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Glass — Viscosity and viscometric fixed points —

Part 2 :
Determination of viscosity by rotation viscometers

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Foreword

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Glass — Viscosity and viscometric fixed points —

Part 2 :
Determination of viscosity by rotation viscometers

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 and field of application

This part of ISO 7884 specifies a method of determining, by means of rotation viscometers, the dynamic viscosity of glass and, in particular, the viscosity-temperature relationship at any temperature or viscosity within the range of measurement. This covers the ranges of viscosity for melting, refining and working of glass.

This method allows for continuous measurements and for measurements under various shearing stresses (i.e. for the determination of flow curves) in order to check whether or not the glass behaves as a Newtonian liquid.

Dependent on the particular viscosity-temperature relationship of the glass tested, the viscosity range covered by this method extends approximately from 10 to 10^8 dPa·s * in the temperature range from about 1 600 to 600 °C, the torque necessary ranging from 0,1 to 20 N·mm according to the construction of the apparatus. The method is applicable if the rotational frequency does not exceed 8 s^{-1}; however, at rotational frequencies above 1 s^{-1} it should be ascertained that inertia forces are negligible.

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

(P is the symbol for poise)

2 Reference

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

3 Definitions

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

3.1 Field of flow, crucible and plunger

3.1.1 field of flow : The whole gap filled by the molten glass sample and the spatial distribution of the flow velocities within it, including its boundaries.

3.1.2 crucible : The outer boundary of the molten glass sample corresponding to the inner surface of the crucible up to the level of the melt.

3.1.3 plunger : The inner boundary of the molten glass sample corresponding to the outer surface of the plunger up to the level of the melt.

3.2 Flow field coefficient and instrument constant

3.2.1 Rotation viscometers are either of the Searle type or of the Couette type, both of which allow the determination of the viscosity according to basic equation (1) :

$$\eta = f\frac{T}{n} \qquad \ldots (1)$$

where

η is the viscosity;

f is the flow field coefficient;

T is the torque applied to the plunger;

n is the rotational frequency either of the plunger (Searle type) or of the crucible (Couette type).

The **flow field coefficient** f is a function only of the geometrical shape of the field of flow and has the dimension of reciprocal cubic length.

The analytical calculation of the flow field coefficient is possible in a restricted number of geometries, e.g. in the following cases (see also annex A).

a) Crucible and plunger are shaped like infinite concentric cylinders; methods of eliminating experimentally the effect of the plane end surfaces are known from literature, but they are difficult to apply in the case of glass melts.

b) Crucible and plunger form two confocal rotation surfaces of the second order, e.g. two half-ellipsoids, cut perpendicularly to the axis of rotation.

If the shapes of the crucible and of the plunger are made up by several rotational surfaces, the boundaries of the perpendicular median cuts being irregular curves (e.g. cylinders with plane or cone-shaped or hemi-spherical ends), the flow field coefficient can be determined only by means of viscometric standard liquids with Newtonian behaviour.[1)]

3.2.2 If, for the torque, the length and/or the rotational frequency, units are used which do not correspond to the unit for the viscosity, and if the resulting factor is combined with the flow field coefficient, a new constant, the **instrument constant** k, can be defined by equation (2):

$$\eta = k \frac{T}{n} \qquad \ldots (2)$$

where

η is the viscosity measured, in decipascal seconds;

k is the instrument constant (numerical value), resulting from equation (3);

T is the torque, in newton millimetres;

n is the rotational frequency, in reciprocal seconds.

The instrument constant k is related to the flow field coefficient f by equation (3), if the flow field coefficient f is expressed in reciprocal cubic millimetres (mm^{-3}):

$$k = 10^7 f \qquad \ldots (3)$$

NOTE — The factor 10^7 in equation (3) results from the relation

$1\ N \cdot s/mm^2 = 10^7\ dPa \cdot s$

3.2.3 In many cases the rotational frequency n and the torque T are not read or recorded directly. For the sake of convenience all the factors needed for their calculation and the flow field coefficient can be combined into a **special instrument constant** k^*. This constant shall be determined by calibration using viscometric standard liquids with Newtonian behaviour.[1)]

3.2.4 The flow field coefficient f and the instrument constants k and k^* are, for Newtonian liquids, independent of the rotational frequency, of the torque and of the viscosity. They are slightly dependent on temperature because of the thermal expansion of the glass melt, of the crucible and of the plunger (see 7.3).

3.3 Torques

3.3.1 driving torque: The torque applied by the drive to the boundary surface rotating with the rotational frequency n.

3.3.2 frictional torque: Torque of the opposite sign to the driving torque, arising from the viscous flow between the two boundaries, the one rotating and the other being at rest.

3.3.3 torque of mechanical losses: The frictional resistance caused by influences other than the glass melt (e.g. friction in bearings, air friction; in the case of rotational vibrations, also the self-damping of the torsion spring).

3.3.4 In the case of stationary rotation, which is essential for obtaining correct results, these three torques add up to zero (action = reaction). Torques of mechanical losses, caused by the particular construction of the instrument, shall be eliminated through a correction.

4 Apparatus (see annex B)

4.1 Rotation viscometer

Two types of rotation viscometer comply with this part of ISO 7884:

a) **Viscometer of the Couette type** with adjustable, revolving crucible. The crucible stands on a turntable which is to be positioned in the furnace through its lower opening; the shaft of the plunger reaches through the upper opening of the furnace up to the torque-measuring device.

b) **Viscometer of the Searle type** with the crucible at rest and the plunger revolving, the revolving movement being induced through a shaft reaching out of the upper opening of the furnace. The torque-measuring device is applied to the shaft of the plunger.

Both types can be operated

a) either at fixed rotational frequencies (e.g. by means of a combination of synchronous motor and gear transmission, the variation range of the rotational frequency n being at

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

least 1:100) with a torque-measuring device (torsion wires, torsion spring or torsion balance) whose possible inaccuracy shall not exceed 2 % at a torque of about 5 N·mm;

b) or at fixed torques (e.g. by means of a weight and pulley system or by means of an electric motor) with a measuring device for the rotational frequency (frequency meter, electronic speedmeter with optical or inductive sensor, microscope for very low rates).

To protect these parts of the apparatus, special screening against heat or, if necessary, water-cooling is recommended.

4.2 Furnace

Electrically heated tube-shaped furnace, designed for a vertical working position, for temperatures up to 1 400 °C (in special cases up to 1 600 °C), with covers for the upper and the lower ends of the tube which shall be made of heat-resistant ceramic material. The temperature in the flow field area or in the adjacent space in the furnace shall be constant to ± 2 °C with respect to time, and the temperature gradient shall not exceed 1 °C/cm.

NOTE — This requirement is achieved by one or more of the following devices: extra heaters at the two ends of the ceramic tube; baffles made from noble metals (e.g. platinum); a suitable cover on the crucible; a thick-walled crucible made of noble metal. Good thermal insulation of the furnace is required in any case.

4.3 Temperature measuring and indicating instruments

4.3.1 The alumina-insulated platinum-10 % rhodium/platinum (type S according to IEC 584-1), or (for extensive use above 1 200 °C) platinum-30 % rhodium/platinum-6 % rhodium (type B according to IEC 584-1) thermocouples 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).

4.3.2 Control thermocouples should be located as near as possible to the furnace winding for fast response. The hot junction of the measurement thermocouple, however, shall be placed in the immediate vicinity of the flow field (the crucible). In accordance with ISO 7884-1, the measurement thermocouple shall be calibrated and the calibration checked regularly.

NOTE — Further improvement in the accuracy of the temperature determination is achieved by dipping the (electrically isolated) measurement thermocouple into the melt and/or by observation of the temperature distribution by means of two further thermocouples placed above and below the crucible. If the construction of the viscometer permits the positioning of the measurement thermocouple at the centre of the plunger, the best assignment of temperature to the shear area, which is mainly responsible for the measured viscosity, is achieved; in that case other special devices (see note to 4.2) may be omitted.

4.3.3 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 1 µV. 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 can be omitted.

4.4 Crucible and plunger

4.4.1 Crucibles

According to their chemical resistance to the glass melt under test, the crucibles shall be made of ceramic material (e.g. alumina) or noble metal. If ceramic crucibles are used (normally they can be used only once), it is recommended that such a crucible be placed in a larger crucible made of thin noble sheet-metal, in order to protect the furnace in case the ceramic crucible breaks. Noble metal crucibles (preferably platinum or platinum-rhodium alloys) require cleaning, e.g. in a hydrofluoric acid bath. The volume of the crucibles (sample volume) shall normally be between 20 and 250 cm^3.

4.4.2 Plungers

The plungers shall be made of noble metal, preferably of platinum-rhodium alloys. They shall be welded to a long thin shaft, reaching out of the furnace to the torque-measuring instrument. The shaft may be made entirely of noble metal, or alternatively only that part of it which plunges into the melt may be of noble metal, the part above the melt being made of ceramic material.

The lower end of a cylindrical plunger shall be cone-shaped, not plane, in order to avoid bubbles adhering to it; this would produce scatter in the results. Moreover, the plunger shall have no sharp edges, as sharp edges would be more easily attacked by the melt. Therefore, the plungers should be spheres, cylinders with hemi-spherical ends, or ellipsoids.

NOTE — The contribution to the torque of flow arising from the shaft can be restricted to less than 15 %.

If the flow field coefficient is to be determined by calculation, the shape of the plunger shall be chosen accordingly.

By means of a set of plungers of different sizes, the ratio of the sizes being for example 1:2, the possible range of measurement can be extended by about one order of magnitude.

4.5 Apparatus for quantifying the test sample

One of the following methods shall be used:

a) a balance and device for determining the density of the glass at room temperature (between 18 and 28 °C); or

b) a device for determining the distance between the surface of the melt and the upper edge of the crucible at a temperature above the melting range of the sample, e.g. at 1 000 °C; or

c) a device for the visual determination of the level of the melt in the crucible.

4.6 Devices for the adjustment of the flow field

4.6.1 Device for the adjustment of the position of the furnace and/or of the torque-meter, if necessary with a facility for swinging the furnace or the viscometer aside and precisely back in the same plane again.

4.6.2 Device for centring crucible and plunger.

4.6.3 Device for checking the concentric running of the plunger or of the crucible; deviations shall not exceed 1/100 of the plunger diameter; such faults normally originate from bends in the plunger shaft.

It is recommended to use a self-centring suspension device, e.g. a universal joint; its effectiveness shall be checked.

When the position of the apparatus is changed or the furnace and/or the measuring device are swung aside, the adjustment of the flow field shall be maintained.

4.6.4 Device to check the relative positions of crucible and plunger in the furnace, e.g. scale or marks or windows in the wall of the furnace.

NOTE — If it is impossible to avoid the use of windows, they should be of minimum size. Take care to minimize disturbance of the temperature homogeneity, e.g. by suitable wiring of the heaters.

4.7 Other equipment

The following may be necessary:

— crucible pincers with platinum points;

— rods of ceramic material, diameter between 3 and 10 mm, length about 400 mm;

— tools for crushing the glass;

— thermal insulating gloves;

— infra-red protective goggles;

— melting furnace.

5 Test specimen

5.1 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).

5.2 The quantity needed shall be determined according to the size of the flow field, known from the calibration experiment.

5.3 The glass specimen shall be melted either in the furnace of the viscometer or in a separate melting furnace; the melt shall be free from bubbles. Normally the bubbles rise quickly enough if the viscosity is about 1 000 dPa·s. The melting temperature shall be chosen accordingly. It shall be taken into account that, with increasing temperature (decreasing viscosity), the sample can change its properties through incongruent evaporation and/or changes in water content. For the treatment of melts, see also ISO 7884-1 : 1987, sub-clause 7.3.

NOTE — For the usual flat glasses a melting temperature between 1 200 and 1 250 °C is sufficient.

Glasses with an unusually high tendency to foam, evaporate or attack chemically the crucible and plunger materials give rise to special requirements for the preparation of the sample and for the materials which come into contact with the melt.

6 Procedure

6.1 Calibration

The rotation viscometer shall be calibrated by means of viscometric standard liquids with Newtonian behaviour, e.g. certified reference glasses (see ISO 7884-1 : 1987, annex B), unless the flow field coefficient and the instrument constant can be determined by calculation. When calibrating with standard liquids other than reference glasses, measurable heating by friction shall be avoided and the torque of mechanical loss shall be taken into account.

Thermocouples shall be calibrated by comparison with a calibrated standard and/or with thermometric fixed points.

Every calibration shall be repeated regularly.

6.2 Preparation

The crucible containing the molten glass test specimen shall be positioned in the heated furnace of the viscometer. The plunger shall be dipped slowly into the melt, down to its desired position relative to the position of the crucible, the viscosity of the melt being less than 1 000 dPa·s.

NOTE — It is advantageous to rotate slowly the plunger — or the cup in the case of a Couette-type device — while dipping the plunger slowly into the melt.

Then, the covers shall be fitted on the upper and lower orifices of the tube of the furnace; if necessary, the extra heaters shall be switched on.

6.3 Measurement

6.3.1 Control the electrical input of the furnace so as to heat the specimen to a certain temperature. Switch on the rotating system of the viscometer. Choose the measurement range for rotational frequency and torque so as to obtain a suitably high response from the instruments. When the temperature of the flow field and the reading of the torque-meter have become constant, read and record the output of the thermocouple, rotational frequency and torque. If necessary, repeat the measurement with other values of rotational frequency and driving torque.

NOTE — It will be difficult to carry out the measurements exactly at the nominated temperatures or viscosities. It is sufficient to measure close to those points and to find the nominated temperatures or viscosities by applying equation (2) given in ISO 7884-1 : 1987 for interpolation.

6.3.2 The measurement may also be carried out at varying temperature, as long as the viscosity can be measured within a sufficiently short time interval. The maximum rate of change of temperature which may be tolerated depends on the construc-

tion and materials of the furnace, crucible and plunger, on the dimensions of the flow field, the position of the thermocouple and the absolute value of the temperature. The tolerable rate of change of temperature shall be determined empirically by comparing these results with the results of the exact measurement carried out according to 6.3.1

6.4 Time schedule for measurements

Unless there is a special agreement, the glass shall be tested in its delivery state.

The time needed for the measurement shall be kept short in order to avoid, as far as possible, any alteration of the chemical and/or physical properties of the glass under test.

In addition to a possible chemical reaction between the glass melt and the materials of crucible and plunger, primarily two effects have to be taken into account.

a) **Selective evaporation**. If heated above 1 300 °C for a few hours, many glasses of bulk-production compositions show a considerable increase in viscosity, especially above 10^5 dPa·s. In this case the glass shall be melted at a temperature corresponding to a viscosity of about 1 000 dPa·s; the viscometric measurements shall then be carried out, first at lower temperatures; afterwards the glass shall be heated again, the repeatability of the measurements shall be checked and finally the range of higher temperatures (lower viscosities) shall be tested.

b) **Devitrification**. If the glass tends to devitrify, the readings for the driving torque and for the rotational frequency change with time at a constant temperature. Slight devitrification can be checked by repetition of measurements after different thermal pretreatments; e.g. temperatures in the range between 750 and 950 °C can be applied either by quickly cooling down from 1 200 °C or after a longer tempering period in the critical temperature range.

6.5 Final check

After the sample has cooled to room temperature, it shall be checked for visible inhomogeneities, e.g. devitrification, traces of dissolution of the crucible and plunger materials, bubbles, discolouration. Any unusual alterations of all materials which have been in contact with the melt shall be taken into consideration.

7 Expression of results

7.1 Measured viscosity

For the evaluation start from equation (2), using throughout the units specified in 3.2.2 and equation (3) for k.

7.2 Correction for frictional losses

If the driving torque T_D is measured or adjusted and if the causes for the torque T_L of mechanical losses lie between the torque-meter in the drive and the rotating plunger (or crucible, depending on the type of apparatus) equation (2) shall be replaced by equation (4):

$$\eta = k \frac{T_D - T_L}{n} \quad \ldots (4)$$

T_L can be developed into a series with increasing powers of the rotational frequency n:

$$T_L = T_{L0} + T_{L1} n + \ldots \quad \ldots (5)$$

If this series can be truncated after the constant term, T_L equals approximately T_{L0}, which can be determined simply by variation of T_D. If it is necessary to determine T_{L1}, this can only be achieved by means of several — at least two — viscometric standard liquids or by means of one standard liquid and the analytical calculation of the flow field coefficient f.

7.3 Dependence of the instrument constant on temperature

If the geometrical shape of the flow field remains constant and if a linear approach to the influence of the thermal expansion is sufficiently accurate, the dependence of the instrument constant k on the temperature ϑ can be calculated according to equation (6):

$$k(\vartheta) = k_0 [1 - 3\alpha(\vartheta - \vartheta_0)] \quad \ldots (6)$$

where

k_0 is the instrument constant at the reference temperature ϑ_0;

α is the coefficient of linear thermal expansion of the materials of crucible and plunger.

If the geometrical shape of the flow field changes with the temperature, α shall be replaced in equation (6) by another constant whose value is to be estimated from experiments.

NOTE — The dependence of the instrument constant k on temperature is illustrated by the following example, which holds true, as to the order of magnitude, for many typical rotation viscometers.

If the temperature rises by 100 °C, the instrument constant decreases by about 0,4 %. This influence is of practical importance and needs to be taken into account if the viscometer is calibrated at room temperature; it can be neglected, however, if the calibration is carried out at elevated temperatures, not too far from the temperature range of the measurements.

7.4 Evaluation of measurements

The output of the thermocouples shall be read or recorded; the corresponding temperatures are to be taken from the calibration (see 6.1). The instrument constant k shall be determined for these temperatures according to 7.3. The instrument constant k and the measured values of the torque T_D and the rotational frequency n shall be inserted in equation (4) and the dynamic viscosity η shall be calculated accordingly.

In many cases, the torque of mechanical losses is negligible. If not, it shall be determined according to 7.2 and inserted in equation (4).

7.5 Presentation of results; precision

The measurements shall be used for the numerical or graphical representation of the viscosity-temperature relationship.

The precision is influenced mainly by the scatter of several readings at constant temperature, the deviation of the measured points from the curve of best fit, the uncertainty in the determination of the temperature and of the mechanical values (torque and rotational frequency) and the uncertainty in the determination of the flow field coefficient or the instrument constant.

Errors should be presented according to ISO 7884-1.

8 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) time and temperature of melting down the test specimen;

f) type of rotation viscometer used;

g) shape, dimensions and materials of crucible and plunger;

h) type and position of the thermocouples;

i) complete time-temperature schedule of the measurement;

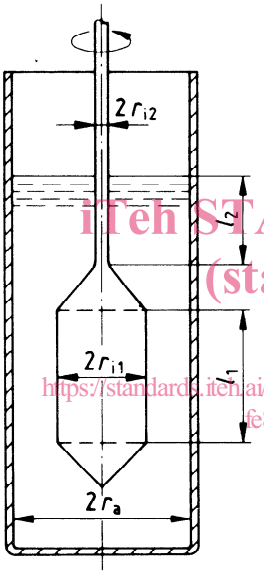
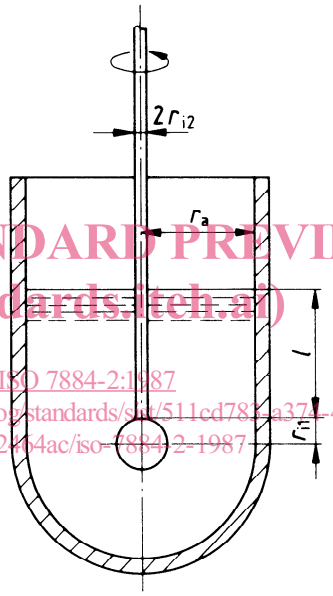
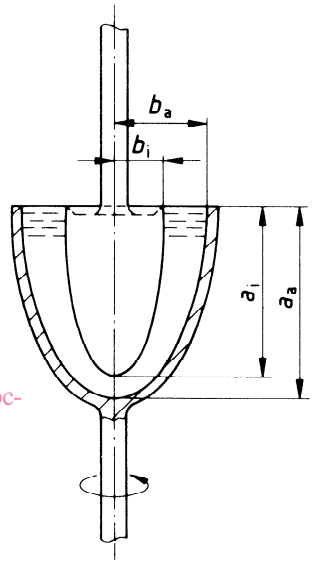
j) viscosity-temperature relationship of the sample as described in ISO 7884-1;

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

Annex A

Typical examples of flow fields in rotation viscometers

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

Viscometer **Plunger** **Crucible**	Searle-type Cylinder with cone-shaped ends, cylindrical shaft Cylinder with plane bottom	Searle-type Sphere at end of long cylindrical shaft Cylinder with hemispherical bottom (Tammann-crucible)	Couette-type Confocal half-ellipsoids (rotation ellipsoids)
Median section of flow field			
Determination of the flow field coefficient f	Relative	Relative; approximately absolute if $r_{i1}/r_{i2} \geqslant 5$ and $r_a/r_{i1} \geqslant 4{,}5$	Absolute or relative
Formulae for calculating or estimating f	**Cylinder and crucible wall:** $f_1 = \frac{1}{8\pi^2} \times \frac{r_a^2 - r_{i1}^2}{l_1 \cdot r_{i1}^2 \cdot r_a^2}$ **Shaft:** $\lim_{r_a \to \infty} f_2 = \frac{1}{8\pi^2 l_2 \cdot r_{i2}^2}$	**Sphere:** $\lim_{r_a \to \infty} f_1 = \frac{1}{16\pi^2 r_{i1}^3}$ **Shaft:** $\lim_{r_a \to \infty} f_2 = \frac{1}{8\pi^2 l \cdot r_{i2}^2}$ $f \approx f_1 f_2/(f_1 + f_2)$	$f = \frac{3A}{16\pi^2 c^3}$ where $c^2 = a_a^2 - b_a^2 = a_i^2 - b_i^2$ and $A = c\left(\frac{a_i}{b_i^2} - \frac{a_a}{b_a^2}\right) - 2{,}302\,6 \lg\left[\frac{b_a (a_i + c)}{b_i (a_a + c)}\right]$ Limit value for large crucibles: $\lim_{\substack{b_a \to \infty \\ b_a/a_a \to 1}} A = \frac{c \cdot a_i}{b_i^2} - 2{,}302\,6 \lg\left(\frac{a_i + c}{b_i}\right)$
Disregarded in calculation	Cone-shaped ends. Transition area cones/cylinder and upper cone/shaft. Thermocouple, if plunged into melt.	Influence of crucible. Transition area sphere/shaft. Thermocouple, if plunged into melt.	Thermocouple, if plunged into melt.