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Glass — Viscosity and viscometric fixed points —

Part 8 :
Determination of (dilatometric) transformation temperature

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 7884-8 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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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 (dilatometric) transformation temperature t_g of a glass by means of the thermal expansion method. This temperature characterizes a certain glass transition range from the elastic brittle (low temperature) state to the viscous (high temperature) state of glass. The transformation temperature has been found useful for specifying cooling programmes and estimating the upper temperature limit of applicability of the respective glassware.

NOTE — The more direct viscometric method of annealing point and strain point determination (see ISO 7884-7) serves similar purposes. The determination of the transformation temperature t_g might be convenient if a suitable device for determining the coefficient of thermal expansion is available in the laboratory.

2 Field of application

This method is applicable to all glasses of normal bulk-production compositions. Generally, the transformation temperature t_g falls in the range from 350 to 800 °C, depending on the type of glass.

3 References

ISO 7991, *Glass — Determination of coefficient of mean linear thermal expansion.*

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

4 Definition

For the purposes of this part of ISO 7884, the following definition applies.

transformation temperature t_g of glass : The temperature corresponding to the point of intersection of two tangents, drawn from the low-temperature branch and the high-temperature branch of the dilatometer curve (see clause 5 and figure 1).

NOTE — The transformation temperature t_g corresponds to a dynamic viscosity of the order of $10^{13,3}$ dPa·s*. No exact relation exists between t_g and the fixed points ϑ_{f3} and ϑ_{f4} according to ISO 7884-7.

For usual silicate glasses, ϑ_{f3} is 5 to 10 °C higher than t_g. For some special cases (e.g. borosilicate glasses with high SiO_2 content) ϑ_{f3} is up to 30 °C higher than t_g. However, ϑ_{f3} can be up to 20 °C lower than t_g (e.g. in the case of crown glasses having an La_2O_3 content). In these extreme cases the annealing point ϑ_{f3} is the more appropriate information about cooling programmes required and the temperature limit of applicability of the respective glassware.

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

(P is the symbol for poise)

5 Principle

The transformation temperature is determined by measuring the change in length, related to the length at initial temperature, of a rod made from the glass under test with temperature. The relative change in length is plotted against the temperature (dilatometer curve).

The dilatometer curve is determined for a defined rate of temperature increase. From this dilatometer curve the transformation temperature is determined by a graphical procedure as shown in figure 1.

6 Apparatus

6.1 Dilatometer, capable of the determination of changes in length of the specimen to $2 \times 10^{-5} \times l_0$ (i.e. 2 µm per 100 mm).

NOTE — Differential dilatometers may also be used. The reference curves may be evaluated directly, taking the first minimum as t_g, or they may be redrawn as shown in figure 1 and evaluated as described in clause 5.

The contact force shall not exceed 1 N. This force shall not act via conical tips or conical end faces of the test specimen, but only via contacts of planes with spherical faces whose radii of curvature shall be not less than the rod diameter of the test specimen.

6.2 Furnace, compatible with the dilatometer assembly, for temperatures up to 50 °C above the expected transformation temperature.

6.3 Furnace control device for the desired rate of temperature increase of (5 ± 1) °C/min in the range above 200 °C. The temperature distribution within the furnace during the increase shall be smoothed to such an extent that deviations over the whole length of the test specimen remain less than 5 °C.

6.4 Temperature measuring device (e.g. thermocouple type E, J or K according to IEC 584-1), capable of determining the mean temperature of the specimen to ± 2 °C.

6.5 Recording device for the change in length and for the temperature.

7 Test specimen

7.1 Shape

The shape of the test specimen depends on the form of the specimen holder of the dilatometer assembly. The test specimen length shall be at least 5×10^4 times the resolution of the extensometer.

The test specimen shall be a rod with a cross-section of about 10 to 25 mm², and in any case of such a value that the stress arising from the contact force of the dilatometer remains less than 0,1 N/mm². This is to avoid viscous or elastic deformations of the rod during the test.

7.2 Pretreatment

The test specimen shall be annealed before the test by heating it to about 30 °C above the estimated transformation temperature and then cooling it to about 150 °C below the estimated transformation temperature at a cooling rate of (2 ± 0,2) °C/min, followed by further cooling in draught-free air to room temperature.

In figure 2 an example of a dilatometer curve is shown for an insufficiently annealed specimen. Such a curve, however, can be used for preliminary estimation of the value of the transformation temperature prior to a repeated annealing procedure as specified in the preceding paragraph.

8 Procedure

The whole of the following procedure shall be performed twice. If no deformation takes place in a test specimen, it may be retested after re-annealing (see 7.2).

Insert the test specimen at room temperature into the dilatometer and set the extensometer.

Heat the test specimen from 200 °C at a constant rate of temperature increase of (5 ± 1) °C/min and record the temperature t and the related change in length.

Correct for a possible temperature difference between the specimen and the hot junction of the thermocouple as caused by the temperature increase.

NOTE — The amount of the correction may be found empirically from appropriate runs of the dilatometer in accordance with ISO 7991, working on a glass with a sufficiently higher transformation temperature t_g.

9 Expression of results

9.1 Evaluation of transformation temperature

If not recorded directly, plot the relative change in length against the temperature as shown in figure 1.

Draw the tangent to the low-temperature branch of the dilatometer curve at a point about 150 °C below the estimated transformation temperature.

NOTE — A slight deviation from that temperature results generally in changes of less than 1,5 °C in transformation temperature.

Draw the tangent to the high-temperature branch of the dilatometer curve at the point of inflection (see figure 1).

Read from the graph the temperature that corresponds to the point of intersection of the two tangents. If the values of this temperature found in both runs of the test differ by not more than 5 °C, then take the arithmetic mean and round it to the nearest entire degree Celsius. This is the transformation temperature.

If the difference between the results of both runs is greater than 5 °C, then repeat the whole test on a new sample.

9.2 Precision

Repeatability : 3 °C

Reproducibility : 6 °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 and form of the test specimens;

f) type of apparatus used;

g) corrections applied;

h) multiple use of test specimens;

i) transformation temperature, t_g, in degrees Celsius;

j) any changes observed in the glass during and/or after measurement.

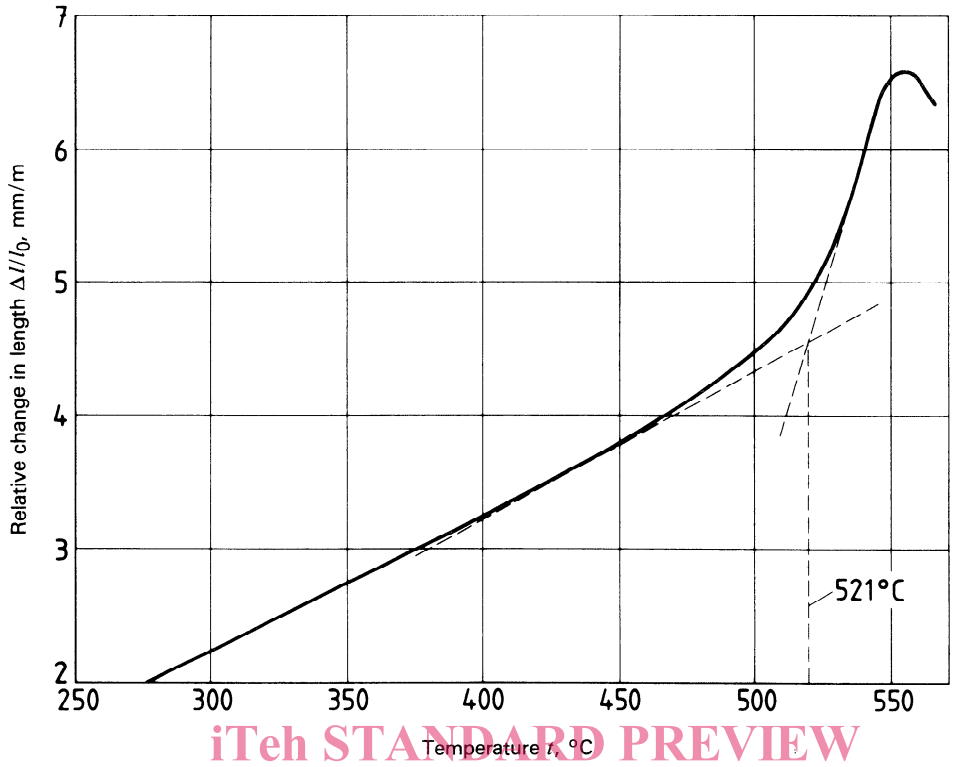

Figure 1 — Example of a dilatometer curve and the evaluation of transformation temperature (521 °C in this case) of a glass

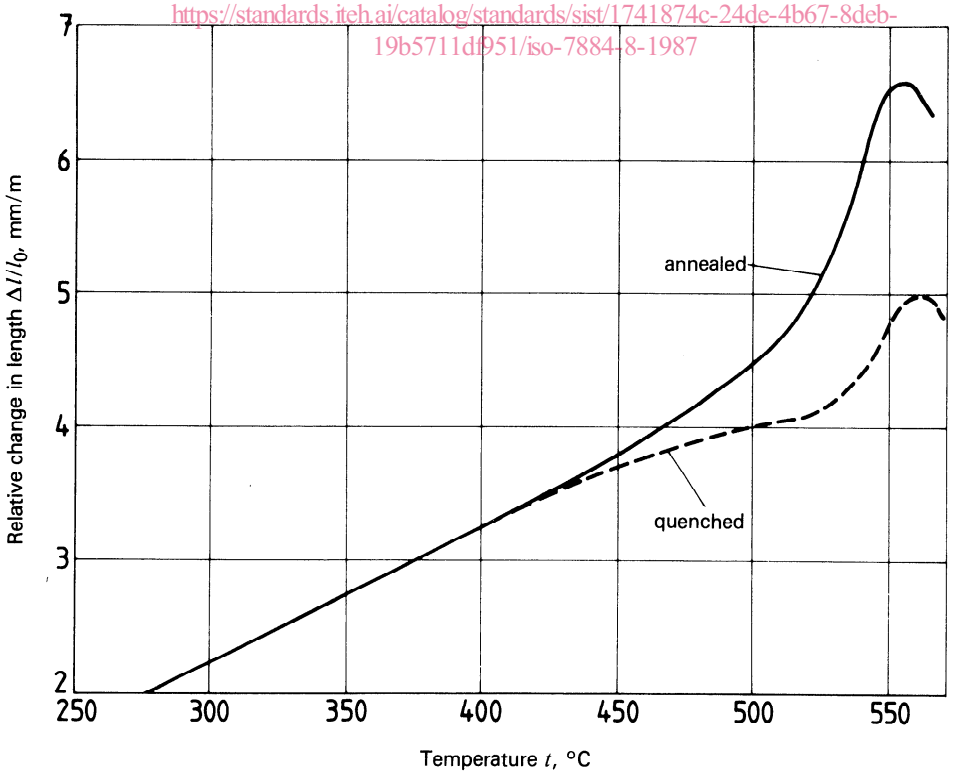

Figure 2 — Example of the dilatometer curve of a glass in the annealed and in a quenched state

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