PURE environnement

Ingénierie & Analyses

Perpignan, the 29th of May 2017

ANALYTICAL REPORT – N° OF201705405_1

Customer Identification	
Name:	**DIAM BOUCHAGE**
Address:	Espace Tech Ulrich – 66400 CERET
Contact:	M. BIZART Patrick

Sample identification:	
Description:	1 sample of white wine + its corresponding stopper
	Dossier CORK JANOSA (17-216)
Sampling mode:	under customer responsibility
Receipt date:	11th of May 2017
State at receipt	Satisfactory for analysis

Identification and description of wine samples submitted to analysis:

Laboratory reference	Customer reference	Characteristics
O1705405-11-1	N° 1	White Wine

Identification and description of cork stopper samples extracted from bottles submitted to analysis:

Laboratory reference	Customer reference	Characteristics
O1705405-11-2	N° 1	DIAM* stopper extracted from bottle reference: O1705405-11-1

* Marking:

Notice – Interpretation :

Analysis of the wine revealed the presence of 2,4,6-trichloroanisole, 2,3,4,6-tetrachloroanisole, pentachloroanisole and 2,4,6-trichloroanisole in grades higher than the perception thresholds for each haloanisoles.
As a reminder, the thresholds of perception (TP) on a still white wine are:

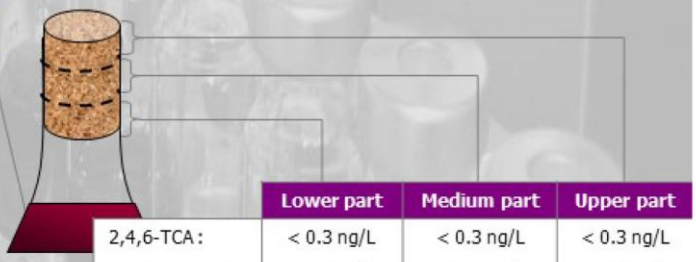
-TP 2,4,6-trichloroanisole: 2 ng/L
-TP 2,3,4,6-tetrachloroanisole: 15 ng/L
-TP 2,4,6-tribromoanisole: 2 ng/L.

Analysis of the corresponding stopper does note reveal contamination of the stopper. The contents of 2,4,6-trichloroanisole, 2,3,4,6-tetrachloroanisole, pentachloroanisole and 2,4,6-trichloroanisole are below the quantification limits.
These compounds are not fed by the stopper. Aerocontamination process can be the cause of this deviation.

Annex : Synthesis data sheet
Wine and matching stoppers analysis

Wine Analysis

Sample n° :	O1705405-11-1
Customer reference:	N° 1
2,4,6-TCA :	< 21.8 ng/L
2,3,4,6- TeCA :	< 14.7 ng/L
PCA :	< 7.1 ng/L
2,4,6-TBA :	< 14.8 ng/L



	Lower part	Medium part	Upper part
2,4,6-TCA :	< 0.3 ng/L	< 0.3 ng/L	< 0.3 ng/L
2,3,4,6- TeCA :	< 1.0 ng/L	< 1.0 ng/L	< 1.0 ng/L
PCA :	< 1.0 ng/L	< 1.0 ng/L	< 1.0 ng/L
2,4,6-TBA :	< 2.0 ng/L	< 2.0 ng/L	< 2.0 ng/L

Stopper analysis

Sample n° : O1705405-11-2

Customer reference : N° 1

Analytical report n° OF201705405

Object of the essay : **Research and quantification of haloanisoles in wine samples**
(operating protocol MO.HAHP.01).

Description of sample preparation

Samples are analyzed on their state at receipt. This report only concerns the analyzed samples.

A 5 mL sample taking is analyzed.

Description of haloanisoles quantification method

- Sample preparation : without matrix modification
- Analysis by headspace-micro-extraction in solid phase
- Quantification by gas chromatography coupled with mass spectrometry
- Internal standardization : 2,4,6-Trichloroanisole deuterium

Results: Haloanisoles in wine samples :

Laboratory reference	Customer reference	Concentration (ng L^{-1})			
		2,4,6-TCA	2,4,6-TBA	2,3,4,6-TeCA	PCA
O1705405-11-1	N° 1	21.8	14.8	14.7	7.1

Limits of quantification (LQ) of analyzed compounds

2,4,6 – Trichloroanisole (2,4,6 – TCA) : *LQ = 0.3 ngL^{-1}*
2,3,4,6 - Tetrachloroanisole (2,3,4,6 – TeCA) : *LQ = 1.0 ngL^{-1}*
Pentachloroanisole (PCA) : *LQ = 1.0 ngL^{-1}*
2,4,6 – Tribromoanisole (2,4,6 – TBA) : *LQ = 2.0 ngL^{-1}*

Object of the essay: **Quantification of releasable haloanisoles on stoppers extracted from wine bottles**

(operating protocol : ISO20752 and MO.HAHP.01)

Description of sample preparation

Samples are analyzed on their state at receipt. This report only concerns the analyzed samples.

Cork stopper extracted from wine bottle is cut in 3 equal parts (Upper – Middle – Lower). The different parts are immersed in a qsf 30 mL of wine simulant at 12% (hydroethanolic solution at 12% acidified at pH 3.6) during 48 hours at room temperature.

Description of haloanisoles quantification method

- Sample preparation : without matrix modification
- Analysis by headspace-micro-extraction in solid phase
- Quantification by gas chromatography coupled with mass spectrometry
- Internal standardization : 2,4,6-Trichloroanisole deuterium

Results: Haloanisoles in stoppers extracted from wine bottles :

Laboratory reference	Customer reference	Concentration (ng L^{-1}) 2,4,6-TCA	2,4,6-TBA	Laboratory reference	Customer reference
O1705405-11-2H	N°1	< LQ	< LQ	< LQ	< LQ
O1705405-11-2M		< LQ	< LQ	< LQ	< LQ
O1705405-11-2B		< LQ	< LQ	< LQ	< LQ

Limits of quantification (LQ) of analyzed compounds

2,4,6 – Trichloroanisole (2,4,6 – TCA) : *LQ = 0.3 ngL^{-1}*
2,3,4,6 - Tetrachloroanisole (2,3,4,6 – TeCA) : *LQ = 1.0 ngL^{-1}*
Pentachloroanisole (PCA) : *LQ = 1.0 ngL^{-1}*
2,4,6 – Tribromoanisole (2,4,6 – TBA) : *LQ = 2.0 ngL^{-1}*

Visa of Analysis Manager

Signature numérique de BOUILLOUX Marylene

Responsable Analyses