

# Compounds causing cork taint and the factors affecting their transfer from natural cork closures to wine – a review

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## Abstract

The compounds causing cork taint and the factors affecting their transmission from cork to wine are discussed. These factors include: the solubilities of the taint compounds in wine, their affinity for the surface and the interior parts of the cork; their location on the surface of and within the closure; the rates at which they can migrate through the cork matrix; the volume of wine in contact with a closure(s); and whether taint transmission is taking place in bottled wine or with corks soaked in wine for screening purposes. 2,4,6-Trichloroanisole (TCA) has been the primary topic of investigations reported in the general literature and is therefore the main focus of this article.

## Abbreviations

TCA 2,4,6-trichloroanisole; MDMP 2-methoxy-3,5-dimethylpyrazine;  
GC/O gas chromatography/olfactometry; FM fungal must

*Keywords:* cork, wine, taint, musty, 2,4,6-trichloroanisole, TCA, 2-methoxy-3,5-dimethylpyrazine

## Introduction

Cork taint in wine is one of the most serious problems confronting the modern wine industry (Amon and Simpson 1986, Simpson 1990, Smith 1992, Baldwin 1993, Shaw 1995, Fuller 1995), causing economic loss and occasionally damaging the reputation of a winery unfortunate enough to incur a major problem. Estimates of the proportion of affected bottles range from 1–5% or even higher (Heiman et al. 1983, Baldwin 1993, Casey 1995, Leske et al. 1995, Pollnitz et al. 1996, Capone et al. 1999, Soleas et al. 2002, Hall et al. 2004), although these estimates are generally based on the collective opinion of assessors with varying experience in assessing taint, rather than conclusive evidence that the wine has been tainted by the cork closures. Cork taint is more difficult to assess in wines with wood flavours or showing oxidation, and in older wines which have developed bottle age aroma. The difficulty of confirming the occurrence of cork taint by aroma assessment is compounded when there is variability in the condition of different bottles of wine due to random oxidation or microbial development.

The occurrence of cork taint in bottled wine in individual wineries is highly variable. Our investigations over many years indicate that in commercial production, sometimes none, or only a very small proportion of bottled wine is affected by cork taint, but very occasionally the proportion of tainted bottles can be greater than 30%.

In attempting to provide reasons for the variability in cork taint occurrence, there are several questions that can be asked: (1) are taint compounds present in all cork,

perhaps in widely different amounts, (2) what are the identities and nature of these compounds, and (3) to what extent and how do they transfer into wine. Because they are the subject of other articles (Simpson 1990, Lee and Simpson 1993, Sponholz and Muno 1994, Pollnitz et al. 1996, Capone et al. 2002), the nature of cork taint and the formation of the compounds implicated in cork taint are only discussed briefly, whereas their location in the cork, their affinity for the cork and the means by which they transfer into wine are the major topics of this review. Knowledge of the factors controlling the transfer of taint compounds into wine in bottle will help explain how cork taint occurs, if the presence of certain compounds in the cork poses a risk of contaminating the wine, and how the corks can be treated to minimise or eliminate their taint capability.

Recent literature on the factors influencing cork closure assessments, which are conducted by many wineries as part of their quality control, has provided valuable insights on the transfer of cork taint compounds into the soak wine used to detect taint in these assessments. This has enabled us to evaluate the usefulness of these closure assessments to the industry, and demonstrates that simple protocols can sometimes identify defective bales of corks, and enable the wineries to reject these bales.

Only natural whole bark, agglomerate and technical cork closures (hereafter referred to simply as 'corks' or 'closures') are considered in this review. Because 2,4,6-trichloroanisole (TCA) is regarded as the most important contributor to cork taint, most research has been con-

ducted on this compound, which is therefore the main focus of this article.

### The nature of cork taint

Cork taint is caused by aroma-intense compounds, present in the cork, transferring into the wine after bottling. However, industry personnel and consumers place restrictions on what is considered to be genuine cork taint and exclude instances of chemical contamination with compounds such as naphthalene (Amon and Simpson 1986), which can occur by aerial migration to the corks during transportation or storage. Essentially, taint in bottled wine needs to have certain characteristics to be considered as cork taint; the defect must occur in different batches of corks, and be due to compounds present in corkwood or the cork cylinders with normal processing or, perhaps, form in the cork before or even after bottling. The description of types of cork taint by Duncan (1995) is helpful in demonstrating that there can be different cork taints. In a sequel to that article (Simpson et al. 2005), the incidence of five distinctly different taints was reported in the closure assessments of more than 145,000 corks over nine years. These were the most frequently-occurring taints but others were also recognised. The closure assessments were made by soaking several corks in wine over 24 h and presenting the soak wine to a group of panellists familiar with the different types of taint. There seems little doubt that different compounds were being extracted into the soak wine and that these were responsible for the taints examined. However, there are some fundamental differences between the conditions for the extraction of taint compounds into wines from corks in bottles, compared to during closure assessments, and these are discussed later in this review.

Changes in the processing of corks can affect the types of cork taint observed. With the phasing out of hypochlorite washing of the cork cylinders to bleach the surface of the cork (primarily for cosmetic purposes), one of the types of taint initially found by Duncan et al. (1995) was no longer present in the corks processed with a different bleaching treatment, although no explanation was given for this change.

Estimates of the proportion of cork taint attributable to TCA are about 80–85% (see below) but there are two factors that could lead to an over-estimation of the importance of TCA. This compound has been considered the major and, at times, even the sole cause of cork taint, and has been the only compound examined in many analyses of tainted wines. At lower concentration, it becomes difficult in sensory assessments to distinguish between the musty/mouldy/earthy compounds often implicated in cork taint and, too frequently, TCA may have been held solely responsible for the taint. In addition, there are several reports of taint compounds co-occurring in wines and their associated corks (Amon et al. 1989, Pollnitz et al. 1996, Simpson et al. 2004), so that more than one compound may be contributing significantly to taint in at least some instances. The co-occurrence of these taint compounds is considered later in this review.

When examining wines suspected of being affected

by cork taint, it is necessary to be aware that some of the musty/earthy compounds implicated in cork taint have been found in contaminated wine but the source of the contamination has not been the cork. Darriet et al. (2002) have identified 1-octen-3-one ('mushroom' odour) in extracts of grapes affected by powdery mildew. They also found geosmin ('earthy' aroma), thought also to be derived from mouldy grapes, in several wines (Darriet et al. 2000). Occasionally, wine can become tainted by TCA (and other haloanisoles) prior to bottling as a result of contact with contaminated processing aids and oak barrels or handling in wineries with a high background of these compounds (Amon et al. 1987, Chatonnet et al. 1994, 2004, Capone et al. 1999). We have observed a wine with a strong earthy aroma caused by 2-methoxy-3-*iso*-propylpyrazine derived from a mouldy barrel, a wine contaminated with TCA from a mouldy filter, and another with a very low level of TCA contamination from an unknown source (unpublished data). When wine is tainted prior to closure, the taint is usually detected by the winemaker and the tainted wine does not then reach the consumer.

Bottle-to-bottle variation in the level of taint has generally been taken as evidence that the cork is responsible for the problem. This was based on the assumption that, had the wine been contaminated prior to bottling, then a uniform level of taint in each bottle might be expected. This assumption was recently tested for TCA by Capone et al. (1999) who spiked 60 bottles of a white wine with deuterium-labelled TCA ( $d_5$ -TCA) (allowing the added TCA to be distinguished from cork-derived TCA) and then sealed the bottles with various types of cork closures. Thirty months later, approximately half of the  $d_5$ -TCA had been absorbed by each of the corks, regardless of supplier, bleaching treatment or whether the corks were natural or agglomerate. Fifteen of the corks, mostly agglomerates, imparted some of their endogenous TCA to the wines. The high variability in the distribution of endogenous TCA between wine and cork was in stark contrast to the relatively uniform distribution of the added  $d_5$ -TCA. These results indicated that a wine contaminated with TCA prior to bottling and sealed with taint-free corks will continue to exhibit similar bottle-to-bottle concentrations of TCA at any one time. Consequently, this investigation confirmed that bottle-to-bottle variation in taint is a clear indication of cork taint, but the identity of compounds responsible for the taint and evidence for the involvement of the cork are also needed.

The reasons for the presence of taint compounds in cork have been reviewed by several authors (Simpson 1990, Lee and Simpson 1993, Sponholz and Munro 1994, Pollnitz et al. 1996, Capone et al. 2002), but are still not fully understood. Essentially, TCA and other musty/earthy components of cork are either microbial natural products or are formed from the transformation of man-made biocides by various micro-organisms. However, the actual origin of the chlorophenolic precursors of TCA, the causative microbiological agents, and the time of formation of the metabolites as well as the factors affecting their retention in the corkwood, are uncertain.

### Cork-derived compounds causing wine taint

The compounds present in at least some corks, and capable of transferring to wine and tainting the wine, are examined in this section. Reports of their presence in wine and corks, their aroma thresholds and chemical stability in wine, and their co-occurrence in wine and corks are reviewed.

#### 2,4,6-Trichloroanisole (TCA)

TCA has long been associated with musty taint in a variety of foodstuffs (e.g. Land et al. 1975, Frijti and Bemelman 1977, Maarse et al. 1985, 1988, Whitfield et al. 1991, Aung et al. 1996). TCA was the first compound to be identified as a cause of cork taint (Buser et al. 1982) and is now regarded as the principal contributor to cork taint in the wine industry (Thevenet et al. undated, Buser et al. 1982, Amon and Simpson 1986, Amon et al. 1989, Duncan 1995, Jäger et al. 1996, Hervé et al. 2004, Simpson et al. 2005). For example, in a survey of commercial wines presented at a wine assessment course, 18 bottles out of 374 (i.e. 4.8%) were assessed by at least 20% of the participants as being affected by cork taint (a similar proportion was found to be tainted in two subsequent courses). TCA was detected by chemical analysis in each of these 18 wines at a concentration of 1 ng/L or higher (Pollnitz et al. 1996). TCA was also found in the corks from the wines. Further bottles of some of these wines were randomly selected and showed considerable bottle-to-bottle variation in TCA content. Hervé et al. (2004) reported that 70–80% of corks rejected during quality assurance screening contained TCA. Peña-Neira et al. (2000) analysed 46 samples of wine with a musty/mouldy off-flavour. TCA was present at a concentration above 4 ng/L in 80% of the samples. Rigaud et al. (1984) analysed 12 wines affected by cork taint and found TCA in all these wines. Also, there was a good correlation between the intensity of the musty taint and the concentration of TCA in the wines. Thevenet et al. (undated) similarly reported a good correlation between TCA and perceived cork taint.

Despite the good correlation between the level of taint and the TCA concentration in the examples cited above, there were some wines with a high level of taint but low concentration of TCA – indicating that other components were also contributing to the observed taint.

A contrary view on the importance of TCA as a major cause of cork taint has been put by Soleas et al. (2002). They investigated wines assessed by a 'panel of experts' from the Quality Assurance Laboratory of the Liquor Control Board of Ontario for cork taint. Of 2400 wines examined, 145 (6%) were considered to be affected by cork taint. Among the white wines, 18% of the Australian wines (number of wines examined,  $n = 99$ ), 15% of the French wines ( $n = 67$ ) and 25% of Spanish wines ( $n = 20$ ) were classified as cork tainted. Each of these 145 wines was analysed for TCA, which was found at a concentration above 2 ng/L in only 49% of the samples and was not found at all in 35% (limit of detection reported as 0.1 ng/L). They also conducted a bottle trial with five different batches of cork closure, each from two suppliers. They

reported that the percentage of bottles considered tainted by the natural and composite corks ranged from 7% to 64%, depending on the closure. For both red and white wines, no TCA was detected in more than half of the wines considered to be affected by cork taint. However, the assignment of tainted wines and the analysis of TCA were questionable in this study in which a wine was categorised as tainted if assigned as such by as few as one of an unknown number of assessors. The analysis was conducted without an internal standard, had no means of ensuring peak homogeneity and was based on a standard addition curve with concentrations three orders of magnitude greater than those determined in real wine samples.

Various group detection and recognition thresholds for TCA in wine have been reported and are listed in Table 1. In most studies, detection thresholds ranged from 1.4 to 4.6 ng/L, depending on the panel, methodology and wine matrix. Recognition thresholds were slightly above these values.

Prescott et al. (2005) determined 'consumer rejection thresholds' of 3.1 and 3.7 ng/L, depending on panel and experimental methodology. McLean (1995) studied the responses of two groups of wine marketing students to TCA in white wine. While only one of the two groups overall preferred the control wine to one spiked with 6 ng/L of TCA (there was no significant difference for the other group), most panellists in each group preferred the unspiked control compared to the same wine spiked with 20 ng/L of TCA.

Aroma detection thresholds can vary substantially among individual panellists. Suprenant and Butzke (1996) reported individual detection thresholds for TCA in a white wine ranging from 1 to 250 ng/L among 23 experienced panellists and from 2.5 to 25 000 ng/L among 15 inexperienced panellists. The group thresholds for the

**Table 1.** Published group detection and recognition threshold values for TCA in wine.

Detection thresholds (ng/L)	Recognition thresholds (ng/L)	Medium	Reference
1.4		Pinot Noir wine	Duerr (1985)
1.5		White wine	Thevenet et al. (undated)
2		Dry white wine	Hervé et al. (2004)
2.1		Chardonnay wine	Prescott et al. (2005)
2.2–4.6		Several white and red wines	Liacopoulos et al. (1999a)
4		Non-aromatic dry white wine	Amon et al. (1989)
17 <sup>a</sup> /210 <sup>b</sup>		White wine	Suprenant and Butzke (1996)
	4.2	White wine	Thevenet et al. (undated)
	6	Dry white wine	Hervé et al. (2004)
	10	Red and white wine	Buser et al. (1982)

<sup>a</sup> Experienced panel <sup>b</sup> Inexperienced panel

experienced and inexperienced panels were 17 and 210 ng/L, respectively. In contrast, the individual aroma detection thresholds, for TCA in a red wine, of ten experienced judges in our laboratory ranged from 1–2 ng/L (Francis et al. unpublished data). The judges used by Francis et al. all had considerable experience in assessing wines for cork taint. Thevenet et al. (undated) reported that some members of a trained and selected panel could detect TCA in white wine at a concentration as low as 0.3 ng/L, while the least sensitive panellists could only detect TCA at a concentration of 15 ng/L. They also reported that among panellists unfamiliar with TCA, some could not detect this compound at mg/L levels! The variability of individual panellists to detect specific odorants has been reviewed by Land (1989), and the responses to TCA reported above are consistent with this more general account of olfactory response.

TCA is a chemically stable substance that will not significantly degrade in wine over time (Capone et al. 1999). Consequently, for the concentration in wine to change with time physical processes must be responsible including desorption from and absorption by the cork. The migration of TCA from the cork and the processes that ultimately determine the concentration of TCA in wine both in bottled wine and in the quality procedures to assess suitability of corks for purchase are considered in detail later. These considerations are extended to other components implicated in cork taint.

#### *Other chloroanisoles*

Wines are occasionally submitted to our laboratory to confirm if they are cork-tainted. 2,4-Dichloroanisole (2,4-DCA), 2,6-dichloroanisole (2,6-DCA), 2,3,4,6-tetrachloroanisole (TeCA) and pentachloroanisole (PCA) are detected frequently, together with TCA, in these wines and their associated corks. In these analyses, which have been conducted over many years, TCA has always been the most important contributor to taint of this group of chloroanisoles.

Although all these chloroanisoles have undesirable aromas, TCA has the lowest aroma threshold in aqueous solution. TeCA has the next lowest threshold and the remaining compounds have thresholds that are comparatively high (Maarse et al. 1988).

Pollnitz et al. (1996) recorded the presence of chloroanisoles in 18 wines selected from a wine assessment course and considered to be affected by cork taint. They also analysed the associated corks. The results from this investigation were consistent with our cork taint investigations of wines submitted by the industry. All 18 wines contained sufficient TCA to cause a taint detectable to the more sensitive panellists (see previous section) and other chloroanisoles were detected instrumentally in the wine or cork in 14 wines. Also, consistent with the laboratory analyses reported above, the chloroanisoles co-occurring with TCA generally were either the dichloroanisoles or TeCA and PCA. The presence of 'low-chlorine' or 'high-chlorine' chloroanisoles provides an indication of the possible origins of these compounds (Simpson 1990, Pollnitz et al. 1996) but the pathways

proposed for their formation have not been validated in corkwood and cork.

Buser et al. (1982) reported the presence of a dichloroanisole, a dichloroveratrole and several chlorophenols in the extracts from hypochlorite-treated corks. 2,4-Dichloro-6-methylanisole (derivable from chlorinated *o*-cresol) and two other methyl ethers of chlorinated cresols were tentatively identified on the basis of gas chromatography/olfactometry (GC/O) analyses of extracts from tainted wines and their corks (Simpson 1990). These compounds were considered as possible contributors to taint but their formation almost certainly was from the simple phenols present in cork that were converted to chlorophenols by the chlorine-bleaching treatment and (in the case of the methyl ethers) to chloroanisoles by microbial methylation. Chlorine bleaching has been largely discontinued in Portugal, which is the main source of corks used in the Australian wine industry, so that these compounds are now unlikely to be found in cork. Consequently, they are not considered further in this article.

In summary, although chloroanisoles other than TCA can cause taint in wine, there remains no compelling evidence that these other chloroanisoles continue to contribute significantly to cork taint.

#### *2-Methoxy-3,5-dimethylpyrazine (MDMP)*

An account of the quality control procedures developed at one of the largest of the Australian wine companies for closures has been given by Duncan (1995), and included a listing of cork taints and their possible origin. A taint that the winery panellists described as 'fungal must' (FM) was considered by the panellists to be an important form of cork taint, second only to TCA. Duncan and colleagues were the first to recognise this taint as distinct from other forms of cork taint (Duncan 1995). The ability of these winery panellists to distinguish FM from other forms of cork taint was confirmed by GC/O assessments of extracts of corks from the company's taint assessment trials.

The cause of FM taint was recently identified by us (Simpson et al. 2004) as 2-methoxy-3,5-dimethylpyrazine (MDMP). The aroma detection threshold of MDMP in a non-aromatic dry white wine was determined as 2 ng/L, a value similar to that for TCA (Table 1). Some of the descriptors used by assessors in the threshold determination closely matched the FM taint characteristics described by Duncan (1995), i.e. 'fungal must', 'aldehydic', 'coffee', 'acrid', 'nutty' taint. Wines used to soak corks from a batch deemed to have an unacceptable level of FM contamination were analysed for MDMP, and those showing FM aroma all contained MDMP at a concentration well above its sensory detection threshold (Simpson et al. 2004).

MDMP was first identified by Mottram et al. (1984) who reported that it was responsible for an obnoxious odour present in certain machine cutting emulsions used in engineering workshops. They isolated an unidentified aerobic, Gram-negative bacterium from these emulsions which gave MDMP when grown in a culture broth. MDMP has also been identified in coffee (Czerny and Grosch 2000), where it was described as having an

'earthy' aroma and an aroma detection threshold in water of 0.4 ng/L. The origin of MDMP in corks is unknown, but is not necessarily bacterial as it has long been recognised that certain aroma-intense microbial metabolites occurring in foods and beverages can be produced by different types of microflora (Simpson et al. 2005).

Although Mottram et al. (1984) concluded that MDMP was likely to be a relatively common cause of off-odour in the environment, there has been no further report in the published literature of this compound as a cause of off-odour until that of Simpson et al. (2004), and only the one report of its occurrence elsewhere (Czerny and Grosch 2000). This may well be because of the difficulty in analysing for this compound and the exceedingly low concentration at which its aroma can be detected. Whatever the reason, the paucity of reports of MDMP in the environment contrasts with the substantial literature on TCA.

Since the identification of MDMP as the cause of FM taint, we have analysed two bottled wines suspected of being affected by this type of cork taint. In one bottle, both TCA and MDMP were present in the wine at similar concentration (8 ng/L TCA, 10 ng/L MDMP), while in the other, the aroma impact of TCA (2 ng/L) was likely to have been nullified by MDMP which was present at a concentration of 42 ng/L. In order to ascertain the importance of MDMP in general, it will be necessary to analyse a large number of cork-tainted wines for both compounds.

#### Other taint compounds

Six taint compounds, TCA, geosmin, 2-methylisoborneol, 1-octen-3-one, 1-octen-3-ol and guaiacol were detected by GC/O in cork-tainted bottled wines and in extracts of the corresponding corks in an earlier investigation by Amon et al. (1989). MDMP was not implicated in cork taint at the time and was therefore not targeted in this study. TCA was found to be the most important cause of taint in these wines. Although moderate GC/O responses were obtained for geosmin, 2-methylisoborneol, 1-octen-3-one and 1-octen-3-ol in several of the wines and the presence of geosmin and 2-methylisoborneol was later confirmed by qualitative GC/MS analysis, they could not be quantified because of the low concentrations present or the absence of suitable diagnostic ions in the mass spectrum. Nevertheless, three of these earthy/musty/mushroom-smelling compounds (geosmin, 2-methylisoborneol and 1-octen-3-one) have extremely low odour detection thresholds (25, 30 and 20 ng/L respectively in dry white wine, Amon et al. 1989). All could contribute to cork taint in at least some wines, although 1-octen-3-one has also been detected in wines considered sound (Amon et al. 1989). A determination of their importance compared to TCA requires quantitative analysis of a large number of tainted wines. Of these compounds, geosmin is least likely to cause serious wine taint as this compound is chemically unstable in wine, having a half-life of eight weeks at pH 3.2 and 25°C (Amon et al. 1989). Geosmin, 2-methylisoborneol, 1-octen-3-one and 1-octen-3-ol are well documented as mould metabolites (Simpson 1990).

McLean (1995) studied the responses of two groups of

wine marketing students to 1-octen-3-one in white wine. When comparing the wine spiked with 60 ng/L to the control wine, there was no clear preference for either sample by either group. One of the two groups preferred the control wine to one spiked with 200 ng/L of this compound, while for the second group there was no preference for either wine.

Amon et al. (1989) were able to quantify guaiacol in the wines they examined, and although detectable by GC/O, the concentration in the wine was generally at or below the odour detection threshold in a white wine of 20 µg/L (Simpson et al. 1986). Furthermore, guaiacol in wine can also be derived from the grape or during oak barrel maturation (Sefton 1998, Chatonnet et al. 1989) and could not be considered as a cause of cork taint in the wines studied by Amon et al. (1989). However, Lefebvre et al. (1983) identified guaiacol as a major metabolite of a *Streptomyces* sp. present in defective corkwood. The defective corkwood acquired a yellow discoloration, enabling it to be recognised and removed during processing. Nevertheless, this compound could still be responsible for isolated incidences of cork taint, although such incidences are likely to be rare (Duncan 1995, Lee and Simpson 1993).

In an earlier investigation of an incident of tainted wine, Simpson et al. (1986) found high and variable amounts of guaiacol in individual bottles of the same wine, and also in the corks taken from the bottles. The wine had clearly extracted the guaiacol from the cork, since cork closures do not absorb guaiacol from wine (Capone et al. 2003). In this instance, it was suggested that the corks had become contaminated chemically during storage or shipping rather than through microbial degradation of cork lignin.

Heimann et al. (1983) reported the formation of several unidentified sesquiterpene hydrocarbons by cultures of the mould *Penicillium roquefortii* which was isolated from corkwood. These sesquiterpenes eluted in a musty smelling region of the gas chromatogram of extracts of these cultures. They have not, however, been demonstrated to be the cause of the musty aromas. Caldentey et al. (1998) also reported a range of sesquiterpenes, together with 1-octen-3-ol, in cultures of micro-organisms isolated from cork. Some of these cultures produced unpleasant mouldy, earthy aromas. Others have also reported such odours in cultures of cork-derived micro-organisms (e.g. Jäger et al. 1996), but have not identified the compounds responsible for these aromas.

Simpson (1990) reported the tentative identification of *cis*-1,5-octadien-3-ol and *cis*-1,5-octadien-3-one as musty odorants in tainted wines.

Allen et al. (1995) found elevated amounts of 2-methoxy-3-*iso*-propylpyrazine in one of several bottles of the same red wine, and speculated that this might have resulted from a contaminated cork.

While most of the taints derived from cork or from cultures of micro-organisms isolated from cork are varyingly described as musty, mouldy, and earthy, some wineries will also reject corks on the basis of excessive 'woody' characters (Linton 1995). The compounds causing these woody characters have not been determined but pentane

extracts of corkwood contain significant amounts of various monoterpene alcohols and ketones that are likely to possess woody aromas (Simpson unpublished data).

The present status of compounds implicated in cork taint is summarised in Table 2. We believe that with this more complete account of the compounds implicated in cork taint and the mechanisms for their transfer from the cork to wine, future investigations of taint in bottled wine will be more thorough and a clearer understanding of the role of the compounds listed in the table will emerge.

#### *Co-occurrence of taint compounds in cork closures*

The co-occurrence of the chloroanisoles has been discussed briefly in the preceding sections and the co-occurrence of TCA with MDMP has been described by Simpson et al. (2005). Sensory data from the screening of more than 150,000 cork closures by a leading Australian wine company indicated that the frequency of the sensory detection of TCA alone by soaking trials was 1.06%, while for FM (i.e. MDMP) alone the incidence was 0.82%. The incidence of taint where both TCA and FM were recognisable in the same cork soak was 0.19%, corresponding to about 9% of the total TCA and FM taint (Simpson et al. 2005). Almost certainly, there would have been corks containing both TCA and MDMP, but with one not recognised because of its lower concentration. Consequently, the proportion of corks containing instrumentally detectable quantities of both of these compounds together was likely to have been higher. As indicated earlier, TCA and

MDMP have been shown to co-occur in some bottled wines.

The six taint compounds – TCA, geosmin, 2-methylisoborneol, 1-octen-3-one, 1-octen-3-ol and guaiacol – examined in the study by Amon et al. (1989) showed considerable co-occurrence in extracts of cork-tainted bottled wines and of the corks taken from those wines. Three or more of the taint compounds were detected together by GC/O assessment of the extracts of wines (57%) and associated corks (73%), although the contribution of the individual compounds to taint was uncertain. Thevenet et al. (undated) reported finding 2-methylisoborneol and 1-octen-3-one in some musty smelling wines, and that these compounds always co-occurred with TCA. Active growth of mixed microflora occurring at particular sites on the corkwood, concentration of metabolites by movement of water or moisture, or storage of cork slabs on the ground, and aerial contamination of the more exposed slabs might explain the tendency of taint compounds to occur together in the corks.

#### **Factors affecting the transmission of taint from cork closures to wine**

The transmission of taint compounds from cork to wine will depend on several factors, including:

- The solubilities of the taint compounds in wine and their affinity for the surface and the interior of the cork.
- The location of the taint compounds on the surface of, or within the closure.
- The rates at which the taint compounds can migrate through the cork matrix.
- The volume of wine in contact with a closure(s).
- Whether taint transmission is taking place in bottled wine or with corks soaked in wine for screening purposes.

There may be differences between the absorptive capacity of the surface and the interior part of the cork due to the contact of wine with the surface and the effect of moisture and alcohol. Also, the surface treatments with silicone and paraffin can be expected to enhance the hydrophobicity of the cork surface and provide greater retention of non-polar taint compounds. These aspects, however, have not been studied previously and are therefore not addressed further in the present article.

Of all the compounds associated with cork-tainted wines, only TCA has been studied in detail. Consequently, the influence of the factors listed above on the transmission of most taint compounds can only be inferred from an understanding of the behaviour of chemically similar compounds.

#### *2,4,6-Trichloroanisole (TCA)*

1. Affinity of natural bark cork for TCA  
Natural bark (and synthetic) closures have a capacity to absorb certain compounds from wine during bottle storage (Capone et al. 1999, 2003). This is particularly evident for non-polar volatile compounds, including chloroanisoles. In bottled wine, which originally con-

**Table 2.** Present status of compounds implicated in cork taint.<sup>1</sup>

Compound	Status
TCA	Established as a major cause of taint.
2,4-DCA/ 2,6-DCA/ TeCA/ PCA	Unlikely to contribute significantly to most instances of cork taint.
MDMP	Primary cause of taint. Importance to cork taint in bottled wine has yet to be determined but prominent in quality control assessments.
Geosmin	Possible primary cause but degrades quickly in wine.
1-Methylisoborneol	Contributor to taint.
1-Octen-3-one/ 1-octen-3-ol	Contributor to taint.
Guaiacol	Probable primary but infrequent cause of taint.
Unidentified sesquiterpenes	Uncertain because these compounds have not been shown to be the cause of musty, mouldy taint in extracts analysed.
<i>cis</i> -1,5-Octadien-3-one/ <i>cis</i> -1,5-octadien-3-ol	Uncertain because these compounds may not be present at sufficient concentration to cause taint.
2-Methoxy-3- <i>iso</i> -propylpyrazine	Possible cause of taint.

<sup>1</sup>Refer to text for references to these compounds.

tained TCA in the wine, half of this TCA was absorbed by both natural and agglomerate closures over 30 months, regardless of the cork supplier or the bleaching treatment (Capone et al. 1999). Tetrachloroanisole was even more efficiently extracted from bottled wine, with nearly 90% being absorbed over 27 months (Capone et al. 1999). TCA was rapidly and strongly extracted from wine by four cork closures soaked in 200 mL of wine. Eighty per cent was absorbed after 24 h, and nearly 90% after 48 h. A single ground cork soaked in a litre of wine for six days extracted a similar high proportion (88%). Hervé et al. (2004) carried out an extensive study of the transmission of TCA between wine and corks in short-term soakings of whole corks for taint screening. Their results also indicate that only a very small portion of TCA is extracted from cork, and that after 24 hours, most of the rapidly releasable TCA is bound to the cork rather than dissolved in the wine (see later for a detailed discussion).

## 2. Location of TCA in cork closures

Few studies have been undertaken to determine where TCA is located in contaminated closures, and because of the limited nature of these studies, it is impossible to determine whether the observations are typical of a significant proportion of wine corks. Hoffmann and Sponholz (1994) used thermal desorption to analyse the TCA content of fragments of a single wine cork taken from a tainted bottle of wine. They found that the TCA was concentrated in the older bark, and was at lowest concentration in the third of the cork that had been in contact with the wine, and highest in the outer third of the cork. They interpreted this result as indicating that the TCA had been extracted from the lower and middle parts of the closure. This contrasts with the result obtained by Taylor et al. (2000) for the distribution of TCA in wine corks taken from four bottles of wine considered to be affected by cork taint. In two of the corks, there was a concentration gradient of high to low TCA from the inner part of the cork (i.e. in contact with the wine) to the outer part. In the other two closures, the TCA was evenly distributed. However, because of the small sample number and the non-random nature of the sampling (samples taken from bottles in which the wines contain TCA could have a high probability of TCA at the wine contact end), no conclusions can be drawn from these data.

Howland et al. (1997) dissected more than 50 corks taken from the unused portion of a single batch of corks that had caused an unacceptably high incidence of taint in a commercial wine less than two months after bottling. Some corks exhibited faint dark patches, typical of mould, on the outside of the cork. There was no evidence of fungal mycelium penetrating into the body of the cork from these dark areas. If such colonies had been responsible for the methylation of 2,4,6-trichlorophenol (TCP) to form TCA, the latter would have been expected to have been concentrated on the outside of the cork. In agreement with Hoffmann and Sponholz (1994), more TCA was found in the older bark. In each of these halves (cut longitudinal through the cork) corresponding to the older and younger bark, there was also more TCA in the outer 2 mm

of the corks than in the rest of the cork. These differences were statistically significant, despite considerable variation among closures. There was no difference in the amount of TCA between the ends and centre part of the cork.

Amon and Simpson (1986) had earlier suggested TCA could be concentrated in the lenticels of some corks. During dissection of the corks by Howland et al. (1997), fungal colonies were also observed in the lenticels of the cork, and these had spread across the internal growth rings from the lenticels. In some corks, one or two of the dark layers separating the growth rings appeared to be particularly conducive to mould growth. Mould appeared to grow along many lenticels, but across only *specific* growth rings. However, analysis of the lenticel and non-lenticel portions of each of 12 corks showed no significant difference in TCA concentration between these two fractions. There was also no significant difference in TCA concentration between the light and dark bands of each of twelve additional corks. The lack of differentiation in TCA concentration between the lenticel and non-lenticel portions did not, however, exclude the possibility that the TCA in the corks had originally formed in the lenticels of these corks, as preferential extraction from this area during subsequent cork processing could have nullified any concentration effect.

The observation of more TCA in the older (i.e. external) bark by Howland et al. (1997) and by Hoffmann and Sponholz (1994) is consistent with the proposition that polychlorinated biocides were applied to the cork trees in the forest, prior to harvesting the bark. In the Howland study, the closures had been chlorine-bleached, and so it is also possible that chlorophenols were formed by this process. In such circumstances, the finding of a greater amount of TCA in the older bark may simply be because the older bark contained more lignin breakdown products, in particular, phenol – the precursor to TCP – than did the younger bark. It was suggested by Howland et al. (1997) that the substantially higher concentration of TCA in the older bark indicated that aerial migration of TCA to the cork cylinders was unlikely to have been the main cause of contamination, as this mechanism might be expected to contaminate both halves of the cork more evenly. Barker et al. (2001) subsequently demonstrated that contamination by this mechanism alone can result in a (slight) concentration of TCA in the older bark in cork closures, perhaps because of the greater density of lenticels. This disproportionation of (airborne) TCA between older and younger bark observed by Barker et al. (2001) was not, however, as great as that observed by Howland et al. (1997).

A more detailed consideration of the factors that might have led to TCA being concentrated in some parts of a cork more than others is beyond the scope of this review. Nevertheless, it is clear from these limited studies that, although most corks contain at least traces of TCA, the location of TCA in the cork can vary from cork to cork and perhaps also from batch to batch. Such variation, combined with the observation that TCA is transmitted through cork relatively slowly (discussed below), explains

why some corks containing TCA will contaminate wine while others, which might contain even more TCA, do not.

### 3. Transmission of TCA through cork

Neto (reported by Casey (1994)) has described the presence of a concentration gradient of TCA in a cork removed from a bottle of wine. The TCA content was highest at the outer face and lowest at the face that had been in contact with the wine. The bottle had been stored in a contaminated environment, and it was concluded that the wine had been tainted by the external source after the bottle was sealed with the cork and stored. For this to happen, it would be necessary for the exogenous TCA to have contaminated the external face of the cork and then permeated down the long axis of the closure, at right angles to the lenticels. Casey (1994) has also suggested that volatile compounds can migrate rapidly throughout the whole cell structure of cork, and showed that when whole corks are soaked in sulfur dioxide solution, the sulfur dioxide penetrates rapidly to the centre of the cork. Similarly, Barker et al. (2001) showed that when dry corks were exposed to an atmosphere containing TCA, most of the TCA absorbed was found on the outer 2 mm of the cork, but a significant proportion had migrated further into the closure after as little as 24 hours exposure. In these last two cases, it is possible that these compounds had been transmitted via the lenticels, which are at right angles to the long axis of the cork. Vasserot et al. (2001) reported transmission, through the lower part of sparkling wine corks, of TCA that was added via a borehole to within approximately 3 mm of the inner disc. However, this interpretation was based on the distribution of TCA in one replicate only of treated and control corks, and their data do not allow the added TCA to be distinguished from any that might have already been present in the cork. Furthermore, no information is given by the authors on what solvent (or its volume) was used to add the TCA, making it impossible to assess whether solvent played a role in aiding the transmission of the added TCA through the discs.

Although the concentration gradient of TCA from the outside of a cork taken from a bottle of wine into the wine itself, described in Casey (1994), appears to indicate that TCA can travel through cork at right angles to the lenticels, such an interpretation is probably incorrect. Capone et al. (2002) added a substantial quantity of TCA (labelled with deuterium to distinguish it from endogenous TCA that might already be present in cork) to the outer end of corks in 48 bottles of wine. The corks (both agglomerate and whole-bark, and bleached in various ways) had already been in bottle for various times. After a further three years of storage, no trace of the added (i.e. labelled) TCA had reached the wine, and very little had permeated beyond the outer third of the closure. Several of the wines were contaminated with TCA, but this was derived from (unlabelled) TCA already present in the corks. In each of these cases, there was a concentration gradient of *total* TCA from the outside of the corks to the wine, but this resulted from a combination of endogenous TCA already present in the cork plus the additional

(labelled) TCA absorbed by the outer part of the cork in the bottle. These results are also supported by Thevenet et al. (undated) who reported that they found no evidence of TCA permeation through cork closures over a nine month period.

Therefore, although TCA can contaminate the outside of cork closures in bottles, if sealed bottles of wine are stored in a contaminated environment, or if mould growth takes place on this surface and generates TCA during bottle storage in a damp environment (Sponholz and Munro 1994), it appears unlikely that TCA will travel through the cork and significantly contaminate the wine inside the bottle during the normal lifetime of bottled wine. However, this does not mean that TCA cannot pass through the discs of sparkling wine corks or technical closures, as these discs are punched parallel to the lenticels.

The inability of TCA to permeate quickly down the long axis of corks is also indicated by several reports on the relationship between the amount of TCA in bottled wine and in corks taken from those wines. Thus, Chatonnet et al. (2003) analysed wine from eight bottles showing various levels of taint, and also analysed extracts of ground corks taken from those bottles. TCA in the wine matched the relative intensity of the perceived taint, but was poorly correlated with the TCA in the corks. In all cases, there was more TCA in the corks than in the wine. Peña-Neira et al. (2000) also found a weak (though statistically significant) correlation between TCA in wine and in extracts of whole corks. Again, there was less TCA in the wine compared to the corks. Amon et al. (1989) and Soleas et al. (2002) also reported a poor correlation only between TCA in commercially bottled wine and ethanol extracts of whole corks taken from those bottles. Hervé et al. (2004) showed that the correlation between TCA readily extractable during wine soaks and total cork TCA was similarly poor.

In our experience, nearly all natural bark closures contain some TCA (Pollnitz et al. 1996, Howland et al. 1997, Capone et al. 1999) but, in most cases, this TCA does not contaminate bottled wine. It appears that only when contaminated parts of the cork are in direct contact with, or in close proximity to, the wine (or headspace above the wine) does transfer of TCA take place. Consequently, the location of TCA in the cork and the orientation of the cork in the bottling, i.e. which end of the cork is inserted into the neck of the bottle, will determine whether that cork will contaminate the wine.

### 4. Formation of TCA in wine after bottling

The possibility of formation of TCA *in situ* in bottled wine has been suggested (Thevenet et al. undated, Smith 1992, Lee and Simpson 1993). For this to happen would presumably require either continued growth of micro-organisms capable of methylating TCP to TCA on the inner surface of the cork (in contact with the wine), or extraction of TCP into wine and then methylation by wine micro-organisms. In our laboratory, deuterium-labelled TCP (1,000 ng/L) was added to six different non-sterilised wines with and without sulfur dioxide and the bottles were then sealed and stored for over two years. These

experiments showed no conversion of the TCP to TCA (Liacopoulos et al. 1999b). While this outcome does not exclude the possibility that TCA formation from TCP could occur in wine in different circumstances, there remains no published evidence that this can take place.

#### *2-Methoxy-3,5-dimethylpyrazine*

Because MDMP has been identified as the cause of FM taint only recently (Simpson et al. 2004), studies of the rate or extent of transfer of MDMP between cork and wine have yet to be undertaken, and any assumptions about these processes must be inferred from the behaviour of structurally related compounds which can be expected to have similar physical and chemical properties. A recent study on the absorption of flavour compounds from bottled wines by closures (Capone et al. 2003) has shown that no significant absorption of the structurally related 2-methoxy-3-*iso*-butylpyrazine from wine by corks took place over a two-year period. It is likely, therefore, that MDMP also has a poor affinity for the cork matrix. If this is the case, then the partitioning of MDMP between cork and wine during cork soaks will eventually favour dissolution in the wine. Although nothing is known of the rate at which this transfer can take place, at least some MDMP can be extracted from cork by wine during the time for cork screening – from a few hours to a few days (Simpson et al. 2004).

#### *Other taint compounds*

Nothing is known about the rate or extent of transfer of most other taint compounds into wine from cork closures. Corks have a poor affinity for guaiacol when in contact with wine (Capone et al. 2003) and so we might expect that any cork-bound guaiacol in direct contact with wine in bottle will eventually be fully extracted into the wine. The partitioning of taint compounds between the wine and the cork is influenced by the affinity of the cork for the compound and its solubility in wine. More polar compounds such as 1-octen-3-ol and 1-octen-3-one are likely to be more soluble in wine and therefore could behave in a similar manner to guaiacol. On the other hand, non-polar substances such as sesquiterpene hydrocarbons can be expected to be less completely extracted. Geosmin and 2-methylisoborneol are likely to behave in an intermediate manner.

### **Screening cork closures for contamination with taint compounds**

In this section, the screening methods for cork closures followed by many wineries are reviewed and the factors influencing the extent of transfer of taint compounds into the soak wine are examined drawing on the implications from the preceding section in which the affinity of these compounds for the cork, their transmission through the cork, and their location within the cork were considered. We are able to glean some understanding of the cork-to-cork variation in the content of extractable TCA in bales of corks which then shows that simple batch assessment of corks can sometimes enable detection of the defective bales.

#### *Screening methods*

Prior to sale or use, many suppliers and users of natural bark closures screen samples of the closures for possible contamination with taint compounds. Such screening methods are usually based on sensory assessments of wine soaks of corks, or of corks placed in a damp environment for several hours. Sensory assessment has the advantage of relatively low cost, the lack of requirement for specialised technical or instrumental skills, and the scope to detect both known and unknown taint compounds. Disadvantages of these methods are variation in the performance of the assessors due to low sensitivity on some or all occasions (health, fatigue, anosmia, etc.) and lack of familiarity with some taints. To save time, and therefore allow a larger number to be screened, corks are most often soaked in batches.

Methods used by Australian wine companies to screen batches of corks for possible contamination with taint compounds have been described by several authors (e.g. Caloghiris 1995, Duncan 1995, Leske et al. 1995, Linton 1995, Shaw 1995). Nearly all companies soaked several corks (commonly five) in wine (nearly always a dry, neutral white wine), and the soaking time usually ranged from a few hours to two days. Longer soaking times can lead to an enhanced woody cork aroma which can interfere with the detection of TCA by smell (Amon and Simpson 1986). Simpson and Veitch (1993) have recommended against using aqueous ethanol for soakings. Usually, the number of corks taken for a sampling is less than that needed to give statistically reliable results (Simpson and Veitch 1993, Fugelsang et al. 1995, Dallavalle 1997), although sufficient to detect corks from severely tainted batches (Hervé et al. 2004, see discussion below). Some companies prepare extracts of many corks in a small volume of wine and then dilute the extract with additional wine to diminish the sensory effect of natural cork components. Unfortunately, while some cork components will be concentrated when multiple corks are soaked in small volumes, TCA is not so concentrated (Hervé et al. 2004, see below) and this methodology is likely to result only in the detection of corks containing a high level of TCA. Each company sets its own rejection criteria, usually based on the number of soaks showing taint and the type of taint detected. Spiked reference samples containing TCA are often included in the panel assessment of soaks to check panellist performance.

Other screening methods have also been proposed. These include instrumental analysis for TCA (Hervé et al. 2004), and aroma assessment of corks stored in sealed vessels in contact with a small amount of water for several days, conditions conducive to mould growth on the cork (Brunner 1989, Casey 1990). This latter test has the capacity to detect TCA that could be formed by mould growth during transportation or storage (providing the corks already contain suitable precursors such as trichlorophenol) as well as TCA already present on the cork, and therefore might prove useful in evaluating corks prior to shipping. Occasionally, cutting corks longitudinally is recommended (Casey 1990), but we regard this as pointless because any TCA that might be present in the inner part

of the closure would not be expected to migrate from whole corks into wine (see earlier) (Capone et al. 2002).

Other than bottling trials, which are completely impractical for everyday use (although valuable as a means to monitor the effectiveness of screening protocols), no cork screening method has been demonstrated to reflect accurately the level of taint in bottle. One reason for this could be that the whole surface of the cork is extracted in cork soaks, while, for bottled wine, only that part of the cork which is in contact with the wine – and not necessarily the part where the taint compounds are located – is extracted in bottled wine. Other reasons for the lack of a clear relationship between the proportion of corks seen as tainted by soaking and by bottling trials are discussed below for individual known taint compounds. Nevertheless, batches of corks that are badly contaminated with taint compounds (including those affected by chemical contamination during transport or storage) can be expected to be detected in almost all tests (see below).

#### *2,4,6-Trichloroanisole (TCA)*

##### 1. Apparent equilibrium concentration in wine soaks

Hervé et al. (2004) have gathered an extremely useful set of data on the partitioning of TCA between cork closures and wine in soakings and in bottles. They showed that when several composite batches of 100 corks were each soaked in 1,500 mL of wine, the concentration of TCA in the wine rose rapidly, reaching a peak after 12–48 h and then remaining relatively constant. They considered that this concentration was due to an equilibrium established between the TCA in the cork and in the wine and that it was determined by the physical characteristics of the cork. They defined this concentration of TCA as 'releasable TCA' and investigated its usefulness in quality assurance of cork closures. However, we are of the view that the equilibrium at this time is apparent rather than real, and that a more complex process is taking place as follows: TCA at, or very close to the surface of the cork or in the lenticels (i.e. wherever the wine comes into contact with the cork) is rapidly extracted into the wine, reaching a maximum value within a day or two. Simultaneously, TCA is re-absorbed by the corks, as shown by Capone et al (1999), until these processes take place at a near-identical rate, so that the concentration of TCA in the wine samples appears constant. The extractible TCA may be highly localised on various parts of corks at the beginning of the soaking, but should be more or less evenly distributed on or near the surface of the cork after one to two days. In redistributing the TCA in this way, an apparently steady concentration can be reached, as indicated by the data of Hervé et al. (2004). Their data gathered over five days appear to show a slight decrease in the concentration of TCA in the wine soak after 24 h, which is also consistent with the proposal that the corks re-absorb part of the TCA initially released. Over time (many months), some of the TCA that is not in immediate contact with the wine (i.e. sub-surface) probably also migrates into the wine. This supposition is supported by experiments in our laboratory described above which show that TCA can diffuse through the cork matrix, but that this process is extremely slow

(Capone et al. 2002). It also explains the observation of Hervé et al. (2004) that the concentration of TCA in bottled wine increases slowly over several months. In their bottling trial, TCA in the wines reached a concentration, for the most contaminated wines, of up to 15 ng/L after 14 months. This indicates that, in the latter part of the bottle storage period, the rate of extraction of TCA from the closures would have been, on average, less than one nanogram per litre per month, a rate that would be too low to be detectable in short-term (five day) kinetic studies.

Notwithstanding the above considerations, the data of Hervé et al. (2004) confirm that the time periods used in most soaking trials are sufficient to determine the TCA extractable by wine from corks in the short term. However, we prefer the term 'rapidly releasable TCA' for the TCA that is detected by these short-term cork soaks. The measurement of the TCA in the cork soaks is a determination of only a proportion of the total rapidly releasable TCA (see below). The larger proportion remains bound to the cork.

##### 2. Effect of wine volume and the number of corks on concentration of TCA in the soak wine

As a consequence of the high affinity of corks for TCA (i.e. most of the rapidly releasable TCA would be expected to remain absorbed on the cork), the concentration of rapidly releasable TCA in a cork should be approximately constant, regardless of the wine volume, within the range normally used for cork soaks. Assuming that the rate of extraction of TCA from the cork into the soaking wine is proportional to the concentration of rapidly releasable TCA in the cork, and the rate of reabsorption of TCA is proportional to the concentration of the TCA in the wine, then when these two rates are approximately equal (as is the case after one to five days of soaking) the concentration of TCA in the wine should also be independent of the wine volume, as indeed has been shown empirically by Hervé et al. (2004). Another consequence of only a small absolute proportion of rapidly releasable TCA being extracted from the cork is that, because the amount of rapidly releasable TCA in the cork is essentially unchanged after each soaking, repeated soaks of the same cork give the same concentration of TCA in the soaks for any given cork or group of corks, as also shown by the data of Hervé et al. (2004).

While the TCA concentration in wine derived from soaking one or several corks is essentially independent of wine volume, the effect on TCA concentration of the number of corks in the soak is important. With more corks, a greater surface area will be available for the retention and re-adsorption of TCA. The kinetic considerations discussed above suggest that dilution of the TCA on the cork surface would result in dilution of the TCA in the wine. Thus, if the rapidly releasable TCA in a single cork was such as to produce 100 ng/L of rapidly releasable TCA in a wine soak, and this single cork was soaked in the presence of 99 other TCA-free corks, the rapidly releasable TCA in the soak should become approximately 1 ng/L. This is based on the finding by Capone et al. (1999) that each cork has similar sorptive capacity. Similarly, if ten

corks, each containing sufficient rapidly releasable TCA to give 10 ng/L of TCA when soaked individually, were combined with 90 TCA-free corks, the rapidly releasable TCA in the soak should also become approximately 1 ng/L due to the additional surface area (10×) provided by the TCA-free corks. These assumptions have effectively been verified experimentally by Hervé et al. (2004) who showed that when 100 corks were soaked individually, the average of the individual rapidly releasable TCA values in the soaks was similar to the rapidly releasable TCA of all 100 corks soaked as a single batch (see later).

One of the difficulties of assessing batches of cork for possible TCA taint is the logistics of examining large numbers of individual corks, either instrumentally or by sensory means. For this reason, many companies choose to assess corks by soaking them in batches. For reasons discussed above, if one out of five corks contains rapidly releasable TCA, then this TCA will be present in higher concentration in an individual soaking than when soaked in the presence of four other non-contaminating corks. This could have the effect of reducing the concentration of TCA in the soak to a value below the limit of instrumental or sensory detection, and is likely to be a one of the reasons why the frequency of TCA detection in industry assessments of corks is less than the frequency reported in bottled wine (Casey 1995, Simpson et al. 2005). Another reason for this discrepancy is that TCA in wine that has been in bottle for several months or years is presumably a combination of some of the rapidly releasable TCA and TCA released more slowly from within the cork.

### 3. Cork-to-cork variation in TCA content in a bale

In their extensive investigations of TCA transfer between cork and wine, Hervé et al. (2004) examined the population distribution of rapidly releasable TCA in corks within a bale. Composite soaks of 100 randomly selected corks from each of 14 bales were analysed for rapidly releasable TCA. Additional samples of 100 corks from the same bale gave similar rapidly releasable TCA concentrations in the composite soaks. A further 100 corks were obtained from each bale, individually soaked, and the soaks analysed. A high correlation between the concentration of TCA in the soaks of the composite sample and the average concentration of the soaks of the individual corks was obtained for each bale. Examination of the population distribution of the rapidly releasable TCA, determined from the soaks of individual corks from the 14 bales, gave a clearer insight into the distribution of TCA within these bales. Most individual corks in the badly contaminated bales contained an elevated level of rapidly releasable TCA even though the corks were presumably derived from diverse origins. The corks in a bale are assembled by the major cork merchants but often supplied by many small processors, who are likely to obtain corkwood from different areas and forests. The study of Duncan et al. (1997) indicated that there were different levels of contamination in the forests but even in the same forest, only a relatively small proportion of the trees (7.5%) carried most of the TCA. Also, TCA was localised, with a higher content in the trunk and concentrated in the lower part of the trunk. Con-

sequently, most badly contaminated corks might originate from a small proportion of corkwood. Consequently, one should expect a considerable number of corks with little or no TCA in a bale initially. One explanation for the high TCA content of the majority of the corks in the defective bales examined by Hervé et al. (2004) is that corks that were initially non-tainting, accumulated TCA from nearby, badly contaminated corks through aerial transfer (Barker et al. 2001). The extent of the accumulation of TCA by individual corks would be dependent on their location in the bale, with greater uptake of TCA by corks in closer proximity to the badly contaminated corks.

While the data of Hervé et al. (2004) indicated that aerial transmission of TCA might have been occurring in the bale, it is not known for how long these bales had been set aside and retained before analysis. The transfer of TCA in these bales could have been more extensive than in bales normally received at wineries in Australia. The extent of secondary contamination, i.e. the increase in the number of contaminated corks in a bale, is likely to depend on the initial number of badly contaminated corks, their content of TCA and the time the corks were held in the bale. Even though secondary contamination of corks may only be a problem with the more severely affected bales, these aspects require further investigation.

Nevertheless, the data of Hervé et al. (2004) indicate that composite soaks of 100 corks could provide an effective way of distinguishing badly contaminated from less contaminated bales, and could enable the removal of the most contaminated closures from circulation at an early stage. A single soak of 100 corks appears to be sufficient for this purpose, as separate batches of 100 corks from the same bale gave near-identical results. It was not shown, however, whether the bales studied were from different sources and/or producers and whether, therefore, all cork batches would necessarily behave in a similar way. Finally, while composite soaks show promise for distinguishing moderately from badly contaminated bales, they may not be able to distinguish, for example, a batch with 1% contamination from a batch with 5% contamination. In the data displayed by Hervé et al. (2004), 5% or more of the corks were contaminated even in the batch with the lowest concentration of rapidly releasable TCA in the composite soaks.

### 4. Benefit of cork taint appraisals in closure quality assurance

All producers and users of cork closures seek cork screening methods that give a reliable indication of whether wines sealed with these closures are going to have an unacceptably high proportion of cork taint. Hervé et al. (2004) have attempted to relate rapidly releasable TCA in cork soaks to TCA released during bottle storage. They took 400 corks from three bales with a high level of contamination, determined the rapidly releasable TCA in soaks of each cork, coated the corks with paraffin and silicone oil and then used these corks (each individually coded) to seal 400 bottles of wine. Substantial numbers of individual bottles were opened after each of one, three, eight and 14 months storage. During this time, the mean

TCA concentration in the bottled wines increased steadily. There was a good correlation between the rapidly releasable TCA initially measured in the cork soaks and TCA concentration in the bottled wine for a short bottle storage period, but this correlation became weaker with longer storage times. Nevertheless, the authors concluded that measured rapidly releasable TCA can help predict bottle TCA.

Unfortunately, while Hervé et al. (2004) had earlier demonstrated that the soaking process would not remove any significant amount of TCA from the corks, they failed to recognise that, whereas the location of the rapidly releasable TCA on the corks *prior* to soaking was unknown (it might be evenly spread, but could just as easily be highly localised), it would almost certainly be distributed over the whole surface of the cork *after* soaking (see above), and that, as a result, both ends of each cork would contain a proportion of the rapidly releasable TCA. Thus every cork containing a significant amount of rapidly releasable TCA would now be able to impart some of that TCA into the bottled wine via direct contact between the end face of the cork and the wine. So the fact that there was a direct relationship between the rapidly releasable TCA measured prior to bottling and the concentration of TCA in the bottled wine after a short bottle storage period is not surprising. Nor is it surprising that this relationship broke down with increasing storage time, as the rapidly released TCA in the wine presumably became augmented with TCA from deeper within the cork matrix. The presence of more slowly released TCA in corks could also help explain why the proportion of bottles affected by TCA in industry appears to be greater than that indicated by industry soaking trials. Some subsurface TCA is certainly present in some corks, as shown by dissection experiments (Howland et al. 1997, Barker et al. 2001). Such more slowly released TCA would not be measured, nor would it be redistributed during the initial soaking. In this case, the location of this latter TCA (i.e. close to or remote from the cork/wine interface) would determine if, and to what extent, it would contribute to TCA in the bottle.

Soleas et al. (2002) also described the evolution of TCA in experimentally bottled wine and the corresponding loss of TCA from the cork closures in the bottles. They reported that little TCA was extracted from the closures in the first three months, the concentration of TCA then increased to a maximum after nine months and finally decreased during months nine to twelve. A substantial decrease in the amount of TCA in the extracts of the corks (between 50 and 95%!) was observed between one and two months of storage (no zero time data was presented), with little change thereafter. The apparent loss of TCA from the closures was much greater than the increase in TCA content of the wines. The authors interpreted these results as indicating metabolic conversion of TCA within the cork to other products or loss by escaping into the atmosphere. Unfortunately, despite the likelihood of substantial variance in the TCA content of these closures and the fact that at each time point, different bottles (two replicates only of each treatment) were opened, analysed

then discarded, no indication of the extent of such variance is given with the mean data. This, combined with our previously discussed concerns regarding the robustness of the analytical method, make it impossible to assess the reliability of these results.

The extent to which measuring rapidly releasable TCA in cork soaks predicts bottle performance is important to determine and has undoubtedly been done by some major wine companies, but such data are usually kept confidential. It would be beneficial to repeat the experiments of Hervé et al. (2004), but using separate batches of corks from the same bale for the soaking and bottling trials. In this case, a substantial number of closures and bottles would need to be examined in order to obtain statistically significant results.

#### *MDMP and other taint compounds*

If MDMP has a poor affinity for the cork, as is likely from the observations of Capone et al. (2003) for 2-methoxy-3-*iso*-butylpyrazine, then the partitioning of MDMP between cork and wine during cork soaks will favour dissolution in the wine, and the concentration of MDMP in cork soaks will be inversely proportional to the volume of wine used in the soaks. This contrasts with the situation with TCA in which TCA concentration in cork soaks is independent of the volume of wine used. Thus, soaking corks in relatively small volumes of wine could exaggerate the sensory impact of MDMP compared to TCA, and could therefore also lead to masking of the latter in cork assessments where the sensory panel is able to distinguish between these two types of taint. The experience of Southcorp Wines suggests that FM taint is more frequently detected in cork soaks than in commercial wine (Simpson et al. 2005) and this is likely to be a result of the smaller volumes of wine used in the cork screenings.

Based on these considerations and an estimation of their polarity, some other taint compounds (guaiacol, 1-octen-3-one and 1-octen-3-ol) might be expected to behave in a similar manner to MDMP, while geosmin and 2-methylisoborneol could behave in an intermediate manner between that of TCA and MDMP.

#### **Removing taint compounds from cork closures**

Having examined the factors that are likely to control the transfer of taint compounds to wine in bottle and in the soak wine used in industry screening of corks, we are able to comment on some of the more recent strategies developed by cork merchants for the removal of taint compounds from corks during manufacture.

Several strategies for dealing with the problem of cork contamination by taint compounds have been suggested. Given that TCA migrates only very slowly through cork (Capone et al. 2002), it can be argued that those processes that are designed for the cork cylinder or for cork granules intended for use in technical closures are most likely to be successful (rather than, for example, the cork slabs where new surfaces, which may be severely contaminated, are generated during manufacture of the cylinders). Such processes presumably do not need to remove all TCA from wine corks, but only that which is at

or near the surface and exchangeable during the lifetime of wine in bottle. Even if TCA in the centre of a cork were to eventually spread evenly through the entire closure (a condition that would presumably not be reached during normal bottle storage), most of this TCA would remain bound to the cork (Capone et al. 1999).

The concentration of TCA in cork can be diminished by simple aeration (Thevenet et al. undated, Barker 2001), and this process can be accelerated by a high moisture content (Casey 1995) and heat. Thus, the Amorim group, a major Portuguese cork producer, uses a steam-cleaning process called ROSA to remove TCA from cork. The effectiveness of this process for cork granules has been independently verified by one of us (Sefton unpublished), and also by Hall et al. (2004). Treatment of contaminated granules by ROSA removed 75–80% of rapidly releasable TCA from the granules. Importantly, Amorim have also conducted bottling trials to determine whether the amount of TCA that is released more slowly into wine from deeper within the cork is similarly reduced, and have reported encouraging results at recent industry seminars.

Taylor et al. (2000) used supercritical carbon dioxide extraction of cork stoppers in a new analytical method to determine TCA in corks. They found that virtually all TCA added to ground cork could be isolated by this technique. Supercritical carbon dioxide extraction is now used by Sabaté to remove TCA from cork granules, a procedure known as the 'Diamant' (diamond) Process (Lumia et al. 2001). Recent closure trials conducted at the Australian Wine Research Institute have found no TCA transfer to wine from prototypes of these closures after two years in bottle (P. Godden, personal communication). Other cork processing techniques designed to diminish cork taint, include microwave treatment (Jäger 1999), the use of enzymes (Conrad et al. 1999), and the use of physical barriers to cover the ends of corks (Vasserot et al. 2001).

Silva Pereira et al. (2000) reported that cultures of *Chrysonilia sitophila* would grow on cork, inhibiting the growth of other moulds and degrading TCP, but would produce neither TCA from TCP, nor other unpleasant smelling metabolites.

Finally, it is important to emphasise the value of bottling trials rather than short term cork soakings to determine the effectiveness of processes to remove taint from cork closures. Such trials can be monitored either by instrumental analysis, particularly for TCA, or by sensory assessment, which has the advantage of also detecting less easily measured or unknown taint compounds.

### Summary

TCA is recognised as the major cause of cork taint in bottled wine. But occasionally, the concentration of TCA present in tainted wine cannot account for the intensity of the taint. Presently, there are about 12 compounds in addition to TCA that are implicated in cork taint, either as a primary cause of cork taint or co-occurring with other taint compounds. All are aroma-intense microbial metabolites. Because TCA has dominated the literature on cork taint, the role of these other compounds has not received adequate attention and is incompletely understood.

The factors affecting the transmission of taint compounds in cork to wine have been considered in detail in this review. TCA has been the primary topic of investigations reported in the general literature and is therefore the main focus of the present article. TCA is strongly retained in the cork whereas most of the other compounds implicated in cork taint have greater polarity and are likely to be more completely transferred into the wine. Part of the TCA in a contaminated cork is more amenable to extraction into the wine; it follows that this TCA must be located on or close to the surface of the cork or within the lenticels where the wine can reach or it can migrate into the wine. In addition, some TCA is less readily released and part of this may never transfer into the wine during normal bottle storage because it is locked away deep within the matrix of the cork. Part of the more slowly extractible TCA that is not accounted for in wine soaks used in taint assessment could nevertheless contribute to TCA measured in wine after months in bottle, because the longer time will allow migration of some of the TCA within the cork to parts of the cork that are extractible by the wine.

Several investigations examining the location of TCA in corks have confirmed that the concentration is not uniform throughout the cork. For example, unused corks from a contaminated batch showed higher concentrations of TCA in the sections corresponding to older bark, suggesting that the corkwood might have been contaminated whilst in the forest. In addition, these corks also contained more TCA at or near the surface of the cork.

The procedures for the assessment of cork taint in the quality assurance of closures have been examined. The proportion of TCA contaminated corks detected in the quality assessments conducted by a major Australian winery of about 1% was much lower than the incidence of cork taint generally observed in Australian bottled wine (about 5%). The importance of TCA, and other non-polar compounds implicated in cork taint such as sesquiterpene hydrocarbons, can be underestimated during cork assessments because of the conditions of the assay, in particular, the short duration of the extraction (usually 24 or 48 h), the presence of several (usually five) corks in each soak and the small volume of wine. A greater proportion of compounds more polar than TCA is likely to be transferred into the soak wine, so these test conditions could then exaggerate their potential to taint wine in the bottle.

Despite the limitations of the test procedures followed by most wineries, the tests are able to detect badly contaminated bales and enable wineries to reject these bales. For this reason, it is strongly recommended that wineries continue these assessments as part of their quality assurance for closures.

Several commercial treatments of cork to remove contamination appear promising, i.e. decontamination of corks by treatment with steam, supercritical fluid extraction, microwave treatment, enzymes, end coating using materials that stop the transmission of TCA into the wine, and use of cultures that degrade TCP without forming TCA. It will be necessary to evaluate these treatments more completely by bottling trials and to assess the

removal not only of TCA, but also other compounds implicated in cork taint. However, treatments that are effective in removing TCA are also likely to be effective in removing other potential taint components that – in most instances – are less strongly retained by the cork.

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