RESOLUTION OIV-OENO 623-2018

DETERMINATION OF RELEASABLE 2,4,6-TRICHLOROANISOLE IN WINE BY CORK STOPPERS – UPDATE OF THE METHOD OIV-MA-AS315-16

WARNING: this resolution amends the following resolution:
- *OIV/OENO 296/2009*

THE GENERAL ASSEMBLY,

CONSIDERING Article 2 paragraph 2 iv of the Agreement of April 3, 2001 establishing the International Organisation of Vine and Wine,

Upon the proposal of the Sub-commission of Methods of Analysis,

CONSIDERING that the proposed modifications can improve the accuracy and precision of the method,

CONSIDERING that the proper internal standard should be chemically similar to the compound(s) that you are analyzing, but is not expected to be naturally present in your sample,

CONSIDERING that whenever possible the internal standard should have similar functional groups, boiling points, and activity as the target compounds.

CONSIDERING that the amount of salt indicated in the Resolution differs significantly from the mass ratio often obtained in the analytical validation processes involving the preparative technique of solid phase microextraction: “Adding 25-30 % (wt./vol.) sodium chloride to the sample prior to extraction can increase the ionic strength of the solution and, in turn, reduce the solubility of some analytes ”.

DECIDES to update the method OIV-MA-AS315-16 (OIV-OENO 296-2009) of the Annex A of the Compendium of International Methods of Analysis of Wine and Must with the following amendments:

DETERMINATION OF RELEASABLE 2,4,6-TRICHLOROANISOLE IN WINE BY CORK STOPPERS

Point 3.3 is replaced with the following text:

Internal standard for GC/MS analysis: 2,4,6-trichloroanisole (TCA)-d5 purity ≥ 98% or 2,3,6-trichloroanisole purity ≥ 99%.

Certified in conformity Punta del Este, 23rd November 2018
The Director General of the OIV
Secretary of the General Assembly
Jean-Marie AURAND

© OIV 2018

Internal standard for GC/ECD analysis; 2,6-dibromoanisole purity ≥ 99% or 2,3,6-trichloroanisole purity ≥ 99%.

Point 6.1, the second paragraph is modified as follows (the part to be deleted is crossed out, the part in bold italic is added)

To increase extraction efficiency and subsequent sensitivity of the method, a quantity of about 1g of sodium chloride (3.2) can be added. **The amount of sodium chloride can be adjusted / optimized by the users of this method, depending on the desired level of sensitivity and possible matrix effects that may occur. For example, a quantity of about 3 g of sodium chloride is** suggested. 50 µL of the internal standard solution at 2.0 µg/L (3.10) are immediately added, then the bottle is closed using a perforated metal capsule fitted with a silicone / Teflon-coated liner. The capsule is crimped. The contents of the bottle are homogenized for 10 minutes by mixing using a stirring system (4.4) or by using an automatic system (4.7).

Point 6.3 is modified as follows (the part to be deleted is crossed out, the part in bold italic is added)

Detection and quantification by mass spectrometry is carried out with a selection of specific ions. for the 2,4,6-trichloroanisole (ions m/z 195, 210, 212), quantified on the m/z 195 ion, and the internal standard 2,4,6-trichloroanisol-d5 (ions m/z 199, 215, 217) quantified on ion m/z 215... **For example, the following ion ratio is suggested**

	Analyte	Interesting ions for detection (m/z):	Ion Quantification (m/z) :
Analysis in SIM mode	2,4,6-TCA	195, 210, 212	195
	(2,4,6-TCA)-d_5	199, 215, 217	215
	2,3,6-TCA	195, 210, 212	212

Certified in conformity Punta del Este, 23rd November 2018
The Director General of the OIV
Secretary of the General Assembly
Jean-Marie AURAND

© OIV 2018

Analysis in tandem mode (MS/MS)	Analyte	Parent ions (m/z):	Daughter ion (m/z) :
	2,4,6-TCA	212	169, 197
		196	167, 169
	(2,4,6-TCA)-d_5	217	171, 199

For the determination by GC/ECD, identify the analyte and internal standard (2,6-dibromoanisole or 2,3,6 trichloroanisole) in the chromatogram, by comparing the retention time of the sample peak corresponding to that of the standard solution peak.

Certified in conformity Punta del Este, 23rd November 2018
The Director General of the OIV
Secretary of the General Assembly
Jean-Marie AURAND

© OIV 2018