



# **OIV-OENO 601A-2021 Revision of the method OIV-MA-AS2-01A 'Density and specific gravity at 20°C**

THE GENERAL ASSEMBLY,

IN VIEW OF the Article 2, paragraph 2 b) iv of the Agreement of 3<sup>rd</sup> April 2001 establishing the International Organisation of Vine and Wine,

AT THE PROPOSAL of the “Methods of Analysis” Sub-Commission,

CONSIDERING that inconsistencies have been noted between the methods relative to density (OIV-MA-AS2-01A) and alcoholic strength by volume (OIV-MA-AS312-01A and OIV-MA-AS312-01B),

CONSIDERING that this mainly consists of the task of restructuring the two methods present in the *Compendium of International Methods of Analysis of Wines and Musts*,  
DECIDES to restructure the methods OIV-MA-AS2-01A and OIV-MA-AS2-01B, giving to the method the new nomenclature OIV-MA-AS2-01 : 2021, and to merge them in one method as follows:

**Density and specific gravity at 20°C**

## **Type I and IV methods**

### **1. Scope of application**

This resolution is applicable for determining the density and specific gravity at 20 °C of wines and musts, using any of the following:

- Pycnometry: Type I Method,
- Electronic densimetry using a frequency oscillator: Type I Method,
- Densimetry using a hydrostatic balance: Type I Method,
- Hydrometry: Type IV Method.

### **2. Definition**

Density is the quotient of the mass of a certain volume of wine or must at 20 °C by this volume. It is expressed in g/cm<sup>3</sup> and its symbol is  $\rho_{20^{\circ}\text{C}}$ .

The specific gravity is the ratio of the density of a substance to the density of a reference material. For the analysis of wine or must, it is typically expressed as the

ratio of the density of the wine or must at 20 °C to the density of water at 20 °C. Its

symbol is:  $d_{20}^{20^{\circ}\text{C}}$  or  $d_{20^{\circ}\text{C}}^{20^{\circ}\text{C}}$

*Note:* It is possible to obtain the specific gravity from the density  $\rho_{20}$  at 20 °C:

$\rho_{20} = 0.998203 \times d_{20^{\circ}\text{C}}^{20^{\circ}\text{C}}$  or  $d_{20^{\circ}\text{C}}^{20^{\circ}\text{C}} = \rho_{20} / 0.998203$  (where 0.998203 is the density of water at 20 °C in g/ cm<sup>3</sup>)

### 3. Principle of the methods

The principle of each method is detailed in the following parts:

Method A: Pycnometry

Method B: Electronic densimetry using a frequency oscillator

Method C: Densimetry using a hydrostatic balance

Method D: Hydrometry

*Note:* For very precise determinations, the density should be corrected to account for sulphur-dioxide action.

$$\rho_{20} \text{ (g/cm}^3\text{)} = \rho'_{20} - 0.0006 \times S$$

$\rho_{20}$  (g/cm<sup>3</sup>) = corrected density

$\rho'_{20}$  (g/cm<sup>3</sup>) = observed density

S (g/L) = total sulphur dioxide

### 4. Preliminary sample preparation

If the wine or must contains notable quantities of carbon dioxide, remove the grand majority by, for example, mixing 250 mL of sample in a 1000-mL vial, or by filtering under reduced pressure on 2 g of cotton placed in an extension tube, or by any other suitable method.

#### Method A: Density at 20 °C and specific gravity at 20 °C measured by pycnometry (Type method)

##### A.1. Principle

The density of the wine or must is measured for a specific temperature using a glass pycnometer. This comprises a flask of known capacity, onto which a hollow ground-glass stopper is fitted equipped with a capillary tube. When the flask is closed, the overflow rises in the capillary. The volumes of the flask and the capillary being known, the density is determined by weighing using precision balances before and after filling of the pycnometer.

##### A.2. Reagents and products

A.2.1. Type II water for analytical use (ISO 3696 standard), or of equivalent purity

A.2.2. Sodium chloride solution (2% m/v)

To prepare 1 litre, weigh out 20 g of sodium chloride and dissolve to volume in water.

### A.3. Apparatus and materials

Current laboratory apparatus, including the following:

A.3.1. Pyrex-glass pycnometer of around 100 mL capacity with a removable thermometer, with ground-glass joint and 10<sup>th</sup>-of-a-degree graduations, from 10 °C to 30 °C. This thermometer should be calibrated (Fig. 1).

Any pycnometer of equivalent characteristics may be used.

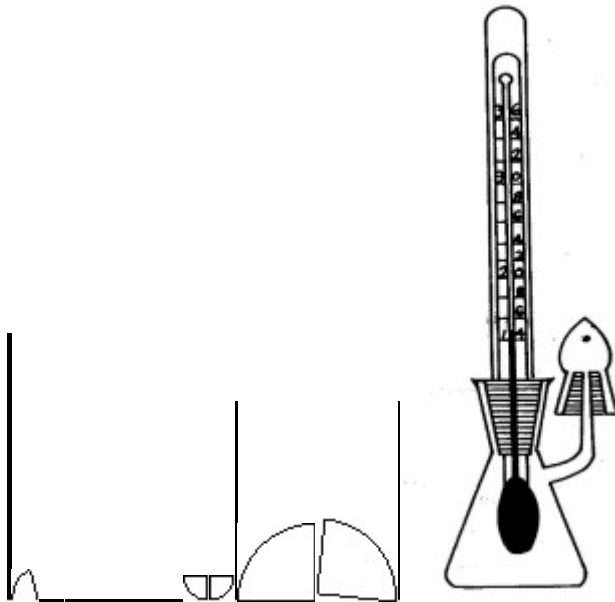


FIGURE 1: Pycnometer and its tare bottle

This pycnometer includes a side tube of 25 mm in length and an inside diameter of at most 1 mm, terminated by a ground-glass conical joint. This side tube may be capped by a 'reservoir stopper' composed of a ground-glass conical tube, terminated by a tapered joint. This stopper serves as an expansion chamber.

The two joints of the apparatus should be prepared with great care.

A.3.2. Tare bottle of the same external volume (to within 1 mL) as the pycnometer and with a mass equal to the mass of the pycnometer filled with a liquid of a density of 1.01 g/mL (sodium chloride solution at 2% m/v)

A.3.3. Thermally insulated jacket that fits the body of the pycnometer exactly.

A.3.4. Twin-pan balance accurate to the nearest 0.1 mg

or

single-plate balance accurate to the nearest 0.1 mg.

A.3.5. Masses calibrated by an accredited body

#### A.4. Procedure

A.4.1. Pycnometer calibration

The calibration of the pycnometer comprises the determination of the following characteristics:

- tare weight,
- volume at 20 °C,
- water mass at 20 °C.

##### 1. *Using a twin-pan balance*

Place the tare bottle on the left-hand pan and the clean, dry pycnometer with its 'reservoir stopper' on the right-hand pan. Balance them by placing weights of known mass on the pycnometer side:  $p$  grams.

Fill the pycnometer carefully with water (A.2.1) at room temperature and fit the thermometer.

Carefully wipe the pycnometer dry and place it in the thermally insulated jacket.

Shake by inverting the container until the thermometer's temperature reading is constant, accurately adjust the level to the upper rim of the side tube, wipe the side tube clean and fit the reservoir stopper.

Read the temperature,  $t$  °C, carefully and if necessary correct for any inaccuracies in the temperature scale.

Weigh the water-filled pycnometer, with the weight in grams,  $p'$ , making up the equilibrium.

Calculations:

Tare of the empty pycnometer:

Tare weight =  $p + m$  where  $m$  (g) = mass of the air contained in the pycnometer

- $m$  (g) =  $0.0012 (p - p')$

Volume at 20 °C in mL:

- $V_{20^{\circ}\text{C}}$  (mL) =  $(p + m - p') \times F_t$



$F_t$  = factor for temperature,  $t^{\circ}\text{C}$ , taken from Table I

$V_{20^{\circ}\text{C}}$  should be known to  $\pm 0.001$  mL

Water mass at  $20^{\circ}\text{C}$ :

- $M_{20^{\circ}\text{C}} (\text{g}) = V_{20^{\circ}\text{C}} \times 0.998203$
- $0.998203 (\text{g}/\text{cm}^3) = \text{water density at } 20^{\circ}\text{C}$

### 2. Using a single-pan balance

Determine:

- the mass of the clean, dry pycnometer:  $P$ ,
- the mass of the water-filled pycnometer at  $t^{\circ}\text{C}$ :  $P_1$  following the instructions outlined in A.4.1.1,
- the mass of the tare bottle,  $T_0$ .

Calculations:

Tare of the empty pycnometer:

Tare weight:  $P - m$  where  $m (\text{g}) = \text{mass of the air contained in the pycnometer}$

- $m (\text{g}) = 0.0012 (P_1 - P)$

Volume at  $20^{\circ}\text{C}$  in mL:

- $V_{20^{\circ}\text{C}} (\text{mL}) = [P_1 - (P - m)] \times F_t$

$F_t$  = factor for temperature,  $t^{\circ}\text{C}$ , taken from Table I

$V_{20^{\circ}\text{C}}$  should be known to  $\pm 0.001$  mL

Water mass at  $20^{\circ}\text{C}$ :

- $M_{20^{\circ}\text{C}} (\text{g}) = V_{20^{\circ}\text{C}} \times 0.998203$
- $0.998203 = \text{water density at } 20^{\circ}\text{C} (\text{g}/\text{cm}^3)$

A.4.2. Determination of the density:

A.4.2.1. *Using a twin-pan balance*

Weigh the pycnometer filled with the test sample following the instructions outlined in A.4.1.1.

Where  $p''$  represents the mass in grams that makes up the equilibrium at  $t^{\circ}\text{C}$ , taking into account that the liquid mass contained in the pycnometer =  $p + m - p''$ , the

apparent density at  $t^{\circ}\text{C}$ , in  $\text{g}/\text{cm}^3$ , is given by the following equation:

$$\rho_{t^{\circ}\text{C}} = \frac{p + m - p''}{V_{20^{\circ}\text{C}}}$$

Calculate the density at  $20^{\circ}\text{C}$  using one of the following correction tables in Annex I, according to the nature of the liquid to be analysed and the type of pycnometer to be used: dry wine and dealcoholized wine (Table II or V), natural or concentrated must (Table III or VI), or liqueur wine (Table IV or VII).

A.4.2.2. *Using a single-pan balance*

Weigh the tare bottle, where  $T_1$  is its mass in g.

Calculate  $dT = T_1 - T_0$

Mass of the empty pycnometer at the time of measurement =  $P - m + dT$  in g

Weigh the pycnometer filled with the test sample following the instructions outlined in A.4.1.1.

Where  $P_2$  represents its mass at  $t^{\circ}\text{C}$ ,

the liquid mass contained in the pycnometer at  $t^{\circ}\text{C} = P_2 - (P - m + dT)$  in g

and the apparent density at  $t^{\circ}\text{C}$ , in  $\text{g}/\text{cm}^3$ , is as follows

$$\rho_{t^{\circ}\text{C}} = \frac{P_2 - (P - m + dT)}{V_{20^{\circ}\text{C}}}$$

Calculate the density at  $20^{\circ}\text{C}$  of the liquid to be analysed: dry wine, natural or concentrated must, or liqueur wine, as indicated in A.4.2.1.

## A.5. Expression of results

The density is expressed in  $\text{g}/\text{cm}^3$  to 5 decimal places.

## A.6. Precision

A.6.1. Repeatability in terms of density:

- for dry and sweet wines, except liqueur wines:  $r = 0.00010 \text{ g}/\text{cm}^3$ ,
- for liqueur wines:  $r = 0.00018 \text{ g}/\text{cm}^3$ .

2. Reproducibility in terms of density:

- for dry and sweet wines, except liqueur wines:  $R = 0.00037 \text{ g}/\text{cm}^3$ ,

- for liqueur wines:  $R = 0.00045 \text{ g/cm}^3$ .

#### A.7. Numerical example

##### A.7.1. Measurement by pycnometer on a twin-pan balance

###### A/Calibration of the pycnometer

###### 1. Weighing of the clean, dry pycnometer:

- Tare = pycnometer +  $p$
- $\rho = 104.9454 \text{ g}$

###### 2. Weighing of the water-filled pycnometer at the temperature $t^\circ\text{C}$ :

- Tare = pycnometer + water +  $p'$
- $p' = 1.2396 \text{ g}$  for  $t = 20.5^\circ\text{C}$

###### 3. Calculation of the mass of the air contained in the pycnometer:

- $m = 0.0012 (p - p')$
- $m = 0.0012 (104.9454 - 1.2396)$
- $m = 0.1244$

###### 4. Parameters to be kept:

- Tare of the empty pycnometer:  $p + m$
- $p + m = 104.9454 + 0.1244$
- $p + m = 105.0698 \text{ g}$
- Volume at  $20^\circ\text{C} = (p + m - p') \times F_{t^\circ\text{C}}$
- $F_{20.50^\circ\text{C}} = 1.001900$
- $V_{20^\circ\text{C}} = (105.0698 - 1.2396) \times 1.001900$
- $V_{20^\circ\text{C}} = 104.0275 \text{ mL}$
- Water mass at  $20^\circ\text{C} = V_{20^\circ\text{C}} \times 0.998203$
- $M_{20^\circ\text{C}} = 103.8405 \text{ g}$

###### B/ Determination of the density at $20^\circ\text{C}$ and the $20^\circ\text{C}/20^\circ\text{C}$ specific gravity of a dry wine:

$p' = 1.2622 \text{ g}$  at  $17.80^\circ\text{C}$



$$\rho_{17.80\text{ }^{\circ}\text{C}} = \frac{105.0698 - 1.2622}{104.0275}$$

- $\rho_{17.80\text{ }^{\circ}\text{C}} = 0.99788\text{ g/cm}^3$

Table II makes it possible to calculate  $\rho_{20\text{ }^{\circ}\text{C}}$  from  $\rho_{t\text{ }^{\circ}\text{C}}$  using the following formula:

$$\rho_{20\text{ }^{\circ}\text{C}} = \rho_{t\text{ }^{\circ}\text{C}} \pm \frac{c}{1000}$$

For  $t = 17.80\text{ }^{\circ}\text{C}$  and for an alcoholic strength of 11% vol.,  $c = 0.54$ :

$$\rho_{20\text{ }^{\circ}\text{C}} = 0.99788 - \frac{0.54}{1000}$$

$$\rho_{20\text{ }^{\circ}\text{C}} = 0.99734\text{ g/cm}^3$$

$$d_{20\text{ }^{\circ}\text{C}}^{20\text{ }^{\circ}\text{C}} = \frac{0.99734}{0.998203} = 0.99913$$

#### A.7.1.2. Measurement by pycnometer on a single-pan balance

##### A/ Establishment of the pycnometer constants

##### 1. Weighing of the clean, dry pycnometer:

- $P = 67.7913\text{ g}$

##### 2. Weighing of the water-filled pycnometer at $t^{\circ}\text{C}$ :

- $P_1 = 169.2715\text{ g}$  at  $21.65^{\circ}\text{C}$

##### 3. Calculation of the mass of the air contained in the pycnometer:

- $m = 0.0012 (P_1 - P)$

- $m = 0.0012 \times 101.4802$

- $m = 0.1218\text{ g}$

##### 4. Characteristics to be retained:

- Tare of the empty pycnometer:  $P - m$

- $P - m = 67.7913 - 0.1218$

- $P - m = 67.6695\text{ g}$



- Volume at 20 °C =  $[P_1 - (P - m)] \times F_{t^{\circ}C}$
- $F_{21.65^{\circ}C} = 1.002140$
- $V_{20^{\circ}C} = (169.2715 - 67.6695) \times 1.002140$
- $V_{20^{\circ}C} = 101.8194$  mL
- Water mass at 20 °C:  $V_{20^{\circ}C} \times 0.998203$
- $M_{20^{\circ}C} = 101.6364$  g
- Mass of the tare bottle:  $T_0$
- $T_0 = 171.9160$  g

B/Determination of the density at 20 °C and 20 °C/20 °C specific gravity of a dry wine:

$$T_1 = 171.9178$$

$$dT = 171.9178 - 171.9160 = 0.0018 \text{ g}$$

$$P - m + dT = 67.6695 + 0.0018 = 67.6713 \text{ g}$$

$$P_2 = 169.2799 \text{ at } 18^{\circ}C$$

$$\rho^{18^{\circ}C} = \frac{169.2799 - 67.6713}{101.8194}$$

$$\rho^{18^{\circ}C} = 0.99793 \text{ g/cm}^3$$

$$d_{20^{\circ}C}^{20^{\circ}C} = \frac{0.99734}{0.998203} = 0.99913$$

#### A.7.2. Measurement by pycnometer on a single-pan balance

##### A/ Establishment of the pycnometer constants

##### 1. Weighing of the clean, dry pycnometer:

- $P = 67.7913$  g

##### 2. Weighing of the water-filled pycnometer at $t^{\circ}C$ :

- $P_1 = 169.2715$  g at  $21.65^{\circ}C$

##### 3. Calculation of the mass of the air contained in the pycnometer:

- $m = 0.0012 (P_1 - P)$

- $m = 0.0012 \times 101.4802$
- $m = 0.1218 \text{ g}$

#### 4. Characteristics to be retained:

- Tare of the empty pycnometer:  $P - m$
- $P - m = 67.7913 - 0.1218$
- $P - m = 67.6695 \text{ g}$
- Volume at 20 °C =  $[P_1 - (P - m)] \times F_{t^{\circ}\text{C}}$
- $F_{21.65^{\circ}\text{C}} = 1.002140$
- $V_{20^{\circ}\text{C}} = (169.2715 - 67.6695) \times 1.002140$
- $V_{20^{\circ}\text{C}} = 101.8194 \text{ mL}$
- Water mass at 20°C:  $V_{20^{\circ}\text{C}} \times 0.998203$
- $M_{20^{\circ}\text{C}} = 101.6364 \text{ g}$
- Mass of the tare bottle:  $T_0$
- $T_0 = 171.9160 \text{ g}$

B/ Determination of the density at 20 °C and 20 °C/20 °C specific gravity of a dry wine:

- $T_1 = 171.9178$
- $dT = 171.9178 - 171.9160 = 0.0018 \text{ g}$
- $P - m + dT = 67.6695 + 0.0018 = 67.6713 \text{ g}$
- $P_2 = 169.2799 \text{ at } 18^{\circ}\text{C}$

$$\rho^{18^{\circ}\text{C}} = \frac{169.2799 - 67.6713}{101.8194}$$

$$\rho^{18^{\circ}\text{C}} = 0.99793 \text{ g/cm}^3$$

**Method B: Density at 20 °C and specific gravity at 20 °C measured by electronic densimetry using a frequency oscillator (Type I method)**

## B.1. Principle

The density of the wine or must is measured by electronic densimetry using a frequency oscillator. The principle consists of measuring the period of oscillation of a tube containing the sample undergoing electromagnetic stimulation. The density is related to the period of oscillation by the following formula:

$$\rho = T^2 \times \left( \frac{C}{4\pi^2 V} \right) - \left( \frac{M}{V} \right) (1)$$

$\rho$  = density of the sample

T = period of induced vibration

M = mass of empty tube

C = spring constant

V = volume of vibrating sample

This relationship is in the form  $\rho = A T^2 - B$ (2), so there is a linear relationship between the density and the period squared. The constants A and B are specific to each oscillator and are estimated by measuring the period of fluids of known density.

## B.2. Reagents and products

### B.2.1. Reference fluids

Two reference fluids are used to adjust the densimeter. The densities of the reference fluids should encompass the densities of the wines or musts to be analysed. A spread of greater than 0.01000 g/cm<sup>3</sup> between the densities of the reference fluids is recommended.

The reference fluids used to measure the density of the wines or musts by electronic densimetry are as follows:

- dry air (unpolluted),
- Type II water for analytical usage (ISO standard 3696), or of equivalent analytical purity,
- hydro-alcoholic solutions, wines or musts whose densities have been determined by a different Type I method, for which the uncertainty does not exceed 0.00005 g/cm<sup>3</sup> at the temperature of 20.00 ± 0.05 °C,
- solutions calibrated with traceability to the International System of Units, with viscosities of less than 2 mm<sup>2</sup>/s, for which the uncertainty does not exceed 0.00005 g/cm<sup>3</sup> at the temperature of 20.00 ± 0.05 °C.

### B.2.2. Cleaning and drying products

Use products that ensure the perfectly clean and dried state of the measuring cell, according to the residues and manufacturer's indications. For example:

- detergents, acids, etc.,
- organic solvents: 96% vol. ethanol, pure acetone, etc.

### B.3. Apparatus and equipment

#### B.3.1. Electronic densimeter with frequency oscillator

The electronic densimeter consists of the following elements:

- a measuring cell consisting of a measuring tube and a temperature controller,
- a system for setting up an oscillation tube and measuring the period of oscillation,
- a digital display and possibly a calculator,
- sample injector syringe, autosampler or other equivalent system.

The densimeter is placed on a perfectly stable support isolated from all vibrations.

#### B.3.2. Temperature control of the measuring cell

Locate the measuring tube in a temperature-controlled system. Temperature stability should be better than  $\pm 0.02$  °C.

It is necessary to control the temperature of the measuring cell when the densimeter makes this possible, because this strongly influences the determination results. The density of a hydro-alcoholic solution with an alcoholic strength by volume (ABV) of 10% vol. is  $0.98471$  g/cm<sup>3</sup> at 20 °C and  $0.98447$  g/cm<sup>3</sup> at 21 °C, equating to a spread of  $0.00024$  g/cm<sup>3</sup>.

The test temperature is 20 °C. Measure the cell temperature with a resolution thermometer accurate to less than 0.01 °C and with traceability to national standards. This should enable a temperature measurement with an uncertainty of better than  $\pm 0.07$  °C.

#### B.3.3. Calibration of the apparatus

The apparatus should be calibrated before using it for the first time, then periodically or if the verification is not satisfactory. The objective is to use two reference fluids to calculate the constants A and B [see formula (2), B.1]. To carry out the calibration in practice, refer to the user manual of the apparatus. In principle, this calibration is

carried out with dry air (taking into account the atmospheric pressure) and very pure water (B.2.1).

#### B.3.4. Calibration verification

In order to verify the calibration, the density of the reference fluids is measured.

Every day of use, a density check of the air is carried out. A difference between the theoretical density and observed density of more than  $0.00008 \text{ g/cm}^3$  may indicate that the tube is clogged. In that case, it should be cleaned. After cleaning, verify the air density again. If the verification is not conclusive, adjust the apparatus.

Check the density of the water; if the difference between the theoretical density and the density observed is greater than  $0.00008 \text{ g/cm}^3$ , adjust the apparatus.

If verification of the cell temperature is difficult, it is possible to directly check the density of a hydro-alcoholic solution of comparable density to those of the samples analysed.

#### B.3.5. Checks

When the difference between the theoretical density of the reference solution (known with an uncertainty of  $\pm 0.00005 \text{ g/cm}^3$ ) and the measured density is above  $0.00008 \text{ g/cm}^3$ , the calibration of the apparatus should be checked.

### B.4. Procedure

Before measuring, if necessary, clean and dry the cell with acetone or absolute alcohol and dry air. Rinse the cell with the sample.

Inject the sample into the cell (using a syringe, autosampler or other equivalent system) so that it is filled completely. While filling, check that all air bubbles have been removed. The sample should be homogenous and not contain any solid particles. Where necessary, filter to remove any suspended matter before analysis.

If there is a lighting system available that makes it possible to verify the absence of bubbles, turn it off quickly after checking because the heat generated by the lamp can influence the measuring temperature (for apparatus with a permanent lighting system, this statement is not applicable).

The operator should ensure that the temperature of the measuring cell is stable.

Once the reading has been stabilised, record the density,  $\rho_{20\text{°C}}$ .

If the apparatus only provides the period, the density can be calculated from the A and B constants (refer to the instructions for the equipment or Annex I of the method OIV-MA-AS312-01A).

### B.5. Expression of results

The density is expressed in  $\text{g}/\text{cm}^3$  to 5 decimal places.

### B.6. Precision parameters

The precision parameters are detailed in Table 4 of Annex II.

Repeatability:

- $r = 0.00011 \text{ g}/\text{cm}^3$

Reproducibility:

- $R = 0.00025 \text{ g}/\text{cm}^3$

## Method C: Density at 20 °C and specific gravity at 20 °C measured using a hydrostatic balance (Type I Method)

### C.1. Principle

The density of wine or musts can be measured by densimetry with a hydrostatic balance following the Archimedes principle, by which any body immersed in a fluid experiences an upwards force equal to the weight of the displaced fluid.

### C.2. Reagents and products

C.2.1. Type II water for analytical usage (ISO 3696 standard), or of equivalent purity

C.2.2. Floater-washing solution (sodium hydroxide, 30 % m/v)

To prepare a 100-mL solution weigh 30 g of sodium hydroxide and fill using 96% vol. ethanol.

### C.3. Apparatus and materials

Normal laboratory apparatus, particularly:

C.3.1. Single-pan hydrostatic balance accurate to the nearest 1 mg

C.3.2. Floater with at least 20 mL volume, specifically adapted for the balance, suspended by a thread with a diameter of less than or equal to 0.1 mm

C.3.3. Cylindrical test tube with level indicator. The floater should be able to fit entirely within the test tube volume below the level indicator; only the hanging thread should break the surface of the liquid. The cylindrical test tube should have an inside diameter at least 6 mm greater than that of the floater.

C.3.4. Thermometer (or temperature-measurement probe) with degree and  $10^{\text{th}}$ -of-

a-degree graduations, from 10°C to 40°C, calibrated to  $\pm 0.06$  °C

C.3.5. Masses calibrated by an accredited body.

#### C.4. Procedure

After each measurement, the floater and the test tube should be cleaned with distilled water, wiped with soft laboratory paper that does not lose its fibres and rinsed with solution whose density is to be determined. These measurements should be carried out once the apparatus has reached a stable level in order to limit alcohol loss through evaporation.

C.4.1. Calibration of the apparatus

C.4.1.1. Balance calibration

While balances usually have internal calibration systems, hydrostatic balances should be calibrated with weights with traceability to the International System of Units.

C.4.1.2. Floater calibration

Fill the cylindrical test tube up to the level indicator with water (C.2.1) whose temperature is between 15 °C and 25 °C, but preferably at 20°C.

Plunge the floater and the thermometer into the liquid, shake, note down the density on the apparatus and, if necessary, adjust the reading in order for it to be equal to that of the water at the measurement temperature.

C.4.1.3. Verification using a solution of known density

Fill the cylindrical test tube up to the level indicator with a solution of known density at a temperature of between 15°C and 25 °C, preferably at 20°C.

Immerse the floater and the thermometer in the liquid, stir, read the density of the liquid indicated by the apparatus and record the density and the temperature where the density is measured at  $t$  °C ( $\rho_t$ ).

If necessary, correct  $\rho_t$  using a  $\rho_t$  density table of hydro-alcoholic mixtures (Table II in Annex I).

The density determined in this way should be identical to the previously determined density.

*Note:* This solution of known density can also replace water for floater calibration.

C.4.2. Determination of the density

Pour the test sample into the cylindrical test tube up to the level indicator.

Plunge the floater and the thermometer into the liquid, shake and note down the density on the apparatus. Note the temperature if the density is measured at  $t$ °C ( $\rho_t$ ).

Correct  $\rho_t$  using a  $\rho_t$  density table of hydro-alcoholic mixtures (Table II in the Annex).

C.4.3. Cleaning of the floater and cylindrical test tube

Plunge the floater into the washing solution in the test tube.

Allow to soak for one hour while turning the floater regularly.

Rinse with tap water, then with distilled water.

Wipe with soft laboratory paper that does not lose its fibres.

Carry out these operations when the floater is used for the first time and then on a regular basis when necessary.

#### **C.5. Expression of results**

The density is expressed in  $\text{g}/\text{cm}^3$  to 5 decimal places.

#### **C.6. Precision parameters**

The precision parameters are detailed in Table 4 of Annex II.

- $r = 0.00025 \text{ g}/\text{cm}^3$
- $R = 0.00067 \text{ g}/\text{cm}^3$

### **Method D: Density measured by hydrometry (Type IV Method)**

#### **D.1. Principle**

The density and specific gravity at 20 °C are determined for the test sample by hydrometry following the Archimedes principle. A weighted cylinder equipped with a graduated stem is more or less immersed into the liquid sample whose density is to be determined. The density of the liquid is read directly on the graduation of the stem at the level of the meniscus.

#### **D.2. Apparatus**

##### **D.2.1. Hydrometer**

Hydrometers should meet ISO requirements relating to their dimensions and graduations.

They should have a cylindrical body and a circular stem with a cross-section of at least 3 mm in diameter. For dry wines, they should be graduated in  $\text{g}/\text{cm}^3$  from 0.983 to 1.003, with graduation marks at every 0.001 and 0.0002 interval. All of the marks at 0.001 intervals should be separated from the next by at least 5 mm. For the measurement of the specific gravity of dealcoholized wines, liqueur wines and musts, a set of 5 hydrometers are to be used, graduated (in  $\text{g}/\text{cm}^3$ ) from 1.000-1.030; 1.030-1.060; 1.060-1.090; 1.090-1.120; 1.120-1.150. These hydrometers are to be graduated for density at 20 °C by marks and intervals of no greater than 0.001 and



0.0005, with all the marks at the 0.001 intervals being separated from the next by at least 3 mm.

These hydrometers should be graduated so that they can be read at 'top of the meniscus'. The indication of the graduation in density at 20 °C or specific gravity at 20 °C, and of the reading at the top of the meniscus, is to be given either on the graduated scale, or on a strip of paper attached to the bulb.

This apparatus should be calibrated with traceability to the International System of Units.

D.2.2. Thermometer graduated to intervals of no greater than 0.5 °C, calibrated with traceability to the International System of Units.

D.2.3. Measuring cylinder with dimensions that allow for the immersion of the thermometer and the hydrometer without contact with the sides, held vertically.

### D.3. Measurement method

Place 250mL of the test sample (4) in the measuring cylinder (D.2.3) and insert the hydrometer and thermometer. Stir the sample and wait 1 minute to allow temperature equilibration, then read the thermometer. Remove the thermometer and, after 1 minute of rest, read the apparent density at  $t^{\circ}\text{C}$  on the stem of the hydrometer.

Correct the apparent density as read at  $t^{\circ}\text{C}$  for the effect of the temperature, using the tables in Annex I applying to dry wines (Table V), natural and concentrated musts (Table VI) and liqueur wines (Table VII).

### D.4. Expression of results

The density is expressed in  $\text{g}/\text{cm}^3$  to 4 decimal places

Annexes

Annex I Tables

TABLE I

F factors by which the mass of the water in the *Pyrex pycnometer* at  $t^{\circ}\text{C}$  has to be multiplied to calculate the volume of the pycnometer at 20 °C

$t^{\circ}\text{C}$	F	$t^{\circ}\text{C}$	F	$t^{\circ}\text{C}$	F	$t^{\circ}\text{C}$	F	$t^{\circ}\text{C}$	F	$t^{\circ}\text{C}$	F	$t^{\circ}\text{C}$	F
10.0	1.000398	13.0	1.000691	16.0	1.001097	19.0	1.001608	22.0	1.002215	25.0	1.002916	28.0	1.003704
.1	1.000406	.1	1.000703	.1	1.001113	.1	1.001627	.1	1.002238	.1	1.002941	.1	1.003731
.2	1.000414	.2	1.000714	.2	1.001128	.2	1.001646	.2	1.002260	.2	1.002966	.2	1.003759
.3	1.000422	.3	1.000726	.3	1.001144	.3	1.001665	.3	1.002282	.3	1.002990	.3	1.003797



.4	1.000430	.4	1.000738	.4	1.001159	.4	1.001684	.4	1.002304	.4	1.003015	.4	1.003815
10.5	1.000439	13.5	1.000752	16.5	1.001175	19.5	1.001703	22.5	1.002326	25.5	1.003041	28.5	1.003843
.6	1.000447	.6	1.000764	.6	1.001191	.6	1.001722	.6	1.002349	.6	1.003066	.6	1.003871
.7	1.000456	.7	1.000777	.7	1.001207	.7	1.001741	.7	1.002372	3	1.003092	.7	1.003899
.8	1.000465	.8	1.000789	.8	1.001223	.8	1.001761	.8	1.002394	.8	1.003117	.8	1.003928
.9	1.000474	.9	1.000803	.9	1.001239	.9	1.001780	.9	1.002417	.9	1.003143	.9	1.003956
11.0	1.000483	14.0	1.000816	17.0	1.001257	20.0	1.001800	23.0	1.002439	26.0	1.003168	29.0	1.003984
.1	1.000492	.1	1.000829	.1	1.001273	.1	1.001819	.1	1.002462	.1	1.003194	.1	1.004013
.2	1.000501	.2	1.000842	.2	1.001286	.2	1.001839	.2	1.002485	1	1.003222	.2	1.004042
3	1.000511	3	1.000855	3	1.001306	3	1.001959	3	1.002508	3	1.003247	.3	1.004071
.4	1.000520	.4	1.000868	.4	1.001323	.4	1.001880	.4	1.002531	.4	1.003273	.4	1.004099
11.5	1.000530	14.5	1.000882	17.5	1.001340	20.5	1.001900	23.5	1.002555	26.5	1.003299	29.5	1.004128
.6	1.000540	.6	1.000895	.6	1.001357	.6	1.001920	.6	1.002578	.6	1.003326	.6	1.004158
.7	1.000550	.7	1.000909	.7	1.001374	.7	1.001941	3	1.002602	.7	1.003352	.7	1.004187
.8	1.000560	.8	1.000923	.8	1.001391	.8	1.001961	.8	1.002625	.8	1.003379	.8	1.004216
.9	1.000570	.9	1.000937	.9	1.001409	.9	1.001982	.9	1.002649	.9	1.003405	.9	1.004245
12.0	1.000580	15.0	1.000951	18.0	1.001427	21.0	1.002002	24.0	1.002672	27.0	1.003432	30.0	1.004275
.1	1.000591	.1	1.000965	.1	1.001445	.1	1.002023	.1	1.002696	.1	1.003459		
.2	1.000601	.2	1.000979	.2	1.001462	.2	1.002044	.2	1.002720	.2	1.003485		
.3	1.000612	.3	1.000993	.3	1.001480	.3	1.002065	.3	1.002745	.3	1.003513		
.4	1.000623	.4	1.001008	.4	1.001498	.4	1.002086	.4	1.002769	.4	1.003540		
12.5	1.000634	15.5	1.001022	18.5	1.001516	21.5	1.002107	24.5	1.002793	27.5	1.003567		
.6	1.000645	.6	1.001037	.6	1.001534	.6	1.002129	.6	1.002817	.6	1.003594		



.7	1.000656	.7	1.001052	.7	1.001552	.7	1.002151	.7	1.002842	.7	1.003621		
.8	1.000668	.8	1.001067	.8	1.001570	.8	1.002172	.8	1.002866	.8	1.003649		
.9	1.000679	.9	1.001082	.9	1.001589	.9	1.002194	.9	1.002891	.9	1.003676		

TABLE II

Temperature corrections, *c*, required for the density of dry wines and dealcoholised wines,

measured using a Pyrex-glass pycnometer at °C, in order to correct to 20 °C.

$$\rho_{20} = \rho_t \pm \frac{c}{1000}$$

- if t°C is lower than 20°C

+ if t°C is higher than 20°C

Alcoholic strength																										
	0	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27		

Temperature in °C	10	1.59	1.64	1.67	1.71	1.77	1.84	1.91	2.01	2.11	2.22	2.34	2.46	2.60	2.73	2.88	3.03	3.19	3.35	3.52	3.70	3.87	4.06	4.25	4.44
	11	1.48	1.53	1.56	1.60	1.64	1.70	1.77	1.86	1.95	2.05	2.16	2.27	2.38	2.51	2.63	2.77	2.91	3.06	3.21	3.36	3.53	3.69	3.86	4.03
	12	1.36	1.40	1.43	1.46	1.50	1.56	1.62	1.69	1.78	1.86	1.96	2.05	2.16	2.27	2.38	2.50	2.62	2.75	2.88	3.02	3.16	3.31	3.46	3.61
	13	1.22	1.26	1.28	1.32	1.35	1.40	1.45	1.52	1.59	1.67	1.75	1.83	1.92	2.01	2.11	2.22	2.32	2.44	2.55	2.67	2.79	2.92	3.05	3.18
	14	1.08	1.11	1.13	1.16	1.19	1.23	1.27	1.33	1.39	1.46	1.52	1.60	1.67	1.75	1.94	1.93	2.03	2.11	2.21	2.31	2.42	2.52	2.63	2.74
	15	0.92	0.96	0.97	0.99	1.02	1.05	1.09	1.13	1.19	1.24	1.30	1.36	1.42	1.48	1.55	1.63	1.70	1.78	1.86	1.95	2.03	2.12	2.21	2.30
	16	0.76	0.79	0.80	0.81	0.94	0.86	0.89	0.93	0.97	1.01	1.06	1.10	1.16	1.21	1.26	1.32	1.38	1.44	1.51	1.57	1.64	1.71	1.78	1.85
	17	0.59	0.61	0.62	0.63	0.65	0.67	0.69	0.72	0.75	0.78	0.81	0.85	0.88	0.95	0.96	1.01	1.05	1.11	1.15	1.20	1.25	1.30	1.35	1.40
	18	0.40	0.42	0.42	0.43	0.44	0.46	0.47	0.49	0.51	0.53	0.55	0.57	0.60	0.63	0.65	0.68	0.71	0.74	0.77	0.81	0.84	0.87	0.91	0.94
	19	0.21	0.21	0.22	0.22	0.23	0.23	0.24	0.25	0.26	0.27	0.28	0.29	0.30	0.32	0.33	0.34	0.36	0.37	0.39	0.41	0.42	0.44	0.46	0.47
	20																								
	21	0.21	0.22	0.22	0.23	0.23	0.24	0.25	0.26	0.27	0.28	0.29	0.30	0.31	0.32	0.34	0.36	0.37	0.38	0.40	0.41	0.43	0.44	0.46	0.48
	22	0.44	0.45	0.46	0.47	0.48	0.49	0.51	0.52	0.54	0.56	0.59	0.61	0.63	0.66	0.69	0.71	0.74	0.77	0.80	0.83	0.87	0.90	0.93	0.97
	23	0.68	0.70	0.71	0.72	0.74	0.76	0.78	0.80	0.83	0.86	0.90	0.93	0.96	1.00	1.03	1.08	1.13	1.17	1.22	1.26	1.31	1.37	1.41	1.46
	24	0.93	0.96	0.97	0.99	1.01	1.03	1.06	1.10	1.13	1.18	1.22	1.26	1.31	1.36	1.41	1.47	1.52	1.58	1.64	1.71	1.77	1.84	1.90	1.97
	25	1.19	1.23	1.25	1.27	1.29	1.32	1.36	1.40	1.45	1.50	1.55	1.61	1.67	1.73	1.80	1.86	1.93	2.00	2.08	2.16	2.24	2.32	2.40	2.48
	26	1.47	1.51	1.53	1.56	1.59	1.62	1.67	1.72	1.77	1.83	1.90	1.96	2.03	2.11	2.19	2.27	2.35	2.44	2.53	2.62	2.72	2.81	2.91	3.01
	27	1.75	1.80	1.82	1.85	1.89	1.93	1.98	2.04	2.11	2.18	2.25	2.33	2.41	2.50	2.59	2.68	2.78	2.88	2.98	3.09	3.20	3.31	3.42	3.53
	28	2.04	2.10	2.13	2.16	2.20	2.25	2.31	2.38	2.45	2.53	2.62	2.70	2.80	2.89	3.00	3.10	3.21	3.32	3.45	3.57	3.69	3.82	3.94	4.07
	29	2.34	2.41	2.44	2.48	2.53	2.58	2.65	2.72	2.81	2.89	2.99	3.09	3.19	3.30	3.42	3.53	3.65	3.78	3.92	4.05	4.19	4.33	4.47	4.61
	30	2.66	2.73	2.77	2.81	2.86	2.92	3.00	3.08	3.17	3.27	3.37	3.48	3.59	3.72	3.84	3.97	4.11	4.25	4.40	4.55	4.70	4.85	4.92	5.17

Note: This table can be used to convert the density  $d_{20}^t$  to  $d_{20}^{20}$

TABLE III

Temperature corrections,  $c$ , required for the density of natural or concentrated musts,

measured using a Pyrex-glass pycnometer at  $t$  °C, in order to correct to 20 °C

$$\rho_{20} = \rho_t \pm \frac{c}{1000}$$

- if  $t$ °C is lower than 20°C

+ if  $t$ °C is higher than 20°C

	Density																					
	1.05	1.06	1.07	1.08	1.09	1.10	1.11	1.12	1.13	1.14	1.15	1.16	1.18	1.20	1.22	1.24	1.26	1.28	1.30	1.32	1.34	1.36

	10	2.31	2.48	2.66	2.82	2.99	3.13	3.30	3.44	3.59	3.73	3.88	4.01	4.28	4.52	4.76	4.98	5.18	5.42	5.56	5.73	5.90	6.05
	11	2.12	2.28	2.42	2.57	2.72	2.86	2.99	3.12	3.25	3.37	3.50	3.62	3.85	4.08	4.29	4.48	4.67	4.84	5.00	5.16	5.31	5.45
	12	1.92	2.06	2.19	2.32	2.45	2.58	2.70	2.92	2.94	3.04	3.15	3.26	3.47	3.67	3.85	4.03	4.20	4.36	4.51	4.65	4.78	4.91
	13	1.72	1.84	1.95	2.06	2.17	2.27	2.38	2.48	2.58	2.69	2.78	2.89	3.05	3.22	3.39	3.55	3.65	3.84	3.98	4.11	4.24	4.36
	14	1.52	1.62	1.72	1.81	1.90	2.00	2.09	2.17	2.26	2.34	2.43	2.51	2.66	2.82	2.96	3.09	3.22	3.34	3.45	3.56	3.67	3.76
	15	1.28	1.36	1.44	1.52	1.60	1.67	1.75	1.82	1.89	1.96	2.04	2.11	2.24	2.36	2.48	2.59	2.69	2.79	2.88	2.97	3.03	3.10
	16	1.05	1.12	1.18	1.25	1.31	1.37	1.43	1.49	1.55	1.60	1.66	1.71	1.81	1.90	2.00	2.08	2.16	2.24	2.30	2.37	2.43	2.49
	17	0.80	0.86	0.90	0.95	1.00	1.04	1.09	1.13	1.18	1.22	1.26	1.30	1.37	1.44	1.51	1.57	1.62	1.68	1.72	1.76	1.80	1.84
	18	0.56	0.59	0.62	0.66	0.68	0.72	0.75	0.77	0.80	0.83	0.85	0.88	0.93	0.98	1.02	1.05	1.09	1.12	1.16	1.19	1.21	1.24
	19	0.29	0.31	0.32	0.34	0.36	0.37	0.39	0.40	0.42	0.43	0.44	0.45	0.48	0.50	0.52	0.54	0.56	0.57	0.59	0.60	0.61	0.62
	20																						
	21	0.29	0.30	0.32	0.34	0.35	0.37	0.38	0.40	0.41	0.42	0.44	0.46	0.48	0.50	0.53	0.56	0.58	0.59	0.60	0.61	0.62	0.62
	22	0.58	0.61	0.64	0.67	0.70	0.73	0.76	0.79	0.81	0.84	0.87	0.90	0.96	1.03	1.05	1.09	1.12	1.15	1.18	1.20	1.22	1.23
	23	0.89	0.94	0.99	1.03	1.08	1.12	1.16	1.20	1.25	1.29	1.33	1.37	1.44	1.51	1.57	1.63	1.67	1.73	1.77	1.80	1.82	1.94
	24	1.20	1.25	1.31	1.37	1.43	1.49	1.54	1.60	1.66	1.71	1.77	1.82	1.92	2.01	2.10	2.17	2.24	2.30	2.36	2.40	2.42	2.44
	25	1.51	1.59	1.66	1.74	1.81	1.88	1.95	2.02	2.09	2.16	2.23	2.30	2.42	2.53	2.63	2.72	2.82	2.89	2.95	2.99	3.01	3.05
	26	1.84	1.92	2.01	2.10	2.18	2.26	2.34	2.42	2.50	2.58	2.65	2.73	2.87	3.00	3.13	3.25	3.36	3.47	3.57	3.65	3.72	3.79
	27	2.17	2.26	2.36	2.46	2.56	2.66	2.75	2.84	2.93	3.01	3.10	3.18	3.35	3.50	3.66	3.80	3.93	4.06	4.16	4.26	4.35	4.42
	28	2.50	2.62	2.74	2.85	2.96	3.07	3.18	3.28	3.40	3.50	3.60	3.69	3.87	4.04	4.21	4.36	4.50	4.64	4.75	4.86	4.94	5.00
	29	2.86	2.98	3.10	3.22	3.35	3.47	3.59	3.70	3.82	3.93	4.03	4.14	4.34	4.53	4.72	4.89	5.05	5.20	5.34	5.46	5.56	5.64
Temperature in °C	30	3.20	3.35	3.49	3.64	3.77	3.91	4.05	4.17	4.30	4.43	4.55	4.67	4.90	5.12	5.39	5.51	5.68	5.94	5.96	6.09	6.16	6.22

Note: This table can be used to convert the density  $d_{20}^t$  to  $d_{20}^{20}$

TABLE IV

Temperature corrections,  $c$ , required for the density of liqueur wines, measured using a Pyrex-glass pycnometer at  $t$  °C, in order to correct to 20 °C

$$\rho_{20} = \rho_t \pm \frac{c}{1000}$$

- if  $t$ °C is lower than 20°C

+ if  $t$ °C is higher than 20°C

	13% vol. wines						15% vol. wines						17% vol. wines								
	Density						Density						Density								
	1.000	1.020	1.040	1.060	1.080	1.100	1.120	1.000	1.020	1.040	1.060	1.080	1.100	1.120	1.000	1.020	1.040	1.060	1.080	1.100	1.120

Temperature in °C	10	2.36	2.71	3.06	3.42	3.72	3.96	4.32	2.64	2.99	3.36	3.68	3.99	4.30	4.59	2.94	3.29	3.64	3.98	4.29	4.60	4.89
	11	2.17	2.49	2.80	2.99	3.39	3.65	3.90	2.42	2.73	3.05	3.34	3.63	3.89	4.15	2.69	3.00	3.32	3.61	3.90	4.16	4.41
	12	1.97	2.25	2.53	2.79	3.05	3.29	3.52	2.19	2.47	2.75	3.01	3.27	3.51	3.73	2.42	2.70	2.98	3.24	3.50	3.74	3.96
	13	1.78	2.02	2.25	2.47	2.69	2.89	3.09	1.97	2.21	2.44	2.66	2.87	3.08	3.29	2.18	2.42	2.64	2.87	3.08	3.29	3.49
	14	1.57	1.78	1.98	2.16	2.35	2.53	2.70	1.74	1.94	2.14	2.32	2.52	2.69	2.86	1.91	2.11	2.31	2.50	2.69	2.86	3.03
	15	1.32	1.49	1.66	1.82	1.97	2.12	2.26	1.46	1.63	1.79	1.95	2.10	2.25	2.39	1.60	1.77	1.93	2.09	2.24	2.39	2.53
	16	1.08	1.22	1.36	1.48	1.61	1.73	1.84	1.18	1.32	1.46	1.59	1.71	1.83	1.94	1.30	1.44	1.58	1.71	1.83	1.95	2.06
	17	0.83	0.94	1.04	1.13	1.22	1.31	1.40	0.91	1.02	1.12	1.21	1.30	1.39	1.48	1.00	1.10	1.20	1.30	1.39	1.48	1.56
	18	0.58	0.64	0.71	0.78	0.84	0.89	0.95	0.63	0.69	0.76	0.83	0.89	0.94	1.00	0.69	0.75	0.82	0.89	0.95	1.00	1.06
	19	0.30	0.34	0.37	0.40	0.43	0.46	0.49	0.33	0.37	0.40	0.43	0.46	0.49	0.52	0.36	0.39	0.42	0.46	0.49	0.52	0.54
	20																					
	21	0.30	0.33	0.36	0.40	0.43	0.46	0.49	0.33	0.36	0.39	0.43	0.46	0.49	0.51	0.35	0.39	0.42	0.45	0.48	0.51	0.54
	22	0.60	0.67	0.73	0.80	0.85	0.91	0.98	0.65	0.72	0.78	0.84	0.90	0.96	1.01	0.71	0.78	0.84	0.90	0.96	1.01	1.07
	23	0.93	1.02	1.12	1.22	1.30	1.39	1.49	1.01	1.10	1.20	1.29	1.38	1.46	1.55	1.10	1.19	1.29	1.38	1.46	1.55	1.63
	24	1.27	1.39	1.50	1.61	1.74	1.84	1.95	1.37	1.49	1.59	1.72	1.84	1.95	2.06	1.48	1.60	1.71	1.83	1.95	2.06	2.17
	25	1.61	1.75	1.90	2.05	2.19	2.33	2.47	1.73	1.87	2.02	2.17	2.31	2.45	2.59	1.87	2.01	2.16	2.31	2.45	2.59	2.73
	26	1.94	2.12	2.29	2.47	2.63	2.79	2.95	2.09	2.27	2.44	2.62	2.78	2.94	3.10	2.26	2.44	2.61	2.79	2.95	3.11	3.26
	27	2.30	2.51	2.70	2.90	3.09	3.27	3.44	2.48	2.68	2.87	3.07	3.27	3.45	3.62	2.67	2.88	3.07	3.27	3.46	3.64	3.81
	28	2.66	2.90	3.13	3.35	3.57	3.86	4.00	2.86	3.10	3.23	3.55	3.77	3.99	4.20	3.08	3.31	3.55	3.76	3.99	4.21	4.41
	29	3.05	3.31	3.56	3.79	4.04	4.27	4.49	3.28	3.53	3.77	4.02	4.26	4.49	4.71	3.52	3.77	4.01	4.26	4.50	4.73	4.95
30	3.44	3.70	3.99	4.28	4.54	4.80	5.06	3.68	3.94	4.23	4.52	4.79	5.05	5.30	3.95	4.22	4.51	4.79	5.07	5.32	5.57	

TABLE IV (continued)

Temperature corrections,  $c$ , required for the density of liqueur wines, measured using a Pyrex-glass pycnometer at  $t$  °C, in order to correct to 20 °C

$$\rho_{20} = \rho_t \pm \frac{c}{1000}$$

- if  $t$ °C is lower than 20°C

+ if  $t$ °C is higher than 20°C

	19% vol. wines							21% vol. wines					
	Density							Density					
	1.000	1.020	1.040	1.060	1.000	1.100	1.120	1.000	1.020	1.040	1.060	1.080	1.100

Temperature in °C	10	3.27	3.62	3.97	4.30	4.62	4.92	5.21	3.62	3.97	4.32	4.66	4.97	5.27	5.56
	11	2.99	3.30	3.61	3.90	4.19	4.45	4.70	3.28	3.61	3.92	4.22	4.50	4.76	5.01
	12	2.68	2.96	3.24	3.50	3.76	4.00	4.21	2.96	3.24	3.52	3.78	4.03	4.27	4.49
	13	2.68	2.96	3.24	3.50	3.76	4.00	4.21	2.96	3.24	3.52	3.78	4.03	4.27	4.49
	14	2.11	2.31	2.51	2.69	2.88	3.05	3.22	2.31	2.51	2.71	2.89	3.08	3.25	3.43
	15	1.76	1.93	2.09	2.25	2.40	2.55	2.69	1.93	2.10	2.26	2.42	2.57	2.72	2.86
	16	1.43	1.57	1.70	1.83	1.95	2.08	2.18	1.56	1.70	1.84	1.97	2.09	2.21	2.32
	17	1.09	1.20	1.30	1.39	1.48	1.57	1.65	1.20	1.31	1.41	1.50	1.59	1.68	1.77
	18	0.76	0.82	0.88	0.95	1.01	1.06	1.12	0.82	0.88	0.95	1.01	1.08	1.13	1.18
	19	0.39	0.42	0.45	0.49	0.52	0.55	0.57	0.42	0.46	0.49	0.52	0.55	0.58	0.61
	20														
	21	0.38	0.42	0.45	0.48	0.51	0.54	0.57	0.41	0.45	0.48	0.51	0.54	0.57	0.60
	22	0.78	0.84	0.90	0.96	1.02	1.07	1.13	0.84	0.90	0.96	1.02	1.08	1.14	1.19
	23	1.19	1.28	1.38	1.47	1.55	1.64	1.72	1.29	1.39	1.48	1.57	1.65	1.74	1.82
	24	1.60	1.72	1.83	1.95	2.06	2.18	2.29	1.73	1.85	1.96	2.08	2.19	2.31	2.42
	25	2.02	2.16	2.31	2.46	2.60	2.74	2.88	2.18	2.32	2.47	2.62	2.76	2.90	3.04
	26	2.44	2.62	2.79	2.96	3.12	3.28	3.43	2.53	2.81	2.97	3.15	3.31	3.47	3.62
	27	2.88	3.08	3.27	3.42	3.66	3.84	4.01	3.10	3.30	3.47	3.69	3.88	4.06	4.23
	28	3.31	3.54	3.78	4.00	4.22	4.44	4.64	3.56	3.79	4.03	4.25	4.47	4.69	4.89
	29	3.78	4.03	4.27	4.52	4.76	4.99	5.21	4.06	4.31	4.55	4.80	5.04	5.27	5.48
	30	4.24	4.51	4.80	5.08	5.36	5.61	5.86	4.54	4.82	5.11	5.39	5.66	5.91	6.16

TABLE V

Temperature corrections,  $c$ , required for the density of dry wines and dealcoholised dry wines, measured using an ordinary-glass pycnometer or hydrometer at  $t$  °C, in order to correct to 20 °C

$$\rho_{20} = \rho_t \pm \frac{c}{1000}$$

- if  $t$ °C is lower than 20°C

+ if  $t$ °C is higher than 20°C

		Alcoholic strength																										
		0	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27			
Temperature in °C	10	1.45	1.51	1.55	1.58	1.64	1.76	1.78	1.89	1.98	2.09	2.21	2.34	2.47	2.60	2.75	2.93	3.06	3.22	3.39	3.57	3.75	3.93	4.12	4.31			
	11	1.35	1.40	1.43	1.47	1.52	1.58	1.65	1.73	1.83	1.93	2.03	2.15	2.26	2.38	2.51	2.65	2.78	2.93	3.08	3.24	3.40	3.57	3.73	3.90			
	12	1.24	1.28	1.31	1.34	1.39	1.44	1.50	1.58	1.66	1.75	1.84	1.94	2.04	2.15	2.26	2.38	2.51	2.63	2.77	2.91	3.05	3.19	3.34	3.49			
	13	1.12	1.16	1.18	1.21	1.25	1.30	1.35	1.42	1.49	1.56	1.64	1.73	1.82	1.91	2.01	2.11	2.22	2.33	2.45	2.57	2.69	2.81	2.95	3.07			
	14	0.99	1.03	1.05	1.07	1.11	1.14	1.19	1.24	1.31	1.37	1.44	1.52	1.59	1.67	1.75	1.84	1.93	2.03	2.13	2.23	2.33	2.44	2.55	2.66			
	15	0.86	0.89	0.90	0.92	0.95	0.98	1.02	1.07	1.12	1.17	1.23	1.29	1.35	1.42	1.49	1.56	1.63	1.71	1.80	1.88	1.96	2.05	2.14	2.23			
	16	0.71	0.73	0.74	0.76	0.78	0.81	0.84	0.87	0.91	0.95	0.99	1.05	1.10	1.15	1.21	1.27	1.33	1.39	1.45	1.52	1.59	1.66	1.73	1.80			
	17	0.55	0.57	0.57	0.59	0.60	0.62	0.65	0.67	0.70	0.74	0.77	0.81	0.84	0.88	0.92	0.96	1.01	1.05	1.10	1.15	1.20	1.26	1.31	1.36			
	18	0.38	0.39	0.39	0.40	0.41	0.43	0.44	0.46	0.48	0.50	0.52	0.55	0.57	0.60	0.62	0.65	0.68	0.71	0.74	0.78	0.81	0.85	0.88	0.91			
	19	0.19	0.20	0.20	0.21	0.21	0.22	0.23	0.24	0.25	0.26	0.27	0.28	0.29	0.30	0.32	0.33	0.34	0.36	0.38	0.39	0.41	0.43	0.44	0.46			
	20	0.21	0.22	0.22	0.23	0.23	0.24	0.25	0.25	0.26	0.27	0.28	0.29	0.31	0.32	0.34	0.35	0.36	0.38	0.39	0.41	0.43	0.44	0.46	0.48			
21	0.43	0.45	0.45	0.46	0.47	0.49	0.50	0.52	0.54	0.56	0.58	0.60	0.62	0.65	0.68	0.71	0.73	0.77	0.80	0.83	0.86	0.89	0.93	0.96				
22	0.67	0.69	0.70	0.71	0.72	0.74	0.77	0.79	0.82	0.85	0.88	0.91	0.95	0.99	1.03	1.07	1.12	1.16	1.21	1.25	1.30	1.35	1.40	1.45				
23	0.91	0.93	0.95	0.97	0.99	1.01	1.04	1.07	1.11	1.15	1.20	1.24	1.29	1.34	1.39	1.45	1.50	1.56	1.62	1.69	1.76	1.82	1.88	1.95				
24	1.16	1.19	1.21	1.23	1.26	1.29	1.33	1.37	1.42	1.47	1.52	1.57	1.63	1.70	1.76	1.83	1.90	1.97	2.05	2.13	2.21	2.29	2.37	2.45				
25	1.42	1.46	1.49	1.51	1.54	1.58	1.62	1.67	1.73	1.79	1.85	1.92	1.99	2.07	2.14	2.22	2.31	2.40	2.49	2.58	2.67	2.77	2.86	2.96				
26	1.69	1.74	1.77	1.80	1.83	1.88	1.93	1.98	2.05	2.12	2.20	2.27	2.35	2.44	2.53	2.63	2.72	2.82	2.93	3.04	3.14	3.25	3.37	3.48				
27	1.97	2.03	2.06	2.09	2.14	2.19	2.24	2.31	2.38	2.46	2.55	2.63	2.73	2.83	2.93	3.03	3.14	3.26	3.38	3.50	3.62	3.75	3.85	4.00				
28	2.26	2.33	2.37	2.41	2.45	2.50	2.57	2.64	2.73	2.82	2.91	2.99	3.11	3.22	3.34	3.46	3.58	3.70	3.84	3.97	4.11	4.25	4.39	4.54				
29	2.56	2.64	2.67	2.72	2.77	2.83	2.90	2.98	3.08	3.18	3.28	3.38	3.50	3.62	3.75	3.88	4.02	4.16	4.30	4.46	4.61	4.76	4.92	5.07				

Note: This table can be used to convert the density  $d_{20}^t$  to  $d_{20}^{20}$

TABLE VI

Temperature corrections, c, required for the density of concentrated musts, measured using an ordinary-glass pycnometer or hydrometer at t °C, in order to correct to 20 °C.

$$\rho_{20} = \rho_t \pm \frac{c}{1000}$$

- if t°C is lower than 20°C

+ if t°C is higher than 20°C

		Density																					
		1.05	1.06	1.07	1.08	1.09	1.10	1.11	1.12	1.13	1.14	1.15	1.16	1.18	1.20	1.22	1.24	1.26	1.28	1.30	1.32	1.34	1.36



Temperature in °C	10	2.17	2.34	2.52	2.68	2.85	2.99	3.16	3.29	3.44	3.58	3.73	3.86	4.13	4.36	4.60	4.82	5.02	5.25	5.39	5.56	5.73	5.87
	11	2.00	2.16	2.29	2.44	2.59	2.73	2.86	2.99	3.12	3.24	3.37	3.48	3.71	3.94	4.15	4.33	4.52	4.69	4.85	5.01	5.15	5.29
	12	1.81	1.95	2.08	2.21	2.34	2.47	2.58	2.70	2.82	2.92	3.03	3.14	3.35	3.55	3.72	3.90	4.07	4.23	4.37	4.52	4.64	4.77
	13	1.62	1.74	1.85	1.96	2.07	2.17	2.28	2.38	2.48	2.59	2.68	2.77	2.94	3.11	3.28	3.44	3.54	3.72	3.86	3.99	4.12	4.24
	14	1.44	1.54	1.64	1.73	1.82	1.92	2.00	2.08	2.17	2.25	2.34	2.42	2.57	2.73	2.86	2.99	3.12	3.24	3.35	3.46	3.57	3.65
	15	1.21	1.29	1.37	1.45	1.53	1.60	1.68	1.75	1.82	1.89	1.97	2.03	2.16	2.28	2.40	2.51	2.61	2.71	2.80	2.89	2.94	3.01
	16	1.00	1.06	1.12	1.19	1.25	1.31	1.37	1.43	1.49	1.54	1.60	1.65	1.75	1.84	1.94	2.02	2.09	2.17	2.23	2.30	2.36	2.42
	17	0.76	0.82	0.86	0.91	0.96	1.00	1.05	1.09	1.14	1.18	1.22	1.25	1.32	1.39	1.46	1.52	1.57	1.63	1.67	1.71	1.75	1.79
	18	0.53	0.56	0.59	0.63	0.65	0.69	0.72	0.74	0.77	0.80	0.82	0.85	0.90	0.95	0.99	1.02	1.05	1.09	1.13	1.16	1.18	1.20
	19	0.28	0.30	0.31	0.33	0.35	0.36	0.38	0.39	0.41	0.42	0.43	0.43	0.46	0.48	0.50	0.52	0.54	0.55	0.57	0.58	0.59	0.60
	20																						
	21	0.28	0.29	0.31	0.33	0.34	0.36	0.37	0.39	0.40	0.41	0.43	0.44	0.46	0.48	0.51	0.54	0.56	0.57	0.58	0.59	0.60	0.60
	22	0.55	0.58	0.61	0.64	0.67	0.70	0.73	0.76	0.78	0.81	0.84	0.87	0.93	0.97	1.02	1.06	1.09	1.12	1.15	1.17	1.19	1.19
	23	0.85	0.90	0.95	0.99	1.04	1.08	1.12	1.16	1.21	1.25	1.29	1.32	1.39	1.46	1.52	1.58	1.62	1.68	1.72	1.75	1.77	1.79
	24	1.15	1.19	1.25	1.31	1.37	1.43	1.48	1.54	1.60	1.65	1.71	1.76	1.86	1.95	2.04	2.11	2.17	2.23	2.29	2.33	2.35	2.37
	25	1.44	1.52	1.59	1.67	1.74	1.81	1.88	1.95	2.02	2.09	2.16	2.22	2.34	2.45	2.55	2.64	2.74	2.81	7.87	2.90	2.92	2.96
	26	1.76	1.84	1.93	2.02	2.10	2.18	2.25	2.33	2.41	2.49	2.56	2.64	2.78	2.91	3.03	3.15	3.26	3.37	3.47	3.55	3.62	3.60
	27	2.07	2.16	2.26	2.36	2.46	2.56	2.65	2.74	2.83	2.91	3.00	3.07	3.24	3.39	3.55	3.69	3.82	3.94	4.04	4.14	4.23	4.30
	28	2.39	2.51	2.63	2.74	2.85	2.96	3.06	3.16	3.28	3.38	3.48	3.57	3.75	3.92	4.08	4.23	4.37	4.51	4.62	4.73	4.80	4.86
	29	2.74	2.86	2.97	3.09	3.22	3.34	3.46	3.57	3.69	3.90	3.90	4.00	4.20	4.39	4.58	4.74	4.90	5.05	5.19	5.31	5.40	5.48
	30	3.06	3.21	3.35	3.50	3.63	3.77	3.91	4.02	4.15	4.28	4.40	4.52	4.75	4.96	5.16	5.35	5.52	5.67	5.79	5.91	5.99	6.04

Note: This table can be used to convert the density  $d_{20}^t$  to  $d_{20}^{20}$

TABLE VII

Temperature corrections, c, required for the density of liqueur wines, measured using an ordinary-glass pycnometer or hydromete at t °C, in order to correct to 20 °C

$$\rho_{20} = \rho_t \pm \frac{c}{1000}$$

- if t°C is lower than 20°C

+ if t°C is higher than 20°C

	13% vol. wines							15% vol. wines							17% vol. wines						
	Density							Density							Density						
	1.000	1.020	1.040	1.060	1.080	1.100	1.120	1.000	1.020	1.040	1.060	1.080	1.100	1.120	1.000	1.020	1.040	1.060	1.080	1.100	1.120

	10	2.24	2.58	2.93	3.27	3.59	3.89	4.18	2.51	2.85	3.20	3.54	3.85	4.02	4.46	2.81	3.15	3.50	3.84	4.15	4.45	4.74
	11	2.06	2.37	2.69	2.97	3.26	3.53	3.78	2.31	2.61	2.93	3.21	3.51	3.64	4.02	2.57	2.89	3.20	3.49	3.77	4.03	4.28
	12	1.87	2.14	2.42	2.67	2.94	3.17	3.40	2.09	2.36	2.64	2.90	3.16	3.27	3.61	2.32	2.60	2.87	3.13	3.39	3.63	3.84
	13	1.69	1.93	2.14	2.37	2.59	2.80	3.00	1.88	2.12	2.34	2.56	2.78	2.88	3.19	2.09	2.33	2.55	2.77	2.98	3.19	3.39
	14	1.49	1.70	1.90	2.09	2.27	2.44	2.61	1.67	1.86	2.06	2.25	2.45	2.51	2.77	1.83	2.03	2.23	2.42	2.61	2.77	2.94
	15	1.25	1.42	1.59	1.75	1.90	2.05	2.19	1.39	1.56	1.72	1.88	2.03	2.11	2.32	1.54	1.71	1.87	2.03	2.18	2.32	2.47
	16	1.03	1.17	1.30	1.43	1.55	1.67	1.78	1.06	1.27	1.40	1.53	1.65	1.77	1.88	1.25	1.39	1.52	1.65	1.77	1.89	2.00
	17	0.80	0.90	1.00	1.09	1.17	1.27	1.36	0.87	0.98	1.08	1.17	1.26	1.35	1.44	0.96	1.06	1.16	1.26	1.35	1.44	1.52
	18	0.54	0.61	0.68	0.75	0.81	0.86	0.92	0.60	0.66	0.73	0.80	0.85	0.91	0.97	0.66	0.72	0.79	0.86	0.92	0.97	1.03
	19	0.29	0.33	0.36	0.39	0.42	0.45	0.48	0.32	0.36	0.39	0.42	0.45	0.48	0.51	0.35	0.38	0.41	0.45	0.48	0.51	0.53
	20																					
	21	0.29	0.32	0.35	0.39	0.42	0.45	0.47	0.32	0.35	0.38	0.42	0.45	0.48	0.50	0.34	0.38	0.41	0.44	0.47	0.50	0.53
	22	0.57	0.64	0.70	0.76	0.82	0.88	0.93	0.63	0.69	0.75	0.81	0.87	0.93	0.99	0.68	0.75	0.81	0.87	0.93	0.99	1.04
	23	0.89	0.98	1.08	1.17	1.26	1.34	1.43	0.97	1.06	1.16	1.25	1.34	1.42	1.51	1.06	1.15	1.25	1.34	1.42	1.51	1.59
	24	1.22	1.34	1.44	1.56	1.68	1.79	1.90	1.32	1.44	1.54	1.66	1.78	1.89	2.00	1.43	1.56	1.65	1.77	1.89	2.00	2.11
	25	1.61	1.68	1.83	1.98	2.12	2.26	2.40	1.66	1.81	1.96	2.11	2.25	2.39	2.52	1.80	1.94	2.09	2.24	2.39	2.52	2.66
	26	1.87	2.05	2.22	2.40	2.56	2.71	2.87	2.02	2.20	2.37	2.54	2.70	2.85	3.01	2.18	2.36	2.53	2.71	2.86	3.02	3.17
	27	2.21	2.42	2.60	2.80	3.00	3.18	3.35	2.39	2.59	2.78	2.98	3.17	3.35	3.52	2.58	2.78	2.97	3.17	3.36	3.54	3.71
	28	2.56	2.80	3.02	3.25	3.47	3.67	3.89	2.75	2.89	3.22	3.44	3.66	3.96	4.07	2.97	3.21	3.44	3.66	3.88	4.09	4.30
	29	2.93	3.19	3.43	3.66	3.91	4.14	4.37	3.16	3.41	3.65	3.89	4.13	4.36	4.59	3.40	3.66	3.89	4.13	4.38	4.61	4.82
Temperature in °C	30	3.31	3.57	3.86	4.15	4.41	4.66	4.92	3.55	3.81	4.10	4.38	4.66	4.90	5.16	3.82	4.08	4.37	4.65	4.93	5.17	5.42

TABLE VII (continued)

Temperature corrections,  $c$ , required for the density of liqueur wines, measured using an ordinary-glass pycnometer or hydrometer at  $t^{\circ}\text{C}$ , in order to correct to  $20^{\circ}\text{C}$ .

$$\rho_{20} = \rho_t \pm \frac{c}{1000}$$

- if  $t^{\circ}\text{C}$  is lower than  $20^{\circ}\text{C}$

+ if  $t^{\circ}\text{C}$  is higher than  $20^{\circ}\text{C}$

	19% vol. wines							21% vol. wines						
	Density							Density						
	1.000	1.020	1.040	1.060	1.080	1.100	1.120	1.000	1.020	1.040	1.060	1.080	1.100	1.120

Temperature in °C	10	3.14	3.48	3.83	4.17	4.48	4.78	5.07	3.50	3.84	4.19	4.52	4.83	5.12	5.41
	11	2.87	3.18	3.49	3.78	4.06	4.32	4.57	3.18	3.49	3.80	4.09	4.34	4.63	4.88
	12	2.58	2.96	3.13	3.39	3.65	3.88	4.10	2.86	3.13	3.41	3.67	3.92	4.15	4.37
	13	2.31	2.55	2.77	2.99	3.20	3.41	3.61	2.56	2.79	3.01	3.23	3.44	3.65	3.85
	14	2.03	2.23	2.43	2.61	2.80	2.96	3.13	2.23	2.43	2.63	2.81	3.00	3.16	3.33
	15	1.69	1.86	2.02	2.18	2.33	2.48	2.62	1.86	2.03	2.19	2.35	2.50	2.65	2.80
	16	1.38	1.52	1.65	1.78	1.90	2.02	2.13	1.51	1.65	1.78	1.91	2.03	2.15	2.26
	17	1.06	1.16	1.26	1.35	1.44	1.53	1.62	1.15	1.25	1.35	1.45	1.54	1.63	1.71
	18	0.73	0.79	0.85	0.92	0.98	1.03	1.09	0.79	0.85	0.92	0.98	1.05	1.10	1.15
	19	0.38	0.41	0.44	0.48	0.51	0.52	0.56	0.41	0.44	0.47	0.51	0.54	0.57	0.59
	20														
	21	0.37	0.41	0.44	0.47	0.50	0.53	0.56	0.41	0.44	0.47	0.51	0.54	0.57	0.59
	22	0.75	0.81	0.87	0.93	0.99	1.04	1.10	0.81	0.88	0.94	1.00	1.06	1.10	1.17
	23	1.15	1.30	1.34	1.43	1.51	1.60	1.68	1.25	1.34	1.44	1.63	1.61	1.70	1.78
	24	1.55	1.67	1.77	1.89	2.00	2.11	2.23	1.68	1.80	1.90	2.02	2.13	2.25	2.36
	25	1.95	2.09	2.24	2.39	2.53	2.67	2.71	2.11	2.25	2.40	2.55	2.69	2.83	2.97
	26	2.36	2.54	2.71	2.89	3.04	3.20	3.35	2.55	2.73	2.90	3.07	3.22	3.38	3.54
	27	2.79	2.99	3.18	3.38	3.57	3.75	3.92	3.01	3.20	3.40	3.59	3.78	3.96	4.13
	28	3.20	3.44	3.66	3.89	4.11	4.32	4.53	3.46	3.69	3.93	4.15	4.36	4.58	4.77
	29	3.66	3.92	4.15	4.40	4.64	4.87	5.08	3.95	4.20	4.43	4.68	4.92	5.15	5.36
	30	4.11	4.37	4.66	4.94	5.22	5.46	5.71	4.42	4.68	4.97	5.25	5.53	5.77	6.02

## Annex II

### *Comparison of results for the methods of measurement of density using a frequency oscillator (Method B) and using a hydrostatic balance (Method C)*

Using samples with densities between 0.992 and 1.012 g/cm<sup>3</sup>, the repeatability and reproducibility were measured using an inter-laboratory test. The densities of the different samples as measured using a hydrostatic balance and using electronic densimetry were compared, including the repeatability and reproducibility values derived from the multi-year inter-comparison tests performed on a large scale.

## 1. Samples

Wines with different densities and alcoholic strengths prepared monthly on an industrial scale, taken from a stock of bottles stored under normal conditions, and supplied anonymously to the laboratories.

## 2. Laboratories

Laboratories participating in the monthly tests organised by *Unione Italiana Vini* (Verona, Italy) according to ISO 5725 (UNI 9225) regulations and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemical Laboratories produced by the AOAC, ISO and IUPAC, and ISO 43 and ILAC G13 guidelines. An annual report is provided by the above-mentioned organisation to all participants.

## 3. Apparatus

1. An electronic hydrostatic balance (with precision to 5 decimal places), equipped if possible with a data-processing device.
2. An electronic densimeter, equipped if possible with an autosampler.

## 4. Analyses

According to the rules for the validation of methods of analysis, each sample was analysed twice consecutively to determine the alcoholic strength.

## 5. Results

Table 1 shows the results of the measurements obtained by the laboratories using a hydrostatic balance.

Table 2 shows the results obtained by the laboratories using an electronic densimeter.

## 6. Evaluation of results

1. The test results were examined for evidence of individual systemic error ( $p < 0.025$ ) using Cochran's and Grubbs' tests successively, according to the procedures described in the internationally accepted Protocol for the Design, Conduct and

## Interpretation of Method-Performance Studies.

### 6.2. *Repeatability (r) et reproducibility (R)*

Calculations for repeatability (r) and reproducibility (R) as defined by the protocol were carried out on the results remaining after the removal of outliers. When assessing a new method, there is often no validated reference or statutory method to compare precision criteria; 'predicted' levels of precision and therefore used to compare the precision data obtained from collaborative tests. These predicted levels are calculated from the Horwitz formula. Comparison of the test results and the predicted levels give an indication as to whether the method is sufficiently precise for the level of analyte being measured. The Horwitz predicted value is calculated from the Horwitz equation.

$$RSD_R = 2^{(1-0.5 \log C)}$$

where C is the measured concentration of analyte expressed as a decimal (e.g. 1 g/100 g = 0.01).

The Horrat value gives a comparison of the actual precision measured with the precision predicted by the Horwitz formula for the method and at the particular level of concentration of the analyte. It is calculated as follows:

$$HoR = RSD_R(\text{measured})/RSD_R(\text{Horwitz})$$

### 6.3 *Inter-laboratory reproducibility*

A Horrat value of 1 usually indicates satisfactory reproducibility, whereas a value of more than 2 usually indicates unsatisfactory reproducibility, i.e. reproducibility that is too variable for analytical purposes or where the variation obtained is greater than that predicted for the type of method employed. Hor is also calculated and used to measure intra-laboratory reproducibility, using the following approximation:

$$RSD_r(\text{Horwitz}) = 0.66 RSD_R(\text{Horwitz}) \text{ (this assumes the approximation that } r = 0.66 R)$$

CrD95 is the critical difference for a 95% probability level. It is calculated according to Resolution OIV-MA-AS1-08.

Table 3 shows the differences between the measurements obtained by laboratories using an electronic densimeter and those using a hydrostatic balance.

### 6.4 *Precision parameters*

Table 4 shows the overall averages for the precision parameters calculated from all monthly tests carried out between January 2008 and December 2010

Table 1: Results obtained by laboratories that conducted tests using a hydrostatic balance (HB)

Sample	Average	Total no. of values	No. of selected values	Repeatability	$s_r$	$RSD_r$	Hor	Reproducibility	$s_R$	$RSD_{Rcalc}$	HoR	No. of repet.	CrD95
01/08	0.995491	130	120	0.0001701	0.0000607	0.0061016	0.0046193	0.0005979	0.0002135	0.0214502	0.0107178	2	0.0004141
02/08	1.011475	146	125	0.0004714	0.0001684	0.0166457	0.0126320	0.0008705	0.0003109	0.0307366	0.0153947	2	0.0005686
03/08	0.992473	174	161	0.0001470	0.0000525	0.0052898	0.0040029	0.0004311	0.0001540	0.0155140	0.0077482	2	0.0002959
04/08	0.993147	172	155	0.0002761	0.0000986	0.0099274	0.0075130	0.0005446	0.0001945	0.0195839	0.0097818	2	0.0003595
05/08	1.004836	150	138	0.0001882	0.0000672	0.0066905	0.0050723	0.0007495	0.0002677	0.0266373	0.0133283	2	0.0005215
06/08	0.993992	152	136	0.0001486	0.0000531	0.0053391	0.0040411	0.0005302	0.0001894	0.0190506	0.0095167	2	0.0003675
07/08	0.992447	162	150	0.0002660	0.0000950	0.0095709	0.0072424	0.0006046	0.0002159	0.0217575	0.0108664	2	0.0004063
08/08	0.992210	162	151	0.0002619	0.0000935	0.0094281	0.0071341	0.0006309	0.0002253	0.0227108	0.0113420	2	0.0004265
09/08	1.002600	148	131	0.0001093	0.0000390	0.0038920	0.0029496	0.0007000	0.0002500	0.0249341	0.0124719	2	0.0004919
10/08	0.994482	174	152	0.0001228	0.0000439	0.0044105	0.0033385	0.0004250	0.0001518	0.0152645	0.0076259	2	0.0002942
11/08	0.992010	136	125	0.0000909	0.0000325	0.0032742	0.0024775	0.0004256	0.0001520	0.0153217	0.0076516	2	0.0002975
01/09	0.994184	174	152	0.0001655	0.0000591	0.0059435	0.0044987	0.0005439	0.0001942	0.0195384	0.0097606	2	0.0003756
02/09	0.992266	118	101	0.0001742	0.0000622	0.0062682	0.0047431	0.0005210	0.0001861	0.0187534	0.0093658	2	0.0003580
03/09	0.991886	164	135	0.0001850	0.0000661	0.0066603	0.0050395	0.0004781	0.0001707	0.0172136	0.0085963	2	0.0003251
04/09	0.993632	180	150	0.0001523	0.0000544	0.0054754	0.0041440	0.0004270	0.0001525	0.0153476	0.0076664	2	0.0002922
05/09	1.011061	116	100	0.0003659	0.0001307	0.0129234	0.0098067	0.0008338	0.0002978	0.0294527	0.0147508	2	0.0005605
06/09	0.992063	114	105	0.0002923	0.0001044	0.0105238	0.0079631	0.0005257	0.0001877	0.0189240	0.0094507	2	0.0003418
07/09	0.992708	172	155	0.0002892	0.0001033	0.0104040	0.0078732	0.0006156	0.0002199	0.0221478	0.0110617	2	0.0004106
08/09	0.993064	136	127	0.0002926	0.0001045	0.0105224	0.0079632	0.0007520	0.0002686	0.0270446	0.0135081	2	0.0005112
09/09	1.005285	118	110	0.0002946	0.0001052	0.0104661	0.0079352	0.0007226	0.0002581	0.0256704	0.0128454	2	0.0004892
10/09	0.992905	150	132	0.0002234	0.0000798	0.0080358	0.0060812	0.0004498	0.0001607	0.0161803	0.0080815	2	0.0002978
11/09	0.994016	142	127	0.0001896	0.0000677	0.0068114	0.0051555	0.0004739	0.0001693	0.0170278	0.0085062	2	0.0003214
01/10	0.994734	170	152	0.0002125	0.0000759	0.0076288	0.0057748	0.0005406	0.0001931	0.0194104	0.0096975	2	0.0003672
02/10	0.993177	120	110	0.0002210	0.0000789	0.0079467	0.0060140	0.0005800	0.0002071	0.0208565	0.0104175	2	0.0003950
03/10	0.992799	148	136	0.0002277	0.0000813	0.0081923	0.0061995	0.0015157	0.0005413	0.0545262	0.0272335	2	0.0010657
04/10	0.995420	172	157	0.0002644	0.0000944	0.0094866	0.0071819	0.0006286	0.0002245	0.0225542	0.0112693	2	0.0004244
05/10	1.002963	120	108	0.0007086	0.0002531	0.0252330	0.0191244	0.0013667	0.0004881	0.0486677	0.0243447	2	0.0008991
06/10	0.992546	120	113	0.0001737	0.0000620	0.0062506	0.0047300	0.0005435	0.0001941	0.0195567	0.0097673	2	0.0003744
07/10	0.992831	174	152	0.0003003	0.0001073	0.0108031	0.0081753	0.0006976	0.0002492	0.0250959	0.0125344	2	0.0004699
08/10	0.993184	144	130	0.0001799	0.0000642	0.0064674	0.0048945	0.0005951	0.0002125	0.0213984	0.0106882	2	0.0004111
09/10	1.012293	114	103	0.0002265	0.0000809	0.0079907	0.0060647	0.0014586	0.0005209	0.0514596	0.0257772	2	0.0010251
10/10	0.992289	154	136	0.0006386	0.0002281	0.0229860	0.0173933	0.0007033	0.0002512	0.0253124	0.0126415	2	0.0003812
11/10	0.994649	130	112	0.0002902	0.0001036	0.0104200	0.0078876	0.0005287	0.0001888	0.0189830	0.0094838	2	0.0003445

Table 2: Results obtained by laboratories that conducted tests using an electronic densimeter (ED)

Table 3: Comparison of results from the hydrostatic balance (BH) and from the

## electronic densimeter (ED)

Density – Hydrostatic balance				Density – Frequency oscillator				Comparison
Sample	Average value	Total values	Selected values	Sample	Average value	Total values	Selected values	$\Delta$ (BH-DE)
01/08	0.995491	130	120	01/08	0.995504	114	108	-0.000013
02/08	1.011475	146	125	02/08	1.011493	132	125	-0.000018
03/08	0.992473	174	161	03/08	0.992491	138	118	-0.000018
04/08	0.993147	172	155	04/08	0.993129	132	120	0.000018
05/08	1.004836	150	138	05/08	1.004892	136	116	-0.000056
06/08	0.993992	152	136	06/08	0.994063	142	123	-0.000071
07/08	0.992447	162	150	07/08	0.992498	136	125	-0.000051
08/08	0.992210	162	151	08/08	0.992270	130	115	-0.000060
09/08	1.002600	148	131	09/08	1.002603	136	121	-0.000003
10/08	0.994482	174	152	10/08	0.994493	128	117	-0.000011
11/08	0.992010	136	125	11/08	0.992017	118	104	-0.000007
01/09	0.994184	174	152	01/09	0.994216	148	131	-0.000031
02/09	0.992266	118	101	02/09	0.992251	104	88	0.000015
03/09	0.991886	164	135	03/09	0.991875	126	108	0.000011
04/09	0.993632	180	150	04/09	0.993654	134	114	-0.000022
05/09	1.011061	116	100	05/09	1.011035	128	104	0.000026
06/09	0.992063	114	105	06/09	0.992104	116	106	-0.000041
07/09	0.992708	172	155	07/09	0.992720	144	140	-0.000012
08/09	0.993064	136	127	08/09	0.993139	110	102	-0.000075

09/09	1.005285	118	110	09/09	1.005276	112	108	0.000009
10/09	0.992905	150	132	10/09	0.992912	122	111	-0.000008
11/09	0.994016	142	127	11/09	0.994031	128	118	-0.000015
01/10	0.994734	170	152	01/10	0.994752	144	136	-0.000018
02/10	0.993177	120	110	02/10	0.993181	108	98	-0.000005
03/10	0.992799	148	136	03/10	0.992665	140	127	0.000134
04/10	0.995420	172	157	04/10	0.995502	142	128	-0.000082
05/10	1.002963	120	108	05/10	1.002851	130	119	0.000112
06/10	0.992546	120	113	06/10	0.992607	106	99	-0.000061
07/10	0.992831	174	152	07/10	0.992871	160	150	-0.000040
08/10	0.993184	144	130	08/10	0.993235	104	93	-0.000052
09/10	1.012293	114	103	09/10	1.012328	112	105	-0.000035
10/10	0.992289	154	136	10/10	0.992308	128	115	-0.000019
11/10	0.994649	130	112	11/10	0.994683	120	108	-0.000035
						average	$\Delta$ (BH-DE)	-0.0000162
						standard deviation	$\Delta$ (BH-DE)	0.0000447

Table 4: Precision parameters

	<i>Hydrostatic balance (BH)</i>	<i>Electronic densimeter (ED)</i>
No. of selected values	4347	3800
min	0.99189 g/cm <sup>3</sup>	0.99187 g/cm <sup>3</sup>
max	1.01229 g/cm <sup>3</sup>	1.01233 g/cm <sup>3</sup>



R	0.00067 g/cm <sup>3</sup>	0.00025 g/cm <sup>3</sup>
s <sub>R</sub>	0.00024 g/cm <sup>3</sup>	0.000091 g/cm <sup>3</sup>
R%	0.067%	0.025%
r	0.00025 g/cm <sup>3</sup>	0.00011 g/cm <sup>3</sup>
s <sub>r</sub>	0.000090 g/cm <sup>3</sup>	0.000038 g/cm <sup>3</sup>
r%	0.025%	0.011%

Key:

*n*: number of selected values

*min*: lower limit of the measurement range

*max*: upper limit of the measurement range

*r*: repeatability

*s<sub>r</sub>*: repeatability standard deviation

*r%*: relative repeatability ( $r \times 100 / \text{average value}$ )

*R*: reproducibility

*s<sub>R</sub>*: reproducibility standard deviation

*R%*: relative reproducibility ( $R \times 100 / \text{average value}$ )

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