

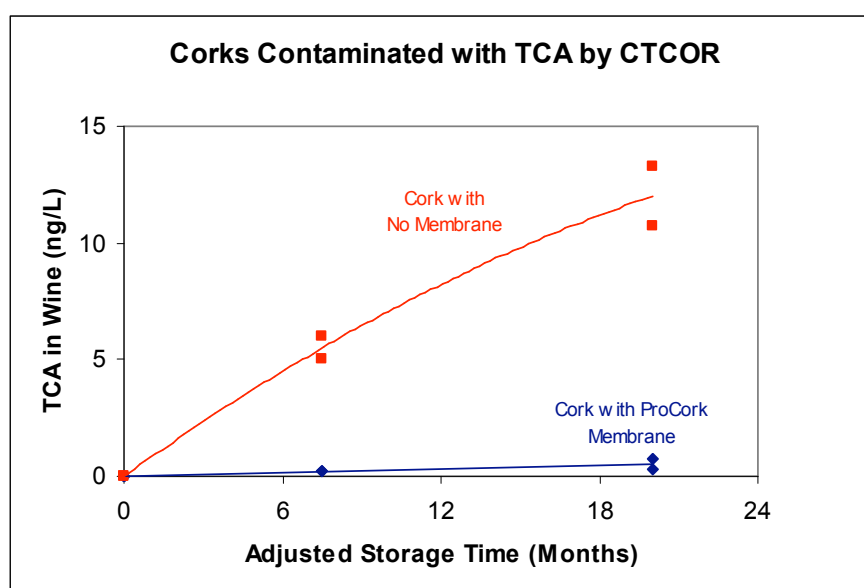


# **Effect of ProCork Membrane on 2,4,6-Trichloroanisole Extraction From Wine Cork**

**20 Months Storage Time (Adjusted)  
April 2004**

## 1. Abstract

Industry standard wine corks were contaminated by *Centro Technologico Da Cortica* (CTCOR) with TCA and then coated with the ProCork membrane Type B. Measurement of TCA by GC/MS (shown in Figure 1 below) in the wine, 20 months (adjusted) after bottling, showed the ProCork membrane reduced TCA contamination levels by 90 to 100%.



**Figure 1: Bottle Performance of ProCork Membrane Cork (20 months (adjusted) storage)**

## 2. Background

In May 2003, industry standard wine corks pre-contaminated with TCA were coated with ProCork membrane to determine the reduction in TCA permeation through wine corks in bottles over time as a result of membrane application. The TCA measurements were independently conducted by the Analytical Service of the Australian Wine Research Institute (AWRI) and Vinpac International. The wine samples were analysed by chemical analysis for chloroanisoles by Gas Chromatography/Mass Spectrometry (GC/MS).

## 3. Materials and Methods

The contaminated corks used in this trial were supplied by CTCOR. The corks were contaminated at CTCOR with a consistent quantity of TCA. The contamination was achieved by placing the corks in a desiccator with TCA compounds injected at the bottom. The desiccator was heated up to allow the



TCA vapour to circulate around the corks and naturally absorb into the corks.<sup>1</sup> The dimension of the corks was 24.2mm ± 0.2 x 45mm ± 0.4.

The extractable TCA in the corks was measured by AWRI and Vinpac International. The amount of TCA extractable from corks at the 7.5 month (adjusted) mark were measured by AWRI by soaking the whole cork in 100ml model wine (10% ethanol potassium tartaric acid) for each cork for 24 hours.<sup>2</sup> The amount of TCA extractable from corks at the 20 month (adjusted) mark were measured by Vinpac International by soaking the cork in 90ml model wine for each cork for 24 hours. The result is given as the concentration of TCA measured in the model wine after the 24 hour soak. The results are tabulated in *Table 1*.

**Table 1: Extractable TCA from Contaminated Corks (24 hour Soak Test)**

Description	Average 2,4,6-TCA ng/l
Cork 1	17.5*
Cork 2	20*
Cork 3	16.9**
Cork 4	13.6**
Cork 5	15.2**

\* Measured by AWRI

\*\* Measured by Vinpac International

The corks were coated with ProCork membrane B according to P83 on 30<sup>th</sup> May 2003 using the Procork 1 pilot machine. The corks were coated with paraffin wax (0.01g/cork) and CAF (0.002g/cork).

All the corks were then vacuum bottled on 24<sup>th</sup> June 2003. The bottles were allowed to stand upright for 2 weeks prior to storage (upside down to allow the cork end to be fully submersed in wine) on 8<sup>th</sup> July 2003.

### **Time Adjustment**

To accelerate the observation of TCA contamination in the wine, the bottles were filled to only 300ml. As a result, the actual time performance in the bottle needs to be adjusted by a factor of 2.5. Therefore, the 3 month data is equivalent to 7.5 months in a full 750 ml bottle and 8 month data is equivalent to 20 months in a full 750 ml bottle.

### **Chemical Analysis**

<sup>1</sup> Information on procedure provided by CTCOR

<sup>2</sup> Information on procedure provided by AWRI



The wine samples were sent to AWRI for TCA analysis after 7.5 months (adjusted) of storage. The chemical analysis conducted by AWRI involved taking a 200ml sub-sample from the bottles of wine sent for TCA analysis, and sealed with a foil seal for analysis for chloroanisoles by GC/MS.<sup>3</sup>

The wine samples from the 20-month (adjusted) storage were sent to Vinpac International. The chemical analysis involved taking a 5ml sub-sample (analyte) from the bottles of wine and analysed by GC/MS using SPME. Deuterated-TCA (dTCA) was used as the internal standard. For each sample, a second replicate was analysed to verify the results.

#### 4. Results

##### 7.5-Month (Adjusted) Data

A summary of the results of the 7.5 month (adjusted) storage is presented in *Table 2*, and shown in *Figure 2*. For full details refer to the AWRI Analytical Service Report, Appendix 1.

The limit of detection on the chemical analysis (GC/MS) is 1ng/l. All of the wines sealed with ProCork membrane B had a reading below the limit (<1ng/l) and considered trace quantity.

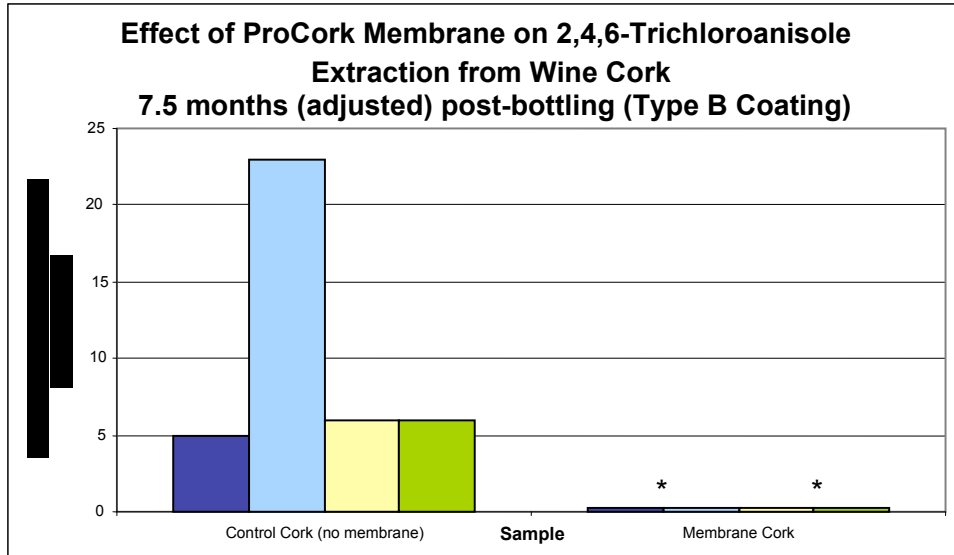
The data 7.5 months (adjusted) post-bottling indicates that the ProCork membrane resulted in a significant reduction in the transmission of 2,4,6-TCA from cork to wine.

**Table 2: TCA Extracted from Contaminated Corks in Bottles (7.5 months (adjusted) post-bottling)**

Description	2,4,6-TCA (ng/l) in wine	
	Control Corks (no membrane)	Membrane Corks
Sample I	5	trace*
Sample II	23	trace*
Sample III	6	trace*
Sample IV	6	trace*

Note: \* indicates that a positive identification has been confirmed; however quantification cannot be confidently cited below the quoted detection limit of 1ng/l.

<sup>3</sup> Modified extract from AWRI Report number FD2801 – FD2824

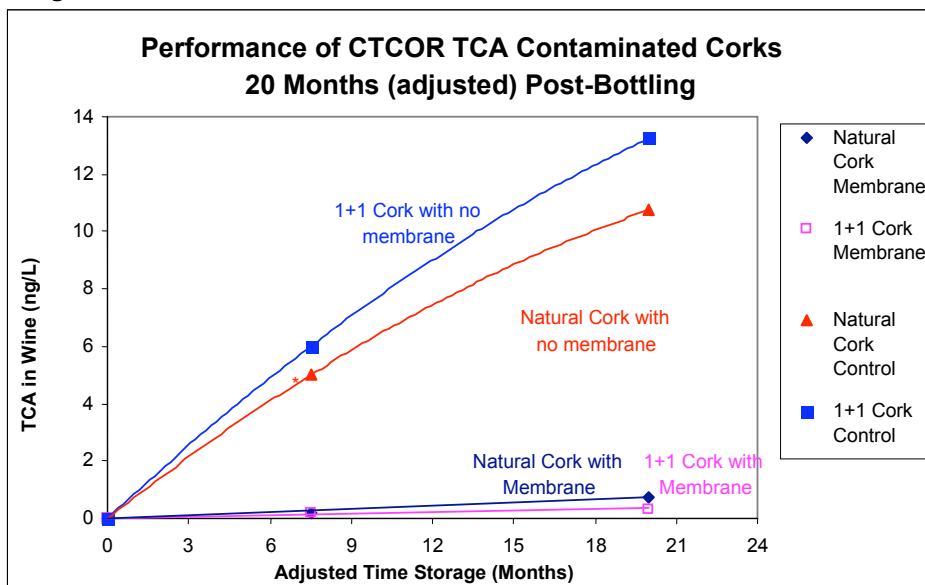


Note: \* indicates trace quantity detected

**Figure 2: Performance of CTCOR Contaminated Corks 7.5 Months (adjusted) Post Bottling**

### 20-Month (Adjusted) Data

A summary of the results from the 7.5 and 20 month storage (adjusted) is shown in Figure 3.



\* Outlier omitted. Control cork without membrane had 23ng/L TCA and was omitted from averaged result.



**Figure 3: Performance of CTCOR Contaminated Corks 20 Months (adjusted) Post Bottling**

The average amount of TCA in wine from bottles sealed with membrane corks remained below 1ng/l 20 months (adjusted) post bottling. The highest level of TCA detected in an individual sample sealed with a membrane cork was 1.5ng/l, and in 50% of the membrane cork bottles no TCA was detected. Refer to Appendix 2.

The amount of TCA in wine bottles sealed with control corks (no membrane) was 13.3ng/l and 10.8ng/l. The graph shows a decrease in TCA migration into wine sealed with membrane cork compared to control corks without membrane, indicating the use of ProCork membrane reduces TCA in the wine by up to 40 fold. (Note that TCA level 23ng/l at 7.5 months (adjusted) was regarded as an outlier. It was found with one of the control corks without membrane. It was omitted from *Figure 3.*)

A summary of the results from the 20-month (adjusted) data are shown in Table 3.

**Table 3: TCA Extraction in Wine and Cork**

<b>Sample</b>	<b>TCA in Wine (ng/l)</b>	<b>TCA in Cork (ng/l)</b>
Control Cork	10.8	15.7
Control Cork	13.3	16.9
Membrane Cork	0.0	16.0
Membrane Cork	1.5	14.7
Membrane Cork	0.0	13.6
Membrane Cork	0.7	15.2