

# Quality counts in battle against TCA

**Dr Miguel Cabral**

Director, Research & Development  
Amorim & Irmãos

Australian and New Zealand winemakers and their customers are benefiting from sweeping changes in cork quality control introduced in the last four years by Amorim, the world's leading cork manufacturer.

Comprehensive quality control using sophisticated analytical methodologies is one of the pillars of the company's successful strategy to defeat the problem of TCA (2,4,6-trichloroanisole) in its cork closures.

Along with other process innovations such as ROSA (Amorim's proprietary TCA-extraction process), the new quality control system has reduced TCA levels in Amorim corks to below sensorily-detectable levels and minimised the risk of cork-related taint in bottled wine.

## An industrial-scale replacement for sensory analysis

The human nose and palate are highly sophisticated instruments for assessing wine quality and identifying the presence of off-flavours such as TCA, the main cause of cork taint in wine. But they cannot match the consistently accurate performance of

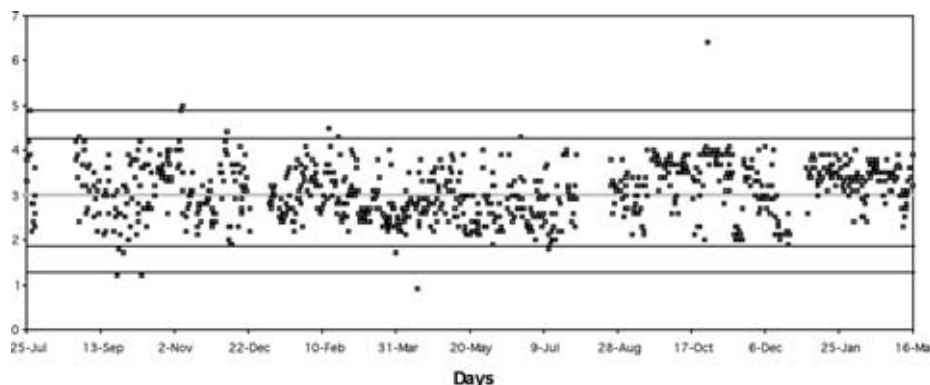


Fig. 1. Results for TCA control samples (GC-ECD machine 1), July 2003–March 2005. If a control sample gives results outside the acceptable range (1.2 to 4.8ng/L for a 3ng/L standard), the day's tests are repeated.

chemical analysis methods when faced with the task of judging large numbers of samples with very low TCA levels over long periods of time.

Given the right conditions, many skilled wine judges and professional assessors can detect TCA at levels as low as one part in a trillion (or one nanogram per litre). However, people vary widely in their sensitivity to TCA, and it is acknowledged that very low levels may be irrelevant to most wine drinkers. Wine styles are also important here, with TCA much easier to detect in delicate white wines than in more robust whites or full-bodied red wines.

A further complication is the sheer size of the task. Amorim alone produces 3.5 billion corks each year so it would be logistically and economically impossible to test each individual cork.

To provide winemakers with the assurances they demand, Amorim and other members of the Cork Quality Council commissioned ETS Laboratories in California to develop an efficient and accurate quality control system that could reliably say whether to accept or reject a batch of corks.

The system combines statistical sampling techniques with standardised chemical analysis to measure 'releasable' TCA levels in cork.

## Sampling protocol for batch testing

The statistically valid and scientifically-proven method involves a sampling protocol based on 'acceptance sampling' methods originally developed by the US military to test bullets during World War II.

Cork 'lots' are made up of bales, each bale typically containing around 10,000 corks of the same grade from the same wash. Corks from the same bale show similar characteristics. The cork protocol, which draws on the results of thousands of analytical tests conducted by ETS, calls for testing of 50 corks per bale.

The Cork Quality Council describes this level of testing as a way of accurately screening out bales that have unacceptable TCA levels so that cork manufacturers can treat or discard these corks. The sample size was decided on the basis of 200 comparisons of the results of 100-cork group soaks and 50-cork group soaks, which showed that the 50-cork samples accurately predicted the 'pass/fail' rate of the 100-cork samples (Peter Weber, CQC, pers. comm., September 2004).



## Revolutionary New Product Universal Cork with Opener

(Patent Pending)

No need for Cork Opener  
Unscrew the cork yourself



## How to Open



Turn to the top line anti-clockwise  
gently press the press wings, and  
turn clockwise till cork is removed  
from wine bottle.



Suitable for natural and  
synthetic corks



REMORK

Contact: Andrew Kromer

Email: [andrew.kromer@remork.com.au](mailto:andrew.kromer@remork.com.au)

Mobile: (61) (0) 403012154

ETS trials also found that the TCA results from group soaks accurately reflected the results that came from soaking the same corks individually. In cases where a few corks had very high TCA levels, the group results were higher than the average of the individual levels. According to the Cork Quality Council, this means that the group score is skewed in favour of detecting particularly bad corks.

### Measuring releasable TCA

The method developed by ETS measures 'releasable' TCA, which is the amount of TCA that the corks release into the soak solution.

ETS trials showed that releasable TCA is a good predictor of TCA in bottled wine, able to explain 90% of the variation in wine TCA. By contrast, total TCA, determined by testing ground-up corks, is not a good predictor of TCA in bottled wine, and the Australian Wine Research Institute has recommended that studies on the viability of TCA-reduction processes focus on releasable rather than total TCA (Capone, Skouroumounis and Sefton, 2002).

Releasable TCA is affected by the alcohol content of the soak solution; ETS results indicate that TCA extraction is optimised by using a 10% ethanol solution. The soak volume does not change the TCA level, ie. soaking the same corks in a smaller or larger volume (or even for a second time) yields the same TCA concentration.

### Standardised chemical analysis at Amorim

The standardised chemical analysis procedure developed by ETS was first introduced at Amorim in 2001 and has almost completely replaced sensory analysis with solid phase micro-extraction (SPME) and gas chromatography (GC) (Herve *et al.* 2000; Evans *et al.* 1997).

Each group sample—consisting of 50 cork stoppers or 250 cork discs or 30 grams of cork granules—is soaked for 24 hours in white wine with 10%. A sample is taken by SPME, which involves using a small polydimethylsiloxane fibre to absorb some of the volatiles above the liquid. The sample is analysed by GC methods that can reliably detect TCA at levels as low as 0.5 nanograms per litre, well below sensory thresholds for almost all wine consumers.

In Portugal, Amorim has six GC machines operating 24 hours a day and analysing more than 10,000 samples a month. Two machines use mass spectrometry (GC-MS) to detect the TCA; the other four use electron capture detection (GC-ECD) (Michel, 1997). All GC machines use phenylmethylsiloxane capillary columns.

The system has been carefully checked to ensure it is reliable, accurate, consistent and precise. Detection limits and quantification limits have been set for each machine. Quantification limits are typically between 1.5 and 2.0ng/L. If a sample yields a value of 2.0ng/L on a machine with a quantification limit of 2.0ng/L and a detection limit of 0.5ng/L, this means the sample has a TCA level of between 0.5 and 2.0ng/L.

The GC output shows a series of peaks corresponding to different chemical compounds, including TCA. Each compound has a characteristic peak. The area under a peak is a measure of how much of that particular compound is present in the sample. The peak area corresponding to TCA is compared with the peak area of an internal standard (deuterated TCA) with a known concentration.

### Validating the system

Regular checks ensure the ongoing validity of Amorim's day-to-day laboratory analysis procedures for quality control of TCA.



# portavin

integrated wine services



### Wine Centres in:

**Victoria**

**South Australia**

**Western Australia**

- ⊙ Featuring fully integrated static bottling and packaging facilities
- ⊙ Tank farm and cellar services
- ⊙ Equipped with wine laboratory for pre and post bottling wine analysis and quality assurance
- ⊙ Warehousing and distribution nationally and overseas for export
- ⊙ Advice on packaging and design
- ⊙ Suppliers of Amcor glass, screw caps and Oeneo closures

### mobile wine bottling and packaging

- ⊙ Offering experience and expertise in Mobile Wine Bottling in Australia since 1988
- ⊙ Covering all wine regions in Australia
- ⊙ Bottle rinsing, low vacuum filling, corking or screw-capping, external bottle washing/drying, capping with PVC or metal caps, self-adhesive labeling, bottle coding, case sealing, carton coding
- ⊙ Choice of smaller lines for full flexibility of bottle shape and size, batch size and property access
- ⊙ Semi-trailers for larger production runs

**enquiries in: Victoria: Melbourne – (03) 9584 7344; Mobile – (03) 5441 7200**

**South Australia Adelaide – (08) 8447 7555**

**Western Australia: Perth – (08) 9437 1033 – Margaret River – (08) 9755 0500**



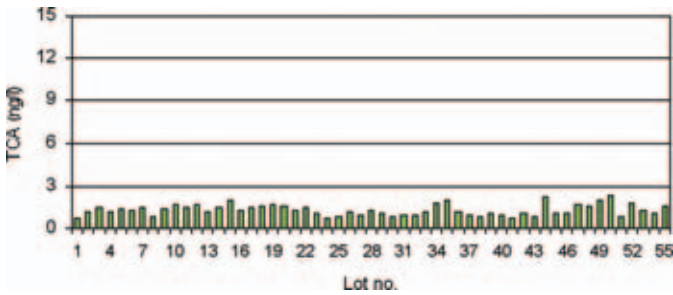


Fig. 2. Average TCA levels in natural cork stoppers before industrial processing, January/February 2005. Average levels of TCA in 55 cork lots from an Amorim industrial unit, before polishing, washing and surface coating.

In addition to the internal GC standards, laboratory staff analyse controls (standard samples made up in the laboratory with known TCA levels of 3 and 6ng/L) twice a day to check the validity of the day's results. If a standard sample gives results outside the range considered acceptable, the day's tests are repeated. For Amorim, the acceptable range is within three standard deviations, which for a standard sample known to contain 3.0ng/L would be between 1.2 and 4.8ng/L.

The validity of Amorim's quality control results is regularly checked by testing Amorim's proficiency in correctly quantifying the TCA levels of samples provided by CEVAQOE laboratories in France and CTCOR in Portugal. In these 'ring tests', each laboratory analyses the same set of samples to ensure that its TCA results remain comparable with those of the other facilities. Statistical analysis of ring test results to date indicates that the Amorim Group is performing the TCA analyses at a high standard.

#### A vital link in the production chain

Amorim routinely uses the GC method to check its natural cork stoppers and the discs and granules used in technical corks (Twin Top and Neutrocork) and Champagne corks. A variable number of samples per lot are analysed in accordance with ISO (International Standards Organisation) sampling standards (ISO2859-1:1999).

Contaminated lots of corks and discs are sent for ROSA treatment. All granules are screened before processing and any highly contaminated lots are sent for other uses such as cork flooring. All lots of granules selected for technical corks go through the ROSA process. Amorim also assesses finished corks before despatch if clients request this.

Figure 2 shows typical GC analysis results for 55 lots of natural cork stoppers, after punching and prior to industrial processing (rectification, washing and surface treatment). Each bar in Figure 2 corresponds to the average of the TCA levels obtained in each lot.

For the last four years ETS Laboratories in California have routinely tested Amorim shipments to the United States, and recent tests results show average TCA levels have fallen to below 1.5ng/L.

Similar results have been recorded in South Africa, where the Institute for Wine Biotechnology at Stellenbosch University now tests all incoming Amorim shipments.

Cork Quality Council director Peter Weber says average TCA levels in US corks have fallen 70% since the introduction of the ETS protocol.

The statistical sampling methods and GC techniques used by Amorim form a highly efficient, accurate and reliable system for day-to-day quality control of the cork manufacturing process. They are also used to validate the effectiveness of innovative new processes and procedures to improve the sensory performance of cork closures.

Such a systematic approach to quality assurance gives winemakers confidence that consumers can enjoy their product as they intend it to be.

#### References

Capone, D.L., Skouroumounis, G.K., Sefton, M.A. (2002) Permeation of 2,4,6-trichloroanisole through cork closures in wine bottles. *Aust. J. Grape and Wine Research*. 8:196-199.

Evans, T., Butzke, C. E., Ebeler, E. (1997). Analysis of 2,4,6-trichloroanisole in wines using solid-phase microextraction coupled to gas chromatography-mass spectrometry. *Journal of Chromatography*. A786:293-298.

Herve, E., Price, S., Burns, G., Weber, P. (2000) Chemical analysis of TCA as a quality control tool for natural corks (<http://www.corkqc.com/asev/asev2-2.htm#PAGE1>)

ISO 2859-1:1999. Sampling procedures for inspection by attributes. Part 1 Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection (<http://www.iso.org/iso/en/ISOOnline.frontpage>)

Michel, G. (1997) Dosage des chloroanisoles dans les vins par microextraction en phase solide (SPME) et chromatographie gazeuse avec detection en capture d'électrons (GC/ECD 63 Ni). *Revue des Oenologues*. 82:24-26. ■

## Ritual research result

A global consumer market research study, conducted by Wine Intelligence, an internationally recognised wine market research firm, shows that 80% of wine-drinking consumers prefer opening wine with a corkscrew. The research also confirms that wine-drinking consumers perceive the cork removal ritual and ceremony as an important aspect of the wine drinking experience.

The research was conducted with approximately 2300 wine-drinking consumers in the UK and USA via an internet survey.

The cork removal ritual (removal, romance, ceremony and "pop") is an important component of the wine drinking ritual.

—Information provided by *Supremecorq*



## Wine Branding...

... in various forms, contributes almost 70% to Mihart Design's bottom line. This is simple testimony to the success of our unique, result-driven solutions. Whether your capacity is 100 cases or 1,000,000, the energy we infuse into a brand – existing or new – is irrefutable even to our strongest competitors. Call Robert Mihart today for your free consultation and brand check-up.

Your brand?  
info@mihartdesign.com



08 8340 2424  
www.mihartdesign.com