



# Cork closures

## Impact on the quality of bottled wines

**Paulo Lopes**

Enologist PhD (Faculté d'Œnologie de Bordeaux)  
WineMBA (BEM, UC Davis, UniSA)

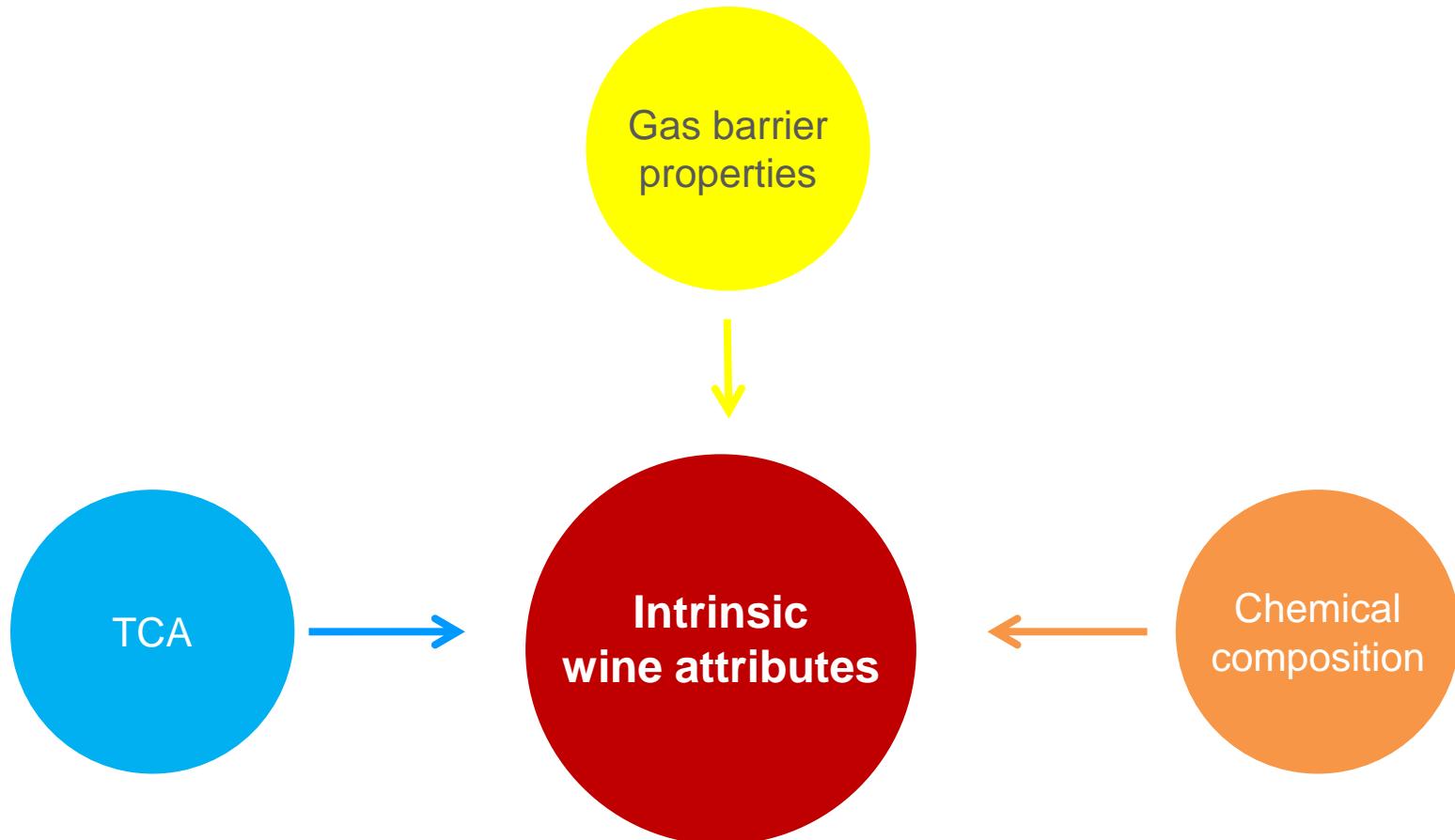
Brock University, 12 November 2012

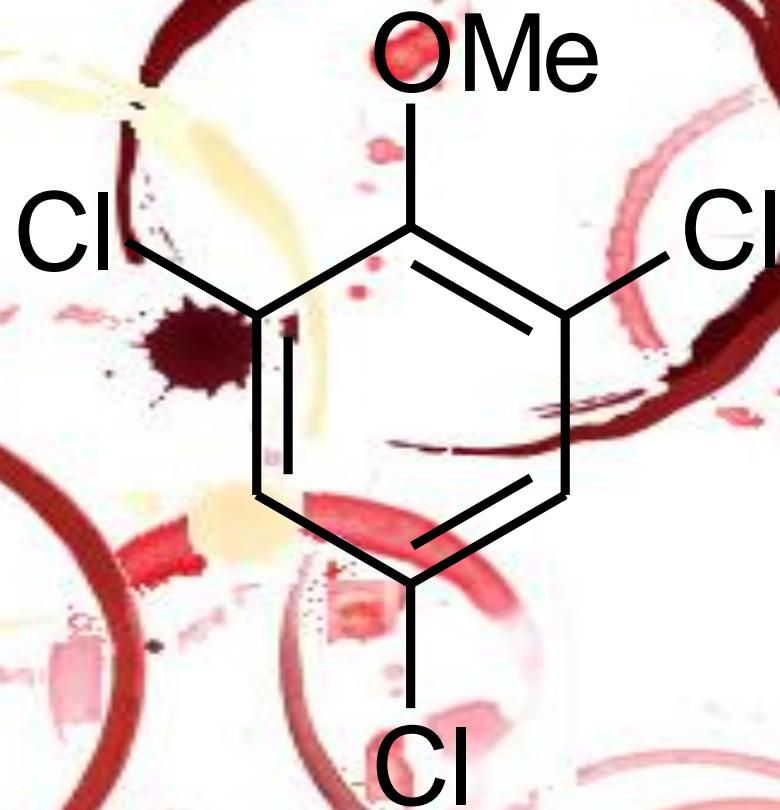


# Impact of closures on wine quality



# How corks can influence wine quality after bottling

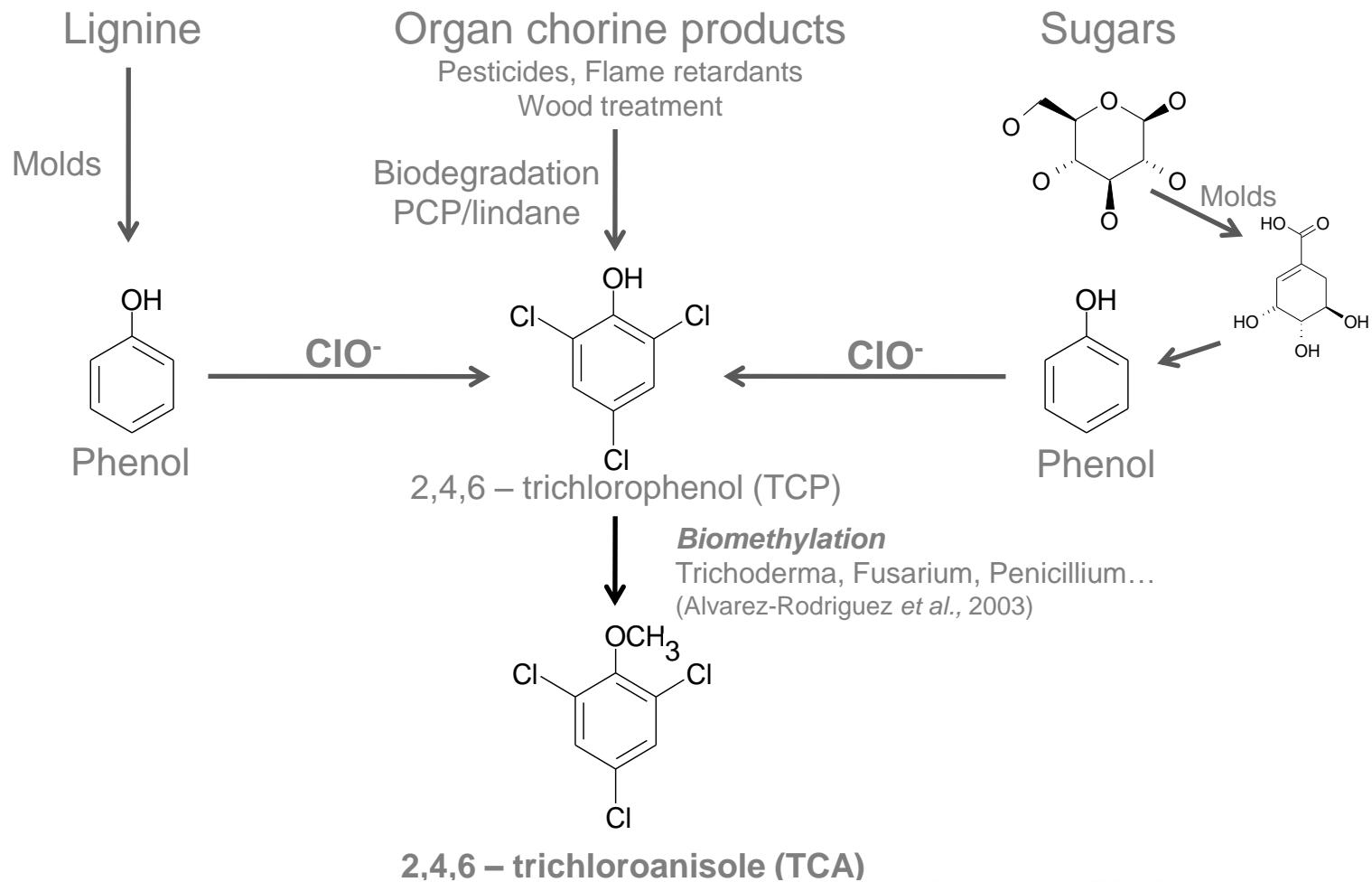




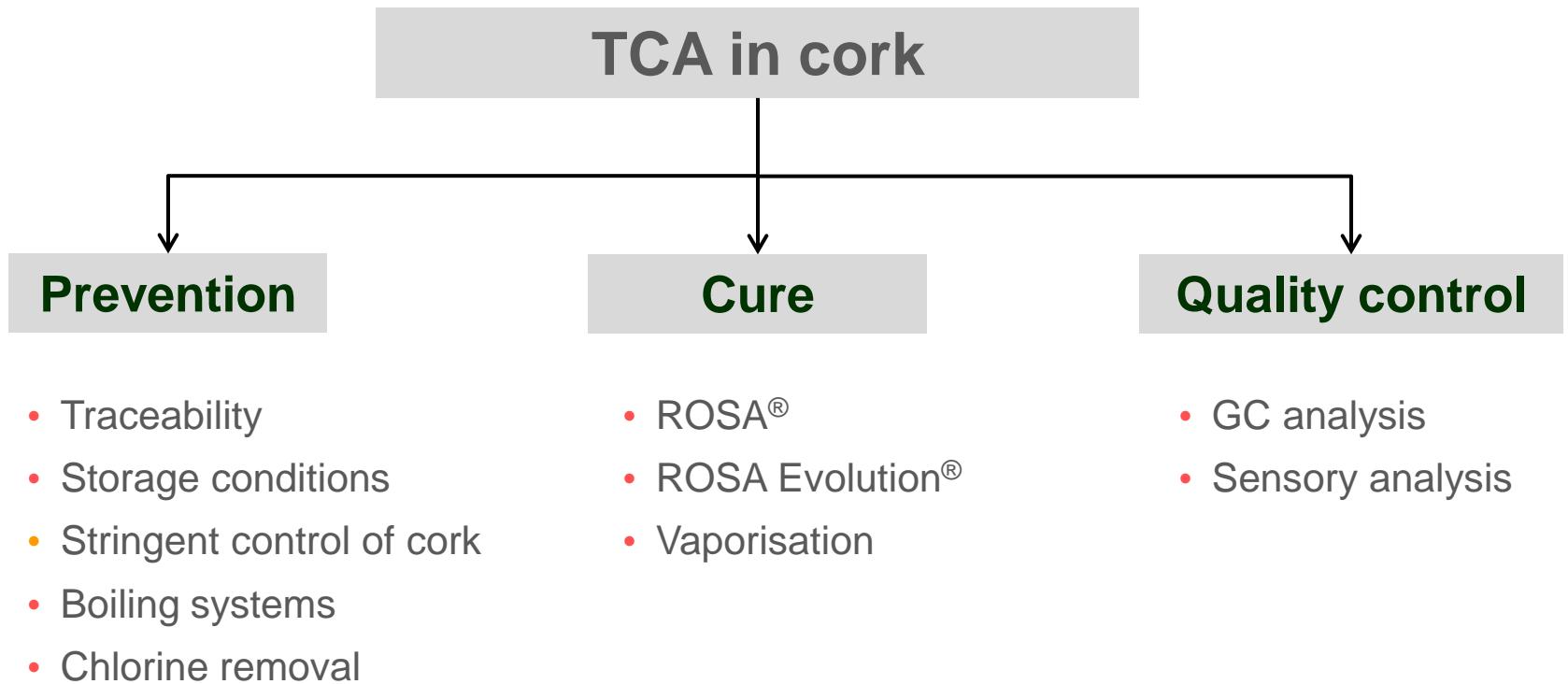
TCA

Current situation and perspectives

# Main mechanism of TCA formation



# Amorim's strategy anti-TCA



# Prevention

## Prevent TCA formation during cork stopper manufacture



Cork lots traceability



Removal of “calços”(10 cm)

Exclusion of yellow stain cork planks



Boiling system that reduces cork storage



Chlorine exclusion

# Descontamination

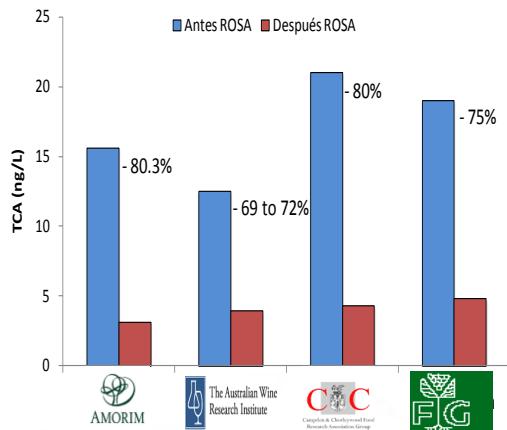
## Steam distillation treatments



**ROSA®**

Patent nº PT103910

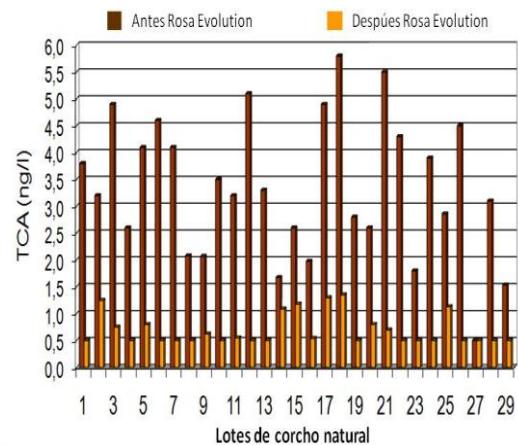
**Cork granules**  
-80 to 90% of TCA



**ROSA Evolution®**

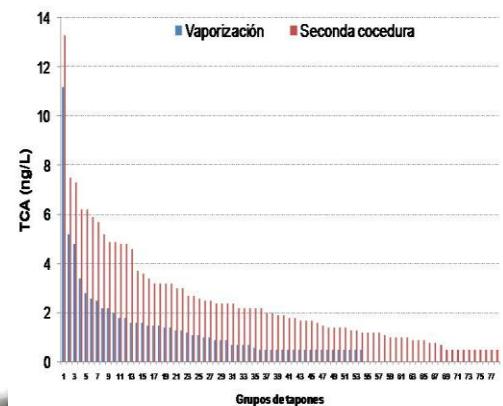
Patent nº PT103910

**Natural cork stoppers**  
-80% of TCA

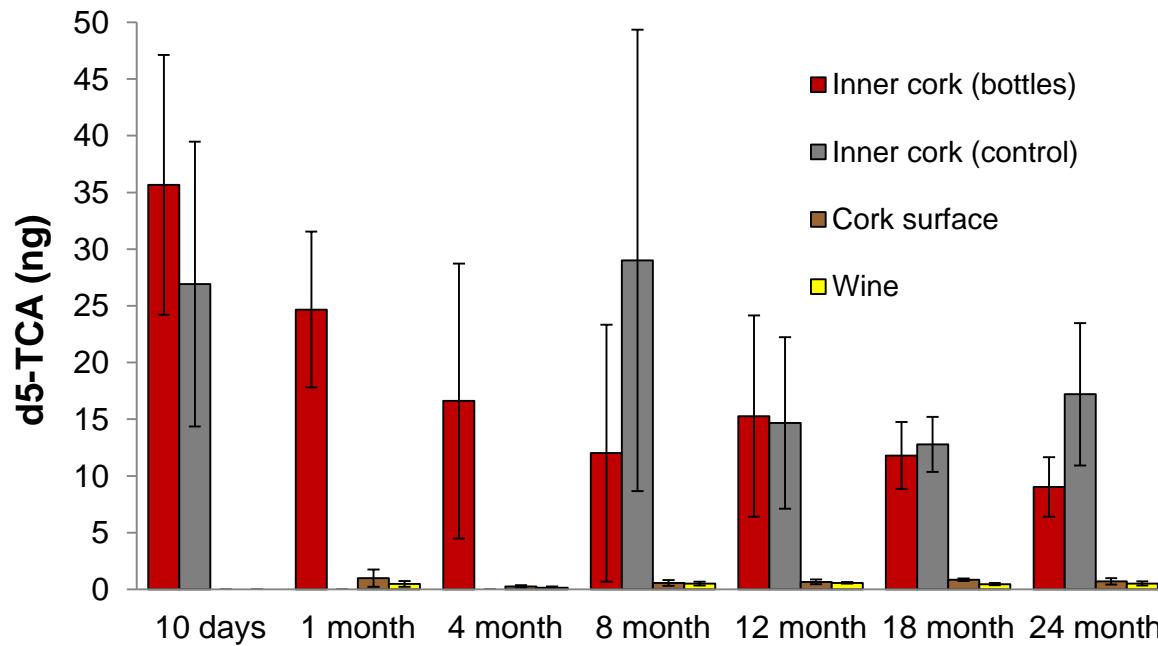
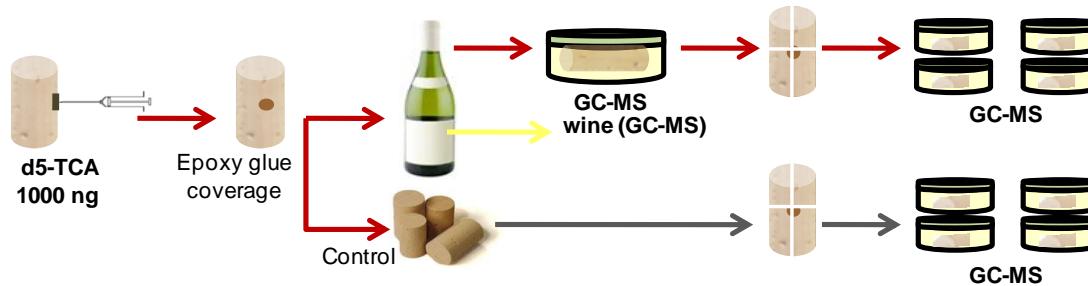


**Vaporization**

**Cork planks**  
-40% of TCA



# Mechanism of TCA migration from cork into wine



Wine contamination occurs by direct contact with cork contaminated surfaces

# Quality control

## GC and sensory analysis



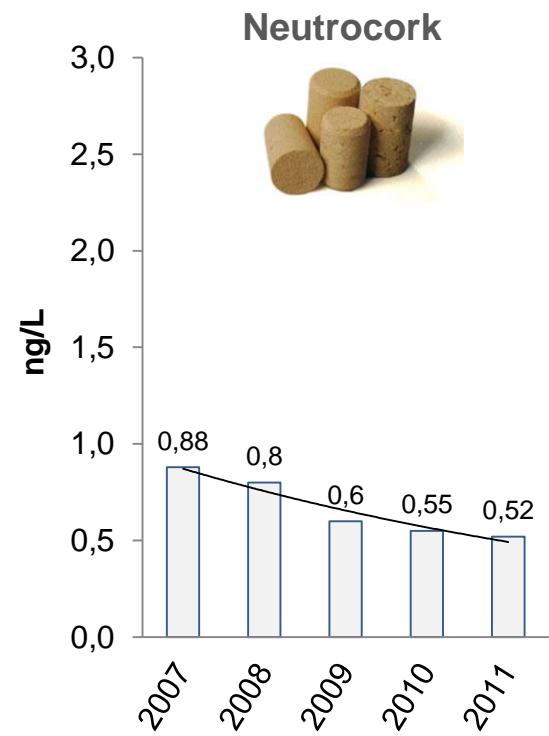
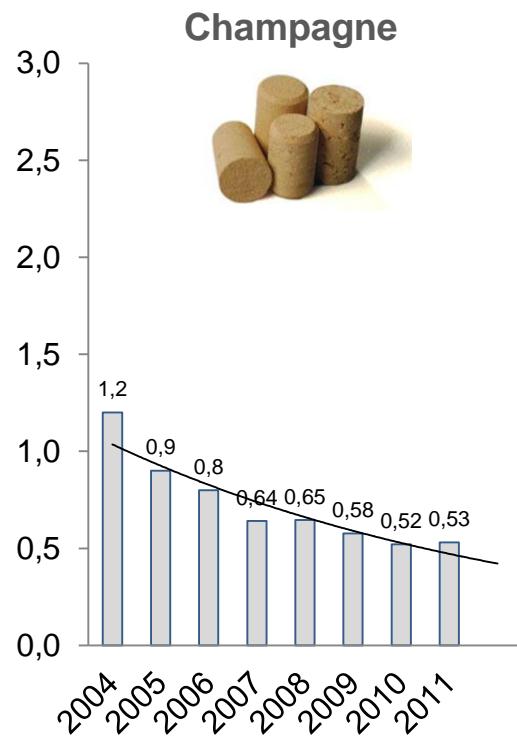
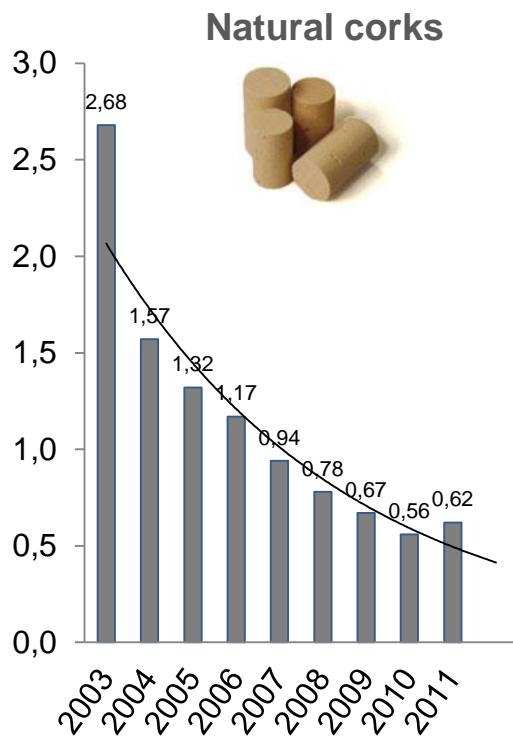
**GC/MS/ECD-SPME** (LD = 0,3 ng/L et LQ = 0,5 ng/L)

13 GCs split by the raw material, cork and R&D\* Units

> 15000 samples per month\*

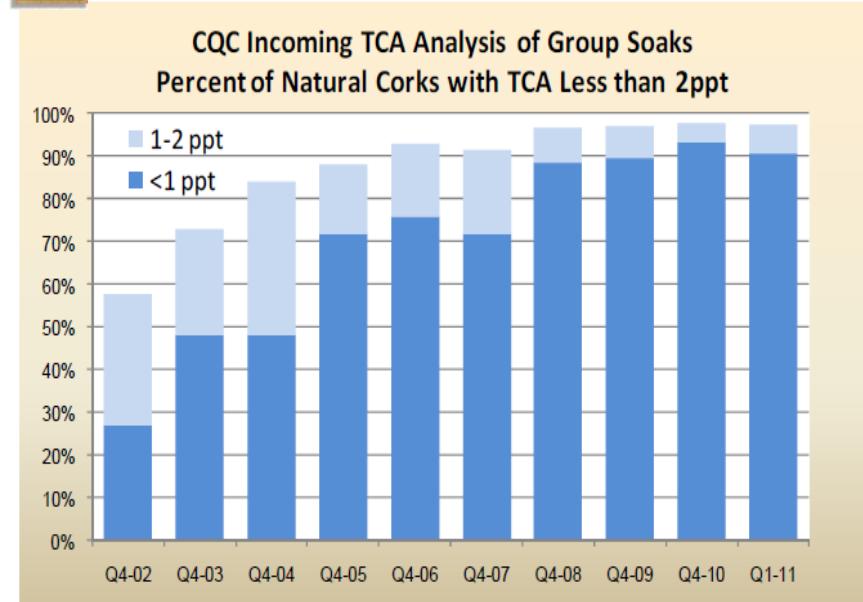
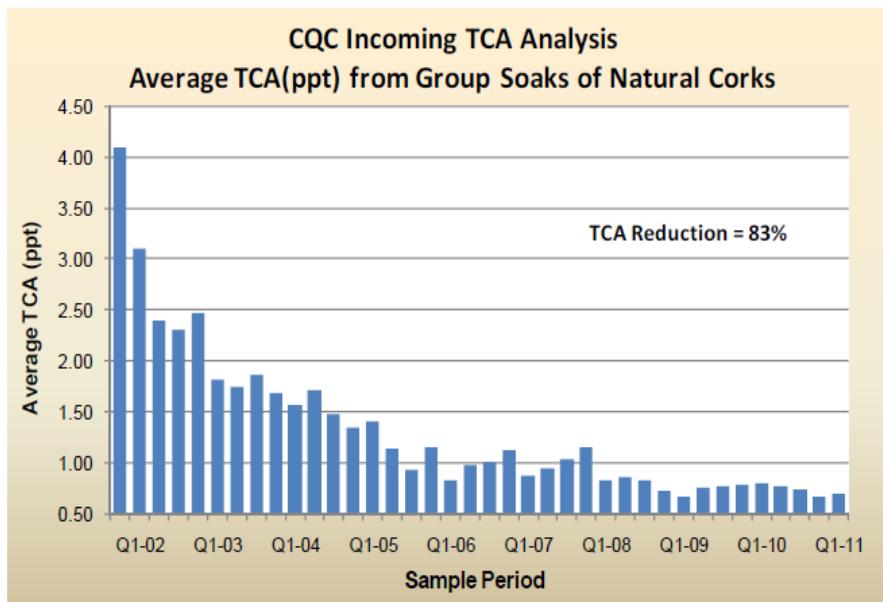
# Strategy results

## Internal



# Strategy results

## External



### The CQC cork sampling program is rigorous.

For a typical lot of 100,000 corks CQC guidelines require a minimum sample of 250 corks taken from a selection of at least five separate bales. These corks are placed in 50-cork wine soaks for 24 hours to extract releasable TCA. Resulting soaks are analyzed at ETS Laboratories using a method that reports TCA at concentrations as low as 1 part per trillion. If one of the five soaks indicates TCA as high as 1.5ppt – the entire cork lot is flagged and withheld from inventory.



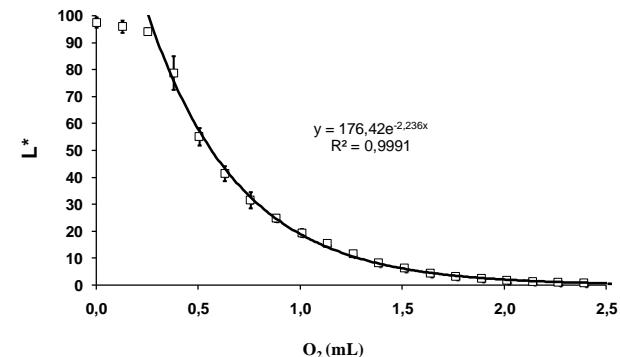
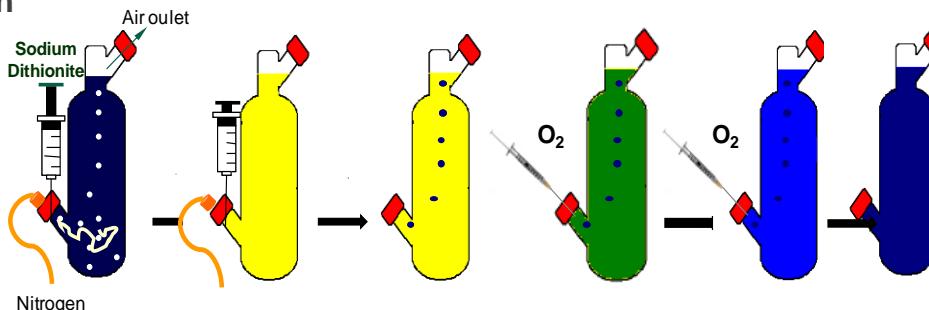
## Closures barrier properties

Impact on wine quality after bottling

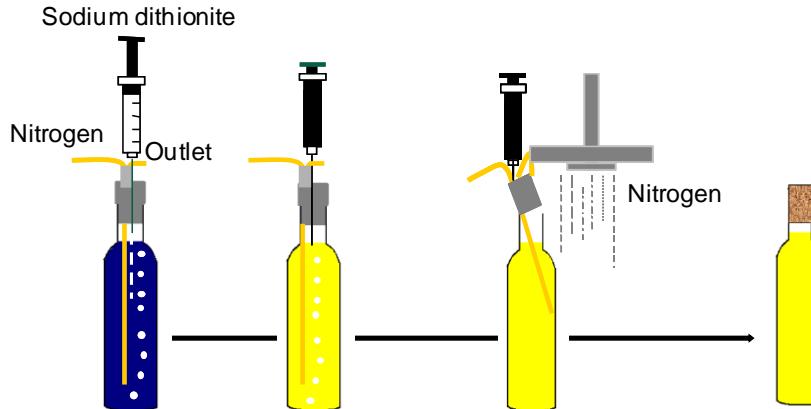
# Measurement of O<sub>2</sub> transfer through closures

Colorimetry: Indicator of oxidation-reduction (Indigo carmine)

## Calibration

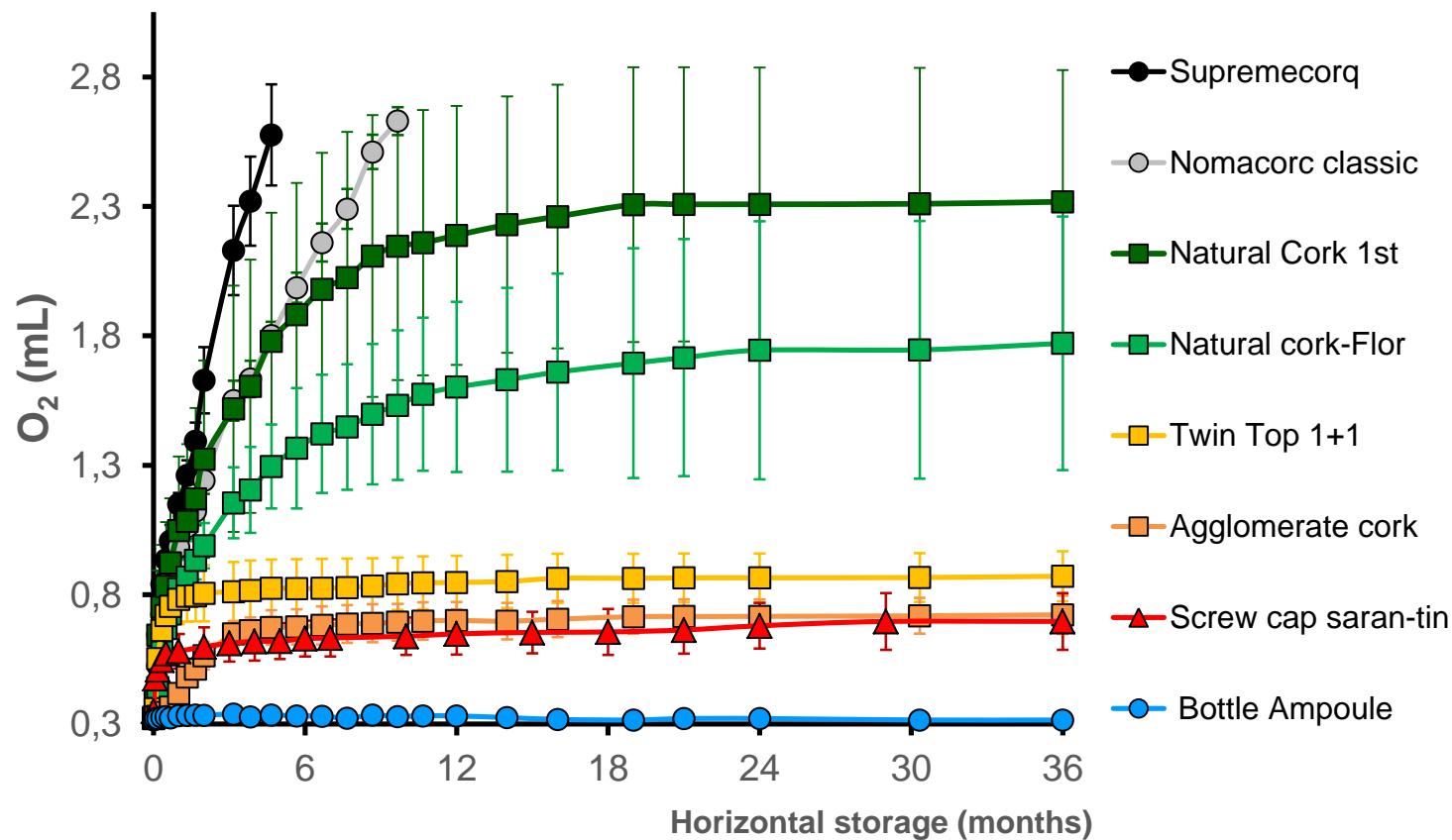


## Bottling & O<sub>2</sub> measurement



LOPES, P.; SAUCIER, C.; GLORIES, Y. Nondestructive colorimetric method to determine the oxygen diffusion rate through closures used in winemaking. *J. Agric. Food Chem.* **2005**, 53, 6967-6973.

# Kinetics of oxygen ingress through different closures

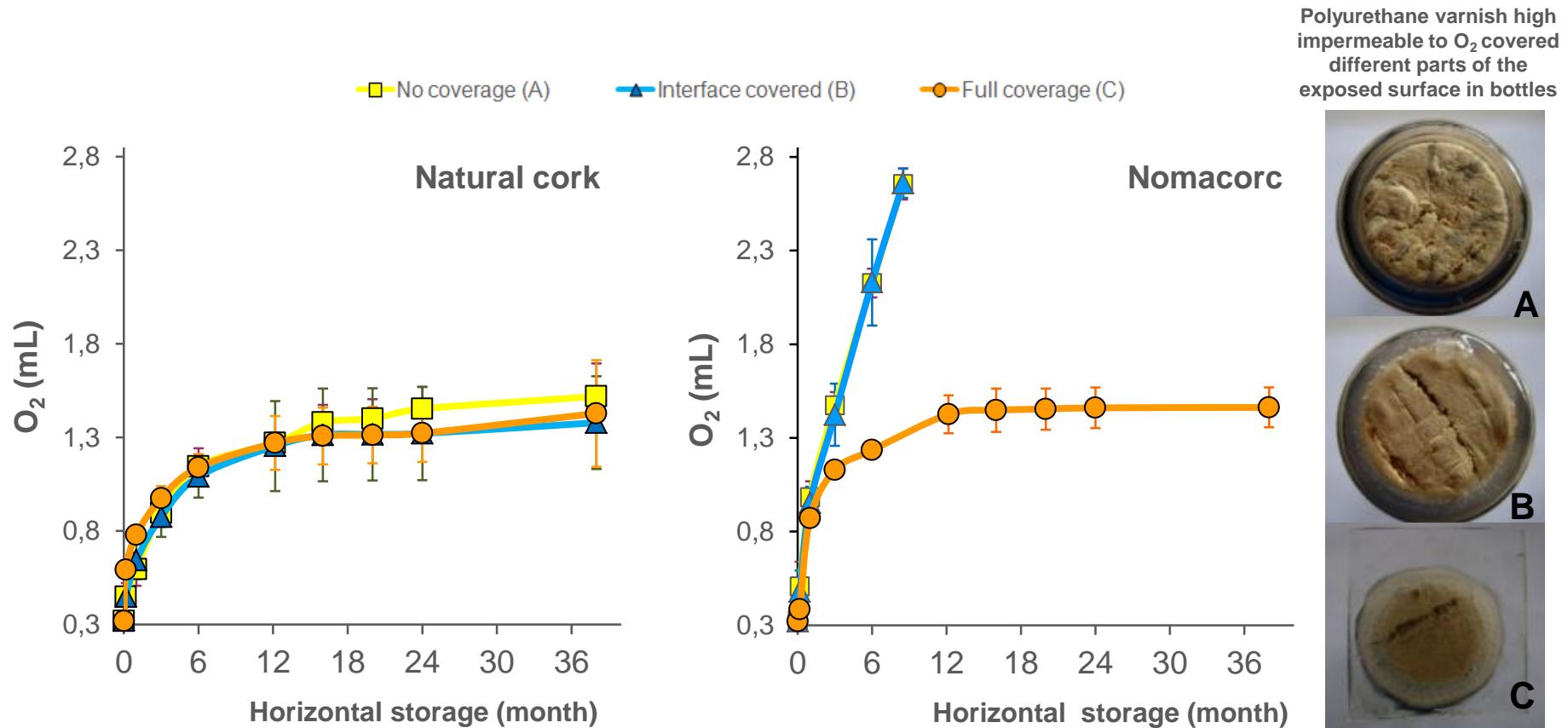


Oxygen ingresses through closures independently of storage position

LOPES, P.; SAUCIER, C.; TEISSEDRE, P.L.; GLORIES, Y. Impact of storage position on oxygen ingress through different closures into wine bottles. *J. Agric. Food Chem.* 2006, 54, 6741-6746



# Main routes by which O<sub>2</sub> enters into bottles



Cork stoppers releases O<sub>2</sub> into wine ... while synthetic are permeable to atm. O<sub>2</sub>

LOPES, P.; SAUCIER, C.; TEISSEDRE, P.L., GLORIES, Y. Main routes of oxygen ingress through different closures into wine bottles. *J. Agric. Food Chem.* **2007**, 55, 5167-5170.



# Total O<sub>2</sub> into wine bottle after bottling

- 1) Dissolved oxygen in wine
- 2) Gaseous oxygen in the bottle headspace
- 3) Oxygen within the closure
- 4) Oxygen that enters throughout the closure

Wine exposition to oxygen before and during bottling are very important, their effect (cumulative) can be only observed during post-bottling

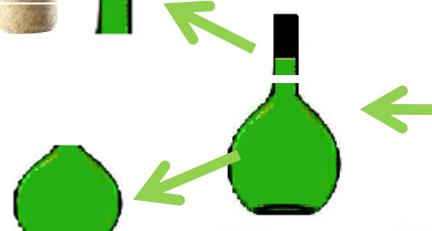


# Sealing effectiveness of closures to volatile compounds

## Storage under contaminated environment

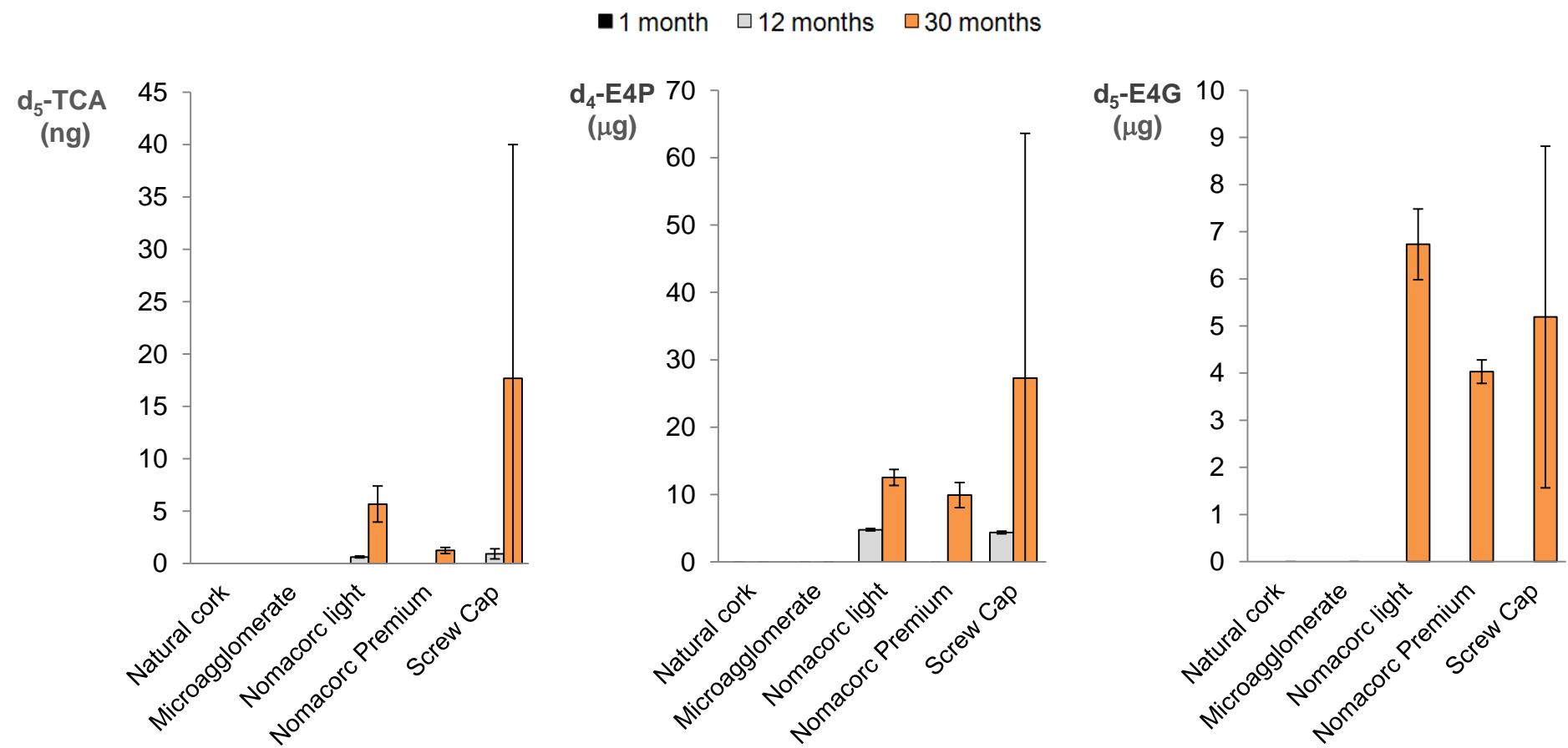


$d_5\text{-TCA}$ : 1,75 µg/L<sub>air</sub>  
 $d_4\text{-E4P}$ : 1,73 mg/L<sub>air</sub>  
 $d_4\text{-E4G}$ : 0,15 mg/L<sub>air</sub>



- Microagglomerate cork
- Natural cork
- Nomacorc light
- Nomacorc premium
- Screw cap saranex

# Sealing effectiveness of closures to volatile compounds



Cork is an effective barrier!!! TCA does not migrate through cork after bottling

PEREIRA, B.; LOPES, P.; MARQUES, J.; PIMENTA, M.; ALVES, C.; ROSEIRA, I.; MENDES, A.; CABRAL, M. Sealing effectiveness of different type of closures to volatile phenols and hanisoles. *American J. Enology and Viticulture*. 2011, submitted.

# Impact of bottling and closure OTR on the chemical & sensory properties of a Sauvignon blanc

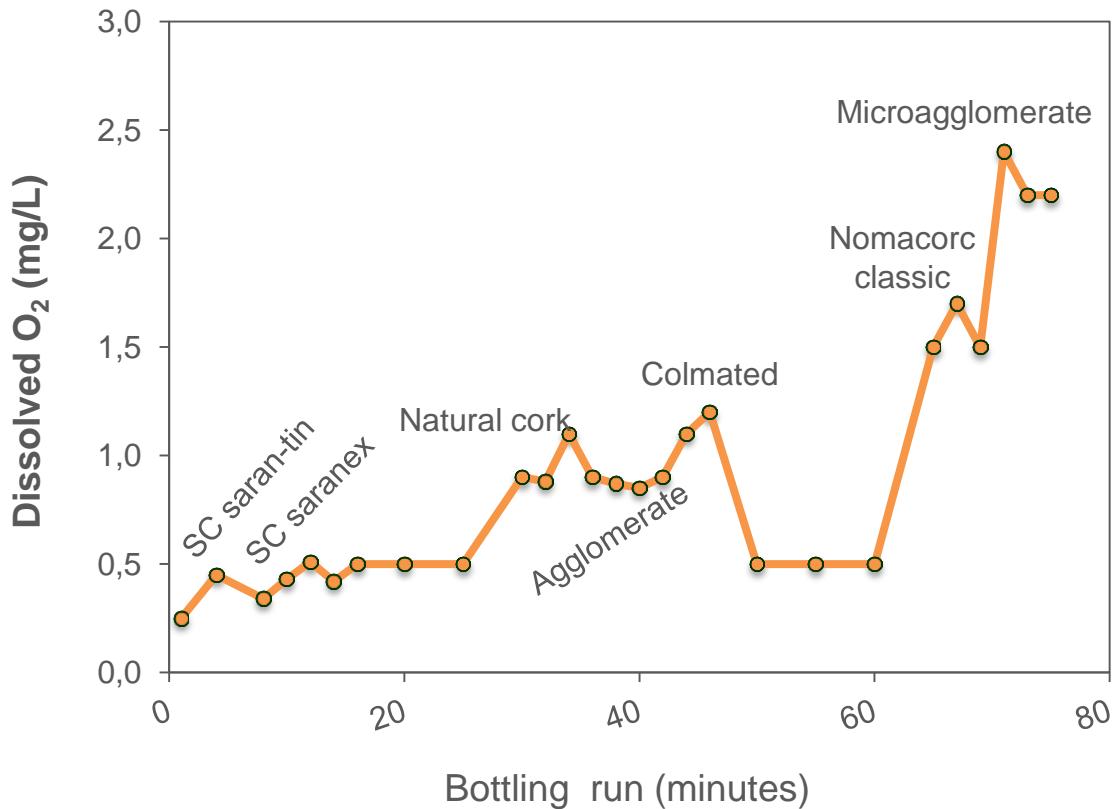
Vin	100% Sauvignon blanc; Côte du Duras 2005								
Composition	TAV 12,2%	pH 3,25	Total acidity 4,27 g/l H <sub>2</sub> SO <sub>4</sub>	Malic acid 3,02 g/L	Sugars (fructose + glucose) 0,40 g/L	Iron 3,5 mg/L	Total SO <sub>2</sub> 132 mg/L	Ascorbic acid 79 mg/L	
			Volatile acidity 0,29 g/l H <sub>2</sub> SO <sub>4</sub>	Tartaric acid 1,40 g/L		Copper 0,4 mg/L	Free SO <sub>2</sub> 41 mg/L		
Bottles	Bordelaise Tradition CETIE						Bordelaise BVS 30H60		Ampole
Closures	Natural cork	Colmated cork	Agglomerate	Micro agglomerate	Nomacorc classic	SC saran-tin	SC saranex		
Analyses	Free SO <sub>2</sub>	Total SO <sub>2</sub>	Ascorbic acid	OD 420 nm	L*a*b*	Oxidation (Sotolon)	Reduction (H <sub>2</sub> S)	Sensory	

Analyses after 2, 12 and 24 months



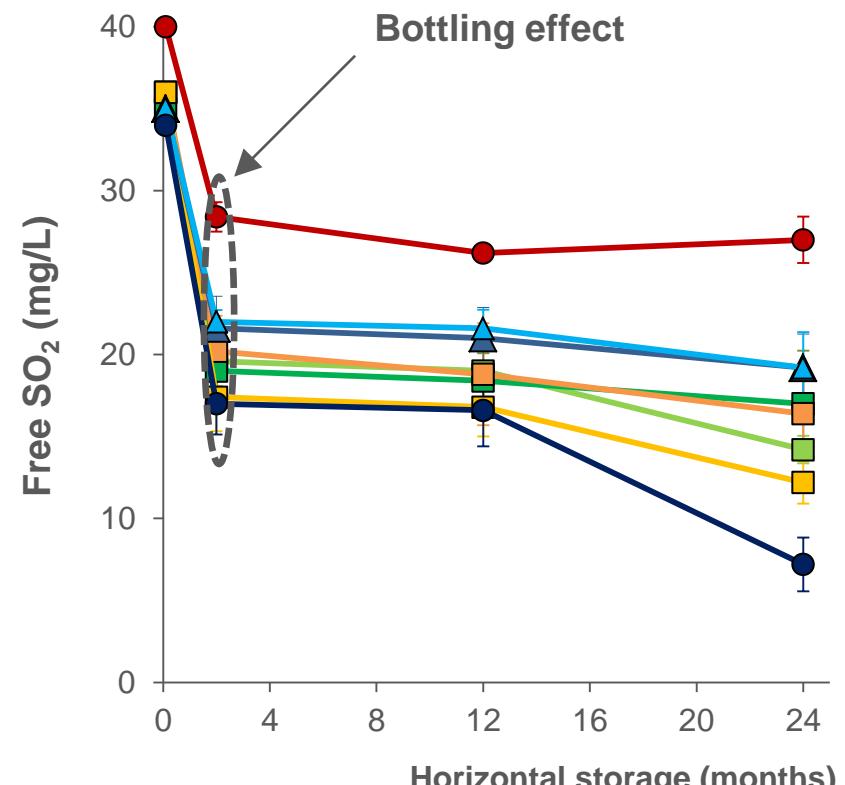
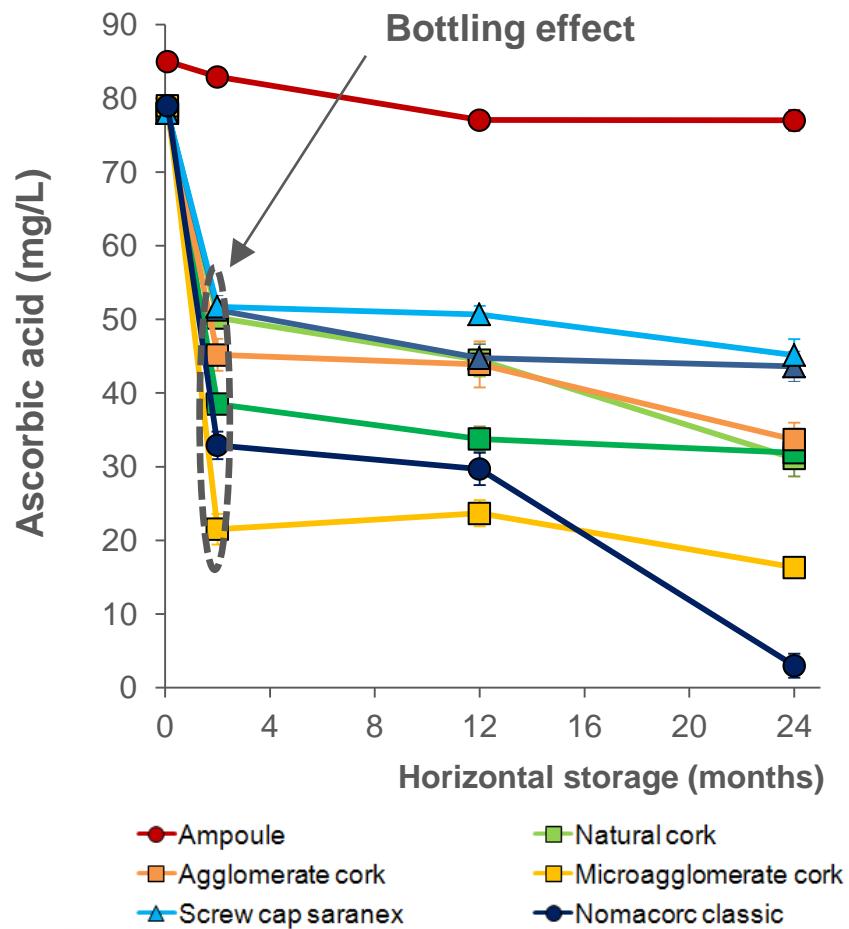
# Impact of bottling and closure OTR on the chemical & sensory properties of a Sauvignon blanc

**Significant variation on O<sub>2</sub> dissolved during bottling**



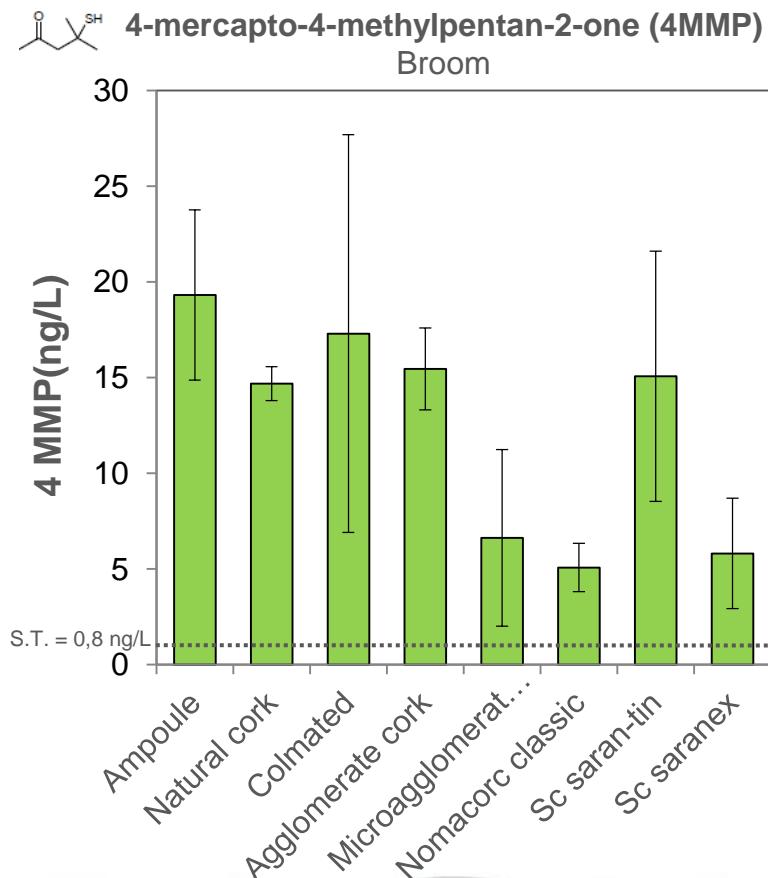
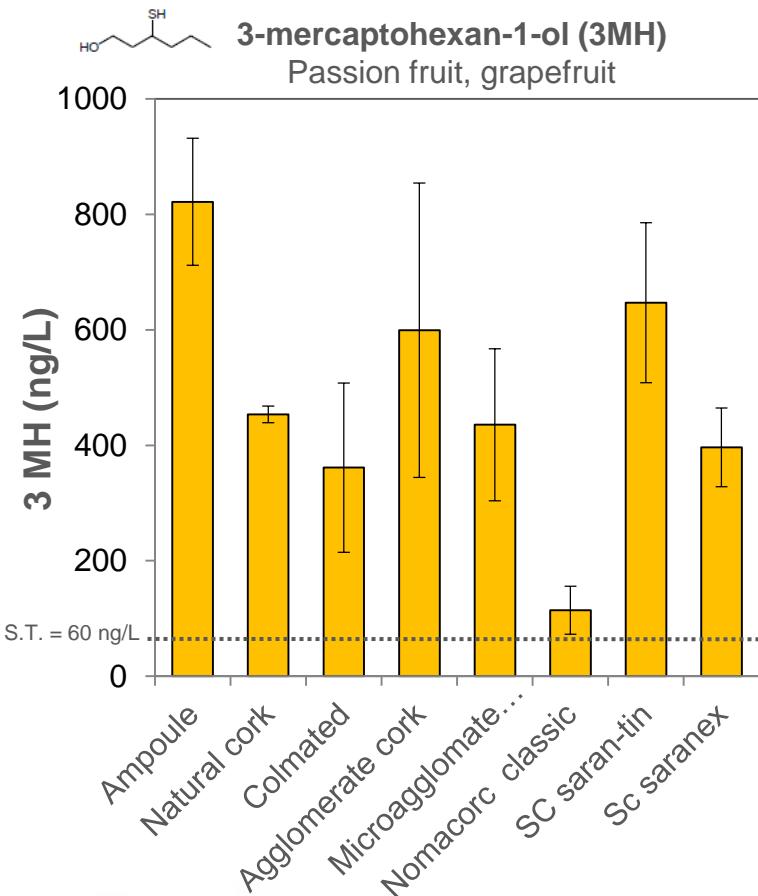
# Impact of bottling and closure OTR on the chemical & sensory properties of a Sauvignon blanc

## Ascorbic acid and free SO<sub>2</sub>



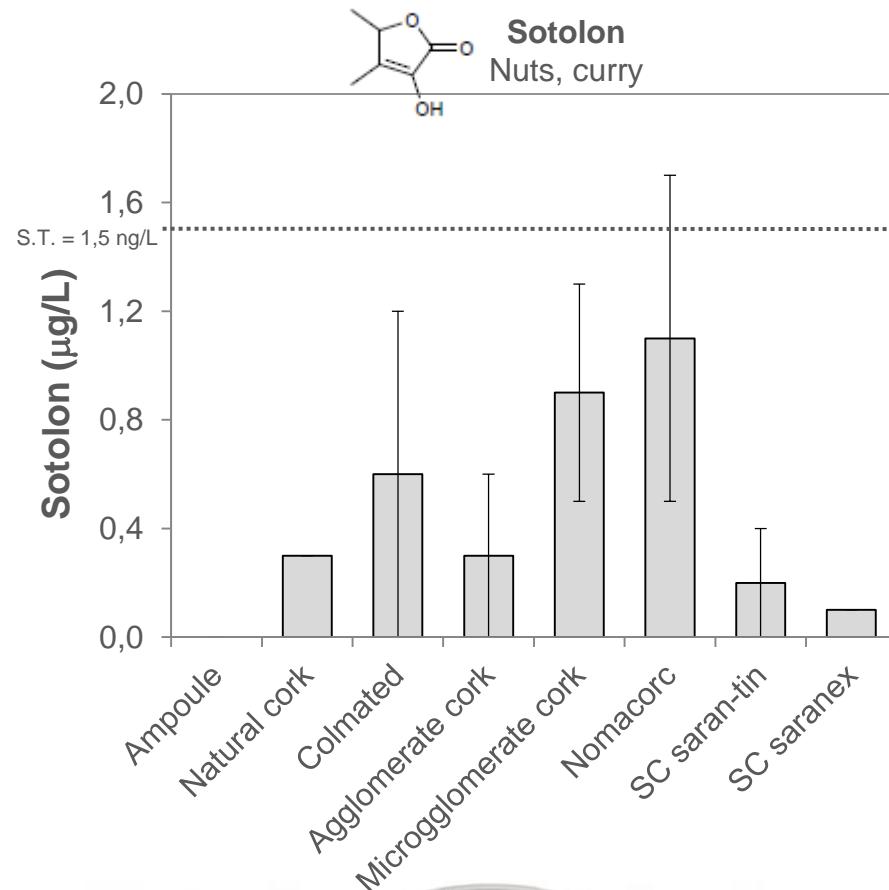
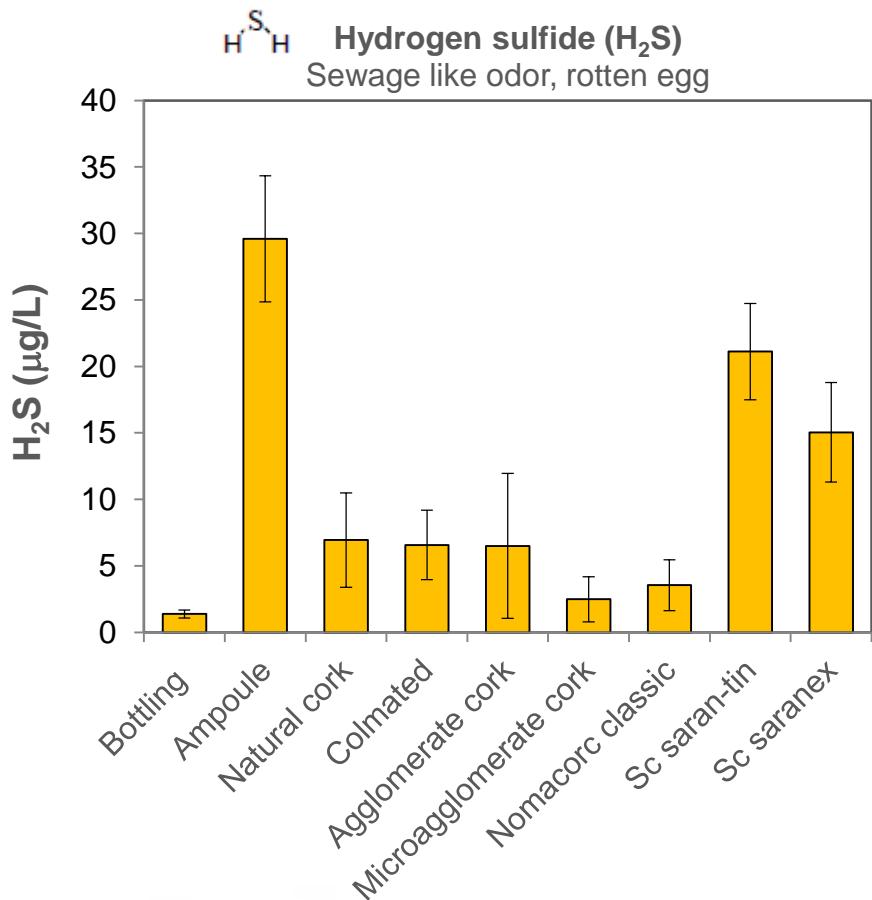
# Impact of bottling and closure OTR on the chemical & sensory properties of a Sauvignon blanc

## Varietal thiols (3MH & 4MMP)



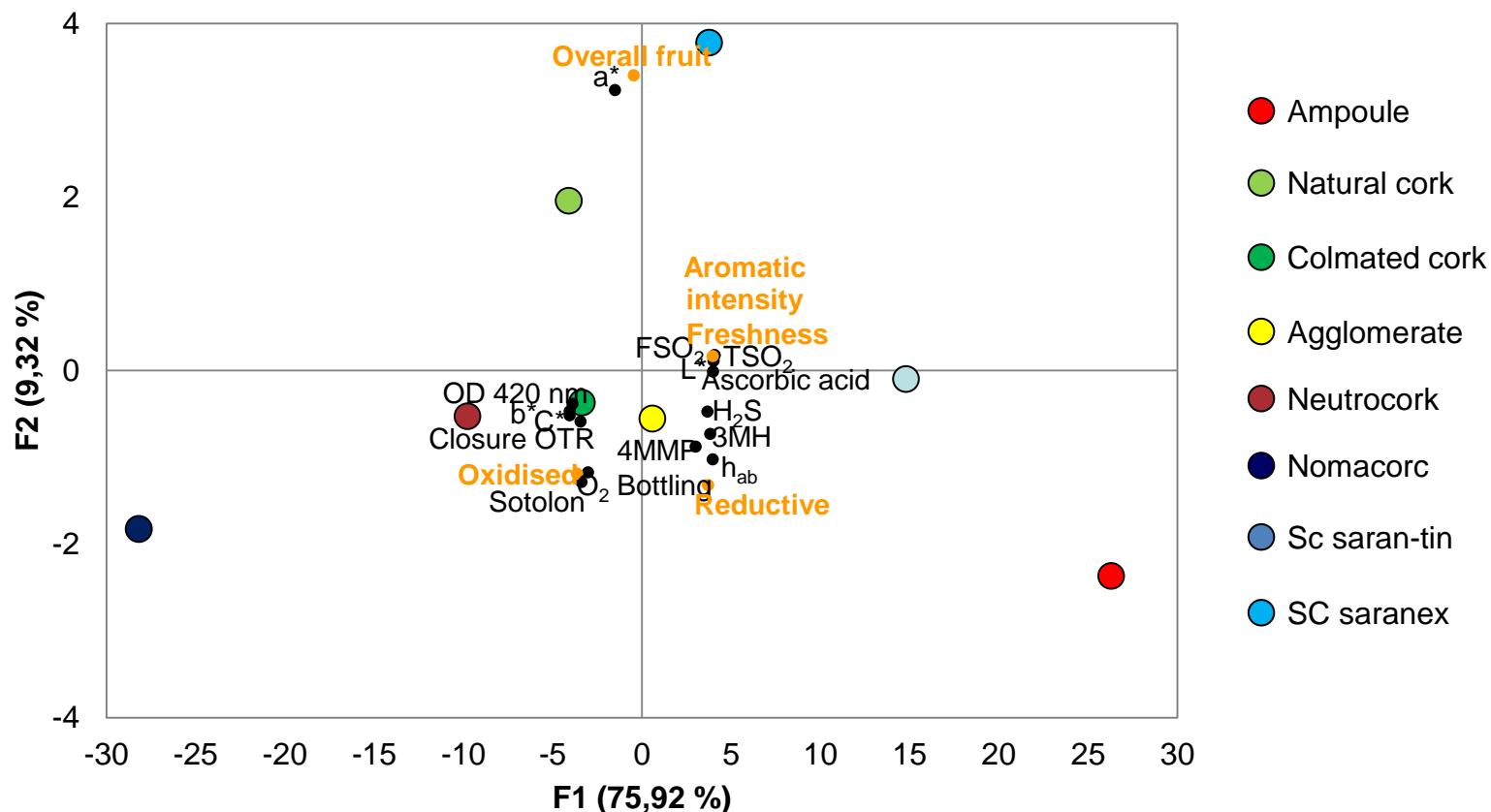
# Impact of bottling and closure OTR on the chemical & sensory properties of a Sauvignon blanc

## Reductive ( $H_2S$ ) and oxidative (Sotolon) characters



# Impact of bottling and closure OTR on the chemical & sensory properties of a Sauvignon blanc

## Compositional & and sensory at 24 months



LOPES, P.; SILVA, C.; TAKATOSHI, T.; LAVIGNE, V.; PONS, A.; SAUCIER, C.; DARRIET, P.; TEISSEDRE, P.L.; DUBOURDIEU, D. Impact of dissolved oxygen at bottling and transmitted through closures on the composition and sensory properties of Sauvignon blanc during bottle storage. *J. Agric. Food Chem.* 2009, 57, 10261-10270.

AMORIM

# Impact of bottling and closure OTR on the chemical & sensory properties of a Merlot

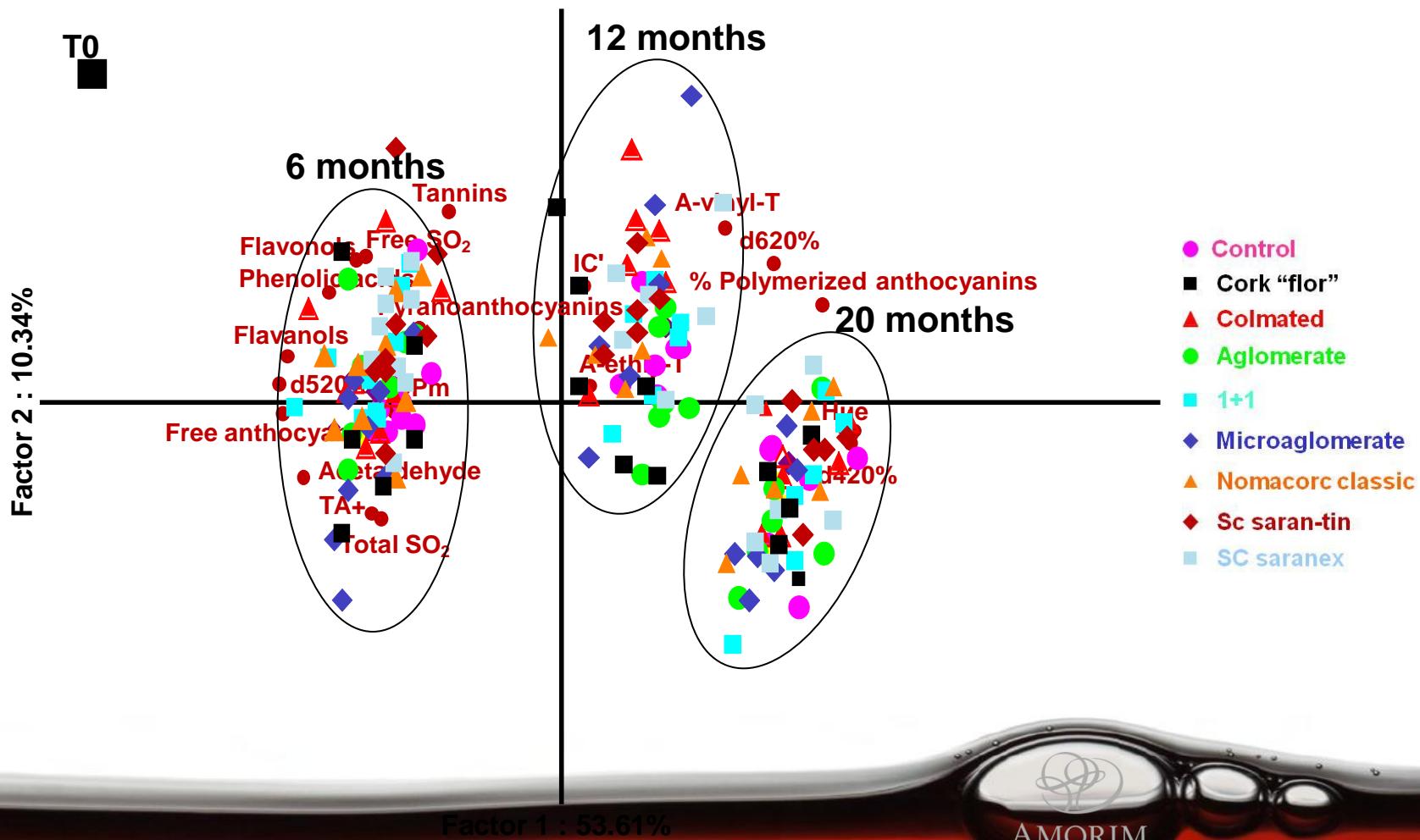
Vin	100% Merlot 2002, UDP Saint Emilion								
Composition	TAV 12.4%	pH 3.5	Total acidity 3.49 g/l H <sub>2</sub> SO <sub>4</sub>	Lactic acid 0.98 g/L	ITP 56.5 Tannins 3.4 g/L	Iron 3.5 mg/L	Total SO <sub>2</sub> 64 mg/L	Sugars 0,22 g/L	
			Volatile acidity 0.37 g/l H <sub>2</sub> SO <sub>4</sub>	Tartaric acid 1.80 g/L	IC' 10.1; tint 0.71	Copper 0.15 mg/L	Free SO <sub>2</sub> 21 mg/L		
Bottles	Bordelaise Tradition CETIE						Bordelaise BVS 30H60		Ampoule
Closures	Natural cork	Colmated cork	Agglomerate	Micro agglomerate	Nomacorc classic	Screw cap saran-tin	Screw cap saranex		
Analyses	Free SO <sub>2</sub>	SO <sub>2</sub> total	Color parameters	Tint	Anthocyanin Tannins phenolics	Polymerized pigments			

Analyses after 6, 12 and 20 months



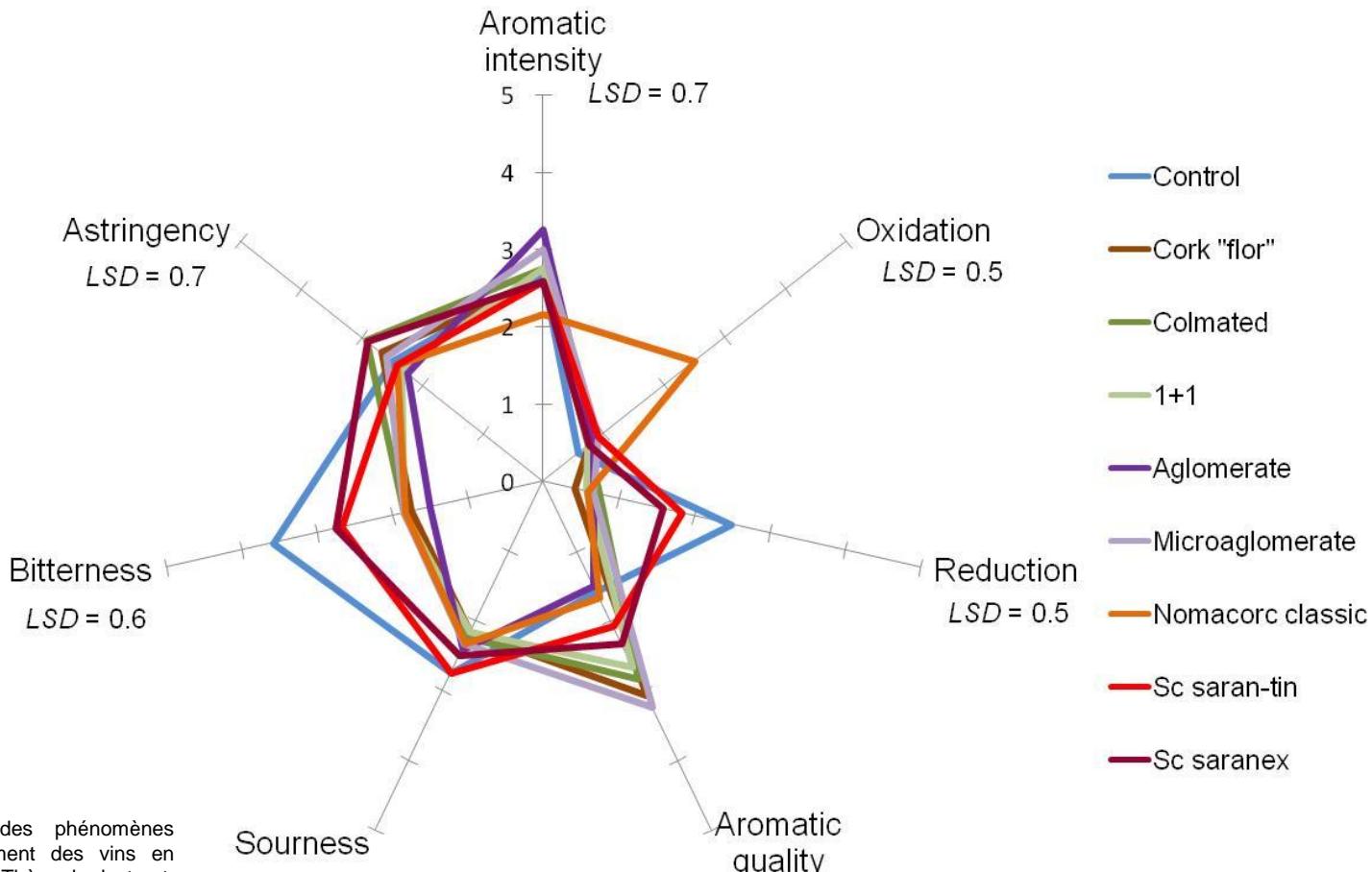
# Impact of bottling and closure OTR on the chemical & sensory properties of a Merlot

## Chemical evolution during 20 months of storage



# Impact of bottling and closure OTR on the chemical & sensory properties of a Merlot

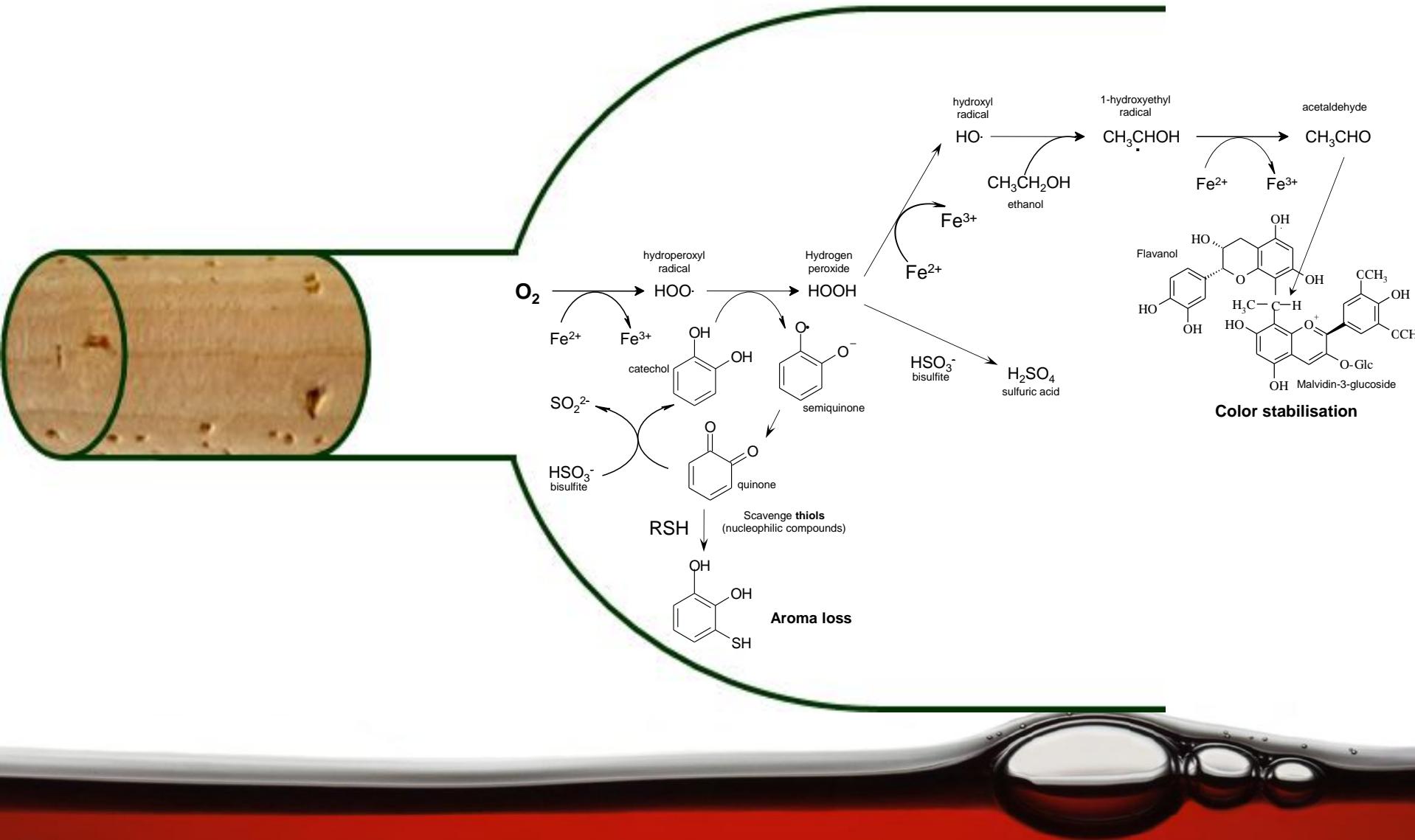
## Sensory assessment at 36 months



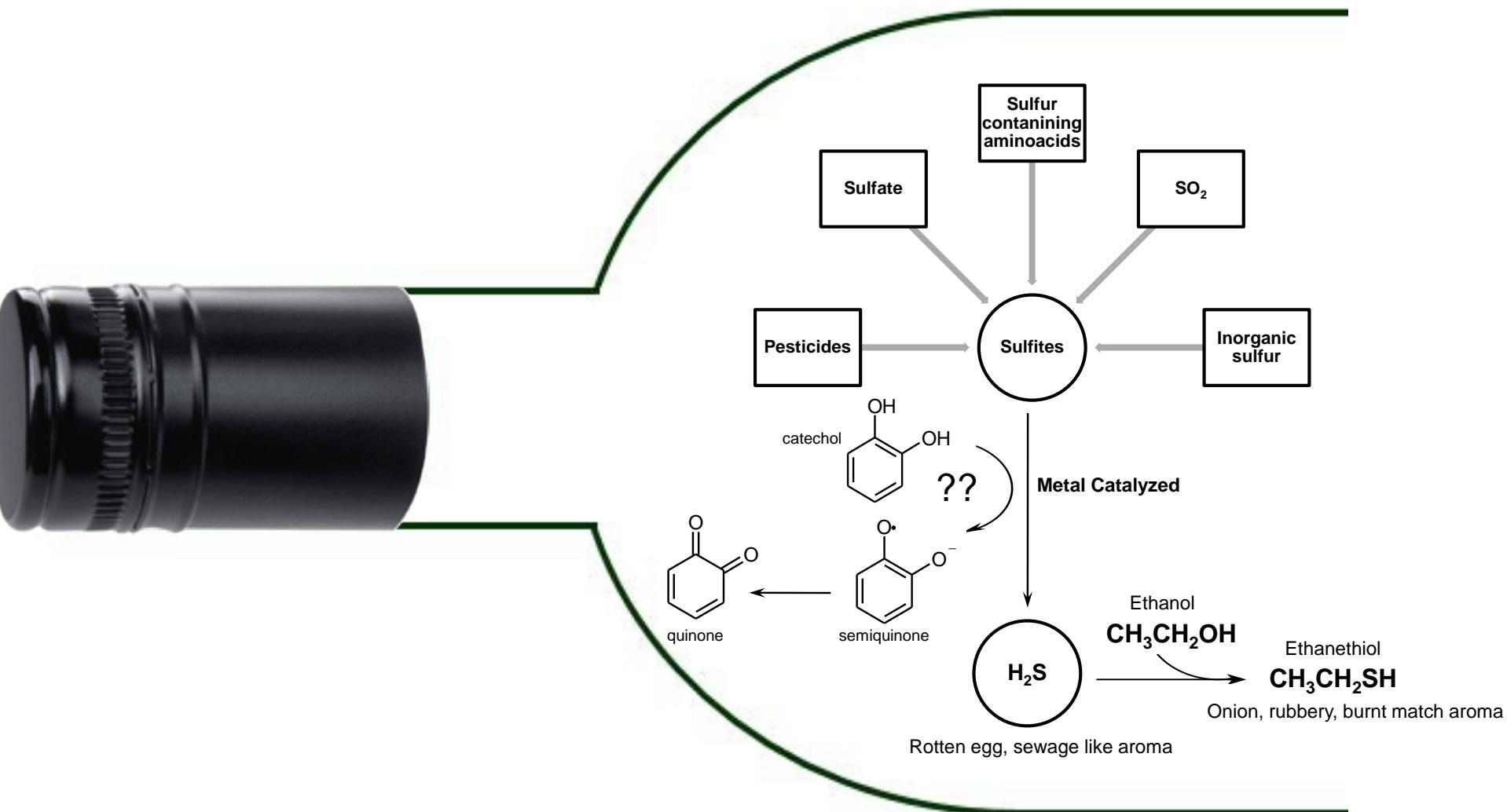
Lopes, P. (2005). L'Etude des phénomènes oxydatifs pendant le vieillissement des vins en bouteille. Role de l'obturateur. Thèse de doctorat. Faculté d'oenologie de Bordeaux



# Metal catalysed wine oxidation mechanism



# Possible post-bottling reduction mechanism



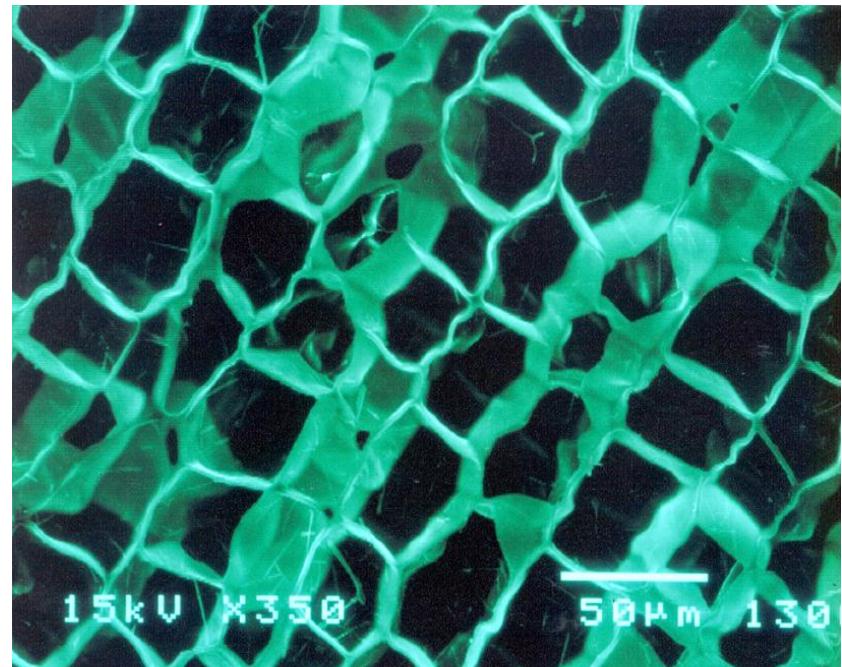
# Chemical composition of cork

## Structural fraction

- 45% suberin
- 27% lignin
- 12% polysaccharides

## Extractible fraction

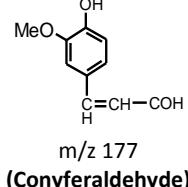
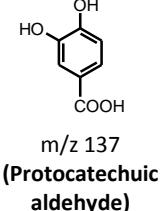
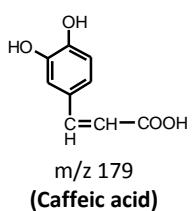
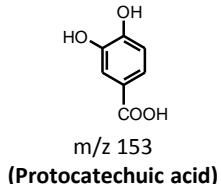
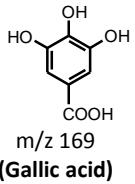
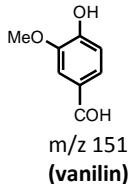
- 6% Triterpenes
- 6% phenolic compounds
- 1% minerals



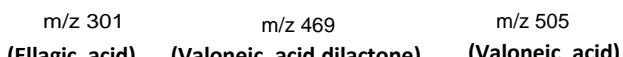
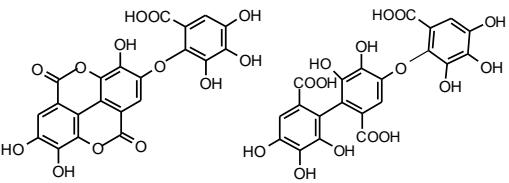
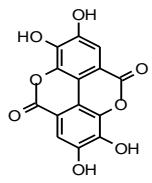
# Chemical composition of cork

## Phenolic compounds

### Phenolic acids and aldehydes

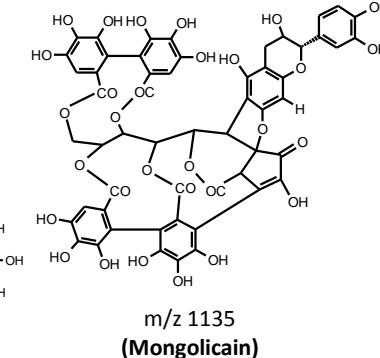
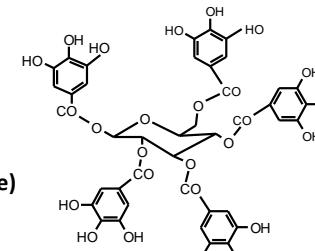
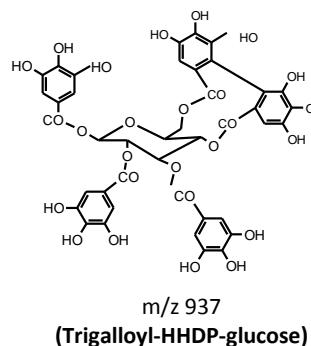
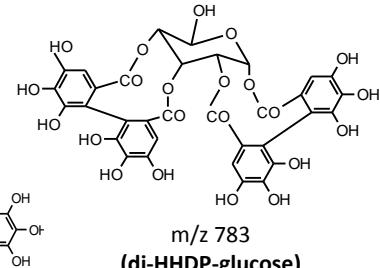
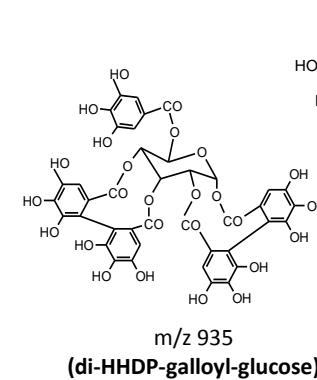
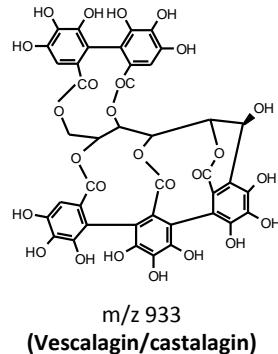


### Gallic and Ellagic acid derivates



FERNANDES, A., MATEUS, N., CABRAL, M.; FREITAS, V. 2011. Analysis of phenolic compounds in cork from *Quercus suber* L. by HPLC-DAD/ESI-MS. *Food Chemistry* 125, 1398-1405

### Ellagictannins



# Chemical composition of cork

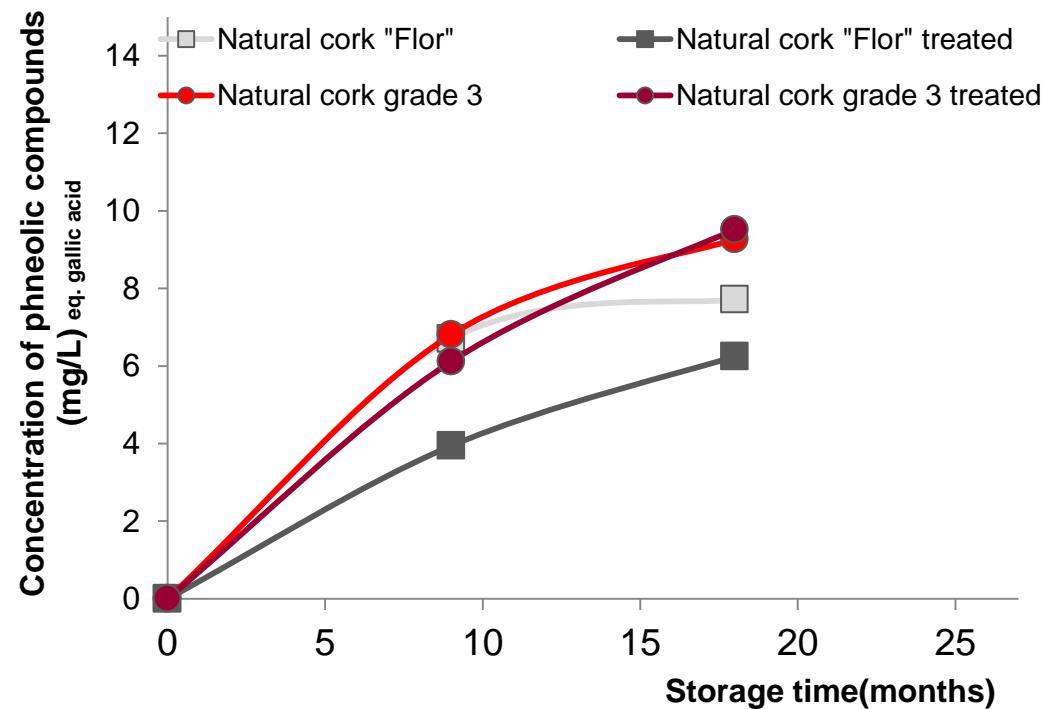
## Migration of phenolic compounds from cork into bottled solutions



On going study for 36 months (30°C)

Natural cork "Flor" grade with and without surface treatment

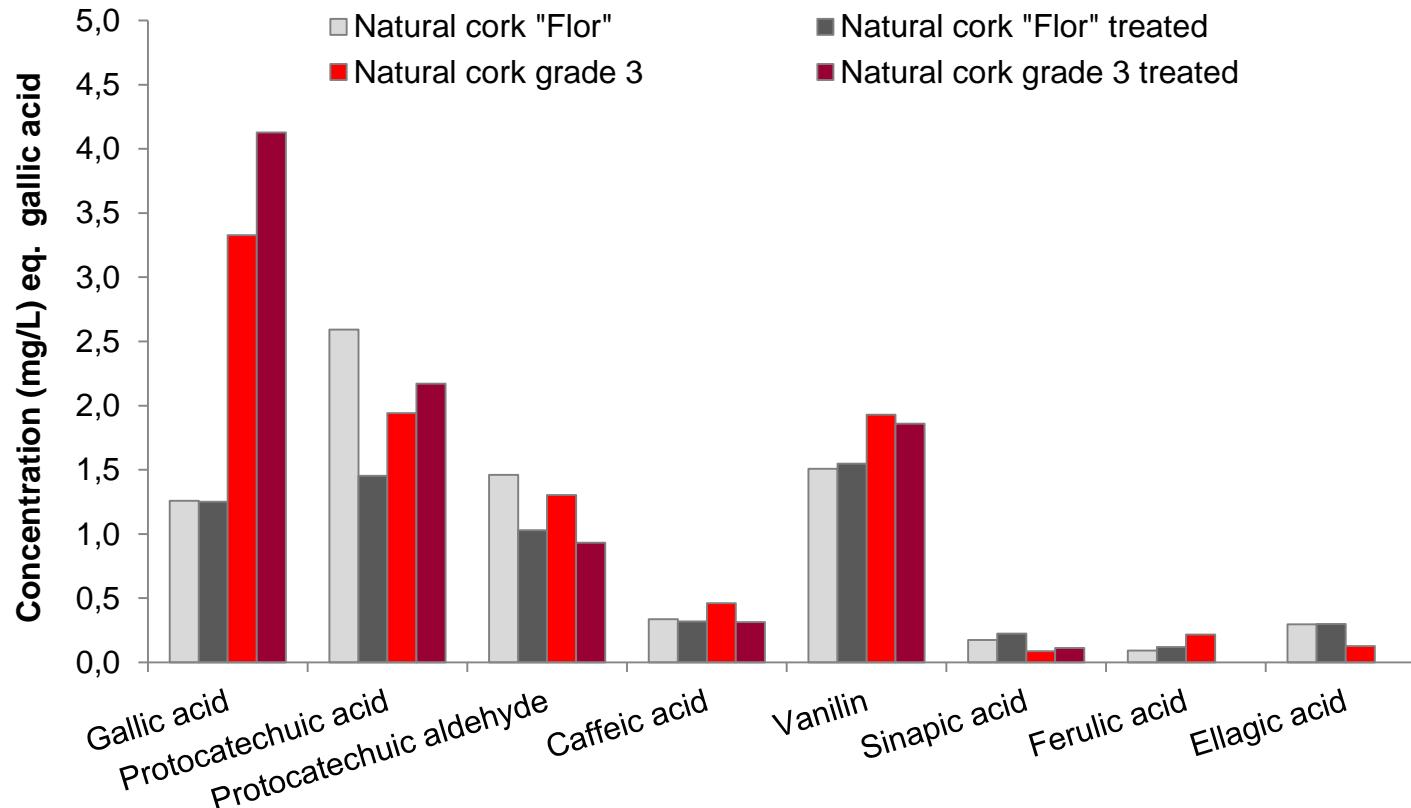
Natural cork grade 3 with and without surface treatment



Very small amounts of phenolic compounds migrate into wine

# Chemical composition of cork

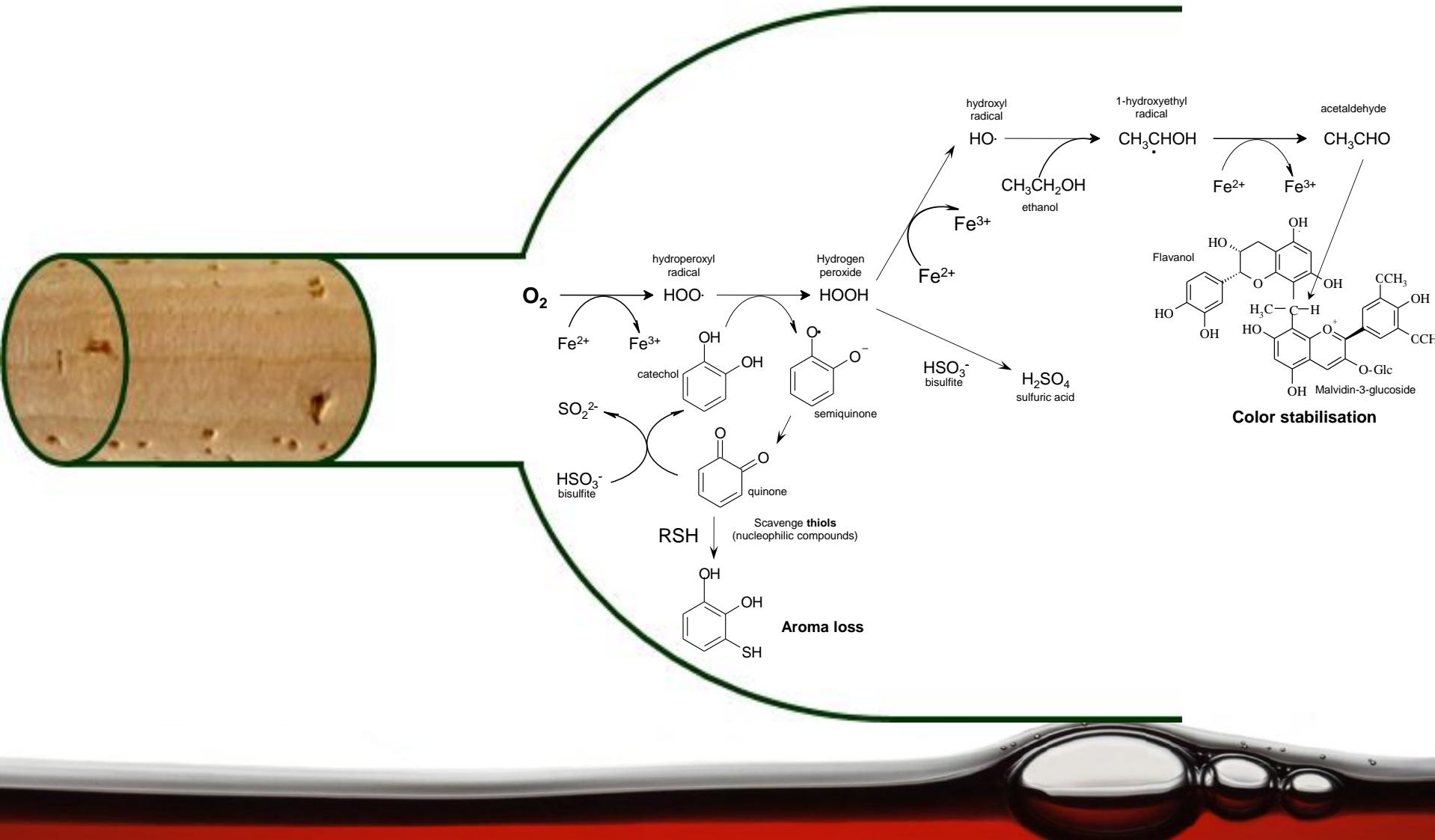
## Migration of phenolic compounds from cork into bottled solutions



The amounts observed phenolic do not impact the wine sensory properties...but do they can contribute to the modulation of oxidative reductive reactions???

# Metal catalysed wine oxidation mechanism

Phenolic compounds have a key role on wine bottle ageing





# Importance of extrinsic wine attributes on the consumer



Wine (red, white, rosé)

Wine variety

Promotion

Brand

Country

Region

Price

Alcohol level

Year

Medals

Green credentials: logo

Packaging:

Type of closure

Type of capsule

Bottle: volume, type, color

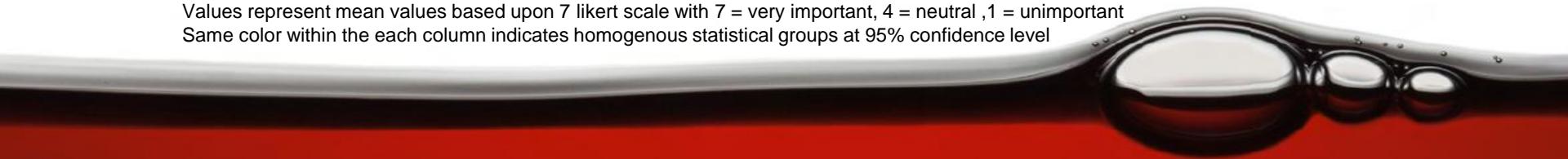
Label: style, type, color

# Importance of extrinsic wine attributes for Canadians purchase

On-line survey  
597 wine consumers  
Ontario; n= 298;  
Québec, n = 299

Attributes		
Price	5.5	5.2
Label information	4.7	4.4
Alcohol level	4.4	4.3
Country of origin	4.2	4.5
Grape variety	4.3	4.4
Region of origin	4.3	4.3
Brand name	4.2	4.3
Type of closure	3.8	3.8
Eco-label logo	3.8	3.7
Capsule material	3.5	3.5
Label pictures	3.5	3.3
Bottle weight	3.4	3.3
Label material	3.2	3.4
Bottle shape	3.3	3.1

Values represent mean values based upon 7 likert scale with 7 = very important, 4 = neutral ,1 = unimportant  
Same color within the each column indicates homogenous statistical groups at 95% confidence level



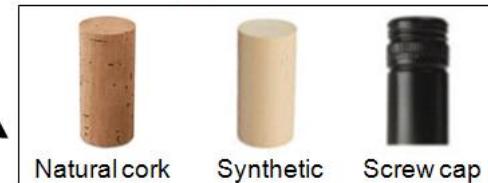
# Consumers attitudes towards wine closures

Canada 

- On-line survey
- 597 wine consumers  
(Ontario; n = 298; Québec, n = 299)
- Technique: Traditional Conjoint Analysis
- 12 graphical representations with the following scenario:
  - « Purchase in retail of an eco-labeled wine to drink by themselves or with other people at home
- Questions:
  - Likelihood of buying this bottle (juster scale)
  - price
  - Realistically intention of purchase (yes/no)



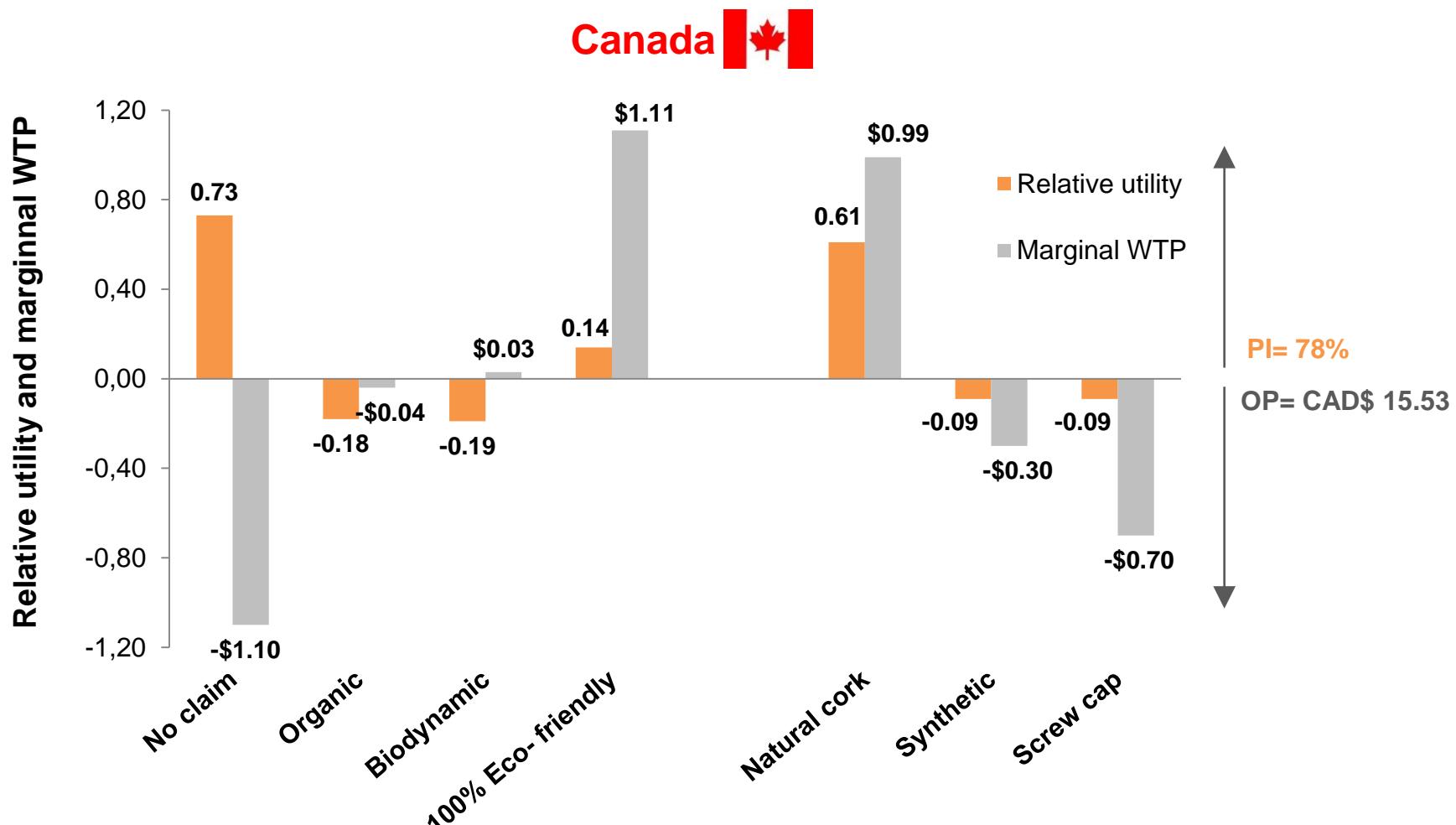
Closure type:



Eco-claim:



# Consumers attitudes towards wine closures



Wine sealed with natural corks enjoys a pricing advantage of CAD\$1.69 and CAD\$1.39 per bottle when compared with screw caps and synthetic

# Key findings

- ✓ TCA is under control; however, there is some room to improvement
- ✓ Gas barrier properties of cork closures are unique, providing an effective barrier against exogenous compounds and releasing low amounts of oxygen that provide a well-balanced wine development
- ✓ Wine development after bottling seems to be rather reductive than oxidative; operations and closures that provide a high and continuous oxygenation are detrimental to wine quality!
- ✓ Phenolic compounds migrate from cork into wine; can they participate on the wine oxidative-reductive reactions ???
- ✓ Natural cork ingrains itself in the minds of consumers as the status quo, while screw caps and synthetic introduce a cognitive dissonance, create poor brand image and thus negative influence the purchase and price



# Thanks very much!!!!

Questions, critics, comments

LinkedIn: Paulo Lopes

