RESOLUTION OIV-OENO 512-2014

DETERMINATION OF THE CARBON ISOTOPE RATIO 13C/12C OF CO_2 IN SPARKLING WINES: METHOD USING ISOTOPE RATIO MASS SPECTROMETRY (IRMS) - MODIFICATION

THE GENERAL ASSEMBLY,

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

ON THE PROPOSAL of the "Methods of Analysis" Sub-Commission,

CONSIDERING the resolution Oeno 7/2005 (Method OIV-MA-AS314-03) "DETERMINATION OF THE CARBON ISOTOPE RATIO 13C/12C OF CO_2IN SPARKLING WINES: METHOD USING ISOTOPE RATIO MASS SPECTROMETRY (IRMS)" appearing in the Compendium of International Methods of Wine and Must Analysis,

DECIDES to modify the method OIV-MA-AS314-03 by changing paragraph 6: Apparatus, and sub-paragraph 7.1: CO_2sampling procedures, as follows:

To paragraph 6: Apparatus, at the end of the sub-paragraph "Continuous-flow systems (CF-IRMS)", the following will be added:

- Gas Sampler-IRMS. A peripheral system may be used for the on-line gas preparation, isolation of CO_2 and introduction of CO_2into the isotope ratio mass spectrometer.

To sub-paragraph 7.1: CO2 sampling procedures, points "d" and "e" will be added as follows:

- d. Refrigerate the sample at 4-5 °C, before quickly transferring the liquid into a vial and sealing it with a Teflon-silicone septum cap. Then 50 μL of liquid is then transferred into a 10 mL vial and analysed. If necessary, the vial should be filled with helium in order to remove the atmospheric CO_2.
- e. After refrigerating the sample, the bottle is opened at room temperature and a sample of 200 µL of liquid is taken using a pipette and placed in suitable vials. The vials are immediately resealed then placed in an ultrasonic bath for 10 min prior to analysis.

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Secretary of the General Assembly
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The statistical results of the inter-laboratory test for sampling procedures 7.1.d and 7.1.e are given in ANNEX E.

To sub-paragraph 9 (Precision) some modifications are added:

9. Precision

Details on the inter-laboratory test on precision of the method are given in Annexes D and E.

To sub-paragraph 9.1 (Repeatability):

9.1. Repeatability

9.1.1 Characteristics of sampling procedures 7.1.a-c

S_r = 0.21 ‰

r = 0.58 ‰

9.1.2. Characteristics of sampling procedures 7.1.d and 7.1.e

S_r = 0.21 ‰

r = 0.56 ‰

To sub-paragraph 9.2 (Reproducibility):

9.2. Reproducibility

9.2.1 Characteristics of sampling procedures 7.1.d and 7.1.e

S_R = 0.68‰

R= 1.91‰

Annexes (A,B,C,D,E)

Annex E was added to the method:

ANNEX E

Statistical results of the inter-laboratory test on sparkling and gasified wines

Sampling procedures 7.1.d and 7.1.e

In accordance with method OIV-MA-AS1-09: R2000, the following parameters were defined as part of an inter-laboratory test conducted with 16 laboratories.

Year of the inter-laboratory test: 2013-2014

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Number of laboratories:16

Type of samples: Sparkling and gasified wines

Number of samples: 3, as blind duplicates

Parameter measured: $\delta^{13}C$

INDICATORS	WINE NO. 1	WINE NO. 2	WINE NO. 3
Number of laboratories	16	14	16
Number of repetitions	2	2	2
Minimum	-32.90	-33.10	-23.64
Maximum	-29.83	-30.97	-20.57
Repeatability variance s_r^2	0.0467	0.0118	0.0648
Inter-group variance s_L^2	0.43853	0,29762	0.51616
Reproducibility variance s_R^2	0.4852	0.3094	0.5810
Overall average	-31.42	-31.83	-22.15
Repeatability standard deviation	0.22	0.11	0.25
r limit	0.612	0.307	0.720
Reproducibility standard deviation	0.70	0.56	0.76
R limit	1.971	1.574	2.157

	Wine No. 1	Wine No. 2	Wine No. 3	Z-score	Z-score	Z-score

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Laboratory Code	A	B	A	B	A	B	Wine No. 1	Wine No. 2	Wine No. 3
Lab 1	-31.40	-31.69	-31.56	-31.88	-21.93	-22.12	-0.18	-0.19	0.16
Lab 2	-31.23	-31.29	-31.43	-31.41	-21.46	-22.04	0.23	-0.73	0.52
Lab 3	-32.65	-32.12	-32.15	-32.13	-23.41	-23.64	-1.39	-0.56	-1.81
Lab 4	-31.55	-31.50	-31.46	-31.66	-22.40	-22.54	-0.15	0.48	-0.42
Lab 5	-31.50	-31.30	-31.80	-31.90	-22.00	-22.30	0.03	-0.04	0.00
Lab 6	-31.46	-31.75	-31.96	-31.75	-22.39	-22.10	-0.27	-0.05	-0.13
Lab 7	-31.48	-30.66	-31.29	-29.35	-21.47	-20.57	0.50	2.71	1.48
Lab 8	-29.83	-30.17	-29.73	-31.35	-21.50	-21.96	2.04	2.31	0.55
Lab 9	-30.96	-30.90	-31.34	-31.21	-22.22	-22.27	0.70	0.99	-0.13
Lab 10	-32.34	-32.29	-32.68	-32.75	-23.25	-23.14	-1.29	-1.60	-1.37
Lab 11	-32.90	-32.70	-33.10	-33.10	-23.00	-23.50	-1.98	-2.29	-1.45
Lab 12	-31.91	-31.68	-32.22	-32.14	-22.58	-22.66	-0.54	-0.63	-0.62
Lab 13	-31.03	-31.10	-31.61	-31.68	-21.78	-21.74	0.51	0.33	0.51
Lab 14	-31.25	-30.93	-31.43	-31.54	-22.01	-22.02	0.57	0.62	0.17
Lab 15	-30.89	-30.88	-31.59	-31.47	-21.08	-21.07	0.76	0.53	1.41
Lab 16	-31.05	-30.98	-31.24	-30.97	-21.090	-21.490	0.58	1.30	1.13

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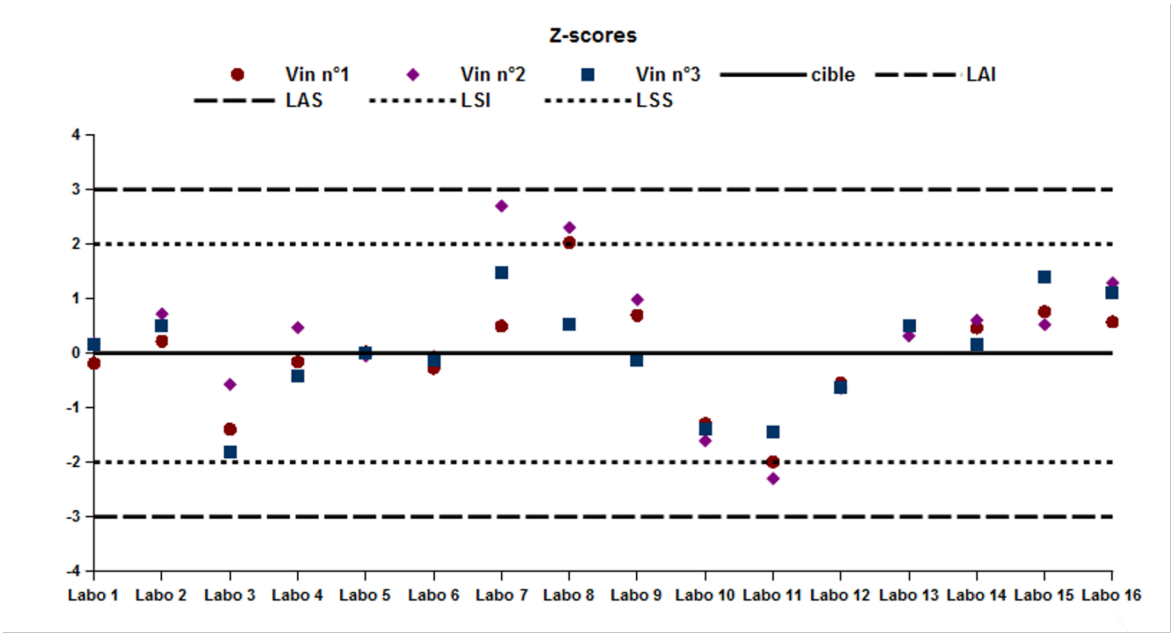

Bibliography

1. Ana I. Cabañero, Tamar San-Hipólito and Mercedes Rupérez, GasBench/isotope ratio mass spectrometry: a carbon isotope approach to detect exogenous CO2 in sparkling drinks Rapid Commun. Mass Spectrom. 2007; 21: 3323–3328.
2. Laetitia Gaillard, Francois Guyon⁄, Marie-Hélène Salagoïty, Bernard Médina, Authenticity of carbon dioxide bubbles in French ciders through multiflow-isotope ratio mass spectrometry measurements. Food Chemistry. 2013, 141: 2103–2107

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