RESOLUTION OIV-OENO 566-2016

BEVERAGES WITH LOW ALCOHOL CONTENT – UPDATE TO METHOD OIV-MA-AS312-01A

THE GENERAL ASSEMBLY,

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

CONSIDERING the work of the "Methods of Analysis" Sub-Commission during its March 2014 session,

DECIDES, on the proposal of Commission II "Oenology", to modify Method OIV-MA-AS312-01A, “Alcoholic strength by volume”, in the Compendium of international methods of wine and must analysis:

DECIDES to add, to paragraph 3, "Method of obtaining distillate", a sub-paragraph regarding beverages with low alcohol content, as per the following modifications:

- Insertion of the following subtitle into the current text under paragraph 3.4: “Procedure for beverages with an ABV greater than 1.5% vol.”, renumbering it as sub-paragraph 3.4.1.

Addition of the sub-paragraph 3.4.2 as follows:

- Procedure for beverages with an ABV lower than or equal to 1.5% vol.
- Take a 200 mL sample of beverage using a calibrated flask. Note the temperature of the beverage. Pour it into the flask of the distillation apparatus or into the bubbler of the steam distillation apparatus. Rinse the calibrated flask four times with 5 mL of water and add this to the apparatus' flask or bubbler.
- Add a 10 mL suspension of 2 M calcium hydroxide and, in the case of distillation, if necessary, a boiling regulator (pumice stone, etc.). Collect, in a 100 mL calibrated flask, a volume of distillate equal to around 75 mL in the case of distillation or 98-99 mL in the case of steam distillation. Make up to 100 mL with distilled water while the distillate is within ± 2 °C of the initial temperature. Carefully mix using a circular motion.

To paragraphs 4A, 4B and 4C, addition of the point "Expression of results" (3.1 for

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paragraph 4A, 1.9 for paragraph 4B and 1.7.6 for paragraph 4C) accompanied by the following phrase:

- The alcoholic strength by volume of a beverage with low alcohol content, with an ABV of less than 1.5% vol., is given by the following formula: ABV = ABVD/2, ABVD being the alcoholic strength by volume of the distillate.
- It is expressed in "% vol.". The result is given to two decimal places.
- The validation parameters for beverages with low alcohol content are annexed.
- Addition of an annex regarding the validation parameters relating to the measurement of the ABV of beverages with low alcohol content:

ANNEX

Validation

This document presents the results of the validation study corresponding to the method for beverages with low alcohol content (updated).

The study was carried out in accordance with the OIV documents MA-F-AS1-08-FIDMET and MA-F-AS1-09-PROPER.

1. Samples

Sample no.	1	2	3	4	5	6
Nature	Grape juice	Beverage obtained by dealcoholisation of wine	Beverage obtained by partial dealcoholisation of wine	Partially fermented grape juice	Cider	Wine-based beverage
Approximate value of ABV (% vol.)	< 0.5	0.5	1.5	2.5	4.5	6.5

The samples were sent to the participating laboratories, applying the double-blind principle.

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2. Analyses

Each of the 12 samples received by the laboratories was analysed by simple distillation or steam distillation, according to the two following procedures:

- OIV reference method involving the use of 200 mL and recovery of 200 mL of distillate,
- alternative method involving the use of 200 mL and recovery of 100 mL of distillate.

3. Participating laboratories

19 laboratories from different countries took part:

Laboratório CVRVV	4050-501 Porto	Portugal
Laboratório de Análises da CVRA	7006-806 Évora	Portugal
Testing Laboratory CAFIA	603 00 Brno	Czech Republic
Laboratório ASAE - LBPV	1649-038 Lisbon	Portugal
Agroscope - Site de Changins	1260 Nyon 1	Switzerland
Labo SCL de Bordeaux	33608 Pessac	France
Labo SCL de Montpellier	34196 Montpellier	France
Laboratorio Arbitral Agroalimentario	28023 Madrid	Spain
Estación Enológica de Haro	26200 Haro La Rioja	Spain
Instituto dos Vinho do Douro do Porto	4050-253 Porto	Portugal

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IVICAM	13700 Tomelloso, Ciudad Real	Spain
INCAVI	08720 Vilafranca del Penedès	Spain
ICQRF Laboratorio di Conegliano/Susegana	31058 Susegana (TV)	Italy
ICQRF Laboratorio di Catania	95122 Catania	Italy
ICQRF Laboratorio di Modena	41100 Modena	Italy
ICQRF laboratorio di Perugia	06128 Perugia	Italy
ICQRF laboratorio di Salerno	84098 Salerno	Italy
ICQRF Laboratorio centrale di Roma	00149 Rome	Italy
Laboratoires DUBERNET	11100 Narbonne	France

4. Results

	Sample No. 1		Sample No.° 2		Sample No. 3		Sample No. 4		Sample No. 5		Sample No. 6	
Laboratory	Position 2	Position 7	Position 4	Position 11	Position 6	Position 12	Position 5	Position 8	Position 9	Position 10	Position 1	Position 3
A	0.21	0.21	0.55	0.55	1.34	1.34	2.58	2.58	4.59	4.60	6.54	6.50
B	0.11	0.14	0.49	0.50	1.32	1.38	2.60	2.57	4.68	4.72	6.52	6.55
C	0.33	0.28	0.68	0.61	1.43	1.35	2.63	2.60	4.63	4.66	6.58	6.51
D			0.62	0.62	1.38	1.36	2.68	2.67	4.69	4.73	6.62	6.64
E	0.20	0.21	0.55	0.56	1.36	1.40	2.61	2.62	4.67	4.68	6.56	6.55
F	0.18	0.12	0.52	0.51	1.31	1.30	2.56	2.56	4.70	4.66	6.51	6.54
G	0.22	0.22	0.55	0.56	1.37	1.37	2.62	2.62	4.68	4.68	6.58	6.57
H			0.41	0.42	1.25	1.27	2.46	2.49	4.57	4.56	**6.39**	**6.40**
I	0.20	0.13	0.54	0.48	1.32	1.28	2.60	2.58	4.62	4.62	6.57	6.55

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J	0.24	0.24	0.58	0.60	1.41	1.37	2.63	2.63	4.69	4.67	6.55	6.55
K	0.22	0.22	0.56	0.55	1.35	1.35	2.63	2.63	4.67	4.68	6.59	6.58
L	0.22	0.23	0.56	0.57	1.38	1.36	2.63	2.61	4.66	4.67	6.56	6.57
M	0.18	0.18	0.53	0.53	1.33	1.29			4.66	4.65	6.53	6.52
N	0.22	0.23	0.56	0.57	1.38	1.41	**2.26**	**2.61**	4.67	4.67	6.51	6.57
O	0.12	0.19	0.53	0.52	1.33	1.33	2.64	2.62	4.67	4.67	6.51	6.55
P	0.25	0.25	0.57	0.58	1.39	1.41	2.66	2.65	4.70	4.68	6.62	6.62
Q	0.22	0.20	0.55	0.59	1.34	1.33	2.61	2.63	4.65	4.63	6.52	6.54
R	0.21	0.21	0.55	0.52	1.29	1.28	2.52	2.55	4.62	4.56	6.50	6.53
S	0.18	0.17	0.41	0.42	1.38	1.37	2.61	2.58	4.63	4.58	6.51	6.48

Results table obtained for a distillation of 200 mL with a recovery volume of 200 mL. The values in bold correspond to the values rejected by the Cochran test (variance outliers) with a significance level of 2.5% (1-tail test) and by the Grubbs test (means outliers) with a significance level of 2.5% (2-tail test).

Note: The values absent were not provided by the laboratory in question.

	Sample No. 1		Sample No. 2		Sample No. 3		Sample No. 4		Sample No. 5		Sample No. 6	
Laboratory	Position 2	Position 7	Position 4	Position 11	Position 6	Position 12	Position 5	Position 8	Position 9	Position 10	Position 1	Position 3
A												
B	0.17	0.18	0.52	0.53	1.34	1.36	2.62	2.62	4.62	4.60	6.48	6.52
C	0.25	0.25	**0.56**	**0.62**	1.35	1.36	2.50	2.46	4.48	4.44	6.12	6.19
D	0.29	0.29	0.63	0.63	1.43	1.42	2.66	2.65	4.68	4.69	6.58	6.59
E	0.24	0.24	0.58	0.58	1.39	1.39	2.64	2.64	4.66	4.67	6.55	6.57
F	0.21	0.18	0.53	0.53	1.31	1.27	2.41	2.48	4.30	4.31	**6.22**	**5.89**
G	0.24	0.24	0.56	0.57	1.35	1.36	2.58	2.57	4.57	4.56	6.46	6.43
H	0.19	0.18	**0.48**	**0.55**	1.33	1.32	2.51	2.55	4.59	4.54	6.38	6.42
I	**0.25**	**0.18**	**0.56**	**0.53**	1.34	1.33	2.62	2.61	4.64	4.64	6.25	6.28
J	0.24	0.24	0.55	0.56	1.31	1.32	2.49	2.53	4.37	4.34	6.14	6.12
K	0.25	0.25	0.57	0.57	1.37	1.38	2.60	2.61	4.60	4.61	6.48	6.38
L	0.24	0.24	0.55	0.55	1.35	1.31	2.52	2.47	4.38	4.31	6.09	6.06
M	0.19	0.20	0.55	0.55	1.34	1.31			4.68	4.67	6.52	6.54
N	0.28	0.26	0.58	0.59	1.28	1.28	2.52	2.47	**4.44**	**4.32**	6.01	6.15
O	**0.19**	**0.25**	0.57	0.57	1.39	1.39	2.63	2.64	4.66	4.66	6.57	6.57

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P	0.25	0.26	0.57	0.57	1.36	1.36	2.58	2.56	4.54	4.53	6.34	6.38
Q	0.24	0.24	0.57	0.57	1.38	1.38	2.63	2.62	4.66	4.67	6.56	6.56
R	0.23	0.23	0.54	0.55	1.32	1.30	2.54	2.56	4.56	4.52	6.40	6.35
S	0.27	0.26	0.55	0.57	1.34	1.34	2.46	2.43	4.53	4.51	6.36	6.36

Results table obtained for a distillation of 200 mL with a recovery volume of 100 mL. The values in bold correspond to the values rejected by the Cochran test (variance outliers) with a significance level of 2.5% (1-tail test) and by the Grubbs test (means outliers) with a significance level of 2.5% (2-tail test).

Note: The values absent were not provided by the laboratory in question.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
No. of laboratories considered	17	19	19	17	19	18
No. of repetitions	2	2	2	2	2	2
Minimum	0.11	0.41	1.25	2.46	4.56	6.48
Maximum	0.33	0.68	1.43	2.68	4.73	6.64
Overall average	0.20	0.54	1.35	2.60	4.65	6.55
Repeatability variance	0.00052	0.00033	0.00050	0.00019	0.00036	0.00047
Reproducibility variance	0.00211	0.00345	0.00190	0.00229	0.00181	0.00147
Inter-laboratory standard deviation	0.043	0.057	0.041	0.047	0.040	0.035
Repeatability standard deviation	0.02	0.02	0.02	0.01	0.02	0.02
r limit	0.06	0.05	0.06	0.04	0.05	0.061
Repeatability CV	11.1	3.3	**1.7**	0.5	0.4	0.3

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Reproducibility standard deviation	0.046	0.059	0.044	0.048	0.043	0.038
R limit	0.130	0.166	0.123	0.135	0.120	0.109
Reproducibility CV	22.5	10.9	3.2	**1.8**	0.9	0.6
Horwitz RSD_r	3.36	2.90	2.52	2.29	2.09	1.99
$Horrat_r$	3.3	1.1	**0.7**	0.2	0.2	0.2
Horwitz RSD_R	5.10	4.39	3.82	3.46	3.17	3.01
$Horrat_R$	4.4	2.5	**0.8**	**0.5**	0.3	0.2

Table: Data obtained for a 200 mL distillate from a 200 mL sample.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
No. of laboratories considered	16	15	18	17	17	17
No. of repetitions	2	2	2	2	2	2
Minimum	0.17	0.52	1.27	2.41	4.30	6.01
Maximum	0.29	0.63	1.43	2.66	4.69	6.59
Overall average	0.24	0.56	1.35	2.56	4.55	6.38
Repeatability variance	0.00006	0.00003	0.00016	0.00050	0.00039	0.00135
Inter-laboratory standard deviation	0.03209	0.02496	0.03752	0.07013	0.12167	0.17621
Reproducibility variance	0.001	0.001	0.001	0.005	0.015	0.031
Repeatability standard deviation	0.01	0.01	0.01	0.02	0.02	0.04
r limit	0.02	0.02	0.04	0.06	0.06	0.104

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Repeatability CV	3.2	1.0	0.9	0.9	0.4	0.6
Reproducibility standard deviation	0.033	0.025	0.039	0.072	0.122	0.178
R limit	0.092	0.071	0.109	0.203	0.347	0.504
Reproducibility CV	13.8	4.5	2.9	2.8	2.7	2.8
Horwitz RSD_r	3.27	2.88	2.52	2.29	2.10	2.00
$Horrat_r$	**1.0**	0.4	0.4	0.4	0.2	0.3
Horwitz RSD_R	4.96	4.36	3.82	3.47	3.18	3.03
$Horrat_R$	2.8	1.0	**0.8**	**0.8**	**0.9**	**0.9**

Table: Data obtained for a 100 mL distillate from a 200 mL sample.

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