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Glass — Viscosity and viscometric fixed points —

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

Reference number
ISO 7884-7 : 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 7884-7 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 7 :
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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 annealing point and the strain point of a glass by beam bending. These values have been found useful for specifying the cooling programme in the production of glassware. The annealing point and strain point include a well-defined temperature decrease during the measurement.

At temperatures corresponding to the annealing and strain points, the viscosity of glass is highly time dependent. Hence, any viscosities that might be derived or inferred from measurements carried out according to this part of ISO 7884 cannot be assumed to represent equilibrium structural conditions. Therefore, the insertion of the strain point into the VFT-equation (see ISO 7884-1) is always impossible. The insertion of the annealing point causes in some cases marked failures.

NOTE — The annealing and strain points by beam bending can also be determined using devices as specified in ISO 7884-4, but these devices are more expensive and the procedures lead to some viscosity-temperature and viscosity-time relationships besides the fixed points. In this part of ISO 7884, however, the device and procedure are restricted to the fixed-point determination.

2 Field of application

This method is applicable to all glasses of normal bulk-production compositions, unless the preparation of suitable test specimens is hindered by special reasons. The method is particularly suited for glasses that for one reason or another are not adaptable for flame-working.

Generally, the annealing point and strain point fall into a range of temperature between 300 and 800 °C, depending on the type of glass.

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 annealing range : The range of temperature in which stresses in glass articles can be relieved at a commercially desirable rate.

For purposes of comparing glasses, the annealing range is assumed to correspond to the temperatures between the annealing point ϑ_{f3} and the strain point ϑ_{f4}. This range corresponds to viscosities around 10^{13} dPa·s* and somewhat higher (see also ISO 7884-1).

4.2 annealing point, ϑ_{f3} : The temperature at which internal stresses in a glass are substantially relieved in a matter of minutes.

During a test in accordance with the requirements of this part of ISO 7884, the rate of viscous deflection of the midpoint of the test beam is measured by an extensometer with suitable magnification during cooling at a rate of (4 ± 1) °C/min. The

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

(P is the symbol for poise)

nominal deflection rate df/dt, expressed in millimetres per second, is at the annealing point ideally given by equation (1) :

$$\left(\frac{df}{dt}\right)_a = \frac{44{,}5 \times 10^{-12} \times l_S^3 m}{I_c} \quad \ldots (1)$$

where

l_S is the support span, in millimetres;

m is the mass of the centrally applied load, in grams;

I_c is the cross-sectional moment of inertia of the test beam, in millimetres to the fourth power (see annex A).

NOTE — The deflection rate df/dt which defines the annealing point by equation (1), corresponds to a viscosity of approximately $10^{13,2}$ dPa·s.

4.3 strain point, ϑ_{f4} : The temperature at which internal stresses in a glass are substantially relieved in a matter of hours.

The strain point is determined by extrapolation of the annealing point data and is the temperature at which the viscous deflection rate is 0,031 6 times that observed at the annealing point.

NOTE — This extrapolated deflection rate corresponds to a viscosity of approximately $10^{14,7}$ dPa·s.

5 Principle

The annealing point is determined by measuring the rate of midpoint viscous bending of a simply loaded glass beam (see annex D). The strain point is subsequently determined by an extrapolation method.

The annealing and strain points shall be obtained following a specified procedure after direct calibration of the apparatus using beams of reference glasses[1] having known annealing and strain points.

6 Apparatus

6.1 Furnace

The furnace shall be electrically heated by resistance-wire windings of suitable alloys capable of maintaining the appropriate temperature.

Dimensions and details of the furnace construction are not critical. Examples are given in ISO 7884-4 and in annex B.

The temperature distribution shall be such that differences in temperature greater than 2 °C do not result over the length of the specimen beam and along the axis of the furnace from the undeflected beam plane to a point 13 mm below.

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 junction of the measurement thermocouple, however, shall be placed within 5 mm of the test specimen near the axis of the furnace. In accordance with ISO 7884-1, the measurement thermocouple 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 Furnace control

Suitable means shall be provided for idling the furnace, controlling the heating rate and, in the case of very hard glasses, limiting the cooling rate to not more than 5 °C/min. Although commercially available programming equipment can be used, a continuously variable transformer with manual control may also be used.

6.4 Specimen support stand and loading rod

A ceramic support stand and a ceramic loading rod shall be provided for supporting the test specimen and applying the load to the test specimen, respectively. The thermal expansion characteristics of both stand and rod materials shall be very similar so as to minimize motion of the loading rod on cooling due to expansion differences (see annex C). A rectangular alumina muffle makes a suitable support stand (see note). The side walls of this muffle can be notched to define the test specimen position. The supporting surfaces of these notches shall be flat and lie in a plane perpendicular to the axis of the furnace. The inside edges of these supporting surfaces define the support span once the test specimen beam starts to deflect. A support span of about 50 mm is recommended. A suitable loading rod can be provided by a single-crystal sapphire rod[2] flame-bent at one end in the form of a shepherd's crook. The arrangement is shown in annex B.

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

2) Sapphire rods according to 6.4 (after ASTM designation C 598-72) may be obtained from Insaco Inc., P.O. Box 422, Quakertown, Pa., USA. This information is given for the convenience of users of this part of ISO 7884 and does not constitute an endorsement by ISO of this product.

NOTE — Vitreous silica is a suitable material for both support stand and loading rod. It is not recommended for temperatures above 900 °C, however.

6.5 Extensometer for measuring midpoint deflection

The means of observing the rate of midpoint deflection of the beam should be such as to indicate reliably over a range of at least 2,5 mm. The graduated scale of the extensometer shall permit direct reading to 0,025 mm and estimates of 0,002 5 mm. Its accuracy shall be such that the error of indication will not exceed ± 0,005 mm for any length change. To ensure this accuracy, the extensometer shall be precalibrated. A linearly variable differential transformer (LVDT) is suitable for this purpose but any device (optical, capacitative, or other) may be used, provided that the length changes are reliably measured as specified. The arrangement with the LVDT is shown in annex B. The core of the LVDT is attached to the end of the loading rod, whereas the coils are attached to the leg of the furnace platform. A screw arrangement is provided in the coil attachment assembly to move the coils vertically for zeroing purposes.

6.6 Micrometer calipers with an accuracy of at least 0,01 mm for measuring specimen dimensions.

7 Preparations

7.1 Preparation of the specimens

7.1.1 Specimens from reference glass

Choose a reference glass whose annealing point lies close to the expected annealing point of the glass under test.

Specimens may either be flame-drawn or centreless ground into cylindrical form, or diamond-saw cut and mill ground into rectangular form. Non-uniformity of any dimension along the length of the specimen shall not exceed 2 %. For a support span of 50 mm, the cross-sectional moment of inertia shall be between 2 and 10 mm^4.

Corresponding ranges for other values of the span may be derived from the relations given in ISO 7884-4.

Prepare a number of specimens (at least two) with different cross-sectional moments of inertia (to be calculated according to annex A), but all within the limits given above.

7.1.2 Test specimens

Prepare the test specimens from the glass under test in the same way as in 7.1.1, second paragraph. Take care that the cross-sectional moments of inertia of the reference glass beams bracket the respective values of the beams from the glass under test.

7.2 Adjustment of the loading device

From the mean of the cross-sectional moments of inertia of all the beams which will be measured, determine an optimum load by means of the graph in figure 1. Choose a weight piece such that the total mass of the loading device — consisting of the loading rod, LVDT core, hooks, fixtures and the weight piece — is close to the optimum load.

This loading mass m shall be used throughout, both for calibration and for test measurements.

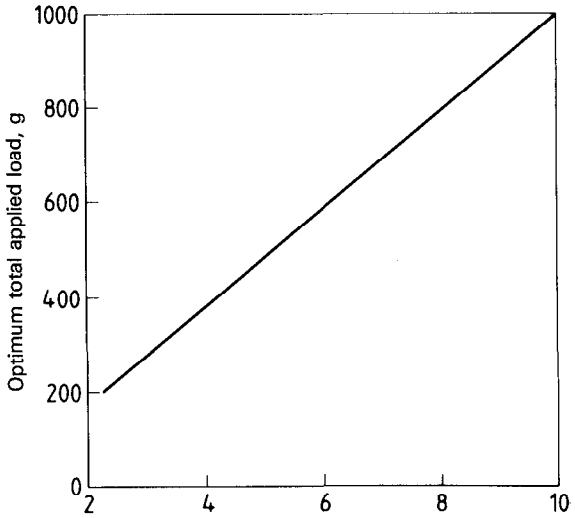


Figure 1 — Optimum load versus cross-sectional moment of inertia for test beams

8 Procedure

8.1 Preparation of a run

All runs, both for calibration (specimens from reference glass) and for determining the annealing and strain point (test specimens), shall be performed in the same manner.

8.1.1 With the furnace at least 25 °C below the estimated annealing point, remove the top plug and place the specimen beam across the support stand at the notch points. Carefully engage the loading rod with the test specimen and centre it using long calipers. Replace the top plug.

8.1.2 Apply the weight piece, chosen according to 7.2, to the hook on the end of the LVDT core as shown in figure 8.

8.1.3 Adjust the position of the extensometer to the lower end of its measuring range. Then start heating the furnace at a convenient rate, preferably at about 5 °C/min. Stop heating and establish a cooling rate of (4 ± 1) °C/min when the specimen midpoint deflection rate, in millimetres per second, reaches

$$\left(\frac{\mathrm{d}f}{\mathrm{d}t}\right)_{\mathrm{e}} = \frac{7 \times 10^{-10} \times l_{\mathrm{S}}^{3}\, m}{I_{\mathrm{c}}} \qquad \ldots (2)$$

where the symbols used are defined below equation (1).

Reset the extensometer to the lower end of its range.

NOTE — This deflection rate, corresponding to a viscosity of 10^{12} dPa·s, guarantees erasure of previous thermal history.

8.1.4 Immediately after cooling has been established, take readings of both the extensometer and potentiometer alternately at 30 s intervals so that each will be read at 1 min intervals. Continue the readings until the temperature is 10 °C below the annealing point. Such a temperature will generally be reached when the extensometer indicates a deflection rate three times less than that expected at the annealing point. If the extensometer goes off range during the test, reset it to the lower end of the range by means of the vertical zeroing screw. Total beam deflections greater than 10 mm are excessive.

8.1.5 Take the change in extensometer readings during each 1 min interval as the rate of midpoint deflection at the temperature recorded for the middle of that minute. Plot it logarithmically against its corresponding temperature, using suitable co-ordinated paper with linear abscissa (about 400 mm) against logarithmic ordinate with three decades (about 250 to 300 mm). The relation should be substantially linear; draw a straight line to represent the plotted points as shown in figure 2.

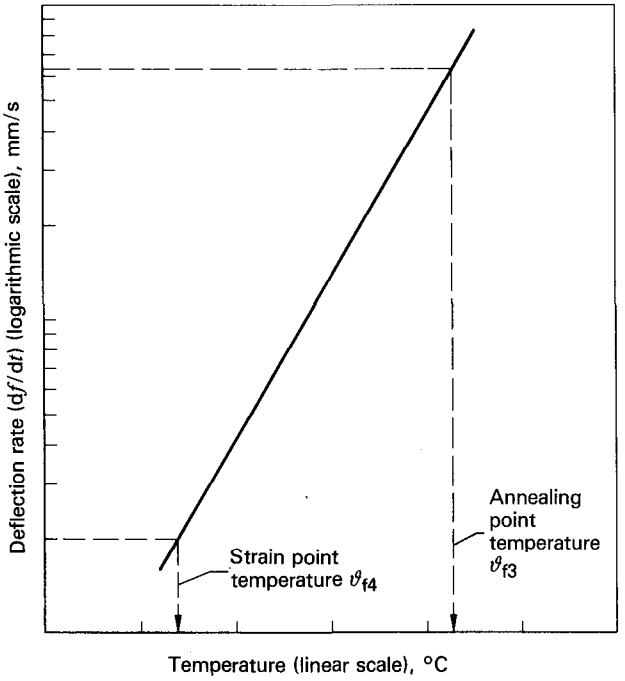


Figure 2 — Graphical method of analysing deflection rate temperature data

8.2 Calibration

Carry out the measurements according to 8.1.1 to 8.1.4 on each reference glass beam prepared according to 7.1.1, and plot the data according to 8.1.5 and figure 2.

From the known annealing point of the reference glass, the related midpoint deflection rate $(df/dt)_a$ is derived from the graph as shown in figure 2 for each beam of that reference glass.

Make a linear diagram as shown in figure 3, plotting the values $(df/dt)_a$ (as found above) against the values of $1/I_c$ (having calculated I_c according to annex A) for each beam of that reference glass.

This is the calibration curve to be used for the test measurements. It is recommended that the apparatus be recalibrated periodically, depending on the incidence of usage.

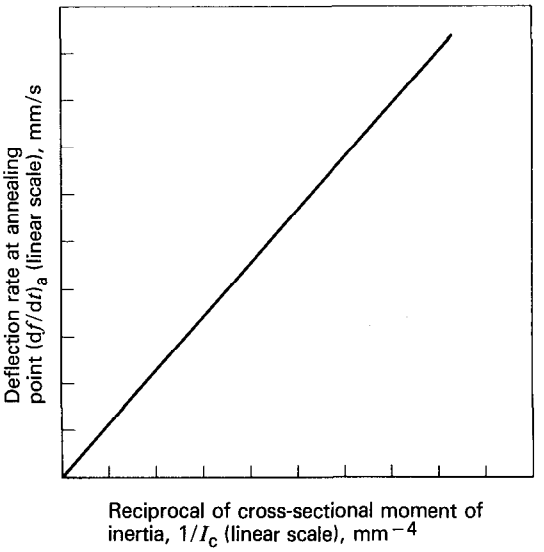


Figure 3 — Graphical calibration plot of deflection rate versus reciprocal of moment of inertia of reference glass test beams

8.3 Test measurement

Carry out the measurements according to 8.1.1 to 8.1.4 on a beam of the glass under test, prepared according to 7.1.2, and plot the data according to 8.1.5 and figure 2.

9 Expression of results

9.1 Evaluation of annealing point

From the known dimensions of the test beam, calculate the cross-sectional moment of inertia I_c according to annex A.

From the values $1/I_c$ find on the calibration curve, as in figure 3 plotted according to 8.2, the related midpoint deflection rate at the annealing point $(df/dt)_a$ for the beam under test.

Then, from the $\lg(df/dt)_a$ versus temperature plot for that beam, drawn according to 8.3 as shown in figure 2, find the related temperature value on the abscissa. This is the annealing point ϑ_{f3} of the glass under test.

9.2 Evaluation of strain point

Calculate the midpoint rate of deflection at the strain point $(df/dt)_s$ for the beam under test by means of equation (3) :

$$\left(\frac{df}{dt}\right)_s = \frac{(df/dt)_a}{31{,}6} \qquad \ldots (3)$$

Extrapolate the straight line on the data plot (as shown in figure 2) for that beam towards lower temperatures.

From the extrapolated data plot, find the related temperature value on the abscissa corresponding to the $\lg(df/dt)_s$ value determined above. This is the strain point ϑ_{f4} of the glass under test.

9.3 Precision and accuracy

This procedure in general will yield annealing points to ± 2 °C (standard deviation) of reference glass values. A strict test of the apparatus is to calibrate with one reference glass and then to measure other reference glasses on the basis of this calibration. If the other reference glass values are within 2 °C of their certification values, excellent performance has been established. If errors arise that increase as the difference in annealing points increases, a temperature measurement or distribution problem could exist. This should be corrected. If attempts to correct such a situation are unsuccessful, an unknown glass should never be measured without calibration with a reference glass as close as possible in annealing point.

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 apparatus used;

g) calibration reference and correction applied;

h) annealing point in degrees Celsius;

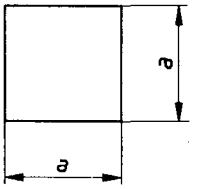
i) strain point in degrees Celsius;

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

Annex A

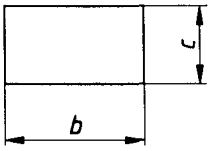
Cross-sectional moment of inertia I_c : formulae for various cross-section geometries

(This annex forms an integral part of the standard.)



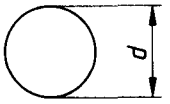
$I_c = a^4/12$

Figure 4 — Square



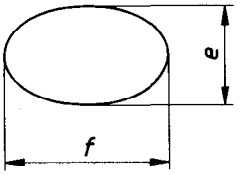
$I_c = bc^3/12$

Figure 5 — Rectangular



$I_c = \pi d^4/64$

Figure 6 — Circular



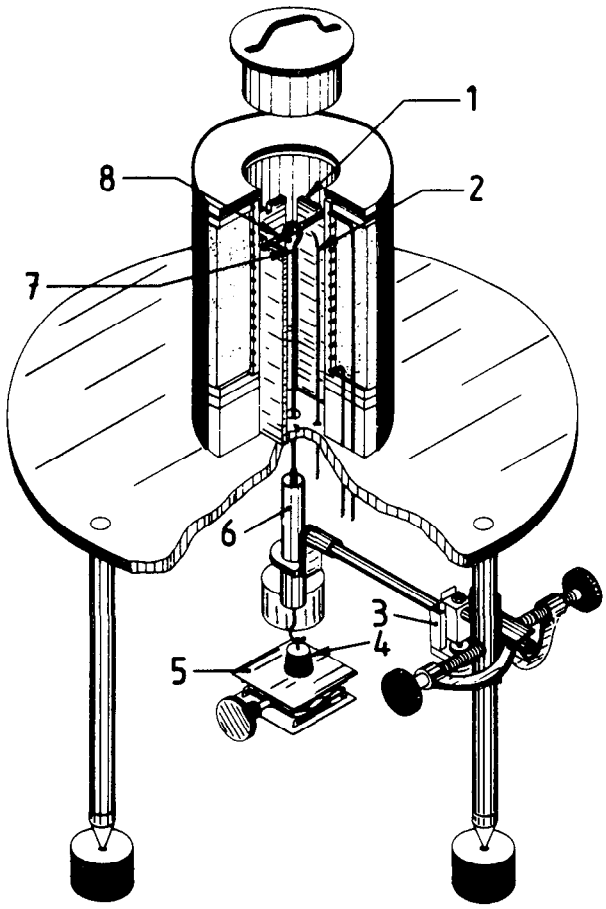
$I_c = \pi f e^3/64$

Figure 7 — Elliptical

Annex B

Example of beam bending apparatus

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



1 Alumina muffle support stand
2 Thermocouple
3 Zero-adjust mechanism for LVDT
4 Weight
5 Laboratory jack
6 LVDT
7 Loading rod
8 Specimen beam

Figure 8 — Cutaway drawing of beam bending apparatus

For the cylindrical furnace a height of 255 mm, outside diameter of 230 mm and inside diameter of 130 mm, and a removable top plug are recommended.

Annex C

Verification of specimen support stand and loading rod

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

To evaluate the effectiveness of matching of the thermal expansion characteristics of materials used for both specimen support stand and loading rod, the following procedure is recommended.

In place of a specimen glass beam, put a single-crystal sapphire rod of 3,18 mm diameter on the support stand. Engage the loading rod and centre it in the usual manner. Place a moderate weight at the end of the LVDT core. Replace the top plug of the furnace and heat to a temperature above the usual operating temperature range. Set the extensometer near to the middle of its range. Establish a cooling rate of (4 ± 1) °C/min and record extensometer readings at intervals of 1 min throughout the temperature range used for annealing point determinations. No motion should result; any motion detected is probably due to expansion differences. Rates above 0,005 mm/min are excessive and should be corrected either by

a) correcting observed rates of deflection during actual testing by the amount measured in the procedure described above, or

b) selecting two materials with a closer expansion match.

Annex D

Bibliography

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

HAGY, H.E. Experimental evaluation of beam bending method of determining glass viscosities in the range 10^8 to 10^{15} poises. *J. Amer. Ceram. Soc.,* 1963 (Vol. 46), pp. 95-97.

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