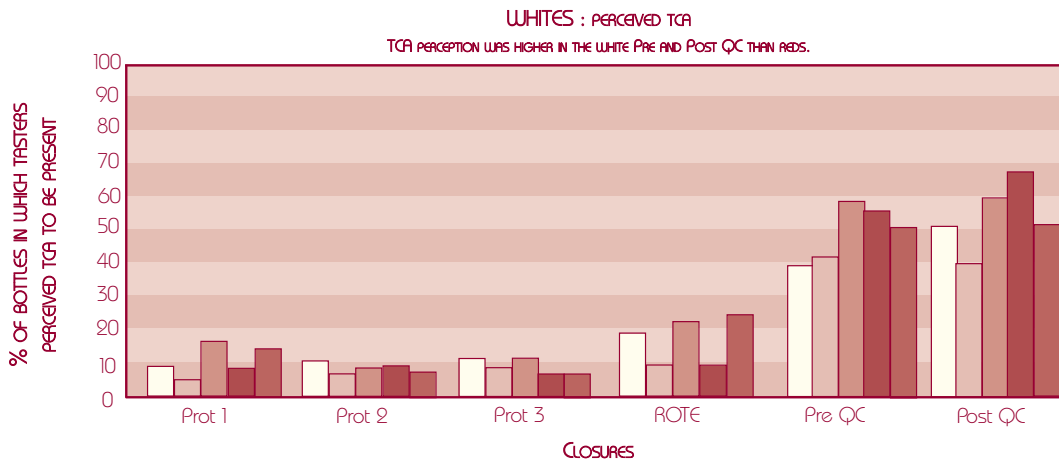
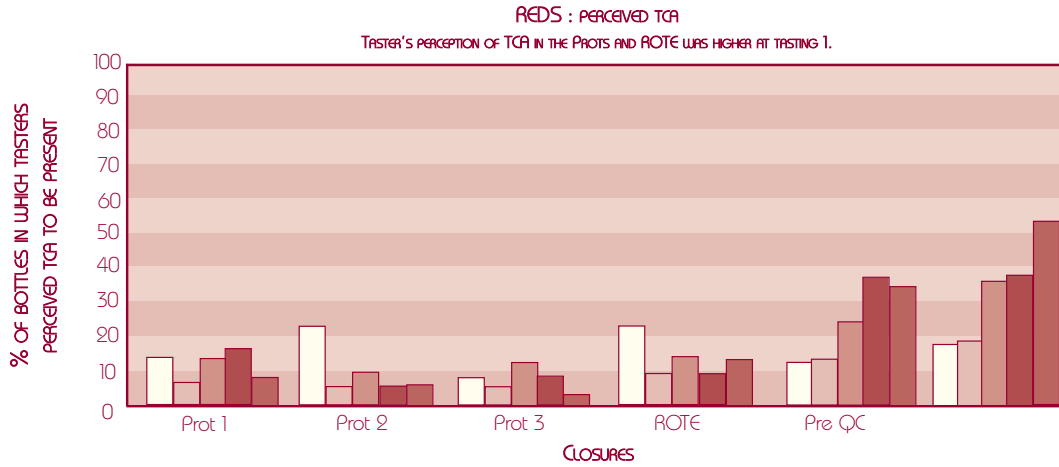
THE PROTS WERE SCORED UNIFORMLY AT TASTINGS 2,4,5.



WHITES: AVERAGE OVERALL

SCORES GIVEN TO THE PROT WHITES AT TASTINGS 2,4,5 ARE MORE DIVERSE.





>>> KEY

- TASTING 1
- TASTING 2
- TASTING 3
- TASTING 4
- TASTING 5

GRAIN OF TRUTH

by Jamie GOODE

(originated HARPER'S 15 November 2002 - Pages 32 to 36)

BY THE END OF THE 1990S, SABATÉ'S ALTEC CLOSURE WAS WIDELY CRITICISED FOR UNACCEPTABLE LEVELS OF TAINT. AFTER MODIFICATIONS, THE FRENCH MANUFACTURER INVITED EXPERTS FROM THE TRADE AND PRESS TO TEST THE PERFORMANCE OF ITS OLD AND NEW CLOSURES. JAMIE GOODE ANALYSES THE RESEARCH MODEL'S OPENNESS, METHODOLOGY AND ITS SURPRISING RESULTS.

NEVER BEGIN WITH AN APOLOGY. This is one piece of advice that budding writers and public speakers would do well to adhere to, but I'm going to disregard it this time. In fact, I'll start with two. First, I apologise for the fact that this is yet another piece of cork taint. It continues to be one of the most important and divisive issues facing the wine trade, albeit one of which many have prematurely grown weary. My second apology concerns the necessarily technical nature of some of the issues discussed here. However, this article represents the first public airing of some extremely important data from a rigorously conducted research study, shedding light not only on the performance of a particular closure type, but also on the nature of human perception of the cork taint culprit, 2,4,6 trichloroanisole (TCA).

The study in question was commissioned by French company Sabaté, the world's second largest closure manufacturer. As well as conventional corks, Sabaté's portfolio includes Altec, a manufactured and standardised cork-based closure. Launched in 1995, Altec represented a novel approach to alternative closures. The problem it addressed was the dissatisfaction of wine producers with the performance of the inexpensive corks that they were using for their mass-market wines. Because of perceived consumer resistance to plastic corks and screwcaps – the opposition is particularly strong in the French market – Sabaté devised what is, in effect, a hybrid closure: part cork, part synthetic, but which looks like a natural product.

This process involves taking raw cork and fragmenting it into tiny particles. These are then sorted and most of the lignin – the hard, woody material that surrounds the lenticels (the tiny pores in the cork) – is discarded. This cork 'flour' is then blended with proprietary polymer microspheres and the whole lot

stuck together with a binding agent. The resulting closures are consistent, and in theory should have lower risk of TCA taint than normal corks because the lignin-rich material that surrounds the lenticels is considered to harbour the majority of the TCA contamination.

Initially, Altec was tremendously successful. Sales were huge: to date, Altec has sealed more than two billion bottles of wine. But a couple of years ago, reports began to circulate that Altec, which had initially been marketed as taint free, was causing unacceptably high level of taint. The situation was particularly bad in the US, where four wineries blamed Altec for tainting large numbers of their wines. This led to legal action that is still ongoing, with a decision due from the courts in January 2003.

More bad news came from the results of the study conducted by the Australian Wine research Institute (AWRI). This scientifically rigorous study is currently monitoring the performance of a number of closure types over time, including Altec. When the 24-month results were published they reported that each of the bottles sealed with an Altec closure was found to have a TCA-like aroma, and follow-up chemical analysis found detectable levels of TCA in each of the Altec samples analysed. Nicolas Serpette, communications manager of Sabaté, confirms that the sales of Altec in 2002 have been affected by this adverse publicity. 'We've been exposed to very negative coverage' he says. 'We have lost a lot of US customers in particular, who are now scared of using Altec.'

SABATÉ'S RESPONSE - Some credit must go to Sabaté for its reaction to this criticism. The typical cork industry response to a problem like this with simply be to treble their marketing and PR budget, rather than tackling the problem itself.

Serpette subtly acknowledges this by insisting on Sabaté's determination to 'communicate transparently', and that this 'positions Sabaté differently of other elements in the cork sector'. He adds that, 'PR isn't the answer; people want facts'.

SABATÉ'S RESPONSE INVOLVED THREE STRATEGIES - First, the company instituted a new quality control standard. Every batch of Altecs leaving the factory was subjected to testing, and the company now ensures that no batch leaves with an average TCA concentration of greater than 3 parts per trillion (part per trillion, or ppt, = nanograms per litre, or ng/l).

Second, Sabaté has invested substantial sums of money developing a new technique, CO₂ extraction, for the removal of TCA from the cork 'flour' that is the basic constituent of Altec. This involves using CO₂ in its supercritical state, when it has properties somewhat in between those of a gas and a liquid. Basically, if you pressurise a gas at a certain point it becomes a liquid. If you then juggle the parameters of pressure and temperature, at a certain point – known as the critical point – the interface between the two disappears, and you then have the penetration power of a gas and the extraction power of a liquid. This is the principle used for decaffeinating coffee, and its claimed to remove any TCA that might be present in the cork microparticles.

Third, Sabaté commissioned an impartial, wine industry-led research programme to look into actual and perceived TCA levels across a number of closures – including pre- and post-quality control Altecs, and the new prototype Altecs made with the CO₂ extraction process – with both red and white wines. To co-ordinate this research effort, Sabaté hired Cube Communications, a relatively new UK-based PR company. Cube's James Gabbani, who was in charge of the project, was initially unsure about whether Cube should associate itself with an issue that carries such a high risk of damaging fallout. Gabbani decided that the only way Cube would get involved was if the 'trial was run in a way that we knew was beyond reproach, so we wouldn't get our fingers burnt'. He adds that, 'Sabaté has held to that all the way through.'

STUDY METHODOLOGY - From a journalistic viewpoint it is entirely appropriate to be somewhat cynical about a study of closure performance funded by the manufacturer of the closure in question. I wasn't expecting a trial funded by the cork industry and put together by a PR company to be terribly scientific. But close scrutiny of the methodology reveals that this is a properly designed, rigorously conducted study, the results of which have been subjected to thorough statistical analysis. At this stage I must state, for the record, that I have no involvement with either Sabaté or Cube, and my perspective here is that of an independent journalist. Cube convened a star-studded panel of 15 wine industry figures to participate in the trial and monitor its progress. As well as helping to decide on the study methodology, the panel chose two wines from an available selection, one white and one red, which were then bottled using a variety of closures. The procedure used for analysis was based largely on the methods used by the AWRI in its benchmark closure trial. Chemical analysis of 2,600 bottles for TCA was carried out by the Cambden and Chorleywood Food Research Organisation (CCFRA) using solid-phase micro-extraction and gas chromatography/mass spectro-

metry to a tolerance of 0.2ppt. Cube emphasises that no other study has been carried out on this scale assessing TCA down to such a minute concentration. A total of 528 of these bottles were also tasted by the panel, to compare the actual versus perceived TCA occurrence. These tastings were spread over four sessions at three-monthly intervals. When wines were tasted, CCFRA staff were on hand to take two samples from each bottle, which were then sealed in individual glass capsules with foil caps, transported to the lab and analysed with minimal delay. The raw data were passed on to an academic statistician, Russel Gerrard of City of London University, for analysis.

RATES OF TAINT : HOW THE CLOSURES PERFORMED - The results provide some welcome news for Sabaté. Most significantly, the new CO₂ extraction process seems effective at removing TCA from the cork 'flour' used in the Altec manufacturing process. For four of the closures, the three prototypes and the ROTE, there is no significant TCA in the wine. There is one slightly anomalous reading for the red wine sealed with the ROTE, where one of the bottles showed a TCA level of 1.4ppt, possibly through airborne contamination during the bottling process. Both the pre- and the post-quality control Altecs show some TCA contamination of both the red and white wines. Interestingly, the post-quality control Altecs have higher levels of TCA than the pre-quality control Altecs, which caused all the problems for Sabaté in the first place. Is this bad news for Sabaté ? It depends. If you look at the levels of TCA involved, they are extremely low – well below 2ppt. For the post-quality control Altecs, it is clear that they meet the new standard of less than 3ppt. With the pre-quality control corks, the likelihood is that the batch used in this study is a good one, and not one of those likely to have caused the taint problems with which Altec has become associated. I asked Nicolas Serpette about this, but he was unable to discuss the taint rates Sabaté found in pre-quality control batches of Altec because of ongoing legal action in the US. Evidently, though, with the current standard of 3ppt, some batches with higher TCA levels are not now leaving the factory, but previously would have.

TASTER THRESHOLDS FOR TCA ?

This raises an important question. At what level does TCA become a problem ? Is there a threshold concentration below which TCA is undetectable by a taster, and above which is it identifiable ? It would be extremely useful for the wine industry if we could identify a cut-off point above which TCA is problematic, and below which it can safely be ignored. So does such a threshold exist ? In short, no. One of the significant general findings from this study is that the data across all tasters show that the notion of a threshold for TCA is not tenable. Russel Gerrard, the statistician who analysed the findings, states that 'the proportion of bottles identified as containing TCA increases steadily as the measured TCA content increases, rather than jumping from a lower value to a higher [value] as the TCA content passes some cut-off point'. Gerrard's statistical analysis reveals some interesting findings. These show that half of tasters will detect TCA when it is present at 1.2ppt in white wines and 2.5ppt in red wines. Three quarters will detect it when it is present at 2.07 ppt in whites and 3.93ppt in reds. Bear in mind, though, that even for this expert group of tasters, for both red and white wines there was more than a 10% chance of them detecting TCA when there was none present, itself a rather startling statistic.

These figures correlate reasonably well with some other recent data on TCA detection and recognition from the Wine and Spirit Association's (WSA's) Musty Flavour Defects in Wine Survey. Although not a focus of the WSA's study, the published report provide some measurements of the participants' sensory thresholds for TCA in white wine, looking at the thresholds for both detection (when participants could spot something was wrong with the wine) and recognition (when they could identify the flaw as TCA). The average values for these were 1.5ppt (range 0.5–10ppt) and 6.5ppt (range 2.5–20ppt) respectively for 28 participants spread over two sessions. What does this say about Sabaté's decision to set its batch quality control level at 3ppt TCA ? For white wines, the data from the current study suggest that the vast majority of tasters would report a wine with 3ppt TCA as being tainted. For reds, it would be just over half the tasters. But bear in mind that the tasters in this study were all experts who were actively looking for TCA. The fact that they were calling clean wines tainted more than 10% of the time suggests they were being overzealous in diagnosing slight differences in the samples as TCA, even when they are not detecting any mustiness. At each session they were simultaneously assessing dozens of samples of the same two wines, red and white, so any differences would be immediately apparent. It would be interesting to see the same data gathered with a range of different wines, and with a non-expert group who were not actively looking for TCA.

THE DIFFERENCE BETWEEN WHITES AND REDS

Another interesting finding is that even expert tasters are significantly better at detecting TCA in white wines than in red wines. Perhaps more surprisingly, though, is that the technical analysis indicated that white wines in this sample actually had more TCA than the reds. The underlying reason for this is a matter for speculation: it may be that there is some sort of chemical interaction between TCA and specific components of red wine. Nonetheless, it is an intriguing finding.

CONCLUSIONS

The key conclusion from this study is that Sabaté's CO₂ extraction process works, and produces closures which are free from TCA. Serpette revealed that Sabaté has recently taken the decision to press on with the commercialisation of the CO₂-extracted prototype closures. 'The process has been validated at the lab and semi-industrial scale, but now we need to build a new factory'. Serpette estimates that the first products should be available 24 months after the first stone is laid, and he anticipates that the new Altecs will be on the market at the beginning of 2005.

In the meantime, what of the post-quality control Altecs ? It seems that without the CO₂ extraction step, it is inevitable that cork-based products such as Altec will have a residual level of TCA. It is not clear whether this level is problematic. Sabaté is convinced that the current taint levels are not significant and will cause no problems. However, the data here on taster detection limits show that wine industry professionals actively looking for TCA in Altec-sealed wines report its presence in approximately 35% of reds and up to 60% of whites, albeit with a high false positive rate. In the absence of further data, wine producers will have to decide for themselves whether they consider this to be a significant risk for their products. A question mark surrounds whether

this level of TCA would be noticeable by the consumer.

One key question that remains to be answered is how well Altecs perform in terms of permeability. Are they as effective as screwcaps in retaining freshness over time ? In this respect it's a shame that the current study didn't involve measurements of free SO₂ levels, a relatively simple test. The AWRI trial found that decline in free SO₂ is a useful surrogate measure for oxidation, and that small differences in SO₂ concentrations at early stages are strong predictors of differences in later sensory performance. The sensory analysis carried out in the tasting component in this study looked at subjective measures of freshness, fruit and finish, and indicated that the Altecs performed well in comparison with screwcaps. These data are backed up by the measurements of free SO₂ in the AWRI trial at the 30-month time point, where Altecs performed as well as screwcaps and better than both the synthetic and natural corks in the study.

In closing, these are clearly important data, answering some questions and raising others. Credit is due to both Sabaté, for the openness with which it has conducted this study and released the data, and to Cube, for overseeing a scientifically rigorous study and ensuring proper statistical treatment of the results.

Could this be a new dawn for the cork industry ?

TAINT NECESSARILY SOLVED

by Jamie GOODE

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SABATÉ CLAIMS TO HAVE COME UP WITH A TECHNIQUE FOR SOLVING CORK TAINT, AN AILMENT THAT ATTACKS 5% OF NATURALLY SEALED WINES. BUT WILL THE PROCESS, SUCCESSFUL IN TESTS, PROVE VIABLE IN MASS PRODUCTION ? JAMIE GOODE REPORTS.

I'll begin with the big news. Sabaté, the second largest cork manufacturer, may have found a cure for cork taint. Its new, supercritical carbon dioxide (CO₂) extraction process, called 'Diamant' ('Diamond', in English), was recently unveiled to a group of 18 journalists – 16 French, one Italian and me – at a press conference held at the high-security research laboratories at the Commissariat à l'Énergie Atomique (French Atomic Energy Commission, CEA) near Montelimar in Provence. Using the Diamond technique, developed in conjunction with the CEA's Supercritical Fluids and Membranes laboratory, Sabaté has successfully removed as much as 97% of 2,4,6-trichloroanisole (TCA), a compound that causes musty taint in wines at even fantastically low concentrations. But although this looks like a highly significant breakthrough, Sabaté will have to wait two years for a factory to be built in which the Diamond process can be operated on an industrial scale. In the meantime, Sabaté has got closures to sell and customers to keep loyal to existing products.

Amorim, the world's largest cork manufacturer, has also got a new industrial process, called 'ROSA'. Unlike Sabaté's Diamond process, ROSA doesn't take out virtually all the TCA but Amorim hopes that the 60-80% reduction it says this process achieves will be enough to cut TCA to levels where the incidence of taint is commercially acceptable. Because of the patent situation, precise details of this technique, which relies on steam treatment, are still confidential. Of course, we have all heard previous claims for TCA-eliminating techniques that have come to nothing, so a degree of caution – if not scepticism – is entirely appropriate. And with such significant commercial interests at stake, solid independent data are needed to back up any claims made by closure manufacturers. With this in mind, it is worth comparing the two processes and assessing whether a cure for cork taint really could be on the horizon. I'll also discuss what sorts of further studies are needed to verify the claims made by Sabaté and Amorim about the

effectiveness of their taint-beating processes.

IS IT TIME TO FORGET ABOUT CORKS ? Faced with the extent of the problem of TCA contamination – which reliable studies put at around 4-5% of all wines sealed with natural corks – shouldn't we just switch to synthetic closures and forget about natural corks ? This is certainly the attitude of certain sectors of the wine industry, most notably in New Zealand and Australia. But synthetic closures are not without their problems. The Australian Wine Research Institute's ongoing closure trial may have found that wines sealed with a range of synthetic closures are taint free, but almost all the wines also showed unacceptable levels of oxidation after just 30 months. This suggests that synthetic corks may be suitable only for wines destined for early consumption. (As an aside, it should be mentioned that different designs of synthetic cork, and not all leading makes were tested in this study; some brands that were not tested may have better long-term sealing properties.) The general challenge is that plastic corks don't have the same elasticity as natural cork, and thus a compromise has to be reached between tightness of seal and ease of extraction. The same study indicates that screwcaps provide a good, taint-free seal, and one that is considerably better than that of natural corks. This sounds like a good thing, but where wines sealed with screwcaps have been compared with the same wines sealed with corks, the consistent verdict has been that those under screwcaps taste fresher. Of course, with some New World wine styles, fresher and fruitier may be better, but the same isn't necessarily the case with Old World classics. Put another way, we like the way that wines age when they are sealed with a natural cork that isn't tainted. No buyer wants to wait 50 years for their newly-purchased First Growth to come round, especially if they are in middle age, but they certainly don't want them to age into something quite different. Get rid of the taint problem and suddenly the market for alternative closures has shrunk dramatically.

However, because cork is a natural product, it is variable, so fine wines sealed with natural corks will evolve at slightly different rates. Despite this variability, it is likely that taint-free natural corks, if they could be produced, would be the closure of choice for most winemakers.

SABATÉ'S DIAMOND PROCESS

So how does Sabaté's 'cure' for cork taint work? The Diamond process uses supercritical CO₂ for selective extraction of volatile compounds from cork. Work on this process was initiated in 1997 and patented in 1999, the patent being shared by Jean-Marie Aracil, Sabaté's head of R&D, and Guy Lumia and Christian Perre of the CEA (whose goals include 'guaranteeing national Independence by designing, manufacturing and maintaining dissuasion tools'). 'Sabaté has exclusive use of this patent in the wine closure industry', says Aracil.

Supercritical CO₂ is a slightly difficult concept to explain in simple terms, but I'll have a go. If you pressurise a gas, at a certain point it becomes a liquid. If you then juggle parameters of pressure and temperature, at a specific combination of these – known as the critical point – the interface between the two disappears, and you then have the penetration power of a gas and the extraction power of a liquid. For CO₂ this point is at 31.1°C and 73 bars of pressure – a conveniently low temperature, even if the pressure is on the high side (73 times atmospheric pressure). Other advantages of using CO₂ are that it is cheap and environmentally friendly. The technique is already used to remove caffeine from coffee and by the perfume industry to extract fragrances. 'The Diamond process shows an efficiency rate of around 97% for TCA extraction', says Aracil.

Initially, the Diamond process was used to treat the cork flour that is the basis of Sabaté's Altec closure – it is later blended with polymer microspheres and stuck together with a binding agent. Wines sealed with prototype Diamond-treated Altecs showed no detectable TCA, both by sensory and chemical analysis, in a rigorous independently conducted panel study carried out in the UK last year. So, it looks like Diamond works.

'For obvious reasons, Sabaté rejects any idea of offering a "zero TCA" guarantee', says Aracil. 'First of all, because TCA can come from sources other than cork, and also because 0% doesn't exist from a scientific point of view.' But he says that the maximum residual level of TCA is 'between the quantification and detection limits of the analytical method; that is, between 0.2 and 0.5ppt [parts per trillion].'

Since then, and significantly, the Diamond process has been extended to treating sheets of raw cork bark, which can then be used to produce what are effectively taint-free natural corks. 'We had to face some difficulties with deformation in the early stages of our trials', says Aracil, 'but we've since been able to optimise the parameters to get satisfactory results with cork planks.' Tests show that the cleaning process doesn't alter the mechanical properties of the cork significantly.

The production capacity of treated corks at the CEA facility is small, at around 100,000 per year – sufficient only to supply test corks to customers. Sabaté has decided to imple-

ment the Diamond process on an industrial scale, but first it has to build a new factory. This will be in Estremadura, in Spain, where it already has a facility, and the first commercial release of Diamond process closures will be in spring 2005 – almost two years away. The overall investment in the project is around 15 million (£10.4 million). Initially, two product lines are envisaged: a range of Altec-like technological corks and a range of high-grade natural corks targeted at ultra-premium producers. If it all works out, then Sabaté will extend the industrial site to increase capacity.

AMORIM'S ROSA PROCESS

'A great part of my mandate is to change the PR culture', says Carlos de Jesus, Amorim's newly-installed marketing and communications director. Many wine journalists have been infuriated by the communication style commonly adopted by cork companies, who have largely denied the existence of cork-taint and sought to deflect attention away from the topic by bringing in issues such as protection of cork forest ecology and consumer preference. 'The traditional response has been to run for cover' agrees de Jesus, 'but when you are confronted with an overwhelming amount of evidence, you have to get your act together. The only way to move forward is to align our interest with that of the wine industry and bring debate to a rational level.' In terms of addressing taint by means of research and development, 'we've done more in the last year and a half than in the previous 30 years', says de Jesus.

'We first became aware of the problem of cork taint in 1978', recalls Antonio de Barros, Amorim's executive vice-president, 'and we've been trying to fight it ever since.' He admits that in the past, the industry tended to ignore cork taint. The problem has become more apparent as wine has become more sophisticated and subtle; winemaking has changed a lot. Over the past ten years we have been doing whatever is necessary to try to control the problem.' De Barros says a key step was to renew Amorim R&D department, bringing in Professor Miguel Cabral, 'with a total focus on TCA control and removal'. Amorim has adopted three approaches to dealing with taint, explains Cabral. 'The first is a new boiling system that has been in place for a few years. It is completely different in several ways, the most significant difference being that the cork planks are extracted with boiling water, plank by plank. In the previous system, the planks were close together, so extraction was not so easy'. Linked to this boiling system, Amorim has devised a process known as Convex (for 'continuous volatile extraction'), by which all the volatile elements present in the water during the boiling process are cleansed at the same time.

Amorim's second approach has been to add chemical analysis for TCA to its quality control process, involving gas chromatography-mass spectrometry/solid phase microextraction (CG-MS/SPME). Amorim has five different machines. 'We can do 400 analyses of cork soaks in 24 hours', reveals Cabral, 'and the threshold is 5 ng/l [1ng/l = 1 ppt] TCA for a soak of 50 corks.' According to Cabral, half the volatiles from the soak would be expected to get into wine after 14 months, so this threshold would correspond to a wine with a TCA level of 2.5ng/l. How many batches of cork fail to meet this threshold? 'An enormous amount of batches are clean', says Cabral, 'but some have 20% to 30% of bales with above threshold levels.'

Thirdly, and most importantly, Amorim has also developed a curative strategy. 'We have tried a few different approaches', says Cabral, 'but the best is ROSA.' ROSA, which stands for 'rate of optimal steam application', is a special method of steam cleaning. Cabral claims that it reduces the TCA in cork granules by between 80 to 85%. 'We've asked two institutes to validate this independently', he reveals: 'the Cambden and Chorleywood Food Research Association (which found a 80% reduction) and Gelsenheim (which showed a 75% reduction).'

Amorim already has two ROSA machines in place, with three more expected to come into operation by the end of the year. 'By July, 80% of granules will be ROSA treated, and all our granules will be treated eventually', says Cabral. The ROSA technique can also be used for discs and corks. 'The reduction in TCA has been very good, but there has been high deformation: we had to change from a continuous to a batch system', says Cabral. Amorim is now doing ROSA tests on an industrial scale. 'We have not yet achieved the same results, but we are not far off, with a 60 to 70% reduction in TCA', he told me. 'There is no problem with the mechanical performance of the treated corks.'

This raises a critical question. What effect will a 60-70% TCA reduction in cork sheets have on the incidence of cork taint? Without proper data, it is almost impossible to tell. This could be enough to bring the level of TCA in the 5% or so of corks that are tainted down to below threshold levels, effectively eliminating taint; or, alternatively, 30-40% of the current TCA level in those corks might still be enough to taint the wine.

WHAT NEXT ?

For the trade and the consumer alike, what matters is the number of bottles that are ruined by cork taint. From this perspective, any measure that results in a reduction in taint is to be welcomed, whether it involves the replacement of natural corks with screwcaps or synthetic corks. The apparent unsuitability of screwcaps and synthetic closures for fine wines destined for ageing makes the prospect of taint-free natural cork an exciting one. If Sabaté manages to produce commercial quantities of effectively taint-free natural corks – as seems possible – then it would be hard to imagine a fine wine producer that could afford not to use these closures. Likewise, if Amorim's ROSA process produces corks that show sufficiently low rate of taint – say, less than 1% - this should be enough to satisfy many producers.

Good independent data on taint rates associated with closures that have been through these treatments are badly needed. Sabaté has set the standard with its independently run, rigorous scientific panel test of wines sealed with a range of Altecs, including prototypes, that have been through the Diamond process. The company needs to repeat some of this work for Diamond-treated natural corks, and Amorim should carry out a similar, transparent and independently verified testing of ROSA-treated closures to prove the efficacy of this process in a real world situation, with bottled wines.

These developments, particularly the Diamond process, are very exciting. But I'm not going to get carried away until I see treated, virtually taint-free corks rolling off production lines in numbers sufficient to bottle significant quantities of

wine. There's always the danger that the added cost – both to the manufacturer and the producer – of eliminating TCA from cork will prove to be a barrier to the large-scale application of these techniques, no matter how effective they are. In some markets, such as France and Italy, consumer acceptance of screwcaps and synthetic is poor, and this, coupled with the low profile of cork taint in these countries, might mean that wine producers are unwilling to pay a premium for closures displaying significantly lower levels of taint.

But I wouldn't want this rather depressing thought to take too much of the gloss off some very promising and highly significant developments. And I'm also looking forward to seeing some rigorous scientific trials of treated corks in the near future.



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