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Glass — Viscosity and viscometric fixed points —

Part 3 :
Determination of viscosity by fibre elongation viscometer

Reference number
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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 7884-3 was prepared by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Glass — Viscosity and viscometric fixed points —

Part 3 :
Determination of viscosity by fibre elongation viscometer

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 dynamic viscosity of glass by measuring the elongation of a glass fibre under a defined uniaxial stress. In addition, the viscosity-temperature relationship and the dependence of the viscosity on the thermal history can be determined.

NOTE — Using this method, all parts of the tested material are subject to the same mechanical stress when the shape of the fibre is correct, the glass is homogeneous and isotropic, and the influence of gravity on the fibre itself can be neglected. The deformation is a pure elongation instead of a simple shear. Without major uncertainty, the determination of the shear viscosity by an elongation experiment is possible only when the material behaves like a Newtonian fluid.

The knowledge of equilibrium viscosities in the low-temperature range is useful for fitting suitable viscosity-temperature formulae over the whole range of glass viscosities and for a more detailed survey on thermal history influences than can be derived from fixed-point methods alone.

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

(P is the symbol for poise)

2 Field of application

The method is applicable mainly in a viscosity range from 10^8 to 10^{13} dPa·s* but special devices might be able to extend the range up to $10^{14.5}$ dPa·s. This corresponds to a range of temperature from 800 to 400 °C for all glasses of normal bulk-production compositions.

NOTES

1 In the viscosity range above 10^{12} dPa·s the thermal equilibrium of the liquid structure is noticeably delayed following a temperature change. For tests within that range it should be agreed whether it is necessary to wait for the final equilibrium viscosity at a given temperature, or to take the viscosity value corresponding to a conventional temperature-time programme. In the latter case a non-equilibrium viscosity is denoted by η_{neq}. The symbol η without index denotes always equilibrium viscosity.

2 The softening point, the annealing point, and the strain point are conventional temperature points (so-called fixed points), which are obtained by special test procedures in appropriate, well-defined apparatus (see ISO 7884-6 and ISO 7884-7). These points characterize the flow behaviour of glass in the range of softening and glass transition, but only approximate viscosity values are assignable.

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 Sample, test specimen, fibre, ball

The **test specimen** is prepared from a molten test portion of the **sample**. The **fibre** is the cylindrical part of the test

specimen with constant cross-section $S = \pi d^2/4$ over its length l. The diameter d of the fibre is small compared with the length l.

Each end of the fibre is melted into a **ball**, which enables the test specimen to be suspended vertically, fixed at its upper end and connected to a loading device at its lower end.

4.2 Load, loading pieces, dead-weight

The **load** consists of all parts of the apparatus on which gravity acts to produce a force on the lower end of the fibre — i.e. the **loading pieces** (variable), and the device for suspending the loading pieces below the specimen (a given value for the apparatus) — plus the weight of the lower ball (to be estimated after preparing the test specimen). The load produces the force F_0 acting on the test specimen in the axial direction.

The **dead-weight** stems from the part of the fibre below the cross-section under consideration (see 9.2). The force due to gravity varies linearly from zero at the lower end to $g \cdot \varrho \cdot l \cdot S$ at the upper end of the fibre (g and ϱ being the acceleration of free fall, and the glass density, respectively).

4.3 Effect of surface tension

The effect of surface tension is to decrease the surface area of the specimen, i.e. to shorten the fibre. Therefore, it is a force which acts upon the fibre in the opposite sense to that of the gravitational force of the load and of the dead-weight.

5 Principle

5.1 Basic relation

Consider the force F_0 of the load acting on a specimen prepared from a glass with Newtonian behaviour and showing no defects. When the effects of dead-weight and surface tension are negligible and all elastic deformations by the loading become constant, there exists to a good approximation an extensional flow at constant volume as described by equation (1):

$$\frac{dl}{dt} = \frac{1}{3\eta} \times \frac{F_0 l^2}{V} \qquad \ldots (1)$$

where

η is the dynamic viscosity;

$V = l \times S$ is the volume of the fibre (the other symbols have the meaning as introduced above).

During the time interval Δt of the measurement, the fibre elongates from the initial length l_1 to the final length l_2. Then the viscosity is calculated using equation (2):

$$\eta = \frac{g}{3} \times m \times \frac{\Delta t}{V} \times \frac{l_2 \times l_1}{l_2 - l_1} \qquad \ldots (2)$$

where

g is the average acceleration of free fall;

m is the mass of the load that produces the force F_0.

In practice it is often more convenient to use Δl, i.e. the change in length $l_2 - l_1$, which is measured using a different device from that for l_1. In this case equation (2) can be re-written as

$$\eta = 32\,700\, m\, \Delta t\, \frac{l_1}{V} \times \frac{1 + (\Delta l / l_1)}{\Delta l / l_1} \qquad \ldots (3)$$

In equation (3) m is expressed in grams; l_1, l_2 and Δl in millimetres; V in cubic millimetres; Δt in seconds; and η in decipascal seconds (dPa·s).

NOTE — In calculating the viscosity, corrections sometimes have to be applied (see 9.2).

5.2 Preliminary estimation of time interval and elongation

The table shows the expected values of the time interval Δt_s for given loads in relation to the viscosity. The values are calculated for a fibre with a diameter $d_1 = 0{,}737$ mm at the beginning of the measurement, and an elongation of 2 % during the time interval Δt_s ($\Delta l / l_1 = 0{,}02$). Between the two heavy stepped lines in the table the sensitivity of the measuring device for the elongation Δl should be increased by the factor 10 (the elongation should be decreased by the factor 1/10) for shortening the time interval Δt_s by the factor 1/10. Below the lower line the factor is 100 instead of 10 (1/100 instead of 1/10).

Table — Time interval Δt_s for an extension $\Delta l / l_1 = 0{,}02$ under a load of mass m (as corrected for dead-weight and surface tension), for a fibre of initial diameter $d_1 = 0{,}737$ mm

Viscosity dPa·s	Time interval Δt_s in seconds for load of mass 1 g	4 g	16 g	64 g	256 g
10^8	25,6	—	—	—	—
10^9	256	64	—	—	—
10^{10}	2 560	640	160	40	—
10^{11}	25 600	6 400	1 600	400	100
10^{12}	256 000	64 000	16 000	4 000	1 000
10^{13}	—	—	160 000	40 000	10 000
10^{14}	—	—	—	—	100 000

For other diameters of the fibre the time interval $\Delta t'_s$ for an elongation of 2 % can be estimated from equation (4):

$$\Delta t'_s = \Delta t_s \cdot f_t \qquad \ldots (4)$$

The value of the dimensionless conversion factor f_t as a function of the diameter is found from the diagram in figure 1.

The estimated values Δt_s and $\Delta t'_s$ derived from the table, figure 1 and equation (4) give only a survey of the expected time required for the measurement and an estimation of the whole elongation of the fibre during a sequence of measurements on the same fibre. For the quantitative evaluation of the elongation experiment, however, the starting point is equation (3), using the actual values (see clause 8).

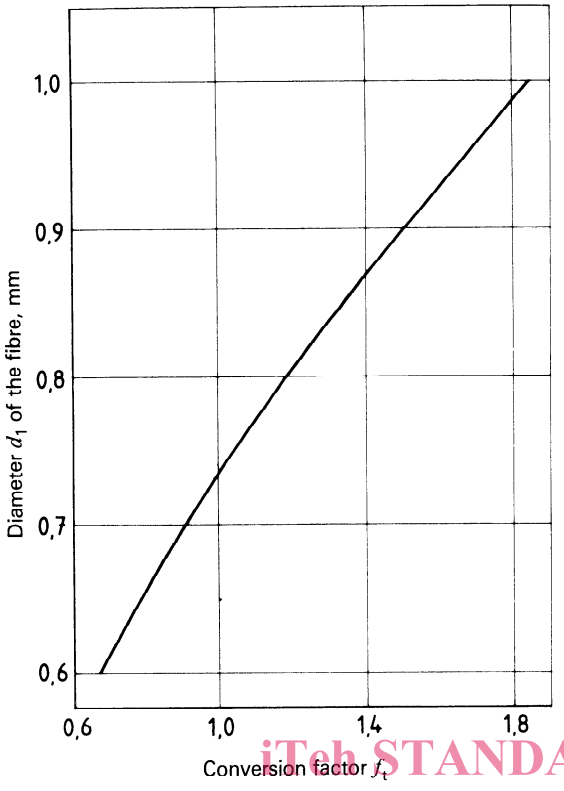


Figure 1 — Conversion factor f_t as a function of the fibre diameter d_1

6 Apparatus

6.1 Viscometer furnace

Electrically heated furnace for temperatures at least up to 900 °C. The furnace shall be capable of accepting thermocouples for measuring the temperature and its distribution along the specimen. The temperature gradient at the locus of the specimen shall not exceed 0,2 °C/cm.

The furnace heaters shall be controlled by a device which ensures that the temperature remains constant with respect to time, within the working space of the furnace, to ± 1 °C or better. Linearly increasing time-temperature programmes with a maximum rate of 6 °C/min shall be achievable by the same device.

The furnace and its control device shall have characteristics such that the desired time-temperature programme is attained at the latest 5 min after starting from a constant initial temperature. The value of the rate shall be held within ± 10 % throughout the determination.

6.2 Temperature measuring and indicating instruments

6.2.1 The alumina-insulated platinum-10 % rhodium/platinum (type S according to IEC 584-1) thermocouples, or nickel-chromium/nickel (type K 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).

6.2.2 Control thermocouples should be located as close as possible to the furnace windings for fast response. The hot junctions of the measurement thermocouple, however, shall be placed in the immediate vicinity of the specimen (see thermocouple A in figure 2). The temperature distribution along the fibre shall be monitored by suitable devices such as

— a high-mass block of nickel or silver around the fibre, with a number of holes for fixed thermocouples; or

— a mobile thermocouple (such as thermocouple B in figure 2).

In this case care should be taken that the thermocouple does not affect the temperature of the fibre (see ISO 7884-1 : 1987, sub-clause 5.3).

In accordance with ISO 7884-1 the measurement thermocouples shall be calibrated and the calibration checked regularly.

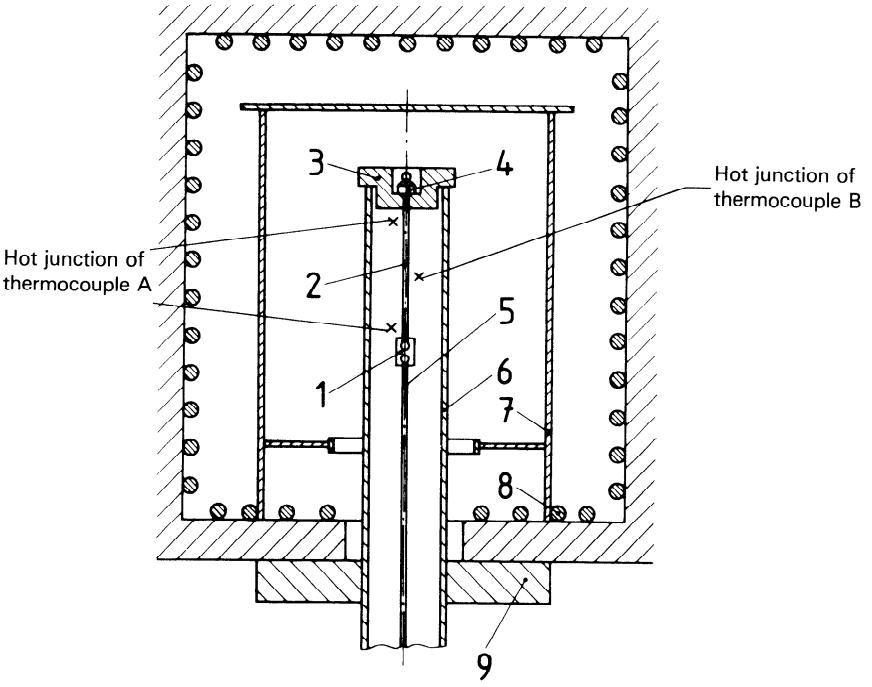
6.2.3 The electrical output of the thermocouples shall be determined at zero current by means of potentiometers, or high-resistance electronic amplifiers having a sensitivity of 1 µV for type S (according to IEC 584-1), or 4 µV for type K (according to IEC 584-1) thermocouples. Precautions shall be taken 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.

6.3 Hanging device for test specimen and load

6.3.1 The construction of the support for the test specimen depends on the type of furnace used. When the top of the working space of a chamber furnace is closed (as shown in figure 2), then a tube of fused silica is used as a support stand. The tube projects into the working space of the furnace from below. The centred support plate on top of the stand is made from ceramic or from stainless steel. The test specimen is suspended by its upper ball which locates in a central recess in the support plate. Because of possible adhesion of the glass, a centrally bored ceramic bead of appropriate dimensions (only to be used once) should be placed between the support plate and the upper ball of the test specimen.

6.3.2 When using a vertically mounted muffle furnace (both ends open), the fibre is connected to an upper rod of fused silica. Examples of devices for connecting the test specimen to the rod (by means of balls) are shown in figures 3 and 4. It is essential that the rod and the fibre hang vertically, coaxially and centrally in the tube of the furnace. Aside from the support, the top of the furnace tube should be covered.

6.3.3 The loading linkage consists of a rod (made of fused silica, about 1 mm in diameter) below the test specimen, with fused balls on both ends and connectors as shown in figure 3 or 4. Below the lower connection (outside the furnace) the attachment of the load carries a suitable indicator for the elongation measurement (marker or transducer core). For the medium range of viscosity the total mass of all parts of the loading linkage device should amount to approximately 4 g. In the range of higher viscosities and heavier loads the diameter of the silica rod should be greater.



1 Connector for the lower ball of the test specimen and the loading device (for connectors offering different magnitudes of load, see figures 3 and 4)

2 Test specimen

3 Support plate

4 Upper ball of the test specimen

5 Loading device (only the rod is shown)

6 Support stand: tube of fused silica

7 Screening between working space and heaters

8 Chamber furnace with heaters

9 Cover ring below the furnace (fixed at the silica tube)

Figure 2 — Fibre elongation method: example of a testing device (schematic)

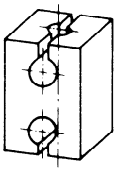


Figure 3 — Connector block (metal) bored to act as hanging device

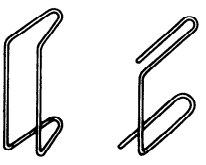


Figure 4 — Connector hooks (wire) to act as hanging devices

6.3.4 When measuring within the range from 10^8 to 10^9 dPa·s, the loading linkage device consists of a connector and lower rod, with a diameter of about 0,5 mm, without further devices below the end of the rod (this end is below the furnace and acts as indicator). The total mass of this device should amount to approximately 1 g.

NOTE — The measurement of the elongation by means of a travelling microscope is facilitated when the lower end of the rod is gently guided with minimum friction.

6.4 Loading pieces

A set of loading pieces made from brass, with masses determined to the nearest 0,01 g. The pieces shall be fitted by suitable attachment devices (e.g. hooks). It is convenient to choose pieces of masses such that the total load corresponds nearly to the values given in the table. When the mass of the loading linkage device (in accordance with 6.3.3) is 4 g, then the appropriate masses of the pieces are 12 g, 60 g and 252 g, respectively.

6.5 Extensometer arrangements

6.5.1 Moving marker below the furnace as an indicator of the elongation of the fibre.

6.5.2 Device for determining the fibre elongation Δl during the time interval Δt.

A minimum sensitivity of 0,02 mm is needed.

The instrument for observing the downwards moving marker shall be locked in position during any one single determination of Δl. It shall be possible to position it in advance so that the whole elongation length Δl in a determination is within the range of observation. Errors arising from incorrect graduation of the scale, or deviations from linearity of the display, shall not exceed 0,01 mm.

NOTE — Suitable devices are for example travelling microscopes with micrometer scales, or linear differential transformers (core and coil separated from each other). The use of different ranges of sensitivity is an advantage.

6.5.3 Device for determining the length l_1 over the whole amount of elongation $(l_1 - l_0)$ from the original length l_0 after preparing the specimen at room temperature (18 to 20 °C) to the initial length l_1 before starting the time interval Δt, see equation (2). The length l_0 shall be determined to the nearest 0,2 mm. The last value of $l_1 - l_0$ at the end of a sequence of measurements should not exceed $0{,}1 \times l_0$.

NOTE — The device might consist of, for example, a cathetometer fitted with scale and vernier, carrying the microscope or the coil of the differential transformer.

6.5.4 Timer for determining the interval Δt [see equations (2) and (3)], ranging from 20 to 2 000 s, with a least count of 0,1 s. The timer shall be corrected for systematic errors greater than 0,2 %.

6.6 Equipment for preparation of test specimens

6.6.1 Blast flame burner or **electrically heated furnace** for melting the sample and drawing fibres.

6.6.2 Two rods made from platinum metal alloys, ceramics or hard glass for flame-working a sample portion.

6.6.3 Platinum crucible and **rods** made from platinum metal alloys or fused silica, when an electrically heated furnace for the preparation is used.

6.6.4 Slide-gauge with vernier graduation 1/10 for the determination of the fibre length.

6.6.5 Micrometer caliper, with a least measurement of 0,005 mm, for measuring the fibre diameters. The surface of the fibre shall not be damaged by the advancement of the spindle head.

Instead of the caliper, a microscope with micrometer scale may be employed by experienced users.

7 Sample and test specimen

The sample shall be uniform, bubble-free and homogeneous.

7.1 Preparation of a test specimen by flame-working

Melt a test portion of about 2 to 3 cm^3 of the sample in the flame. By turning the test portion between the rods, form a ball. Remove the test portion from the flame and draw out the ball to a long fibre. Proceed quickly because of possible evaporation of the more volatile components of the glass.

NOTE — To produce sufficient regularity of the diameter along the fibre, a certain experience of the preparation technique is required, especially with respect to the

- temperature at the beginning of drawing;
- temperature-viscosity relationship of the glass;
- force and speed of drawing;
- uniformity of drawing; and
- conclusion of drawing at the correct time.

7.2 Preparation of a test specimen in a furnace

The method applies to glasses with a tendency to crystallize.

A test portion of about 100 g of the sample is melted in a platinum crucible in the electrically heated furnace. The glass may be delivered in any form, but the diameter for grains should not be less than 3 mm. A cylindrical rod of platinum, ceramics or fused silica is dipped into the almost bubble-free molten test portion of the glass. After the rod tip is wetted by the glass, it is drawn upwards (see note to 7.1). The rod may be drawn by hand or any suitable device.

7.3 Finishing the test specimen

The length of the test specimens should be between 50 and 100 mm before beginning the measurement. A suitable piece of the drawn fibre (see 7.1 or 7.2), 20 to 30 mm longer than the desired fibre length of the test specimen, is selected on the basis of uniform diameter and roundness. By flame-working, both ends are melted down to balls about 2 mm in diameter. Take care that the balls are centred on the fibre axis.

After the test specimen has cooled, the length of the fibre (between the balls) is determined to the nearest 0,2 mm. Generally, the diameter shall be between 0,6 and 0,8 mm. For measuring viscosities ranging from 10^8 to 10^9 dPa·s, diameters up to 1 mm are acceptable. A number of determinations of the diameter shall be made, equally distributed over the length of the fibre, in directions perpendicular to the fibre axis. The maximum and minimum diameters shall not differ by more than 2,5 % of the mean. The arithmetic mean of the diameters measured shall be used for calculation of the volume V.

That end of the fibre which has the larger diameter shall be chosen as the upper (fixed) end of the test specimen. The test specimen shall be free from scratches, striae, devitrifications or other inhomogeneities. There shall be no necking of the fibre at either end adjacent to the balls.

7.4 Etching of the test specimen

The surface layer resulting from the drawing process of the glass fibre shall be removed by etching the test specimen in an aqueous solution of hydrochloric, sulfuric or nitric acid [$c(HCl)$, $c(1/2H_2SO_4)$, or $c(HNO_3)$ = 2 mol/l], containing 3 % (m/m) of hydrofluoric acid, for 0,5 to 1 min at room temperature. The test specimen shall be rinsed with distilled water, then dried.

7.5 Special treatment

For glasses whose properties might be altered very easily, conditions should be agreed for the treatment of the sample, e.g. the type of burner flame, exclusion of the application of any flame in the preparation of the test specimen, the use of an inert gas atmosphere.

8 Procedure

8.1 Calibration

Generally, the fibre elongation measurement is an absolute determination of the viscosity, i.e. the viscosity is calculated (see 5.1 and 9.2) from the dimensions of the finished test specimen. The thermocouples shall be calibrated by comparison with a certified standard thermocouple. Repeat the calibration regularly.

8.2 Determination of the viscosity at constant temperature

8.2.1 Heat the viscometer furnace to the desired measuring temperature. Suspend the specimen and the loading linkage from the support, centre and adjust for free hanging with respect to the wall of the furnace. Observe as soon as possible the position of the indicator by means of the cathetometer. Assign this position to the original length l_0 of the test specimen.

8.2.2 When viscosities from 10^8 to 10^9 dPa·s are to be determined by using the loading device described in 6.3.4, wait until there is a nearly constant elongation rate and take that value for the viscosity calculation. The fibre should not elongate by more than 2 % during one test.

NOTE — During a 2 % elongation of the fibre, the instantaneous speed of the descending indicator increases by 4 %. The time for a 2 % fibre elongation is estimated in the second column (for a load m = 1 g) of the table.

Establish the next measuring temperature as soon as possible. Wait once more until there is a nearly constant elongation rate, and so on. When viscosities less than 10^9 dPa·s are to be determined by using the loading device described in 6.3.3 without loading pieces [see the third column (for a load m = 4 g) of the table], use the same procedure as described above.

8.2.3 For measuring viscosities not less than 10^9 dPa·s, the loading pieces have to be used (see the fourth to sixth columns of the table). First heat the specimen to the required temperature without loading it with the loading pieces. When the indicator is descending at nearly constant speed, or does not apparently change its position, attach the loading pieces and determine the rate of elongation once an approximately constant elongation rate is observed. The elongation shall not exceed 2 % in one test. By preference smaller elongations, as determined by more sensitive measuring devices, should be determined if possible. Remove the loading pieces during every change of the measuring temperature.

If viscosities not less than 10^{11} dPa·s are to be measured, the delayed elasticity of the glass under test has to be taken into consideration. Therefore, wait once more for a constant rate of elongation after attaching the loading pieces.

8.3 Determination of the viscosity at increasing temperature

When a heating rate of $\Delta\theta/\Delta t$ (in degrees Celsius per minute) is provided, bring the furnace to a temperature which is less than the desired starting temperature of the increase by an amount equal to five times the value of $\Delta\theta/\Delta t$. Then introduce the test specimen, temper for 10 min, load and start the temperature-time programme. After 5 min begin to measure the elongation and repeat the measurement at equal time intervals. Measure the temperature using the same intervals but displaced against the elongation readings by half an interval. (The continuous registration of both readings may be preferred.)

The method applies to initial temperatures equal to or some degrees higher than the transformation temperature or the annealing point.

NOTE — When the viscosity-temperature behaviour is completely unknown, make a preliminary determination, choosing higher rates of temperature increase, starting from the transformation temperature or annealing point.

When this method is applied, a difference between the reading and the true temperature of the test specimen is possible,

depending on the rate of heating. Within the range of equilibrium viscosity, i.e. below 10^{12} dPa·s, the difference can be determined experimentally by the procedures described in 8.2, and the measurements shall be corrected for this purpose. Within the range in which non-equilibrium viscosities occur, i.e. above 10^{12} dPa·s (see note 1 to clause 2), different laboratories shall agree on the time-temperature programme chosen.

8.4 Termination of the tests

Finish the test (if not terminated before) when the fibre has elongated by more than 10 % of its original length l_0. Remove the test specimen from the furnace, allow it to cool and inspect it for devitrification, striae, scratches, and neckings. Determine once more the length as well as the diameter and its longitudinal and circumferential variations. The difference between the maximum and minimum diameters shall not exceed 4 % of the mean.

9 Expression of results

9.1 Fibre volume

The increase in the fibre volume by thermal expansion never exceeds 2 %. With respect to the precision of the temperature measurement, this error in volume of the fibre can be neglected.

9.2 Correction of the load

When the loading mass is less than 4 g, corrections shall be made, to take into consideration the influence of the deadweight and the surface tension of the glass, using equations (5) to (8):

$$\eta = 32\,700 \times \Delta t \times \frac{l_1}{V} \times \frac{1 + (\Delta l / l_1)}{\Delta l / l_1} \times m^* \qquad \ldots (5)$$

where

$$m^* = m + f_\varrho - f_\sigma \qquad \ldots (6)$$

$$f_\varrho = 0{,}000\,5 \times \varrho \times V \qquad \ldots (7)$$

$$f_\sigma = 0{,}000\,18 \times \sigma \times \sqrt{\frac{V}{l_1}} \qquad \ldots (8)$$

where

ϱ is the density of the glass, in grams per cubic centimetre;

σ is the surface tension of the glass, in millinewtons per metre;

η, Δt, l_1, V and Δl are explained in 5.1.

When the value of the surface tension is unknown, estimate its influence by putting $\sigma \approx 300$ mN/m.

9.3 Evaluation of the measurements at constant temperatures

Insert in equation (3) the values of the fibre volume V in cubic millimetres, of the time interval Δt in seconds, of the related elongation Δl in millimetres, and of the initial length l_1 in millimetres. Apply the corrections explained in 9.2 if necessary.

9.4 Evaluation of the measurements at increasing temperature

The elongation $l(t) - l(t = 0)$, and the temperature ϑ are plotted against the time as registered, or taken from the readings. Select some elongations Δl and assign the respective initial length l_1. Take the mean of the temperature for the related time interval Δt. Evaluate these data as in 9.3.

9.5 Representation of the results; precision

The results consist of a number of temperatures and the related viscosities, calculated according to 9.3 or 9.4. The viscosity-temperature relationship and the temperature coefficient of the viscosity are represented (numerically or graphically) according to ISO 7884-1.

The precision is influenced mainly by the following items:

a) scatter of readings;

b) deviation of the particular measuring points from the fitting curve;

c) uncertainty of the fibre volume, initial length, correction of the load.

Errors should be represented according to ISO 7884-1.

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) test procedure (8.2 or 8.3);

g) time-temperature programme of the measurement;

h) corrections applied;

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

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