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Glass — Viscosity and viscometric fixed points —

Part 4 :
Determination of viscosity by beam bending

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 7884-4 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 4 : Determination of viscosity by beam bending

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 the dynamic viscosity of glass on a rod-shaped test specimen (called a beam) supported at its ends. The viscous deflection rate of the beam is measured under a given load at the midpoint between the supports. In addition the viscosity-temperature relationship and the dependence of the viscosity on the thermal history of the sample can be determined.

The viscosity range covered by this method extends from 10^9 to 10^{15} dPa·s*, corresponding to measuring temperatures between about 900 and 400 °C for all glasses of normal bulk-production compositions.

The procedures are limited to small deflections and to small deflection rates (see 3.6).

NOTE — During beam bending, elongation flows of both signs occur (zero passage within the neutral plane). The determination of shear viscosity is possible only with Newtonian or linear-viscoelastic behaviour of the glass. The procedures are sensitive to interference by devitrification of the sample. With viscosities above 10^{12} dPa·s the adjustment of the structure equilibrium within the glass is perceptibly delayed with respect to the temperature setting. For tests within this 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 (see 6.3).

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

(P is the symbol for poise)

2 Reference

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

3 Principle

3.1 Beams

For this test procedure, rod-shaped test specimens, called beams, are prepared from the sample. Along their length l they have a constant cross-sectional area (see figure 1) which may be

a) rectangular, of thickness h, and width b;

b) circular, of diameter d.

3.2 Supports, span

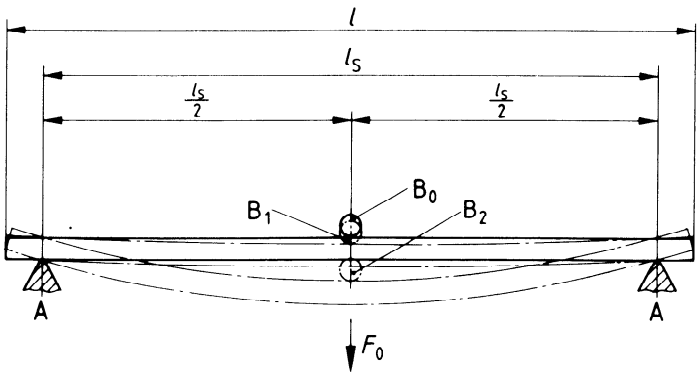
The beam is placed horizontally on two supports; the beam axis and the supports are perpendicular.

For rectangular cross-section beams the supports are horizontal and have straight edges.

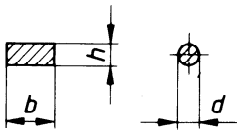
For circular cross-section beams the support edges may be semi-circles or notches.

The distance l_S between the supports is called the span. The beam juts out only little beyond the supports, satisfying equation (1):

$$1{,}1 \leqslant \frac{l}{l_S} \leqslant 1{,}15 \qquad \ldots (1)$$



Cross-sections of the beams



A supports

B_0 bending edge, unloaded

B_1 bending edge, loaded (elastically deformed beam)

B_2 bending edge, sagged position Δf after measuring time Δt (Δf is the interval between B_1 and B_2)

l beam length

l_S span; position of bending edge at $l_S/2$

F_0 force of the load

b beam width } (rectangular cross-section)

h beam thickness }

d beam diameter (circular cross-section)

Figure 1 — Principle of viscosity determination by beam bending

3.3 Load, loading pieces, bending edge

The load consists of all parts of the measuring device on which gravity acts to produce a force on the beam by means of the bending edge, i.e. the loading pieces (variable) and the loading rod together with yoke and bending edge (given for the individual measuring device). The load exerts a vertically downward directed force F_0 upon the central cross-sectional area of the beam (distance $l_S/2$ from both supports). The bending edge is horizontal and parallel to the supports.

3.4 Dead-weight

The dead-weight stems from the beam; it can be taken into account by calculation — see equations (13) to (15). Within the span, the force of the dead-weight acts in the same sense as that of the load. The dead-weight of the overhanging parts of the beam produces a force component opposed to the force of the load; this part of the dead-weight can be neglected if equation (1) is respected.

3.5 Flow

When the force of the load (disregarding the dead-weight) acts upon a beam free from defects and showing Newtonian or linear-viscoelastic behaviour, and all elastic deformations after applying the load have faded out and thereupon the sag is sufficiently small, the flow is described by equation (2) as follows:

$$\frac{df}{dt} = \frac{l_S^3 F_0}{144\, I_c \eta} \qquad \ldots (2)$$

where

df/dt is the midpoint deflection rate, with which the bending edge moves downward (see figure 1);

I_c is the cross-sectional moment of inertia of the beam;

l_S is the span;

η is the dynamic viscosity of the glass.

NOTE — The factor 144 comprises the Trouton ratio 3 and some integration factors.

The cross-sectional moment of inertia for beams with a rectangular cross-section is described by equation (3):

$$I_c = \frac{h^3 b}{12} \quad \ldots (3)$$

and that for beams with a circular cross-section, by equation (4):

$$I_c = \frac{\pi d^4}{64} \quad \ldots (4)$$

During the measuring time Δt the beam sags below the bending edge for a distance Δf. The viscosity is calculated according to equation (5):

$$\eta = 681 \frac{l_S^3 \Delta t m}{I_c \Delta f} \quad \ldots (5)$$

where

η is the viscosity in decipascal seconds;

Δf is the sag of the beam in millimetres during measuring time Δt;

I_c is the cross-sectional moment of inertia in millimetres to the fourth power;

Δt is the measuring time in seconds;

m is the mass of the load in grams;

l_S is the span in millimetres.

When calculating the viscosity it may be necessary to take corrections into account (see 7.1 to 7.3).

3.6 Range of applicability of the simplified calculations

Equations (2) and (5) hold only for very thin beams and very small deflections. That range is characterized by the support ratio q according to equation (6) or equation (7):

$$q = \frac{l_S}{h} \quad \ldots (6)$$

$$q = \frac{1{,}2 l_S}{d} \quad \ldots (7)$$

and also by the relative midpoint deflection z:

$$z = \frac{f}{l_S} \quad \ldots (8)$$

In equation (8) f is the total midpoint deflection of the beam, i.e. f is the sum of the deflection Δf during the measuring time Δt according to equation (5) together with the elastic deflection of the beam caused by the load and — if necessary — the deflections during previous flows.

Measuring devices with $q < 13$ shall be checked by means of a beam made from a reference glass[1] and the results empirically corrected, if necessary.

Relative deflections $z > 0{,}05$ are not permissible. Beams deflected down to this limiting value may be turned over for a further run (see also 6.3.3).

NOTES

1 The correcting calculations known from the statics of an elastic beam with moderate support ratios $q \approx 10$ are subject to the condition that supports are freely movable against one another in the direction of the span. Using the test set-up this is not possible for the flow; therefore mathematical corrections are not available.

2 The dimensions and loads recommended in ISO 7884-7 are taken into account. In view of a more uniform temperature distribution, shorter beams are proposed. The essential difference in comparison with ISO 7884-7 is that:

a) viscosities can be calculated from the bending rates (therefore only considerably smaller relative midpoint deflections are admitted);

b) the viscosity of the delivered sample having its own thermal history is determined, if necessary (therefore the sample is not heated up to 10^{12} dPa·s, and furthermore no viscosities are determined for decreasing temperatures).

4 Apparatus

The requirements for components of the beam bending testing device are given in 4.1 to 4.6. Figure 2 shows an example of a testing device.

4.1 Viscometer furnace

Electrically heated furnace for temperatures up to about 900 °C. The introduction of thermocouples for the determination of temperature and temperature distribution along the beam shall be possible. Temperature differences within the beam shall not exceed 1 °C.

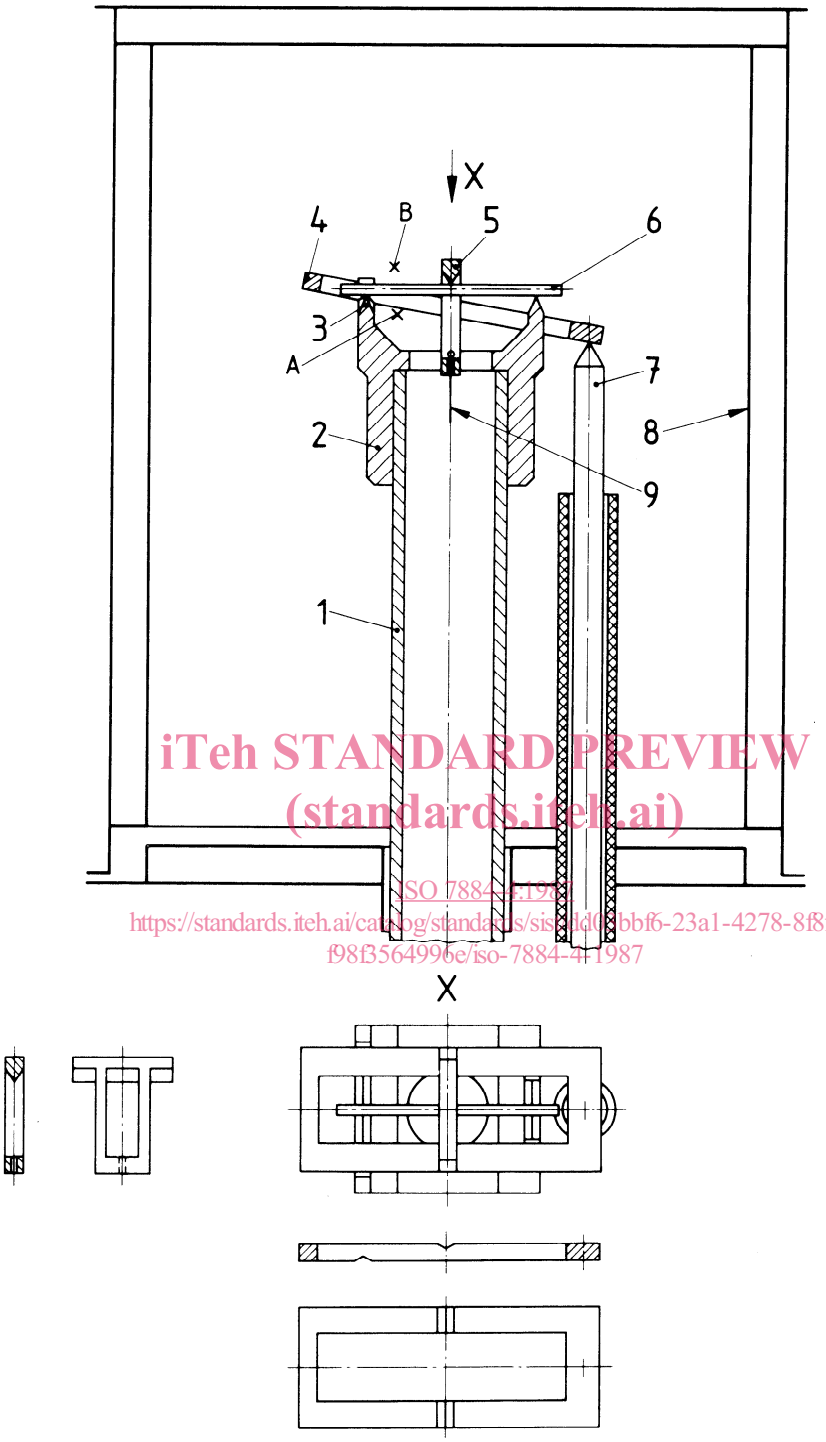
The furnace shall be controlled by a device for maintaining a constant temperature within ±1 °C or better within the working space of the furnace and for the adjustment of linear temperature-time programmes with heating rates up to 6 °C/min.

The furnace and its control device for the temperature-time programme shall be such that the furnace, starting from a constant temperature level, reaches the required heating rate at the latest 5 min afterwards and maintains it to ±10 %.

4.2 Temperature measuring and indicating instruments

4.2.1 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 be not greater than 0,5 mm). The wires shall have a sufficient length within the furnace (with respect to heat conduction along the wires).

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



1 Support stand, made from vitreous silica

2 Frame, made from a suitable temperature-resistant low-expansion metal alloy

3 Supports

4 Locking counterpoise, made from a suitable temperature-resistant low-expansion metal alloy

5 Yoke with bending edge, locking edges and suspension of the loading rod, made from a suitable temperature-resistant low-expansion metal alloy

6 Test specimen (beam)

7 Locking rod, made from vitreous silica

8 Upper part of viscometer furnace: vertically movable cap

9 Loading rod, made from vitreous silica

A and B: Hot junctions of thermocouples (see 4.2)

Figure 2 — Example of a testing device for the beam bending method

4.2.2 Control thermocouples should be located as close as possible to the furnace windings for fast response. The hot junction of the measurement thermocouples, however, shall be placed in the immediate vicinity of the beam (see A in figure 2). The axial temperature distribution along the beam shall be monitored by further thermocouples (see B in figure 2). In accordance with ISO 7884-1, the measurement thermocouples shall be calibrated and the calibration checked regularly.

4.2.3 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.

4.3 Loading pieces

A set of loading pieces with masses from about 10 to 200 g (for arrangements according to 6.2.3, up to 1 000 g) made from brass, nickel-plated or equivalent material. The masses of the loading pieces shall be determined to 0,01 g. The mass of the loading rod including the core of the displacement pick-up together with the yoke and the bending edge can be limited to about 10 g.

4.4 Beam support

4.4.1 Frame

The frame shall be sufficiently rigid against bending and torsion and be made from a suitable temperature-resistant low-expansion metal alloy or hard porcelain. The front sides of the frame bear sufficiently broad (10 to 15 mm) supports with a radius of curvature of about 0,5 mm, the surfaces of the supports being ground and polished. The span, i.e. the distance between the two lines of contact to the bottom surface of the beam, shall be determined to 0,05 mm. Parallelism deviations of the two lines of contact should not exceed 0,05 mm, after the frame has been annealed. After prolonged use, span and parallelism shall be checked.

To prevent sticking of the beam to the support, strips of platinum or nickel foil (about 0,01 mm thick) may be interposed.

4.4.2 Support stand

The support stand bears the frame upon its upper front surface. In the example shown in figure 2 it is set up separately from the furnace. The stand shall be equipped with an adjustment device for the horizontal support of the beam.

The support stand is made from vitreous silica. If temperatures between 750 and 900 °C are often applied, and/or if alkali contamination is suspected, alumina refractory as a material for the support stand is a useful alternative.

NOTE — Another example for a possible construction of the beam support is shown in ISO 7884-7. In that case the supports are machined directly into the top of the stand tube.

4.5 Loading device

4.5.1 Yoke and loading rod

The yoke and bending edge are made from chromium-nickel alloy or hard porcelain. The radius of curvature of the bending edge can vary between 0,5 and 2 mm; the cylindrical surface is ground and polished.

To prevent sticking of the beam to the bending edge, strips of platinum or nickel foil (about 0,01 mm thick) may be interposed.

NOTE — A greater radius of curvature is advantageous for multiple use of the beam after turning it over.

The loading rod connects the yoke within the viscometer furnace to the loading pieces underneath. The loading rod shall be made from the same material as the support stand tube (see 4.4.2) with respect to similar thermal expansion characteristics.

4.5.2 Locking the yoke

A device is needed for lowering the bending edge onto the beam and for lifting it after the test. The device shall ensure that the parallelism between the lowered bending edge and the supports is better than 1° and that the edge is less than 0,5 mm from the median plane.

NOTE — A detailed device which is able to fulfil these requirements is given as an example in figure 2.

4.6 Equipment for the determination of the midpoint deflection rate

4.6.1 Moving indicator (including transducer core) as point of observation for the determination of the deflection rate, placed beneath the viscometer furnace.

4.6.2 Device for the determination of the midpoint deflection Δf during the measuring time Δt according to equation (5). Deflections of 0,1 mm shall be determined to 1 % (see also tables 1 and 2).

4.6.3 Device for the determination of the total beam deflection f monitoring the limiting condition for the relative deflection $z \leqslant 0{,}05$ with a sensitivity of 0,1 mm.

NOTE — Linearly variable differential transformers (LVDTs) with a removable core are suitable for the deflection determination. A measurable elevation adjustment of the coil is then sufficient for the determination of f, whilst the measurement of Δf (see 4.6.2) is achieved by means of the electronic meter of the LVDT having several sensitivity ranges. With this testing method, recording of the values measured should be aimed at. Alternatively, a measuring microscope with scale micrometers (for Δf) and mounted upon a cathetometer base (for f) may be used.

4.6.4 Measuring device for time intervals ranging from 10 to 10 000 s for the determination of the measuring time Δt according to equation (5). Systematic deviations of the measuring device shall be determined to 0,2 % and shall be taken into account.

4.7 Devices for measuring the beam dimensions

4.7.1 Sliding gauge (vernier division 1/10 is sufficient) for the determination of the beam length l.

4.7.2 Micrometer caliper for the determination of beam diameter d or beam thickness h and width b.

5 Preparation of test specimens

5.1 State of delivery

The supplied glass sample shall be uniform, bubble-free, homogeneous and annealed. It shall consist of pieces large enough to permit the preparation of the test specimens.

5.2 Preparation of the beams

Rectangular beams shall be made from the sample by cold working, e.g. diamond-saw cut and mill ground. Cylindrical beams shall be either flame drawn or centreless ground.

The beams shall not have any scratches or defects. The dimensions shall be within the ranges specified in 6.2.1, 6.2.2 and 6.2.3 and figures 3 and 4.

5.3 Determination of beam dimensions and glass density

5.3.1 Beam length

It is sufficient to determine the length of the beam to 0,5 mm.

5.3.2 Rectangular beam cross-section

The beam thickness h shall be measured at nine points altogether: viewed in the longitudinal direction, at the ends and at the middle; viewed in the transverse direction, near to both edges and at the centre. From these values the arithmetic mean shall be taken. The interval between the highest and lowest measured values shall not exceed 0,02 mm.

The beam width b shall be determined near the ends and at the middle, and the arithmetic mean taken from these values. The interval between the highest and lowest measured values shall not exceed 0,05 mm.

5.3.3 Circular beam cross-section

The beam diameter shall be measured in three different circumferential directions, near each end and at the centre, respectively. The interval between the highest and lowest measured value shall not exceed 0,02 mm.

5.3.4 Beam density

The beam shall be weighed to the nearest 0,1 g. The density shall be calculated from the dimensions and the result of weighing.

5.4 Special requirements

Special requirements concerning the treatment of sample and beam shall be agreed upon, especially in the following cases:

a) for samples delivered in the form of grains, the conditions for melting and annealing the rod (as-drawn or cast from the melt), from which the test specimen will be prepared;

b) for specially annealed samples, the highest temperatures to which the beams can be exposed without affecting this annealing treatment.

6 Procedure

6.1 Calibration of the testing device

Generally, the beam bending measurement is an absolute determination of the viscosity, i.e. the viscosity is calculated (see 3.5 and 7.2) from the dimensions of the finished test specimen and from the span l_S. An examination is recommended by means of a beam of equal dimensions and made from a reference glass of known viscosity. This is necessary for support ratios $q < 13$ (see 3.6 and 7.3).

The calibration includes the adjustment of the frame for horizontal positioning of the plane defined by both supports. Subsequently the highest temperature is set that might occur in this testing device, and after cooling down it is checked that the horizontal position has been retained.

To evaluate the influence of the thermal expansion of the different materials used for the apparatus (mainly the support stand and the loading rod), the following procedure is recommended. In place of a specimen glass beam, place a rod of vitreous silica of similar dimensions to the test specimen on the supports, engage the loading rod, attach a moderate loading piece, and prepare the furnace for heating up in the usual manner. Heat the furnace to 400 °C and set the device for the determination of the midpoint deflection (4.6.2) near the middle of its range. With a defined heating rate, increase the temperature to the highest measuring temperature.

As a consequence of expansion differences of the different parts of the apparatus, the moving indicator shows an apparent (positive or negative) midpoint deflection f_s; this is determined as a function of temperature and is used, if necessary, for corrections (see 7.1.2).

The thermocouples shall be calibrated by comparison with a standard thermocouple. This calibration shall be checked at suitable intervals.

6.2 Preliminary estimation of time interval and midpoint deflection

The expected deformation as a function of load and time interval shall be estimated to ensure that the permissible limiting values of the relative midpoint deflection $z = 0{,}05$ are not exceeded during a measurement or a series of measurements. For this purpose tables 1 to 3, in connection with figures 3 and 4, shall be used (but shall not be applied when evaluating the real measurements).

The deformation caused by the dead-weight during the tempering time shall be considered if necessary (see tables 1 and 2).

6.2.1 Measuring device for the medium range of viscosity

The following applies to devices for determining viscosities at constant temperatures within the range 10^{11} to 10^{13} dPa·s.

6.2.1.1 Beams with rectangular cross-section

The values expected for the time interval Δt_1 in the range above 10^{10} dPa·s for recommended loads and as a function of viscosity are given in table 1. The values apply to a beam on a span l_S = 40 mm having a width b = 5 mm and a thickness h = 3 mm (q = 13,3). The cross-sectional moment of inertia of this device is I_c = 11,25 mm^4. For a glass having a density ϱ = 2,5 g/cm^3 the influence of the dead-weight for this design corresponds to an additional load at the bending edge of 1 g.

6.2.1.2 Beams with circular cross-section

A cylindrical beam having the same cross-sectional moment of inertia as the rectangular beam in 6.2.1.1 (11,25 mm^4) has a diameter of d = 3,89 mm. Apart from the differences in the dead-weights, the values of table 1 hold for that cylindrical beam on a support span l_S = 40 mm. The support ratio according to equation (7) is q = 12,3.

For glass having a density ϱ = 2,5 g/cm^3 the influence of the dead-weight for this device corresponds to an additional load at the bending edge of 0,75 g.

6.2.1.3 Allowance for other dimensions

For beams having other dimensions, preliminary values $\Delta t'_1$ can be estimated from equation (9):

$$\Delta t'_1 = \Delta t_1 f_l f_I \quad \ldots (9)$$

In equation (9) the correction factor f_l takes into account spans l_S other than 40 mm. The correction factor f_I, however, takes into account other cross-sectional dimensions. To achieve this, the cross-sectional moment of inertia has to be calculated according to equations (3) and (4); this is the abscissa value in figure 4. The values for the correction factors f_l and f_I have to be taken from the ordinates in figure 3 or figure 4.

NOTE — Figures 3 and 4 also cover the measuring ranges of the devices according to 6.2.2 and 6.2.3; these diagrams may not be used except in connection with table 1.

Table 1 — Preliminary values for the estimation of the time interval Δt_1 and for a midpoint deflection Δf = 0,1 mm, valid for the medium viscosity range according to 6.2.1

These values apply to a beam having a cross-sectional moment of inertia I_c = 11,25 mm^4 (for example of dimensions, see 6.2.1.1 and 6.2.1.2). For a span l_S = 40 mm the largest permissible relative midpoint deflection z = 0,05 is attained within 20 time intervals Δt_1.

Dynamic viscosity η dPa·s	Time interval Δt_1, in seconds, for a midpoint deflection Δf = 0,1 mm under the action of							
	dead-weight exclusively		total load comprising dead-weight and external load of mass					
	circular cross-section	rectangular cross-section	10 g	15 g	20 g	30 g	50 g	100 g
10^{10}	210	280	23,5	16,2	12,3	—	—	—
10^{11}	2 100	2 800	235	162	123	83,4	50,7	25,6
10^{12}	—	—	2 350	1 620	1 230	834	507	256
10^{13}	—	—	—	—	—	—	5 070	2 560

For midpoint deflections Δf = 0,01 mm and Δf = 0,001 mm, and for a ten-fold and hundred-fold sensitivity respectively of the carrier frequency bridge (belonging to the LVDT) the Δt_1-values of this table indicate viscosities respectively 10 and 100 times as high. In this range the limit for $z \leqslant 0,05$ is satisfied more easily.