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RECOMMENDATION

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Glass capillary viscometers for the measurement of kinematic viscosity - Verification method

Viscosimètres à capillaires, en verre, pour la mesure de la viscosité cinématique - Méthode de vérification

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ORGANISATION INTERNATIONALE DE MÉTROLOGIE LÉGALE

INTERNATIONAL ORGANIZATION OF LEGAL METROLOGY

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GLASS CAPILLARY VISCOMETERS
for the MEASUREMENT of KINEMATIC VISCOSITY

VERIFICATION METHOD

0. Scope

This Recommendation sets out the method and means for initial and subsequent verifications of glass capillary viscometers (ordinary instruments), free liquid flow, intended for the measurement of kinematic viscosity of liquids.

The principle of verification consists in determining the constant of the viscometer, using a suitable and stable reference material [(*)] of viscosity, the viscosity of which was measured using standard or reference viscometers.

The operations making it possible to determine the viscosity of the reference materials of viscosity and the constant of the viscometer should be carried out simultaneously, in the same thermostatic bath.

It is however admissible to carry out the verification of viscometers using reference materials of which the values of viscosity have been given by another laboratory. The verification of viscometers shall then be carried out at the temperature of measurement of the viscosity of the reference materials in question.

Other verification methods are accepted provided that they give similar results.

1. Verification operations

The following operations shall be carried out during verification :
— external inspection (point 4.1),
— determination of viscometer constant C (point 4.4).

2. Verification equipment

Standard or reference viscometers and auxiliary equipment, as indicated in Appendix I, shall be used during verification.

3. Verification conditions

Verification of viscometers shall be carried out under the following conditions :

— the viscometers shall be immersed in a thermostatic bath ; temperature instability shall not exceed 0.01 °C in relation to the given temperature,

(*) BIML note : in some countries, the reference materials used for official verifications have to be « certified » reference materials ; for such countries, the qualifier « certified » must be introduced in this text wherever appropriate.

— the viscometers shall be placed in the bath in such a way that the capillary tubes are vertical, except for those types of viscometers for which another position is stipulated ; verticality shall be checked using a plumb bob.

4. Verification procedure

4.1. External inspection

During external inspection, the condition of the viscometer is checked and the presence of the type designation, of the serial number and of the manufacturer's mark is verified ; the markings shall be such that they cannot be defaced during use of the viscometer. The viscometer shall be rejected if it does not comply with all these requirements.

4.2. Washing and drying

4.2.1. The viscometers which are not contaminated with oil products shall be washed in hot water and remain filled with a mixture of potassium dichromate and sulphuric acid so called « chromic acid » (*), for not less than 2 hours. The viscometers shall then be washed in distilled water and dried in a desiccator or in a vacuum chamber at a temperature not exceeding 120 °C. It is recommended to wash the viscometers in rectified ethyl alcohol or in acetone to accelerate drying.

4.2.2. The viscometers which are contaminated with oil products shall be carefully washed in white spirit (or any other suitable solvent) and then in hot water until the white spirit odour is removed. After that, the viscometers shall remain filled with chromic acid for at least 6 hours. The next washing and drying shall be carried out as indicated in point 4.2.1.

4.2.3. The viscometers contaminated with other products shall be washed successively in an appropriate solvent, then in water and then they shall be washed and dried as indicated in point 4.2.1.

4.3. Preparation of reference materials of viscosity

4.3.1. The necessary volumes of component liquids shall be measured using a measuring glass or measuring cylinder and carefully mixed in a dry bottle.

4.3.2. The liquids having a viscosity less than 20 mm^2/s shall, after being agitated, be filtered through a glass filter with a porous plate of 75 μm pore size ; to accelerate filtration, a vacuum flask and vacuum pump are used. More viscous liquids shall, after having been heated, be filtered through a Büchner funnel, with paper filter, using a vacuum flask and pump.

For the filtration of liquids with a viscosity exceeding 2 000 mm^2/s the use of wire gauze with a mesh size not exceeding 0.6 mm is acceptable.

4.3.3. The filtered liquids shall be poured into clean dry bottles and retained for 2 or 3 days in a darkened place and then their viscosity shall be determined as indicated in Appendix II.

(*) 60 g of potassium dichromate dissolved in 1 dm^3 of distilled water and 1 dm^3 of sulphuric acid carefully added. Attention, it is a dangerous product.

4.4. Determination of viscometer constant C

4.4.1. It is desirable to determine the constant C for each viscometer using at least two reference materials of viscosity. Before each filling the viscometers shall be washed and dried as indicated in point 4.2.

4.4.2. The reference materials of viscosity shall be chosen so that the ratio of their viscosities is between 2 and 5. The flow time of the liquid, in any type of viscometer to be verified, shall not be less than 200 s. It is desirable to> have a minimum flow time of the liquid greater than 200 s for suspended-level viscometers having a constant $C \leq 0.05$ mm^2/s^2, to eliminate any correction for kinetic energy.

4.4.3. The viscometer with the reference material of viscosity shall be placed in the thermostatic bath at required temperature and the flow time of the liquid shall be determined not less than 60 min later.

The level of the liquid in the thermostatic bath shall be from 15 to 20 mm above the upper line which marks the limit of the measuring bulb of the viscometer.

At least 5 measurements of the flow time of the liquid shall be carried out for the different types of viscometers, except for viscometers intended for the measurement of viscosity of opaque liquids, for which the number of measurements of the time of filling of the measuring reservoir (bulb) shall not be less than 3 (the viscometer shall be washed and dried as described in point 4.2, before each filling).

The flow times of the liquid shall be measured with an accuracy of 0.2 s. The difference between maximum and minimum values of the flow times of the liquid shall not exceed 0.2 % of the mean flow time (0.3 % for viscometers intended for the measurement of viscosity of opaque liquids). If the differences between the flow times exceed these values, the verification shall be repeated after careful washing and drying of the viscometer.

5. Calculation of the viscometer constant C and presentation of the results of verification

5.1. The constant C shall be determined as the arithmetic mean of the values C_1 and C_2 with not less than 4 significant figures.

5.2. The values C_1 and C_2 shall be calculated using the formulae :

$$C_1 = \frac{\nu_1}{t_1} \cdot \frac{g_n}{g} \qquad C_2 = \frac{\nu_2}{t_2} \cdot \frac{g_n}{g}$$

where :

C_1 and C_2 are the values of the viscometer constant, determined using two reference materials of viscosity (mm^2/s^2),

ν_1 and ν_2 are the kinematic viscosities of the reference materials of viscosity, determined using standard or reference viscometers (mm^2/s),

t_1 and t_2 are the arithmetic means of the flow times for the reference materials of viscosity in the viscometer to be verified (s),

g_n is the standard acceleration due to gravity (9.806 65 m/s^2),

g is the acceleration due to gravity at the place of determination of the constant (m/s^2).

The value g may be determined using the formula :

$$g = 9.780\ 318\ (1 + 0.005\ 302\ 4 \sin^2\varphi - 0.000\ 005\ 9 \sin^2 2\varphi) - 2 \cdot 10^{-6} h$$

where :
φ is the latitude (°),
h is the altitude (m).

5.3. The variation of the values of the constants shall not exceed 0.2 % and 0.4 % of the arithmetic mean for viscometers intended for measurement of the viscosity of transparent and of opaque liquids respectively.

5.4. The verification of the viscometer shall be repeated if this variation exceeds the values indicated in point 5.3.

5.5. The viscometer shall not be admitted for use if, in the second series of measurements, this variation again exceeds the values indicated in point 5.3.

5.6. The arithmetic mean of C_1 and C_2 shall be taken as the value of the viscometer constant C.

5.7. Verification certificates shall be issued for viscometers which have passed successfully the verification procedure. These certificates shall include the following information :

— type of viscometer,

— serial number,

— formula used for calculation during measurement of viscosity using the instrument in question,

— temperature at which the verification of the viscometer was carried out.

The certificate shall also include, in the form of a note, the confirmation that the constant was determined on the basis of the value 1.003 8 mm^2/s, adopted for the viscosity of water at 20 °C.

The obtained value for the constant (reduced to standard acceleration due to gravity) may be marked on the viscometer, using indelible ink or other methods. If the constant differs from that already shown by less than 0.2 %, the new value does not need to be marked.

APPENDIX I

EQUIPMENT for the VERIFICATION

of GLASS CAPILLARY VISCOMETERS

- Standard or reference viscometers of the Ubbelohde, Cannon (NPL), U-tube (NPL), normal flow U-tube (NRLM) etc., types, the constant of which is known with an error not exceeding ± 0.1 %.
- Reference materials of viscosity : transparent Newtonian liquids, of stable viscosity (silicone liquids are not recommended) (See Appendix III).
- Thermostatic baths, with temperature control devices ensuring a constant temperature during measurement, the variations not exceeding ± 0.01 °C.
- Devices for the measurement of flow time of the liquid in a standard or reference viscometer with an error not exceeding ± 0.01 s and in a viscometer to be verified with an error not exceeding ± 0.2 s.
- Thermometers for the measurement of the temperature in the thermostatic bath with an error not exceeding ± 0.01 °C.
- Water jet pump, or other type of suction pump.
- Desiccator.
- Laboratory glassware (beakers, flasks, funnels, stirrers, etc.).
- Liquids for washing the viscometers : distilled water, chromic acid, white spirit, rectified ethyl alcohol, acetone and other solvents.
- Small diameter rubber or plastic tubes.
- Lighting fixtures with negligible thermal radiation.

APPENDIX II

DETERMINATION of the VISCOSITY of REFERENCE MATERIALS of VISCOSITY

The viscosity of the reference materials of viscosity shall be determined by means of two or more standard or reference viscometers, chosen so that the flow times of the reference materials of viscosity is not less than 200 s.

Two or more standard or reference viscometers shall be filled with the appropriate volume of the reference material of viscosity, placed in a thermostatically controlled bath in a vertical position as indicated in point 3 and maintained for at least 30 min at the given temperature (it is desirable to do this at the same time with the viscometers to be verified).

At least 5 measurements shall be made on each viscometer and the arithmetic mean of the flow times shall be calculated with an accuracy of 0.01 s. Individual flow times shall not differ from each other by more than 0.1 % of the arithmetic mean.

The kinematic viscosity of the reference materials of viscosity shall be calculated with at least 5 significant figures, based on the mean flow time in the standard or reference viscometer, using the formula :

$$\nu = C \cdot t - \frac{E}{t^2} \qquad E = \frac{0.016\,6\,V^{3/2}}{L \cdot (C \cdot d)^{1/2}}$$

where :

V is the volume of the reservoir (mm^3),
L is the length of the capillary tube (mm),
C is the viscometer constant (mm^2/s^2),
d is the diameter of the capillary tube (mm).

The arithmetic mean of the measurement results obtained with two standard or reference viscometers shall be taken as the value of kinematic viscosity for the reference material of viscosity, provided that the variation between the individual values and the arithmetic mean does not exceed :

0.2 % for viscosities below 20 mm^2/s,
0.3 % for viscosities between 20 and 2 000 mm^2/s,
0.4 % for viscosities over 2 000 mm^2/s.

If this condition is not met, the viscometer shall be carefully washed and dried, as indicated in point 4.2, and the liquid shall be filtered and the measurement repeated until results are obtained which comply with the above-mentioned condition.

The value of the viscosity of the reference material of viscosity can only be guaranteed for 6 months from the date of the measurement.

APPENDIX III

List of reference materials recommended for the verification of glass capillary viscometers

Designation of the reference material	Nominal value of the kinematic viscosity (mm^2/s) at a temperature t (°C)								
	– 53.89	– 40	20	25	37.78	40	50	98.89	100
3	300	80	4.6	4.0	3.0	2.9	—	1.2	1.2
6	—	—	11	8.9	6.0	5.7	—	1.8	1.8
20	—	—	44	34	20	18	—	4.0	3.9
60	—	—	170	120	60	54	—	7.4	7.2
200	—	—	640	450	200	180	—	17.5	17
600	—	—	2 400	1 600	600	520	280	33	32
2 000	—	—	8 700	5 600	2 000	1 700	—	78	75
8 000	—	—	37 000	23 000	8 000	6 700	—	—	—
30 000	—	—	—	81 000	27 000	23 000	11 000	—	—

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