OIV-MA-BS-05 Reference method for the determination of real alcoholic strength by volume of spirit drinks of viti-vinicultural origin: Measurement by densimetry using hydrostatic balance

Type II method

1. **Principle**

The alcoholic strength of spirits can be measured by densimetry using a hydrostatic balance based on Archimedes' principle according to which a body immersed in a liquid receives a vertical upward thrust from the liquid equal to the weight of liquid displaced.

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. Float cleaning solution (sodium hydroxide, 30 % w/v)

To prepare 100 ml weigh 30 g sodium hydroxide and make up to volume using 96 % volume ethanol.

3. **Apparatus and Equipment**

Usual laboratory apparatus and in particular the following.

3.1. Single-pan hydrostatic balance with a sensitivity of 1 mg.

3.2. Float with a volume of at least 20 ml, specially adapted to the balance, suspended with a thread of diameter not exceeding 0.1 mm.

3.3. Measuring cylinder bearing a level mark. The float must be capable of being contained completely within the volume of the cylinder located below the mark; the surface of the liquid may only be penetrated by the supporting thread. The measuring cylinder must have an internal diameter at least 6 mm larger than that of the float.

3.4. Thermometer (or temperature-measuring probe) graduated in degrees and tenths of a degree from 10 to 40 °C, calibrated to 0.05 °C.

Weights, calibrated by a recognised certifying body.

Note 1: Use of a twin-pan balance is also possible; the principle is described in the Manual of Analysis Methods for Wines of the OIV.

4. **Procedure**

The float and measuring cylinder must be cleaned between each measurement with distilled water, dried with soft laboratory paper which does not shed fibres and rinsed with the solution whose density is to be determined. Measurements must be made as soon as the apparatus has reached stability so as to restrict alcohol loss by evaporation.

4.1. Calibration of the balance

Although balances usually have an internal calibration system, the hydrostatic balance must be capable of calibration with weights checked by an official certifying body.

4.2. Calibration of the float

4.2.1. Fill the measuring cylinder to the mark with double-distilled water (or water of equivalent purity, e.g. microfiltered water with a conductivity of 18.2 M□/cm) at a temperature between 15 °C and 25 °C but preferably at 20 °C.

4.2.2. Immerse the float and the thermometer, stir, read off the density of the liquid from the apparatus and, if necessary, correct the reading so that it is equal to that of the water at measurement temperature.

4.3. Control using a water-alcohol solution

4.3.1. Fill the measuring cylinder to the mark with a water-alcohol mixture of known strength at a temperature between 15 °C and 25 °C but preferably at 20 °C.

4.3.2. Immerse the float and the thermometer, stir, read off the density of the liquid (or the alcoholic strength if this is possible) from the apparatus. The alcoholic strength thus established should be equal to the previously determined alcoholic strength.

Note 2: This solution of known alcoholic strength can also be used to calibrate the float instead of double-distilled water.

4.4. Measurement of the density of a distillate (or of its alcoholic strength if the apparatus allows)

4.4.1. Pour the test sample into the measuring cylinder up to the graduation mark.

4.4.2. Immerse the float and the thermometer, stir, read off the density of the liquid (or the alcoholic strength if this is possible) from the apparatus. Note the temperature if the density is measured at t °C ($□_t$).

4.4.3. Correct $□_t$ to 20 using the table of densities □T for water-alcohol mixtures in the Manual of Analysis Methods for Wines of the OIV.

4.5. Cleaning of float and measuring cylinder

4.5.1. Immerse the float in the float cleaning solution in the measuring cylinder.

4.5.2. Allow to soak for one hour spinning the float periodically.

4.5.3. Rinse with copious amounts of tap water followed by distilled water.

4.5.4. Dry with soft laboratory paper which does not shed fibres.

Carry out this procedure when the float is first used and then regularly as required.

4.6. Result

Using the density ρ_{20} 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.

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		12	
Number of samples		6	
Samples	**A**	**B**	**C**
Number of laboratories retained after eliminating outliers	12	10	11
Number of outliers (Laboratories)	-	2	1
Number of accepted results	24	20	22
Mean value ($\overline{X}$)%vol.	23.80	40.09	40.29
	26.51*		

Repeatability standard deviation (s_r) % vol.	0.048	0.065	0.042
Repeatability relative standard deviation (RSD_r) (%)	0.19	0.16	0.10
Repeatability limit (r) % vol.	0.13	0.18	0.12
Reproducibility standard deviation (s_R) % vol.	0.060	0.076	0.073
Reproducibility relative standard deviation (RSD_R) (%)	0.24	0.19	0.18
Reproducibility limit (R) % vol.	0.17	0.21	0.20

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	12	11	9
Number of outliers (Laboratories)	-	1	2
Number of accepted results	24	22	18
Mean value ($\overline{X}$)%$vol.$	39.26	42.38	57.16
	43.09*	45.89*	63.44*
Repeatability standard deviation (s_r) % vol.	0.099	0.094	0.106
Repeatability relative standard deviation (RSD_r) (%)	0.24	0.21	0.18
Repeatability limit (r) % vol.	0.28	0.26	0.30
Reproducibility standard deviation (s_R) % vol.	0.118	0.103	0.125
Reproducibility relative standard deviation (RSD_R) (%)	0.29	0.23	0.21

Reproducibility limit (R)	% vol.	0.33	0.29	0.35

Sample types

D Grappa; split level*

E Aquavit; split level*

F Rum; split level*