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Glass — Viscosity and viscometric fixed points —

Part 5 :
Determination of working point by sinking bar viscometer

Reference number
ISO 7884-5 : 1987 (E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7884-5 was prepared by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus.*

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0 Introduction

International Standard ISO 7884, *Glass — Viscosity and viscometric fixed points*, consists of the following separate parts :

Part 1 : Principles for determining viscosity and viscometric fixed points.

Part 2 : Determination of viscosity by rotation viscometers.

Part 3 : Determination of viscosity by fibre elongation viscometer.

Part 4 : Determination of viscosity by beam bending.

Part 5 : Determination of working point by sinking bar viscometer.

Part 6 : Determination of softening point.

Part 7 : Determination of annealing point and strain point by beam bending.

Part 8 : Determination of (dilatometric) transformation temperature.

1 Scope

This part of ISO 7884 specifies a method of determining the working point of glass by means of the sinking bar viscometer. This method has been found useful for characterizing the low-viscosity range of glass working. This viscometric fixed point can be used in determining the viscosity-temperature relationship (see ISO 7884-1).

2 Field of application

This method is applicable to all glasses of normal bulk-production compositions unless devitrification or evaporation of volatile components takes place during the preparation or testing of the specimen.

The working points range between 800 and 1 200 °C, depending on the type of glass.

* 1 dPa·s = $1 \frac{dN \cdot s}{m^2}$ = 1 P

(P is the symbol for poise)

3 Reference

IEC Publication 584-1, *Thermocouples — Part 1 : Reference tables.*

4 Definitions

For the purposes of this part of ISO 7884, the following definitions apply.

4.1 working point : Temperature at which the melt of usual silicate glasses possesses an equilibrium viscosity of 10^4 dPa·s*.

4.2 sinking bar temperature : Working point as determined by means of the sinking bar method.

4.3 sinking bar method : Simple procedure for determining rapidly the viscosity within a range of about $10^{3,7}$ to $10^{4,5}$ dPa·s as explained in clause 5.

5 Principle

A vertically positioned narrow metal rod (the bar) of diameter d is allowed to sink under a force F (its own weight) into the melt. From the rate of sinking of the bar into the melt, the dynamic viscosity η of the melt is calculated. Under the geometrical conditions specified in 6.1 and 6.3, the deviations of this method from equation (1) are small compared with the experimental uncertainties as specified in 9.2.

$$\eta = C \cdot m \cdot \frac{t}{l^2} \qquad \ldots (1)$$

where

C is a constant;

l is the depth of sinking into the melt;

m is the mass of the bar;

t is the sinking time corresponding to l.

If fixed values of the depth l and the mass m are specified, a constant K for the bar may be defined according to equation (2):

$$\eta = K \cdot t \qquad \ldots (2)$$

In this part of ISO 7884, the depth of sinking and the mass of the bar are specified as l = 20 mm and m = 0,902 g, respectively.

NOTE — The buoyancy acting upon the immersed part of the bar is taken into account for the density of the reference glass used for calibration (see 6.1). The buoyancy correction for other values of glass density is neglected.

6 Apparatus

6.1 Sinking bar

Bar made from platinum-rhodium alloy with a mass fraction of rhodium between 0,2 and 0,3, with diameter 0,5 mm and mass 0,902 g (corresponding to a length between 240 and 245 mm depending upon the density of the platinum-rhodium alloy used). The ends of the bar shall be hemispherical.

NOTE — With such a bar, for a viscosity of 10^4 dPa·s the time for sinking to a depth of 20 mm is 60 s. This corresponds to a bar constant K of about 140 to 170 dPa·s/s.

For routine measurements it is convenient to have available several bars made from the same batch of the alloy. Used bars shall be gently cleaned of adherent glass residues by sudden heating and chilling, or by dissolving the glass in hydrofluoric acid. Afterwards they shall be straightened before re-use.

6.2 Furnace

Electrically heated tube furnace capable of achieving temperatures up to 1 200 °C. The length of the heated tube shall be about 200 mm, and its inner diameter at least 30 mm. Within a zone of 40 mm in length in which the crucible is placed, the temperature shall be constant within 3 °C.

6.3 Crucible

The inner diameter of the crucible at the surface of the melt shall be at least 23 mm. Its depth shall be 30 to 40 mm. Crucibles made from platinum or platinum alloys are preferred. For single use, crucibles made from non-porous refractory may also be used.

6.4 Temperature measuring and indicating instruments

6.4.1 The alumina-insulated platinum-10 % rhodium/platinum thermocouples (type S according to IEC 584-1) shall exhibit low thermal inertia (the diameter of the wires should not be greater than 0,5 mm). The wires shall have a sufficient length within the furnace (with respect to heat conduction along the wires). Platinum-metal-sheathed thermocouples should be preferred.

6.4.2 Control thermocouples should be located as close as possible to the furnace windings for fast response. The measurement thermocouple is placed near the wall of the crucible, dipping about 1 mm into the melt (see figure 1). In accordance with ISO 7884-1 the measurement thermocouple shall be calibrated and the calibration checked regularly.

6.4.3 The electrical output of the thermocouples shall be determined at zero current by means of potentiometers or high-resistance electronic amplifiers having a sensitivity of 10 µV or better. Precautions shall be taken that the ice-bath for the cold junction is maintained at 0 °C throughout the test. If the temperature measuring equipment is fitted with automatic cold junction compensation, the ice-bath may be omitted.

6.5 Timer

The sinking time shall be measured by means of a stopwatch having scale intervals of 0,1 s, or by an equivalent timer.

6.6 Other equipment

The following equipment is necessary.

a) **Clamp** for handling and holding the bar.

b) **Ammeter** for indicating the position when the bar tip touches the surface of the melt.

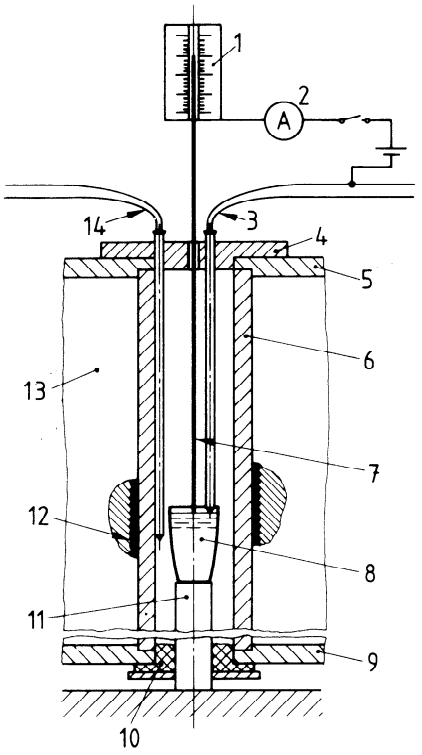
c) **Mirror scale** for parallax-free reading for measuring the depth of sinking of the bar; a groove should be ground in the mirror scale for gently guiding the upper end of the bar with minimum friction.

In place of the devices listed under b) and c) other suitable devices may be used.

Further devices may be useful for the adjustment of the zero position, e.g. cathetometer, adjustable clamping device to achieve central and vertical positioning of the bar (admittable maximum tilt, 5°), etc.

6.7 Assembly of apparatus

Figure 1 shows an example of the test assembly. It is convenient if the crucible is set on a fixed stand whereas the furnace can be moved downwards; this facilitates the filling of the crucible and removal of the test specimen. The measurement thermocouple (positioned according to 6.4.2) or the platinum alloy crucible serve at the same time as an electrode for the ammeter; the other electrode is the bar whilst it is fastened in the clamp. Draught within the furnace tube should be diminished by closing the orifice in the bottom of the furnace (e.g. by means of ceramic wool).



1 Mirror scale with groove (see also figure 2)
2 Ammeter with circuit
3 Measurement thermocouple
4 Cover
5 Furnace platen made of ceramics
6 Furnace tube made of ceramics
7 Bar
8 Crucible containing glass melt under test
9 Furnace platen made of ceramics
10 Ceramic wool (draught protection)
11 Crucible stand (e.g. made of ceramics)
12 Heater
13 Insulation
14 Control thermocouple

Figure 1 — Schematic example of the sinking bar viscometer

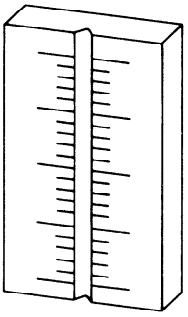


Figure 2 — Mirror scale with groove

7 Preparation of test specimens

The sample of the glass to be tested shall consist of pieces larger than 3 mm. If the pieces do not fit into the crucible, they shall be crushed carefully, avoiding any contamination (see ISO 7884-1 : 1987, sub-clause 7.1).

If a platinum crucible is used, the test specimen (about 20 to 30 cm^3) of the homogeneous glass sample is melted in that crucible. If a refractory crucible is used, it is more convenient, because of possible corrosion, to melt the test specimen in another furnace and to cast it into a mould having a form similar to that of the measurement crucible.

Ensure that no air bubbles are included in the molten sample. However, avoid melting during too long periods, especially in the case of a glass which devitrifies easily.

NOTE — If the working point lies above 1 150 °C, a simple preparation furnace for temperatures higher than 1 200 °C is convenient. In this furnace the glass can be melted sufficiently fast to produce a bubble-free sample, before it is introduced into the testing furnace.

8 Procedure

8.1 Calibration

Calibrate the test assembly by means of a reference glass[1], carrying out the test procedure described in 8.2 at that temperature which corresponds to the viscosity 10^4 dPa·s of the reference glass. The compositions of the reference glass and of the glass under test should be similar.

Calculate the bar constant K from the measured sinking time t_c by means of equation (3) :

$$K = \frac{10^4}{t_c} \qquad \ldots (3)$$

where

K is expressed in decipascal seconds per second;

t_c is expressed in seconds.

Repeat the calibration regularly and after any change in the apparatus.

The constant K determined as above is used in equation (2) for the determination of the viscosity of other glasses.

8.2 Measurement

Heat the specimen in the furnace to that temperature which is expected to correspond to a viscosity of 10^4 dPa·s.

NOTE — In the case of soda-lime glass, this temperature is about 1 000 °C.

1) See for example ISO 7884-1 : 1987, annex B, "Examples of certified reference glasses for viscometric calibration".

Before beginning the test, the temperature of the furnace shall be constant for about 10 min. Then hold the bar for about 1 min in the clamp just above the melt surface.

Keeping the bar as vertical as possible and in the axis of the crucible, lower it slowly in order to find the point of contact with the surface by means of the ammeter indication. In doing so, the upper end of the bar is guided in the groove of the mirror scale. Take the point of contact as the zero point of the upper end of the bar on the mirror scale to determine the depth of sinking *l*.

At the same time, release the bar from the clamp and start the timer. Stop the timer when the upper end of the bar has sunk 20 mm below the zero point on the mirror scale.

9 Expression of results

9.1 Determination of the sinking bar temperature

The viscosity is calculated from the measured sinking time by means of equation (2). Only occasionally will the viscosity be 10^4 dPa·s. Therefore, after the bar has been removed, a second run in accordance with 8.2 and using a new bar shall be carried out at a temperature which is estimated to be more appropriate. This process shall be repeated until the temperature corresponding to 10^4 dPa·s can be found by linear interpolation with respect to temperature and the logarithm of viscosity; the interval of interpolation shall not exceed 30 °C within the range of sinking times between 40 and 90 s.

9.2 Precision

The repeatability (one observer using the same apparatus) is 3 °C. The reproducibility (different observers and different apparatus) is 8 °C.

10 Test report

The test report shall include :

a) reference to this part of ISO 7884;

b) description of the sample;

c) method of sampling;

d) number of test specimens;

e) method of preparation;

f) type of sinking bar apparatus used;

g) calibration temperature;

h) sinking bar temperature, in degrees Celsius;

i) any change in the glass, observed during and/or after the test.

Annex

Bibliography

(This annex does not form an integral part of the standard.)

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