



## Report

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**Written by:** Marine Douguet

**Approved by:** Charlotte Tournier

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## Your project

*Triangular test to evaluate the inertness of a multilayer polymer membrane designed to coat natural cork on wine bottles*

### CLIENT CONTACT

**Dr Gregor Christie**  
CEO ProCork  
Tel: +61 419 599 597  
Email: [gregor@procorktech.com](mailto:gregor@procorktech.com)

**ProCork**  
Suite 768 585 Little Collins Street Melbourne  
VIC 3000



### SENSENET CONTACT

**Marine DOUGUET**  
Sensory and Molecular Analysis Consultant  
SENSENET France  
Tél: +33 (0)2 99 55 17 95  
Mobile: + 33 (0)7 69 45 39 84  
Email: [mdouguet@sensenet.net](mailto:mdouguet@sensenet.net)

**SENSENET France**  
By ODOURNET  
3 allée de Bray  
35510 CESSON SEVIGNE  
[www.sensenet.net](http://www.sensenet.net)  
[www.odournet.com](http://www.odournet.com)

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## 1 Introduction and purpose

The Procork company developed a membrane technology to control the rate of oxygen entering the wine bottles when closed with natural cork. This membrane made of 5 different layers allows selective permeation of oxygen and thus micro-aeration of grape and oak barrel tannins while blocking bitter cork lignins and taints. The company wants to confirm the inertness of the membrane towards the wine it is intended to be in contact with.

To evaluate the food neutrality of the Procork membrane, Sensenet proposed to conduct a triangular test according to NF EN ISO 4120, to compare a synthetic wine which has been in contact with the membrane for three days and the “control” synthetic wine.

This document is the report summarizing and analysing the results related to this study.

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## 2 Services summary

Triangular test to evaluate the inertness of a multilayer polymer membrane designed to coat natural cork on wine bottles		
Experimental Plan		
Number of samples	Two samples: <ul style="list-style-type: none"><li>- 1 synthetic wine which has been in contact with the membrane</li><li>- 1 control synthetic wine</li></ul>	
Parameters	The two samples have been compared using sensory analysis triangular test.	
Sampling		
Protocol	The synthetic wine has been left in contact with the membrane for 3 days. The size of the membrane has been adapted to maintain the real use contact ratio.	
Analyses		
Sensory analyses		
Parameters	Methodologies	Details
Triangular test	According to NF EN ISO 4120	15 naive panellists (duplicate)

## 3 Experimental

### 3.1 Planning

The below planning has been followed for sample preparation and sensory analysis testings :

	Monday 13 <sup>th</sup> May 2019	Tuesday 14 <sup>th</sup> May 2019	Wednesday 15 <sup>th</sup> May 2019	Thursday 16 <sup>th</sup> May 2019
Synthetic wine solution preparation				
Sample storage at room temperature	(from 12 am)			(to 9 am)
Sensory analysis				

### 3.2 Protocol for sample preparation

One master batch of three liters of synthetic wine solution has been prepared on the 13<sup>th</sup> of May 2019 according to the recipe detailed in Annex 1.

The solution was then poured into 2 different glass jars (1.5 liter in each).

To mimic the standard in use ratio (3.14 cm<sup>2</sup> of membrane in contact with 750 mL of wine), a piece of Procork membrane of 1.8 cm x 1.8 cm (6,48 cm<sup>2</sup> in total) was introduced in one of the jar containing 1.5 L of solution.

Glass jars were left at room temperature (18-20°C) during 3 days.



### 3.3 Triangular test principle

This simple sensory analysis test allow detection of presence or absence of sensorial differences between two products. The principle of the test is as follows :

- Objective is to compare two different products named A and B.
- During a triangular test, each panellist is simultaneously presented with three samples of which two are equal and one is different (for example AAB or ABB).
- The panellists evaluate blindly the three samples and must state which sample is different,
- By counting the number of correct choice results and according to ISO 4120 reference tables, we can identify whether or not a detectable difference exists between two samples.
- The method is statistically more efficient than the duo-trio test but has limited use with products that have a strong and/or lingering flavor.

In the present study the objective is to compare the two following samples :

- A synthetic wine which has not been in contact with Procork membrane (W/O)
- A synthetic wine which has been in contact with Procork membrane (W/)

### 3.4 Triangular test protocol

The sensory analyses have been performed on the 16<sup>th</sup> May 2019 within our Cesson-Sévigné, France laboratory. The two sensory analysis sessions have been conducted by our lab technician, Oréane Varasse.

The testings have been done with 15 naïve panellists who evaluated the samples in duplicate, in accordance with ISO 4120 which recommends a minimum of 30 answers. Panellists were instructed not to drink coffee or eat any mentholated product, smoke, eat or wear fragrances or lipstick during the 30 minutes preceeding the testing.

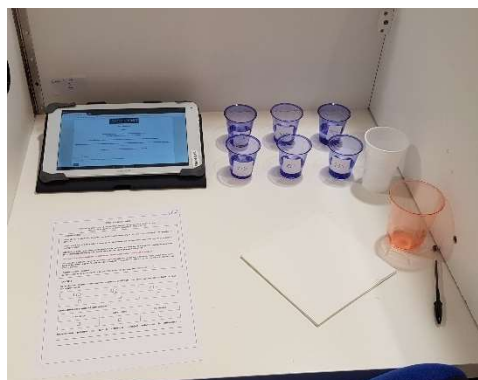
The 2 synthetic wines to be compared have been poured into standardized blue plastic glasses containing each 3 cc of solution just before the sessions. Each sample was coded using a random three digit code eliminating presentation bias. The samples were randomly presented according to a balanced experimental test design to eliminate any placement order bias.

Two series of three samples (one different from the others) were presented to the 15 panellists. The product different from the others (W/O or W/) and the three digit code changed from one serie to another to avoid any bias.

The panellists have been instructed to evaluate both odour and taste of each sample. They were told to smell and palate the solution. They could not swallow it but expectorate. Between two samples, mineral water was used for palate cleansing.

Panellists answered to the question “which sample is different from the other?” for both repeats using a digital tablet equipped with EyeQuestion® sensory analysis software. The panel members were forced to guess and choose when they could not identify the different sample. On a second page of the questionnaire they were asked to tick the level of confidence corresponding to their choice (guess, inkling, certain) and to detail any criteria they used to differentiate the samples.

EyeQuestion® sensory analysis software has been used for test design generation, test questionnaire definition and data collection.



## 4 Results and discussion

The detailed results are presented in Annex 3.

The table below synthetises the answers given by the panellists.

The results are coded as follows :

“+” when the panellist correctly identified the different sample among the three,

“0” when the panellist did not correctly identified the different sample among the three.

SAMPLES CODING :	
Synthetic wine WITHOUT Procork membrane (W/O)	214, 392, 415, 175
Synthetic wine WITH Procork membrane (W/)	968, 871, 504, 723

PANELIST CODE	REPEAT 1						REPEAT 2					
	SAMPLE	SAMPLE	SAMPLE	DIFFERENT SAMPLE	CHOICE RESULT	LEVEL OF CONFIDENCE	SAMPLE	SAMPLE	SAMPLE	DIFFERENT SAMPLE	CHOICE RESULT	LEVEL OF CONFIDENCE
NLG	214	968	871	W/O	0	inkling	392	504	415	W/	+	inkling
KLE	175	871	214	W/	+	inkling	504	415	723	W/O	0	guess
RCE	214	392	871	W/	0	inkling	968	504	175	W/O	+	inkling
CGU	723	415	392	W/	0	inkling	968	214	175	W/	0	guess
CPI	871	415	723	W/O	+	inkling	214	392	504	W/	0	inkling
CIO	968	871	175	W/O	0	guess	415	723	504	W/O	0	guess
LOI	968	214	871	W/O	+	inkling	723	392	415	W/	0	inkling
GPE	214	504	968	W/O	+	certain	415	175	723	W/	+	guess
CPE	214	392	968	W/	0	inkling	723	415	871	W/O	0	certain
SHE	968	175	214	W/	0	inkling	392	723	415	W/	0	inkling
PCO	504	968	175	W/O	0	inkling	214	871	723	W/O	0	guess
MAP	392	968	415	W/	0	inkling	504	723	175	W/O	+	inkling
LLE	392	871	968	W/O	+	certain	175	504	723	W/O	0	inkling
EWA	214	871	392	W/	+	guess	175	415	968	W/	+	inkling
OVA	392	214	723	W/	+	guess	415	504	175	W/	0	guess

Number of correct choice results "+" : 12/30

Number of correct answers after combination with confidence level : 2/30

The “different sample” has been correctly identified during 12 of the 30 sensory evaluations.

As per ISO 4120 reference table (refer to Annex 2), a maximum number of 13 correct answers can be given by the panel to rest assured at 95% that at maximum 40% of the subjects can distinguish the two samples tested.

In a triangle test the probability to get the correct answer by guessing is 1/3. This is why, the panel members were also asked to mention if they were certain, having an inkling or guessing their answer.

Combining the “choice result” with this “level of confidence” gives an additional level of interpretation of the results. For only 2 of the 12 correct answers the panellists mentioned they were certain of having the right answer. For 3 of the 12 correct answers the panellists guessed the answer. What is more only two panellists correctly identified the “different sample” for both repeat 1 and repeat 2 but both of them guessed the answer during one the repeat. In other terms, no panellists identified the correct answer for both repeats and with certainty.

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## 5 Conclusion

Following the triangular test conducted, we can conclude at 95% that at maximum 40% of the subjects can distinguish the two samples tested. With those statistical risk levels, the sensory study conducted confirmed the inertness of ProCork membrane towards synthetic wine.

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## Annex 1: Synthetic wine recipe

To avoid all the complications linked to flavours migrating from the cork and to wine oxidation it has been decided to use a synthetic wine.

The recipe used to prepare the synthetic wine solution is the following one :

Alcohol	12,5%
Tartaric Acid	1 g/L
Sucrose	1 g/L
Mineral water	qsp

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## Annex 2: Maximum number of correct answers needed to conclude that two samples are similar during a triangular test (based on ISO 4120)

ISO 4120:2004(F)

Tableau A.2 — Nombre maximal de réponses correctes nécessaire pour conclure que deux échantillons sont similaires, sur la base d'un essai triangulaire

n	β	p <sub>0</sub>					n	β	p <sub>0</sub>				
		10 %	20 %	30 %	40 %	50 %			10 %	20 %	30 %	40 %	50 %
18	0,001	0	1	2	3	5	66	0,001	14	18	22	26	31
	0,01	2	3	4	5	8		0,01	16	20	25	29	34
	0,05	3	4	5	6	8		0,05	19	23	28	32	37
	0,10	4	5	6	7	8		0,10	20	25	29	33	38
	0,20	4	6	7	8	9		0,20	22	26	31	35	40
24	0,001	2	3	4	6	8	72	0,001	15	20	24	29	34
	0,01	3	5	6	8	9		0,01	18	23	28	32	38
	0,05	5	6	8	9	11		0,05	21	26	30	35	40
	0,10	6	7	9	10	12		0,10	22	27	32	37	42
	0,20	7	8	10	11	13		0,20	24	29	34	39	44
30	0,001	3	5	7	9	11	78	0,001	17	22	27	32	38
	0,01	5	7	9	11	13		0,01	20	25	30	36	41
	0,05	7	9	11	13	15		0,05	23	28	33	39	44
	0,10	8	10	11	14	16		0,10	25	30	35	40	46
	0,20	9	11	13	15	17		0,20	27	32	37	42	48
36	0,001	5	7	9	11	14	84	0,001	19	24	30	35	41
	0,01	7	9	11	14	16		0,01	22	28	33	39	45
	0,05	9	11	13	16	18		0,05	25	31	36	42	48
	0,10	10	12	14	17	19		0,10	27	32	38	44	49
	0,20	11	13	16	18	21		0,20	29	34	40	46	51
42	0,001	6	9	11	14	17	90	0,001	21	27	32	38	45
	0,01	9	11	14	17	20		0,01	24	30	36	42	48
	0,05	11	13	16	19	22		0,05	27	33	39	45	52
	0,10	12	14	17	20	23		0,10	29	35	41	47	53
	0,20	13	16	19	22	24		0,20	31	37	43	49	55
48	0,001	8	11	14	17	21	96	0,001	23	29	35	42	48
	0,01	11	13	17	20	23		0,01	26	33	39	45	52
	0,05	13	16	19	22	26		0,05	30	36	42	49	55
	0,10	14	17	20	23	27		0,10	31	38	44	50	57
	0,20	15	18	22	25	28		0,20	33	40	46	53	59
54	0,001	10	13	17	20	24	102	0,001	25	31	38	45	52
	0,01	12	16	19	23	27		0,01	28	35	42	49	56
	0,05	15	18	22	25	29		0,05	32	38	45	52	59
	0,10	16	20	23	27	31		0,10	33	40	47	54	61
	0,20	18	21	25	28	32		0,20	36	42	49	56	63
60	0,001	12	15	19	23	27	108	0,001	27	34	41	48	55
	0,01	14	18	22	26	30		0,01	31	37	45	52	59
	0,05	17	21	25	29	33		0,05	34	41	48	55	63
	0,10	18	22	26	30	34		0,10	36	43	50	57	65
	0,20	20	24	28	32	36		0,20	38	45	52	60	67

NOTE 1 Les valeurs du tableau sont exactes car elles sont fondées sur la distribution binomiale. Pour les valeurs de n non incluses dans le tableau, calculer la limite supérieure de confiance 100 (1 - β) % pour p<sub>0</sub> en utilisant l'approximation normale de la loi binomiale, comme suit:

$$\left[ 1,5 \left( \frac{x}{n} - 0,5 \right) + 1,5 z_{\beta} \sqrt{\frac{x}{n} - \frac{x^2}{n^2}} \right] / n^3$$

où x est le nombre de réponses correctes, n le nombre de sujets et z<sub>β</sub> varie comme suit:

0,84 pour β = 0,20; 1,28 pour β = 0,10; 1,64 pour β = 0,05; 2,33 pour β = 0,01; 3,09 pour β = 0,001.

Si la valeur calculée est inférieure à la limite sélectionnée pour p<sub>0</sub>, déclarer les échantillons similaires au niveau de signification β.

NOTE 2 Les valeurs de n < 30 ne sont généralement pas recommandées pour les essais triangulaires de similitude.

NOTE 3 Adapté de la Référence [11].

## Annex 3: Detailed results of the triangular test

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<b>SAMPLES CODING :</b>	
Synthetic wine WITHOUT Procork membrane (W/O)	214, 392, 415, 175
Synthetic wine WITH Procork membrane (W/I)	968, 871, 504, 723
<b>ANSWER CODING :</b>	
+: correct choice result	
0: incorrect choice result	

REPEAT 1														REPEAT 2													
PANELIST CODE	SAMPLE		SAMPLE		DIFFERENT SAMPLE		CHOICE RESULT	LEVEL OF CONFIDENCE	Criteria mentioned by the panelist to explain the difference	SAMPLE		SAMPLE		DIFFERENT SAMPLE		CHOICE RESULT	LEVEL OF CONFIDENCE	Criteria mentioned by the panelist to explain the difference									
	214	968	871	W/O	0	inkling				odour less intense	392	504	415	W/	+				inkling	sourer							
NLG	175	871	214	W/	+	inkling	stronger taste	504	415	723	W/O	0	guess	more acid													
KLE	214	392	871	W/	0	inkling	less strong odour and taste	968	504	175	W/O	+	inkling	stronger taste and odour, bitter													
RCE	723	415	392	W/	0	inkling	more salty	968	214	175	W/	0	guess	more pungent													
CGU	871	415	723	W/O	+	inkling	stronger, more pungent	214	392	504	W/	0	inkling	less strong, less pungent													
CPI	968	871	175	W/O	0	guess	sourer	415	723	504	W/O	0	guess	N/A													
CIO	968	214	871	W/O	+	inkling	different taste, aromas and acidity	723	392	415	W/	0	inkling	different taste, aromas, alcohol content and													
LOI	214	504	968	W/O	+	certain	no difference in smell, acidity	415	175	723	W/	+	guess	None													
GPE	214	392	968	W/	0	inkling	different smell and aftertaste	723	415	871	W/O	0	certain	stronger odour, more acidity													
CPE	968	175	214	W/	0	inkling	more acid and sour	392	723	415	W/	0	inkling	more acid and sour													
SHE	504	968	175	W/O	0	inkling	more alcohol	214	871	723	W/O	0	guess	less alcohol and sugar													
PCO	392	968	415	W/	0	inkling	stronger odour	504	723	175	W/O	+	inkling	more pungent													
MAP	392	871	968	W/O	+	certain	more acid, pungent	175	504	723	W/O	0	inkling	slightly less acid													
LLE	214	871	392	W/	+	guess	more sugar, no difference in smell	175	415	968	W/	+	inkling	less alcohol													
EWA	392	214	723	W/	+	guess	more alcohol	415	504	175	W/	0	guess	more alcohol													
OVA																											

Number of correct choice results "++": 12/30

Number of correct answers after combination with confidence level : 2/30