

## **2,4,6-TRICHLOROANISOLE: A CONSUMER PANEL EVALUATION**

### **2,4,6-TRICHLOROANISOL: AVALIAÇÃO POR UM PAINEL DE CONSUMIDORES**

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*(Manuscrito recebido em 11.07.06. Aceite para publicação em 23.12.06)*

#### **SUMMARY**

The “cork taint” is a major problem in wine industry and may be caused by the several sources of contamination although wine cork stoppers are usually subject to suspicion: 2,4,6-trichloroanisole (TCA) has been frequently associated to this off-flavour.

In this work, a consumer panel performed “forced choice” triangular tests in order to measure Odour Detection Thresholds (ODT) and Taste Detection Thresholds (TDT) of TCA in water, hydro-alcoholic solutions (11.5% and 18% ethanol) and white and red wines.

A paired preference test was also performed by the panel in order to measure Odour Rejection Threshold (ORT) in white and red wines spiked with TCA.

Results obtained show that the ODT and the TDT for TCA were respectively: 0.2 and 0.3 ng/L in water, 0.9 and 1.7 ng/L in red wine and 1.5 and 1.0 ng/L in white wine. ODT were 4 and 10 ng/L respectively for hydro-alcoholic solutions with 11.5% and 18% ethanol. ORT for white wines was 10.4 ng/L and for red wines 16.0 ng/L.

These results suggest that although this group of non trained assessors detected very low concentrations of TCA in wines, they did not reject the wine at these low concentration values.

**Key words:** TCA; Cork taint; wine; sensory evaluation

**Palavras chave:** TCA; gosto a rolha; vinho; avaliação sensorial

## INTRODUCTION

The so called “cork taint” is an off-flavour often associated to wine quality, and it is responsible for important losses in wine and cork industries, as consumers tend to reject a wine when this off-flavour is detected. About 2% to 7% of bottled wines are contaminated (Waterhouse et al., 2001; Casey, 1999) with an estimated cost of 1000 million Euros/year to wine industries (Moore et al., 2003; Sanvicens et al., 2003). In Europe cork taint affects between 0.1 to 10% of bottled wines (Riu et al., 2006).

The mechanisms involved in the development of “cork taint” in wine have been related with the presence of chloroanisoles. A wine contaminated with these compounds will be characterized by a musty, mouldy, earthy odour prevailing over its characteristic aroma notes (Pereira et al., 2000, Waterhouse et al., 2001; Casey, 1999). It has been pointed out that 2,4,6-trichloroanisole (TCA) is the main responsible for this defect due to its particularly low sensory threshold, however other compounds have been claimed to be involved as other chloroanisoles: 2,3,4,6-tetrachloroanisole (TeCA) and pentachloroanisole (PCA) (Callejon et al., 2006; Insa et al., 2006) and degradation products of TCA the 2,4-dichloroanisole and 2,6-dichloroanisole (Callejon et al., 2006). Other compounds such as guaiacol, 1-octen-3-one, 1-octen-3-ol, 2-methylisoborneol (Riu et al., 2006) resulting from the metabolism of certain filamentous fungi and more recently, 2,4,6-tribromoanisole (TBA) (Chatonnet et al., 2004; Lorenzo et al., 2006; Ezquerro et al., 2005) were detected in wines with a significant musty character. TBA off-flavour was perceptible on smelling at concentrations as low as 4ng/L (Chatonnet et al., 2004).

The presence of TCA is usually attributed to wine cork stoppers but other important contamination sources should be considered as the wine bottle, materials used during the wine processing or storage and the surrounding atmosphere in cellar (Bertrand, 1998; Bertrand et al., 1994; Chatonnet et al., 1994; Chatonnet et al., 1995; Tindale et al., 1989). PCA and TCA, may be produced by the biochemical breakdown of certain pesticides containing 2,3,4,6-tetrachlorophenol (TCP) or pentachlorophenol (PCP), contaminating wines that have not been in contact with cork (Chatonnet et al., 2004).

Due to the low levels at which these compounds contaminate wines, often lower than detection limits of the analytical techniques used, the analysis of these compounds includes preconcentration steps and the use of sensitive detectors.

With a low detection threshold by humans, compounds responsible for off-flavours are usually detected by the human nose and palate at very low concentrations (a few ng/L), sensory evaluation is an important tool to detect

the presence of TCA and is often used in quality control (QC). The ability to detect this sensory defect depends on the concentration of the compound, type and chemical composition of wine contaminated and also on the sensitivity of the assessors involved (Moore et al. 2003; Waterhouse et al. 2001; Casey, 1999). It is known that, although a wine may not have a distinct musty/mouldy character at very low concentration, its odour can be acrid or dusty masking somehow its flavour, but for the ordinary consumer, this may diminish the enjoyment of the wine without recognition of the defect (Casey, 1999).

Selected and trained assessors will be able to detect low concentrations of TCA but some consumers may also be very sensitive to “cork taint”. However, this ability to detect the off-flavour does not mean that the consumers will reject wines contaminated with these concentration levels (Prescott *et al.*, 2005).

The detection threshold (DT) values, defined as the minimum value of a sensory stimulus needed to give rise to a sensation (ISO Standard 5492:1992), reported for TCA are, in water, 0.03-5 ng/L for the odour detection threshold (ODT) and 0.001-0.1 ng/L for the taste detection threshold (TDT) (Curtis et al. 1974; Griffiths, et al., 1974; Ochiai et al., 2001; Montiel et al., 1999; Cantagrel et al., 1990; Suprenant et al., 1997). ODT values described in the literature for red wine are between 4–50 ng/L and 2 ng/L for TDT (Soleas et al., 2002; Fischer et al., 1997). For white wines values described are somehow similar to red ones: 4-10 ng/L for the ODT and 2 ng/L for the TDT (Soleas et al., 2002; Ribéreau-Gayon et al., 1998; Fischer et al., 1997). For cognac with 40% alcoholic content, Cantagrel et al. (1990) reported values between 300 and 1500 ng/L for the ODT.

In this work, odour rejection thresholds (ORT) for TCA in white and red wines were evaluated and the odour detection thresholds (ODT) and/or taste detection thresholds (TDT) were determined in: water, hydro-alcoholic solutions, white and red wines. Hydro-alcoholic solutions at 11.5% and 18% were used in order to simulate different kind of wines (table and fortified wines) often consumed in Portugal.

## **MATERIALS AND METHODS**

### **Assessors**

Non trained assessors (ISO Standard 5492:1992), students and staff from the laboratory, were recruited to participate in sensory tests. Some questions were asked in order to characterize the population participating in the tests: gender, age, consumption and preferences for different type of wines and also to evaluate their knowledge about the cork taint. Assessors were aged between

22 and 66 years. Considering the gender 8 assessors were male and 36 female. The majority of the assessors were regular consumers of Portuguese wines of different types and quality patterns (table wines, table wines with geographical indication and quality wines with designation of origin). Considering their preferences, 21 assessors preferred red wines and the frequency of consumption was at least once a week. Red wines were consumed at least once a month by 11 assessors and another 3 consumed white wines.

The number of participants (non trained assessors) in each session varied between 26 and 44.

### **Samples**

A slightly acid (pH = 5.68) Portuguese spring water (Cabril) was used to prepare solutions for assessment of the ODT and TDT in water.

Portuguese medium priced red and white wines (Terras Durienses Regional wine) with a bag-in-box closure system were used to assess the ODT, TDT and ORT. This type of system was used in order to be sure there was no previous contact between wine and cork stoppers although, as mentioned above, other contamination sources may occur in wines.

The concentrations used in each test were chosen according to values described in the literature for the detection threshold (DT) of TCA in water, red and white wine. The same concentrations were used for tests with hydro-alcoholic solutions.

A 0.100 g/L stock solution of TCA (Aldrich) was used to prepare solutions with different concentrations of this compound in spring water: 0.0625, 0.125, 0.25, 0.50, 1.0, 2.0 and 4.0 ng/L. Hydro-alcoholic solutions at 11.5%, 18% and spiked wines, were prepared at the following concentrations: 0.50; 1.0; 2.0; 4.0; 8.0; 16; 32 ng/L.

### **Procedures for sensory tests**

“Forced choice” triangular tests were used for the determination of the ODT and TDT of TCA. Sets of three coded samples were prepared: two samples were identical and the other was different (odd sample).

For the evaluation of odour, assessors were asked to take a deep breathe of each of the three samples and then had to choose the sample that was different from the other two. Assessors were supposed to rest a few minutes between each test.

For taste evaluation each assessor placed the sample in the mouth, moved the liquid around for a few seconds and after that the sample was spit. The

assessors were not allowed to swallow samples. After each test, assessors rinsed their mouths with spring water and rested a few minutes.

For each subject, tests were performed individually in different days and each session was performed during a long period, in order to give time for assessors to have a rest to attenuate the eventual tiring effect resulting from a continuous evaluation.

The order of presentation of the samples was randomised in each set of samples. Assessors started evaluating the set with the lowest concentration of TCA and were asked to identify the odd sample. If the sample was not identified correctly, a new set with a higher concentration of TCA was presented to the assessors until a correct identification was obtained. When a correct answer was given the test was repeated. If the subject again identified correctly the different solution, it was considered that he had reached its DT. If the identification was not correct, a new set with a higher concentration of TCA was given for evaluation. If the subject failed all the concentrations tested, it was considered that his DT was higher than the concentration range used in the test, and the result was assigned as: “does not detect”.

Paired comparison preference tests were performed for the determination of rejection thresholds for TCA in white and red wines. Series of paired comparison tests were used: each pair consisted in one sample of wine and one sample of wine spiked with increasing concentrations of TCA: 0.50; 1.0; 2.0; 4.0; 8.0; 16; 32 ng/L. Assessors were asked to choose the sample they preferred in terms of odour and taste, from the pair presented. Between sets assessors were supposed to rest a few minutes.

In all tests performed, the TCA solutions were presented in ascending order, randomised across each series and the sample volume distributed to each assessor was 15 mL.

All tests were performed in a quiet room, free from odours and noises, illuminated with natural light, and situated not far from the assessors' work places.

The criteria used for the significant detection (percent of assessors that identified the sample containing TCA) or rejection (percent of assessors that have chosen the sample without TCA) as a function of TCA concentrations, were based on the binomial distribution tables for triangular and paired comparison tests included in ISO Standard 4120:2004 and ISO Standard 5495:2005, respectively. The significance at the 5% level was considered for the number of assessors ( $N$ ) participating in each test performed.

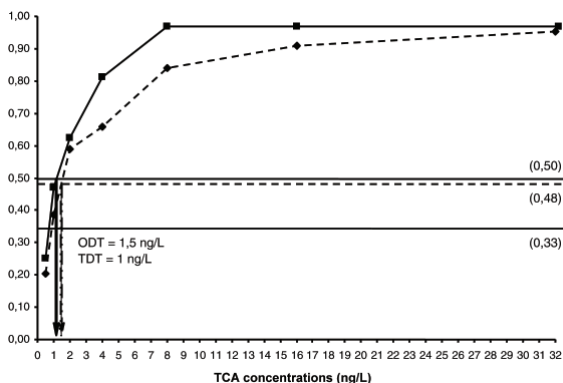
## **RESULTS AND DISCUSSION**

The sensory tests used in this work, triangular and paired comparison tests,

are simple to perform with non trained assessors, as evaluations are based on comparisons of a small number of samples. As previously reported (Casey, 1999) the recognition of low levels of TCA is enhanced when the subject has a sample for comparison and under these circumstances, tasters become quite sensitive to very low levels of cheoroanisoles.

The total number of assessors ( $N$ ) used in each sensory test was taken into account to calculate the minimum number of correct answers required to establish there was a significant difference among samples, when triangular tests were performed. The number of correct answers for each test performed at each concentration was counted and the value obtained compared with the binomial distribution tables according to procedure above described.

For ODT determination in white wine, 44 assessors performed the triangular test. The curve defined by the cumulative proportion of these assessors selecting correctly the sample with TCA for each concentration value is presented in figure 1. The minimum number of correct answers at the 5% level of



**Fig. 1** – Cumulative proportion of assessors identifying the white wine containing TCA at each concentration tested for determination of ODT (—) and TDT (---). The line at 0.33 represents the proportion of results obtained by random response; the lines at 0.48 and 0.5 indicate respectively the proportion of results for the 5% significance criterion using the binomial distribution for triangular tests for the ODT ( $N = 44$ ) and the TDT ( $N = 32$ ) determination

*Proporção cumulativa do número de consumidores que identificou correctamente o vinho branco contaminado com TCA para cada concentração testada para a determinação do LDO (---) e LDG (—). A linha a 0,33 representa a proporção de resultados obtidos por resposta alietória; as linhas 0,48 e 0,5 indicam para o LDO ( $N = 44$ ) e o LDG ( $N = 32$ ) a proporção de resultados para um critério de significância dos resultados de 5% considerando uma distribuição binomial para o teste triangular*

significance using the binomial distribution for triangular tests is 21 (ISO

Standard 4120:2004), corresponding to 0.48 of the total number of assessors. The concentration value corresponding to the detection threshold of TCA was calculated from the interception between the curve presented and the line at 0.48 corresponding to the cumulative proportion of assessors selecting correctly TCA sample. The line at 0.33 corresponds to the minimum cumulative results expected for a random test.

The same methodology was used to evaluate data obtained with the other samples studied and Table 1 summarizes all the results obtained for tests performed by the assessors (N) using water, hydro-alcoholic solutions (11.5% and 18%) and white and red wines.

TABLE I

Threshold values for TCA (concentration in ng/L) accessed in different matrices. ODT: odour detection threshold; TDT: taste detection threshold; ORT: odour rejection threshold; N: number of assessors; n.p.: not performed.

*Limiares determinados para o TCA (concentração em ng/L) em diferentes matrizes. LDO: limiar de detecção olfativo; LDG: limiar de detecção gustativo; LRO: limiar de rejeição olfativo, N- número total de consumidores; n.p.- teste não efectuado*

Threshold	Water	Hydro-alcoholic solution at 11.5%	Hydro-alcoholic solution at 18%	White wine	Red wine
ODT	0.2 (N = 40)	4.0 (N = 31)	10.0 (N = 39)	1.5 (N = 44)	0.9 (N = 30)
TDT	0.3 (N = 31)	n.p.	n.p.	1.0 (N = 32)	1.7 (N = 28)
ORT	n.p.	n.p.	n.p.	10.4 (N = 26)	16.0 (N = 26)

Results obtained show that ordinary consumers can detect TCA concentrations in water as low as 0.2 and 0.3 ng/L by odour or taste evaluation.

Odour threshold obtained when hydro-alcoholic solutions were tested are higher, as the alcohol content must influence the capacity to detect this off-flavour: the ODT value increased when ethanol content increased from 11.5 to 18%.

The DT values obtained in tests performed with white and red wine are lower than those obtained for the corresponding hydro-alcoholic solution (11.5%). It seems that somehow the complex matrix of a wine makes easier for the assessors to detect the “cork taint”. When assessors were asked to describe

the differences noticed among the samples (wines and wines spiked with TCA), they said that spiked samples were different although they were not really able to detect the musty, mouldy odour.

The TDT was lower for white wine than for red wine. This can be explained by the fact that red wines have more complex matrices with a higher polyphenolic content, contributing to some astringency of wine and masking the TCA off-flavour sensation. Some astringency was expected in sensorial evaluation of the young red wine used in these experiments.

Red wines presented lower ODT values than white wines. These results can be explained by the higher contents of fruity flavours usually present in white wines that could make detection of TCA more difficult. As the number of assessors used in the tests was not the same, this fact could also be responsible for the different values obtained.

The ODT value for water solutions, 0.2 ng/L, is in agreement with the values reported in literature (Ochiai *et al.*, 2001; Cantagrel *et al.*, 1990) and TDT values, 0.3 ng/L, is slightly higher than the values mentioned by Montiel *et al.*, 1999.

For the red wine the ODT value calculated, 0.9ng/L, is much lower than the values mentioned in the literature (Fischer *et al.*, 1997) and the TDT value, 1.7ng/L, is in agreement with values previously reported (Soleas *et al.*, 2002). For the white wine, the ODT and TDT values calculated, 1.5 and 1.0 ng/L respectively, are lower than the ones reported in the literature (Ribéreau-Gayon *et al.*, 1998; Fischer *et al.*, 1997; Soleas *et al.*, 2002).

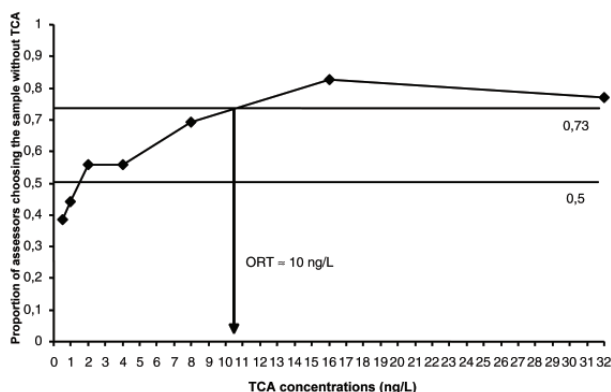
As expected, the ODT obtained for hydro-alcoholic solutions at 18% ethanol (10.0 ng/L) was higher than the corresponding value for 11.5% hydro-alcoholic solution (4.0 ng/L). There were no results published in literature for these concentrations of ethanol although the use of hydro-alcoholic cork lixiviates is a current practice to access the presence of off-flavours in cork stoppers.

When dealing with consumers, the determination of the rejection thresholds can be much more important for industry purposes, than the determination of their detection thresholds. Therefore a paired comparison test was also performed in order to determine the odour rejection threshold (ORT).

For each concentration the data corresponding to the proportion of assessors choosing the sample without TCA is shown in Figure 2, illustrating the determination of ORT for TCA in white wine. The lines corresponding to the minimum number of assessors at the 5% level of significance in a two sided test, and to the random response (0.5) are marked in the figure. The results for the red wine were treated in the same way.

The ORT values obtained (see Table 1) are in agreement with data published





**Fig. 2** – Cumulative proportion of assessors choosing the white wine sample without TCA added, at each concentration used. The line at 0.5 represents the results obtained by random response and the line at 0.73 indicates the 5% significance criterion for determination of ORT using the binomial distribution for a paired two-sided test ( $N = 26$ )

*Proporção cumulativa do número de consumidores que escolheu o vinho branco sem adição de TCA, para cada valor de concentração estudado. A linha a 0,5 representa os resultados obtidos por resposta alietória e a linha a 0,73 indica para o LRO a proporção de resultados a obter para um critério de significância dos resultados de 5% considerando uma distribuição binomial para um teste par bilateral ( $N = 26$ )*

for white wines (Prescott *et al.*, 2005) which refer that although the TCA has very low DT it will only cause a real defect in wine detected by a significant number of assessors in concentrations above 10–20 ng/L.

These results lead us to believe that, for the ordinary consumer, the presence of low concentrations of TCA in wines or alcoholic drinks does not affect them as much as it would be expected. Results obtained showed that the assessors could discriminate between two stimuli: the wine and the wine spiked with TCA. The results from the questionnaire performed to the assessors showed that the majority of them knew that cork taint is an off-flavour associated to the aging and the presence of fungi, but only 40% of them used descriptors associated with this off-flavour to characterize wines spiked with TCA.

Although results presented here refer to white and red wines from the Douro region, they can be taken as representative of Portuguese table wines.

The use of Rejection Threshold (RT) may be a more efficient tool to establish wine quality control guidelines related with rejection of wines due to contamination with TCA, and may give more important information about consumer's preferences than the Detection Threshold (DT). To ascertain the utility of rejection threshold values, these studies should continue with a

larger number of consumer participants as it is well known that sensory analysis results are rather subjective.

Complementary experimental work should be done with other wines, such as wines from other zones of the country, presenting distinct organoleptic characteristics, in order to conclude about the influence of the matrix in the evaluation of the effect of TCA on the preference for different wines. It is well known that taste and odour interactions may occur when complex stimuli are used (Delwich, 2004).

The same consumers should participate in all the tests performed as important differences may occur among detection threshold for each subject: 20% of the assessors detected odour differences among wines spiked with 0.5 ng/L of TCA and 4% did not detect the difference when the TCA content was 32 ng/L.

## CONCLUSIONS

Results obtained in this work show that, for detection of TCA, the concentrations in wines must be higher than in water. The detection of low concentrations of this compound is more difficult in wine which has a more complex matrix than water.

Higher concentrations of TCA are necessary to detect this off-flavour in hydro-alcoholic solutions with increasing concentration of ethanol. These results confirm that in wines with higher content of ethanol it may become more difficult to detect this off-flavour.

Lower concentrations of TCA were detected in red wines, when the odour test was performed, although it was expectable that the detection threshold should be higher in these samples, as they have a more complex matrix. These results may be due to the higher content of fruity flavours in white wines or to the different number of assessors used in both tests. For taste detection, it was easier to detect the “cork taint” off-flavour in white wines.

The results obtained show that the detection of a difference due to compounds such as TCA, does not mean necessarily that a non trained assessor considered as an ordinary consumer will reject the product.

The methodology used in this work based in the establishment of consumers Rejection Thresholds (RT) instead of Detection Thresholds (DT) may be a useful tool to evaluate the real impact of TCA and other compounds, in wine and other food products. However, when dealing with trained assessors performing Quality Control analysis, we have to rely on their detection thresholds to make sure they are able to detect the presence of TCA at very low levels.

## RESUMO

### 2,4,6-Tricloroanisol: avaliação por um painel de consumidores

O denominado “gosto a rolha” é um problema de grande importância para a indústria vitivinícola e constitui um defeito que aparece por contaminação dos vinhos. Esta contaminação é normalmente atribuída à rolha de cortiça e o 2,4,6-tricloroanisol (TCA) é o composto mais frequentemente associado a este “off-flavour”.

Neste trabalho, um painel de consumidores efectuou ensaios triangulares de resposta obrigatória, com vista à determinação dos Limiares de Detecção Olfactivos (LDO) e Limiares de Detecção Gustativos (LDG) do TCA em água, soluções hidroalcoólicas (11,5 e 18% etanol) e vinho de mesa branco e tinto.

Um ensaio de comparação por pares foi utilizado pelo painel para avaliar o Limiar de rejeição Olfactivo (LRO) em vinhos brancos e vinhos tintos aos quais foi adicionado TCA.

Os resultados obtidos mostram que o LDO e LDG em água correspondem respectivamente a 0,2 e 0,3 ng/L, em soluções hidroalcoólicas a 11,5 e 18% o LDO foi de 4 e 10ng/L respectivamente. Nos vinhos tintos o LDO e LDG foi de 0,9 e 1,7 ng/L e nos vinhos brancos foi respectivamente 1,5 e 1,0. O LRO obtido para o vinho branco foi de 10,4ng/L e para o vinho tinto 16,0 ng/L. Os resultados obtidos indicam que, embora este grupo de consumidores tenha detectado a presença de TCA em baixas concentrações nas diferentes amostras testadas, não rejeitaram as amostras de vinho que apresentavam estes teores.

## RÉSUMÉ

### 2,4,6-Tricloroanisol: évaluation par un group de consommateurs

Le «goût de bouchon» est un problème important dans l'industrie vinicole et est provoqué par la contamination des vins. Cette contamination est habituellement attribuée aux bouchons de liège et le 2,4,6-trichloroanisole (TCA) est l'un des composés la plupart du temps associés à ce goût.

Dans ce travail, un panel de consommateurs a effectué des essais triangulaires de choisi «forcé» afin de mesurer les seuils de détection d'odeur (SDO) et les seuils de détection de goût (SDG) de TCA en eau, solutions hydroalcooliques (éthanol de 11.5% et de 18%) et vins blancs et rouges.

Un épreuve de préférence par paire a été également exécuté par le panneau afin de mesurer le seuil de rejet d'odeur (SRO) en vin blanc et rouge pointu avec TCA.

Les résultats obtenues pour les SDO et les SDG pour TCA dans l'eau étaient 0.2 et 0.3 ng/L, respectivement. Dans les solutions hydroalcooliques avec de l'éthanol de 11.5% et de 18% les SDO étaient 4 et 10 ng/L respectivement. En vin rouge les SDO et les SDG étaient 0.9 et 1.7 ng/L et en vin blanc était 1.5 et 1.0 ng/L respectivement. SRO pour le blanc était 10.4 ng/L et pour les vins rouges 16.0 ng/L.

Ces résultats suggèrent que bien que ce groupe de consommateurs ait détecté des concentrations très basses de TCA en vins, ils n'aient pas rejeté le vin à ces valeurs basses de concentration.

## ACKNOWLEDGEMENTS

The authors thank to all the assessors that have participated in the present work.

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