



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 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.

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The General Director of the OIV
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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.

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.

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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 |
| 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

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| Laboratory | Sample No. 1 | | Sample No.° 2 | | Sample No. 3 | | Sample No. 4 | | Sample No. 5 | | Sample No. 6 | |
|------------|--------------|------------|---------------|-------------|--------------|-------------|--------------|-------------|--------------|-------------|--------------|-------------|
| | 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 |
| 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.

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| Laboratory | Sample No. 1 | | Sample No. 2 | | Sample No. 3 | | Sample No. 4 | | Sample No. 5 | | Sample No. 6 | |
|------------|--------------|-------------|--------------|-------------|--------------|-------------|--------------|------------|--------------|-------------|--------------|-------------|
| | 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 |
| 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.

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| | 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 |
| 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.

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| | 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 |
| 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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