# OIV-MA-BS-04 Reference method for the determination of real alcoholic strength by volume of spirit drinks of viti-vinicultural origin: measurement by electronic densimetry (based on the resonant frequency oscillation of a sample in an oscillating cell)

**Type II method**

1. **Principle**

The liquid's density is determined by electronic measurement of the oscillations of a vibrating U-tube. To perform this measurement, the sample is added to an oscillating system, whose specific oscillation frequency is thus modified by the added mass.

2. **Reagents and Materials**

During the analysis, unless otherwise is stated, use only reagents of recognised analytical grade and water of at least grade 3 as defined in ISO 3696:1987.

2.1. Acetone (CAS 666-52-4) or absolute alcohol

2.2. Dry air.

3. **Apparatus and Equipment**

Usual laboratory apparatus and in particular the following.

3.1. Digital display densimeter

Electronic densimeter for performing such measurements must be capable of expressing density in g/ml to 5 decimal places.

Note 1: The densimeter should be placed on a perfectly stable stand that is insulated from all vibrations.

3.2. Temperature regulation

The densimeter's performance is valid only if the measuring cell is connected to a built-in temperature regulator that can achieve the same temperature stability of ± 0.02 °C or better.

Note 2: The precise setting and monitoring of the temperature in the measuring cell are very important, for an error of 0.1 °C can lead to a variation in density of the order of 0.0001 g/mL.

3.3. Sample injection syringes, auto sampler, or other equivalent system.

4. **Procedure**

4.1. Calibration of the densimeter

The apparatus must be calibrated according to the instrument manufacturer's instructions when it is first put into service. It must be recalibrated regularly and checked against a certified reference standard or an internal laboratory reference solution based on a certified reference standard.

4.2. Determination of sample density

4.2.1. If required prior to measurement clean and dry the cell with acetone or absolute alcohol and dry air. Rinse the cell with the sample.

4.2.2. Inject the sample into the cell (using a syringe, autosampler, or other equivalent system) so that the cell is completely filled. During the filling operation make sure that all air bubbles are completely eliminated. The sample must be homogeneous and must not contain any solid particles. Any suspended matter should be removed by filtration prior to analysis.

4.2.3. Once the reading has stabilised, record the density $\rho_{20}$ or the alcoholic strength displayed by the densimeter.

4.3. Result:

When the density $\rho_{20}$ is used, calculate the real alcoholic strength using the alcoholic strength tables identified below:

The table giving the value of the alcoholic strength by volume (% vol.) at 20 °C as a function of the density at 20 °C of water-alcohol mixtures is the international table adopted by the International Legal Metrology Organisation in its Recommendation No. 22 (Table IVa).

## 5. **Method performance characteristics (Precision)**

5.1. Statistical results of the interlaboratory test

The following data were obtained from an international method performance study carried out on a variety of spirit drinks to internationally agreed procedures.

| | | | |
|---|---|---|---|
| Year of interlaboratory test | | 1997 | |
| Number of laboratories | | 16 | |
| Number of samples | | 6 | |
| **Samples** | **A** | **B** | **C** |

| | | | |
|---|---|---|---|
| Number of laboratories retained after eliminating outliers | 11 | 13 | 15 |
| Number of outliers (Laboratories) | 2 | 3 | 1 |
| Number of accepted results | 22 | 26 | 30 |
| Mean value $(\overline{X})$% *vol.* | 23.81 | 40.12 | 40.35 |
| | 26.52* | | |
| Repeatability standard deviation ($s_r$) % vol. | 0.044 | 0.046 | 0.027 |
| Repeatability relative standard deviation ($RSD_r$) (%) | 0.17 | 0.12 | 0.07 |
| Repeatability limit ( r ) % vol. | 0.12 | 0.13 | 0.08 |
| Reproducibility standard deviation ($s_R$) % vol. | 0.054 | 0.069 | 0.083 |
| Reproducibility relative standard deviation ($RSD_R$) (%) | 0.21 | 0.17 | 0.21 |
| Reproducibility limit ( R ) % vol. | 0.15 | 0.19 | 0.23 |

Sample types
A Fruit liqueur ; split level*
B Brandy ; blind duplicates
C Whisky ; blind duplicates

| **Samples** | **D** | **E** | **F** |
|---|---|---|---|
| Number of laboratories retained after eliminating outliers | 16 | 14 | 13 |
| Number of outliers (Laboratories) | - | 1 | 2 |
| Number of accepted results | 32 | 28 | 26 |
| Mean value $(\overline{X})$% *vol.* | 39.27 | 42.39 | 56.99 |
| | 43.10* | 45.91* | 63.31* |

| | | | |
|---|---|---|---|
| Repeatability standard deviation ($s_r$) % vol. | 0.079 | 0.172 | 0.144 |
| Repeatability relative standard deviation ($RSD_r$) (%) | 0.19 | 0.39 | 0.24 |
| Repeatability limit ( r ) % vol. | 0.22 | 0.48 | 0.40 |
| Reproducibility standard deviation ($s_R$) % vol. | 0.141 | 0.197 | 0.205 |
| Reproducibility relative standard deviation ($RSD_R$) (%) | 0.34 | 0.45 | 0.34 |
| Reproducibility limit ( R ) % vol. | 0.40 | 0.55 | 0.58 |

Sample types

D Grappa; split level*

E Aquavit; split level*

F Rum; split level*