INTERNATIONAL STANDARD

ISO 7884-6
First edition
1987-12-15

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Glass — Viscosity and viscometric fixed points —

Part 6 : Determination of softening point

Reference number
ISO 7884-6 : 1987 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 7884-6 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.

Glass — Viscosity and viscometric fixed points —

Part 6 : Determination of softening point

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 softening point of a glass. It has been found useful as a control test to indicate changes in composition, for specification acceptance and for providing information in research and development work with glass.

2 Field of application

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

The softening points range between 370 and 1 000 °C, depending on the type of glass.

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

(P is the symbol for poise)

3 Reference

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

4 Definition

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

softening point, ϑ_{f2} : Approximately the temperature above which the glass is capable of most forming operations.

NOTES

1 A viscosity of about $10^{7,6}$ dPa·s* may be assigned to the softening point for glass of density ϱ = 2,5 g/cm^3 and surface tension σ = 300 mN/m. (For glasses with other values of density or surface tension, see ISO 7884-1.)

2 The softening point is also called the Littleton temperature.

5 Principle

Determination of the temperature at which a round fibre of the glass, nominally 0,65 mm in diameter and 235 mm long with specified tolerances, elongates under its own weight at a rate of 1 mm/min when the upper 100 mm of its length is heated in a specified furnace at the rate of (5 ± 1) °C/min.

NOTE — In principle, the device used in this method corresponds to a fibre elongation viscometer (see ISO 7884-3). Closely defined requirements for the test specimen, procedure and apparatus lead to a good repeatability of the specified temperature point. The related viscosity value, however, is of less certainty.

6 Apparatus

6.1 Furnace

The furnace shall conform in all essential respects to the requirements shown in the annex. Equivalent material may be employed, where available.

6.2 Furnace stand

A means shall be provided for supporting the furnace so that the fibre hangs below it. This stand shall be provided with a levelling device such as three screws. The stand shown in the annex is convenient when used with either a cathetometer or a telescope and scale.

6.3 Heating rate controller

Suitable controls shall be provided for maintaining the furnace heating rate at (5 ± 1) °C/min.

NOTE — A continuously adjustable transformer has proved effective for controlling the heating rate.

6.4 Temperature measuring and indicating instruments

6.4.1 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 placed in a double-bore porcelain tube in accordance with the diagram in the annex shall exhibit low thermal inertia (the diameter of the wires should not be greater than 0,5 mm).

6.4.2 The measurement thermocouple shall be placed in the furnace core in accordance with the specifications in the annex (No. 4 and 17). In accordance with ISO 7884-1, the measurement thermocouples 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 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.

6.5 Fibre elongation measurement equipment

The fibre elongation shall be measured using a device capable of measuring the position of the end of the fibre within 0,02 mm throughout the entire elongation period.

NOTE — Suitable devices that have proved effective for measuring the elongation are of both optical and electronic types. For some devices it may be convenient if they are fixed with the furnace stand, e.g. by a short optical bench.

6.6 Fibre diameter measurement equipment

The fibre diameter shall be measured by a suitable device (e.g. a micrometer) with 0,01 mm divisions.

6.7 Timer

A timing device with a least count and accuracy of 1 s shall be used.

7 Preparation of test specimens

7.1 The fibre specimen used for the test shall meet the following requirements :

a) it shall be round;

b) it shall be smooth and shall contain no voids or foreign matter;

c) its average diameter shall be (0,65 ± 0,1) mm and the maximum diameter shall not exceed the minimum diameter by more than 0,02 mm over the entire length of the fibre;

d) it shall be (235 ± 1) mm in length, not including the top bead.

7.2 A test fibre conforming to these requirements may be drawn by attaching a clean sample of the glass under test between two non-fusible rods (such as platinum-group alloys, porcelain or fused silica) and flame-working the sample until the glass is sufficiently fluid to be drawn into a fibre. If the sample is in long stick form, it may be flamed-worked directly without attaching it to a handle. Acceptable fibre sections may then be broken from the fibre and a bead formed on one end of the fibre section by fusing in the flame. The opposite end shall then be broken to the specified length and the tip may be fire polished if desired. Fibres may also be drawn by any suitable device.

8 Procedure

8.1 Calibration with reference glass

Calibration of the apparatus shall be carried out by measuring in duplicate the softening point of appropriate reference glass(es)[1], the softening points of which are near to that of the test glass. Compute the difference between the average measured softening point and the certified softening point for the reference glass(es)[1], and the average of these differences. If the average difference from the certified values is greater than 1 °C, add or subtract this difference as a correction to the measured softening points of the glasses to be tested.

8.2 Measurement

8.2.1 To equalize the heat distribution of the furnace, heat the furnace to about 30 °C above the expected softening point of the glass under test. Then cool the furnace to about 20 °C below the expected softening point, and determine the settings on the heating rate controller that will give a heating rate of (5 ± 1) °C/min.

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

8.2.2 Again, cool and hold the furnace about 20 °C below the expected softening point, and insert the fibre in the furnace by placing the bead end in the furnace sample holder. Check the fibre to ensure that it is hanging freely in the centre of the furnace, and re-level the furnace if necessary. Prepare the temperature measuring equipment and adjust the elongation measuring equipment.

8.2.3 Set the furnace control for a heating rate of (5 ± 1) °C/min. Observe the fibre as the furnace heats, and when it begins to elongate at the rate of approximately 0,1 mm/min, start recording the fibre length to within 0,02 mm. Take a length reading at the end of each minute and take the temperature of the furnace at each 1 min interval, displaced with respect to the length readings by 0,5 min. Continue to read and record the length and the temperature until the elongation becomes 1,2 mm or greater in a 1 min interval. When the elongation exceeds 1,2 mm in a 1 min interval, remove the fibre and cool the furnace in readiness for a duplicate run.

An alternative acceptable method is to read the length at the 0,5 and 1 min points, and the temperature at the 0,25 and 0,75 min points, continuing to read and record length and temperature until the elongation becomes 0,6 mm or greater in a 0,5 min period.

9 Expression of results

9.1 Method of calculation

Determine the temperature at which the elongation is 1 mm/min. This may be done by using any reliable method, one of which is as follows. Plot the data on semi-log paper, with the potentiometer or temperature readings on the linear scale, and the difference between length readings per unit time on the logarithmic scale. The point where a straight line drawn through the data points crosses the 1 mm/min line shall be taken as the indicated softening point. Make the calibration corrections as specified in 8.1, if necessary.

If the difference between the results for two fibres evaluated is greater than 2 °C, repeat the complete test with two new fibres.

9.2 Precision and accuracy

This method in general will yield softening points having a repeatability of 1 °C.

The accuracy of results should be checked by determining the softening point of appropriate reference glasses as indicated in the calibration procedure (8.1).

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) type of softening point apparatus used;

g) method of calculation (9.1);

h) corrections applied;

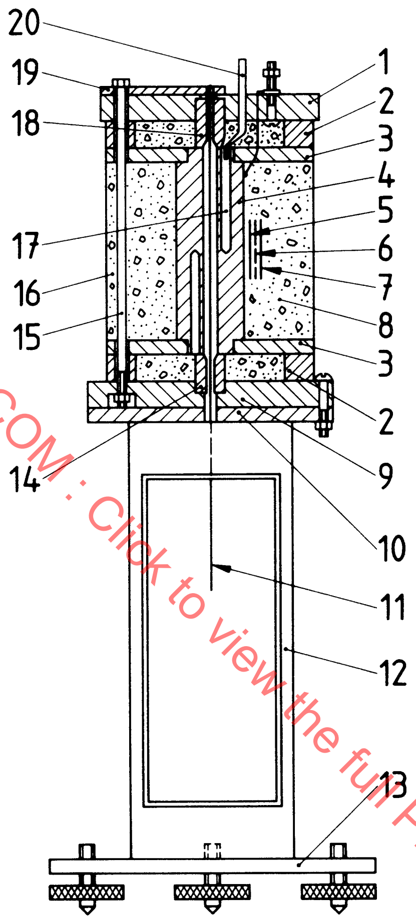
i) softening point in degrees Celsius, i.e. the arithmetic mean of both runs;

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

Annex A

Details of softening point furnace

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



1 Furnace top, 12,7 mm thick by 100 mm outside diameter, having a hole of 12,7 mm diameter at the centre and three holes (for tie rods) 5,6 mm in diameter spaced 120° apart on an 82,5 mm diameter circle; also, two small holes, suitably placed, for thermocouple wires and two binding posts, with small holes nearby, for heater wires. Material to be heat-resistant fibre-reinforced cement. One required.

2 Spacer rings, 12,7 mm thick by 94 mm outside diameter by 70 mm inside diameter, having holes for tie rods (see 1). Material to be heat-resistant fibre-reinforced cement. Two required.

3 Webs, 6,5 mm thick by 94 mm outside diameter, having a hole of 19 mm diameter at the centre and six holes 19 mm in diameter spaced 60° apart on a 51 mm diameter circle; also, holes for tie rods (see 1). Material to be heat-resistant fibre-reinforced cement. Two required.

The upper web shall have two small holes, suitably placed, for the heater wires.

4 Furnace core, 95 mm high by 29 mm outside diameter, with a 6,5 mm length at each end turned to 19 mm outside diameter. Having a bore of 5,6 mm diameter at the centre throughout its entire length and two symmetrical holes 3,2 to 5,6 mm in diameter drilled (one from each end) adjacent, parallel and as near as possible to the central bore to a depth of 47,5 mm (from either end respectively). Material to be nickel. One required.

5 Core wrapping, of double-thickness mica, or fibre ceramics paper.

6 Round resistance wire, wound equidistantly over the whole length of the furnace core. The wire shall exhibit a resistance of 1,76 Ω at room temperature, corresponding to a power of 1 kW at the maximum voltage of 42 V (e.g. an 80 % Ni-20 % Cr wire, 1,6 mm in diameter, with approximately 32 windings).

7 Alundum cement coating.