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Plastics — Phenolic resins in the liquid state or in solution — Determination of viscosity

Plastiques — Résines phénoliques liquides ou en solution — Détermination de la viscosité

Reference number
ISO 9371:1990(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9371 was prepared by Technical Committee ISO/TC 61, *Plastics*.

Annexes A and B form an integral part of this International Standard.

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Plastics — Phenolic resins in the liquid state or in solution — Determination of viscosity

1 Scope

This International Standard specifies four methods for the determination of the viscosity of phenolic resins, either as a liquid or in solution.

It describes the following two types of method:

a) Reference methods:

1) Determination using a viscometer with a definite speed gradient

2) Determination using an Ubbelohde capillary viscometer

b) Comparative test methods:

1) Determination using a rotary viscometer

2) Determination using a Hoeppler dropping-ball viscometer

Other methods may be used as long as it is verified that the same test results are obtained.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2555:1989, *Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method.*

ISO 3105:1976, *Glass capillary kinematic viscometers — Specification and operating instructions.*

ISO 3219:1977, *Plastics — Polymers in the liquid, emulsified or dispersed state — Determination of viscosity with a rotational viscometer working at defined shear rate.*

3 General (valid for all four methods)

Test temperature

Unless specified otherwise, determinations shall be performed at 23 °C within the tolerance allowed in the method used.

Dissolving solid resins

The solvent used and the concentration chosen shall be agreed between the parties concerned.

Expression of results

Dynamic viscosities shall be expressed in millipascal seconds (mPa·s), kinematic viscosities in square millimetres per second (mm^2/s).

4 Reference methods

4.1 Viscosity determination using a viscometer with definite speed gradient

The specifications of ISO 3219 shall be followed, either to determine the viscosity at a definite speed gradient, or to perform a rheological study of the resin and, in particular, to obtain a curve of viscosity as a function of the speed gradient.

4.2 Determination using an Ubbelohde capillary viscometer

4.2.1 Scope

This sub-clause specifies a method of test, for reference purposes, for the determination of the kinematic viscosity of liquid phenolic resins, or resins dissolved in an appropriate solvent at a given concentration chosen by agreement between the parties concerned. The method is suitable for determining kinematic viscosity over the range 2 mm^2/s to 10 000 mm^2/s.

NOTE 1 Kinematic viscosity is a property of newtonian fluids. As resins and resin solutions are non-newtonian, the determination of the viscosity by this method will give an apparent viscosity which will depend on the specific conditions used.

4.2.2 Normative reference

ISO 653:1980, *Long solid-stem thermometers for precision use*.

4.2.3 Principle

The determination involves measuring the time required by a fixed volume of liquid resin or resin solution, contained in the bulb of a glass viscometer, to flow under gravity through a calibrated capillary under a reproducible driving head of liquid at a closely controlled temperature.

4.2.4 Apparatus

4.2.4.1 Ubbelohde viscometer, with design details and construction as shown in figure 1. Table 1 gives the approximate C-constants (see annex A), the diameter of the capillaries and the corresponding viscosity ranges. Bulb C has a volume of 4 ml for a viscometer whose constant is between 0,01 mm^2/s^2 and 0,05 mm^2/s^2, 5 ml for a constant from 0,2 mm^2/s^2 to 1,0 mm^2/s^2 and 6 ml for a constant between 3,0 mm^2/s^2 to 10,0 mm^2/s^2.

The viscometer shall be fitted with supports designed to hold the various tubes which make up the viscometer in a vertical position as required by the method.

The filler marks (G and H) on bulb A indicate the minimum and maximum quantities providing the pressure necessary for correct functioning.

4.2.4.2 Thermoregulated bath, of such a design that the viscometer can be immersed in it so that bulb C above the top of the capillary is at least 30 mm below the surface of the liquid in the bath, but the tube and thermometer are both visible. A rigid support shall be provided to maintain the tube vertically to within 1°.

The bath shall be fitted with a heater and stirrer capable, between them, of maintaining the bath at a temperature of 23 °C ± 0,1 °C along the entire length of the viscometer and from one viscometer tube to another.

Table 1 — Dimensions, approximate C-constants and kinematic-viscosity ranges of the Ubbelohde viscometer

Approximate C-constant mm^2/s^2	Internal diameter of capillary mm (± 2 %)	Volume of bulb C ml (± 5 %)	Utilisation range Kinematic viscosity mm^2/s *)
0,01	0,58	4	2 to 10
0,03	0,78	4	6 to 30
0,05	0,88	4	10 to 50
0,1	1,03	5	20 to 100
0,3	1,36	5	60 to 300
0,5	1,55	5	100 to 500
1,0	1,83	5	200 to 1 000
3,0	2,43	6	600 to 3 000
5,0	2,75	6	1 000 to 5 000
10,0	3,27	6	2 000 to 10 000

*) 1 mm^2/s = 1 cSt

Dimensions in millimetres

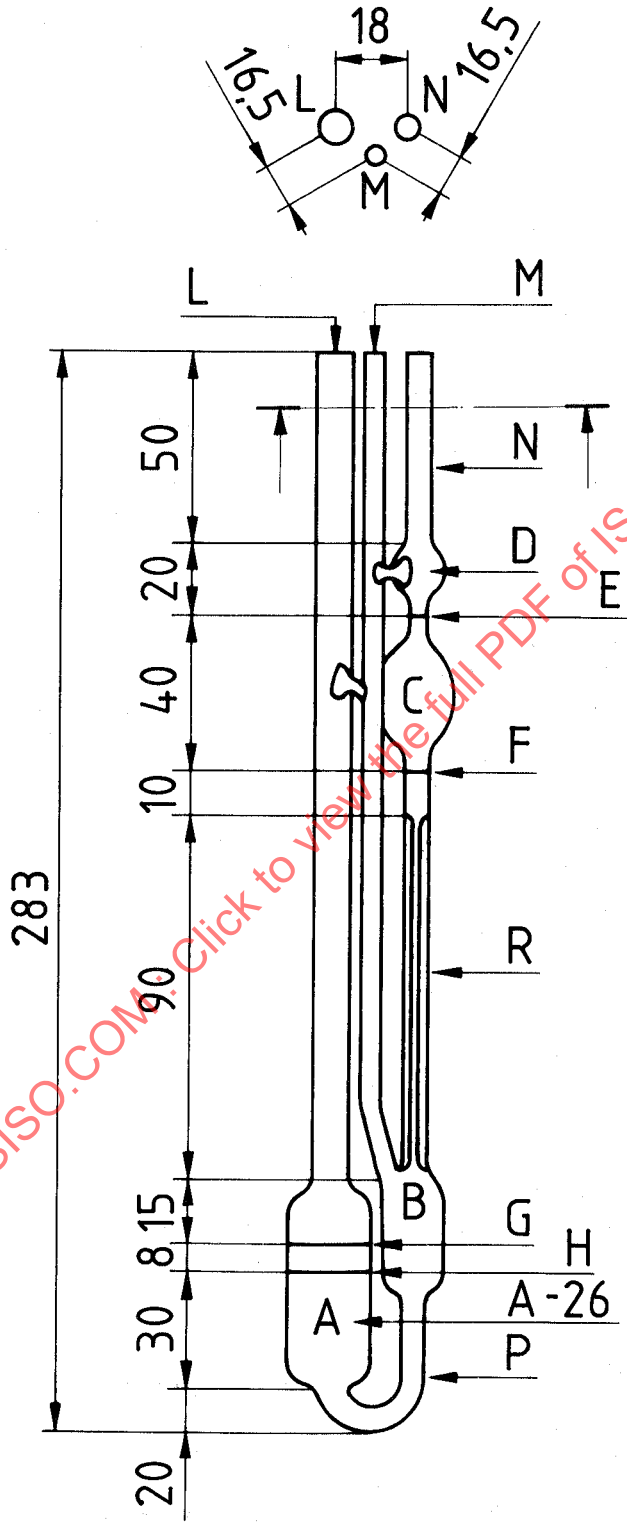

Figure 1 — Details of Ubbelohde viscometer

4.2.4.3 Filter funnel, with sintered-glass disc, medium porosity (average pore diameter between 40 µm and 50 µm).

4.2.4.4 Stopwatch, accurate to 0,1 s.

4.2.4.5 Long-stem thermometer, covering the range from 20 °C to 45 °C, graduated at intervals of not more than 0,1 °C, in accordance with ISO 653 (STL/0,1/20/45).

4.2.4.6 Means of applying sufficient vacuum, to the top of tube N to raise the resin or resin-solution level to the level in bulb C required by the test procedure (see 4.2.5.3).

4.2.5 Procedure

4.2.5.1 Viscometer calibration

Calibrate the viscometer (4.2.4.1) before using it for the first time, then periodically (see annex A). Constants are usually provided with viscometers, but it is advisable to check them.

4.2.5.2 Viscometer cleaning

Initially, then periodically, clean the viscometer with a chromic acid cleaning solution. The cleaning solution shall be kept in the viscometer for at least 12 h at ambient temperature, although this time may be shortened if the temperature is higher. Then empty the viscometer and rinse it first with deionized distilled water followed by acetone, finally drying it with stream of dry, filtered air.

Clean the viscometer thoroughly between measurements by rinsing with a suitable highly volatile solvent. Dry the viscometer by passing a slow stream of dry, filtered air through it for 2 min, or until all traces of solvent are eliminated.

4.2.5.3 Determination of viscosity

Fill the viscometer (4.2.4.1) with resin or resin solution, tilting the instrument about 30° out of vertical, keeping bulb A (see figure 1) below the capillary, and passing the liquid through the sintered-glass filter funnel (4.2.4.3).

Introduce sufficient liquid into tube L to raise the level to the lower filler mark (H), but below the upper filler mark (G), when the viscometer is returned to the vertical. The U-tube (P) at the bottom of the viscometer should then be completely full. It shall not contain any air bubbles.

Immerse the viscometer in the bath (4.2.4.2), using the support to ensure the viscometer is vertical.

Maintain the bath temperature at 23 °C ± 0,1 °C. Allow the filled viscometer to remain in the bath long enough to reach 23 °C ± 0,1 °C. Place a finger over tube M and apply suction to the top of tube N to draw liquid up tube N until it reaches the centre of bulb D.

Disconnect the suction from tube N, remove the finger from tube M and place it immediately on tube N until the liquid in and below tube M has drained clear from the bottom end of the capillary (R). Remove the finger from tube N and, using the stopwatch (4.2.4.4), measure, to within 0,1 s, the time required for the meniscus to pass from the first mark (E) to the second (F). A flow time of at least 200 s is required. If the flow time is less than 200 s, select a viscometer with a finer capillary and repeat the test. Run the determination in duplicate with different test samples.

4.2.6 Expression of results

The kinematic viscosity ν, expressed in square millimetres per second, is given by the equation

$$\nu = C(t_m - \Delta t)$$

where

C is the viscometer constant, expressed in square millimetres per second squared (see clause A.1);

t_m is the flow-time, expressed in seconds;

Δt is the flow-time correction, expressed in seconds (see clause A.2).

Calculate the arithmetic mean of the two determinations. In the case where they differ by more than 5 % in relative value, repeat the determination in duplicate with different test samples.

5 Comparative test methods

5.1 Viscosity determination using a rotary viscometer

Proceed in accordance with ISO 2555.

Table 2 gives the range of viscosities measurable with each of seven spindles at a spindle speed of 50 rev/min.

Table 2 — Correspondance between spindle No. and viscosity range for a rotary viscometer

Spindle No.	Measurable viscosities mPa·s*)
1	40 (20) to 190 (95)
2	190 (24) to 760 (95)
3	760 (38) to 1 900 (95)
4	1 900 (47,5) to 3 800 (95)
5	3 800 (47,5) to 7 600 (95)
6	7 600 (38) to 19 000 (95)
7	19 000 (24) to 76 000 (95)

*) The figures in parentheses indicate how much of a scale from 0 to 100 is covered by each viscosity range, the upper limit of the viscosity range always being 95 on the scale.

For resins used in the manufacture of adhesives, a speed of 20 rev/min is recommended.

If it is desired to compare the viscosities of two resins, the same spindle shall be used, even if this is not in agreement with table 2. This shall be stated in the test report.

5.2 Determination using a Hoeppler dropping-ball viscometer

Until such time as an International Standard is available, use the method described in annex B of this standard.

6 Test report (valid for all four methods)

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the sample of resin tested;

c) the solvent used and the concentration chosen when the resin is tested in solution;

d) the test temperature used;

e) the method employed;

f) if a rotary viscometer was used, the number of the spindle;

g) the results, expressed in millipascal seconds (mPa.s), calculated in accordance with the appropriate "Expression of results" clause for the particular method used (see the relevant International Standard, or sub-clause 4.2.6 or clause B.6 of this standard);

h) the date of the test.

Annex A
(normative)

Calibrating Ubbelohde viscometers

Each viscometer shall be calibrated prior to use since, although viscometers may have similar constants, they may have geometrical differences that require kinetic-energy corrections.

A.1 Determination of the viscometer constant C

Determine, to within 0,1 s, at a temperature of 23 °C ± 0,1 °C, the flow time of a standard reference oil[1], the viscosity of which is such that the flow time is greater than 200 s.

Under these conditions, the kinetic-energy correction required is negligible and the viscometer constant C can be calculated using the equation

$$C = \frac{\nu}{t}$$

where

ν is the kinematic viscosity of the standard reference oil at 23 °C, expressed in square millimetres per second;

t is the flow time, expressed in seconds.

A.2 Determination of the flow-time correction Δt for low-viscosity liquids

Using the same viscometer at the same temperature as in clause A.1, determine the flow times, in seconds, of at least three standard reference oils having a viscosity lower than that of the reference oil used for the determination of the viscometer constant. The viscosities of these three reference oils shall be such that the flow times are between 60 s and 200 s.

Calculate, for each oil, the correction Δt, representing the difference between the measured flow time and the flow time in the absence of hydrostatic energy losses as kinetic energy, using the equation

$$\Delta t = t_m - \frac{\nu}{C}$$

where

t_m is the flow time, expressed in seconds;

ν is the kinematic viscosity of the standard reference oil at 23 °C ± 0,1 °C, expressed in square millimetres per second;

C is the viscometer constant, expressed in square millimetres per second squared, determined in clause A.1.

Using the pair of values $\{t_m, \Delta t\}$ obtained for each of the oils, plot the curve

$$\Delta t = f(t_m)$$

This curve enables Δt to be determined for each sample of phenolic resin tested from the flow time t_m measured for that resin.

Knowing Δt enables the viscosity of the test sample of the phenolic resin to be calculated (see 4.2.6).

1) These standard reference oils are supplied by official organisations in various countries.

Annex B
(normative)

Determination of the viscosity using a Hoeppler dropping-ball viscometer

B.1 Measurement range and temperature range

Viscosity-measurement range: 0,6 mPa·s to 250 000 mPa·s

Dropping time for the ball:

> 60 s for ball No. 1

> 50 s for balls 2-4

> 30 s for balls 5 and 6

Temperature range: −thinsp;20 °C to +120 °C.

B.2 Principle

A ball is allowed to travel down an inclined cylindrical tube filled with the liquid under test (the ball will execute a rolling/sliding motion), and the time taken for the ball to cover the distance between two fixed points is measured. The dynamic viscosity of the liquid, expressed in millipascal seconds (mPa·s), is calculated from this time.

B.3 Apparatus

B.3.1 Dropping-ball viscometer (see figure B.1).

The apparatus consists of an inclined measurement tube, filled with the liquid under test, down which a ball travels. The tube is made of thermally stabilized borosilicate glass with a linear coefficient of expansion of $3{,}3 \times 10^{-6}\ °C^{-1}$. Six balls having diameters ranging from 15,81 mm to 11 mm (see table B.1) are provided with the apparatus. Balls 1 and 2 are made of the same material and thus have the same coefficient of expansion as the tube.

Since the constant K of the apparatus depends on the diameter of the tube, the values given in table B.1 are only valid for a tube 15,94 mm in diameter. In addition, the shorter the measurement distance, the greater the influence of any flaws in the tube or the ball on the result. With the specified tube diameter of 15,94 mm, the diameter of ball No.1 shall not be larger than 15,82 mm in order to keep errors within acceptable limits.

The measurement tube has two circular marks denoting the limits of the measurement distance. The tube is surrounded by a thermal jacket comprising a glass tube filled with a suitable liquid, and measurement tube plus jacket are mounted on a stand in such a manner that, while the determination is in progress, the axis of the tube is inclined at an angle of $10° \pm 1°$ to the vertical. The measurement tube and the jacket can be inverted by rotating them together about their mounting point on the stand, thus enabling the ball to be brought back to its starting position. The ends of the measurement tube are closed by two plugs, one of which contains a capillary joined to a hollow space. This type of closure prevents unacceptable pressure variations occurring, as well as the entry of air due to temperature fluctuations. The liquid under test is completely surrounded by the jacket and plugs, thus avoiding the risk of evaporation and the formation of a skin. The stand is equipped with a spirit-level and feet with adjusting screws to level the apparatus. A removable thermometer (B.3.2) is provided, with a suitable range and suitable precision.