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Glass — Viscosity and viscometric fixed points —

Part 1 : Principles for determining viscosity and viscometric fixed points

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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 7884-1 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 1 : Principles for determining viscosity and viscometric fixed points

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 gives rules for characterizing glass as a liquid (or liquid-analogue deformable) material with respect to its dynamic viscosity η and viscosity-temperature relationship, if it behaves as a Newtonian fluid.

NOTE — Non-Newtonian behaviour may be observed sometimes in opaque glasses, vitreous enamels or highly crystallizing glasses (glass ceramics).

2 Reference

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

3 Definitions

For the purposes of this part of ISO 7884, the following definitions apply.

3.1 viscosity : The property of resistance to flow under stress. In the case of Newtonian behaviour, the rate of deformation is proportional to the stress.

Following internationally used convention, the preferred unit for the viscosity of glass is the SI sub-unit decipascal second (dPa·s) *.

3.2 Ranges of viscosity

With respect to practical application, three ranges of viscosity measurement can be distinguished :

a) **melting range** : up to 10^3 dPa·s

b) **working range** : about 10^3 to 10^8 dPa·s

c) **annealing range** : about 10^{13} to 10^{15} dPa·s

3.3 Viscometric fixed points

It is convenient to specify the following five temperatures to characterize the viscosity-temperature behaviour of a glass.

NOTE — The expression "fixed point" does not denote any relationship to thermodynamical fixed points.

3.3.1 working point ϑ_{f1} : The temperature corresponding to a viscosity

$$\eta_{f1} = 10^4 \text{ dPa·s}$$

to be determined by one of the methods described in ISO 7884-2 or ISO 7884-5.

3.3.2 softening point ϑ_{f2} : The temperature determined by the method described in ISO 7884-6. The corresponding viscosity is estimated by the following equation :

$$\eta_{f2} = 2{,}1 \times 10^7 \times \left(\varrho - \frac{\sigma}{520}\right) \qquad \ldots (1)$$

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

(P is the symbol for poise)

where

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

σ is the surface tension in millinewtons per metre.

For $\varrho = 2{,}5$ g/cm³ and $\sigma = 300$ mN/m the viscosity is

$\eta_{f2} = 10^{7{,}6}$ dPa·s

In most cases η_{f2} lies close to that value; it shall be used if the values of ϱ and σ are unknown. In extreme cases η_{f2} ranges from $10^{7{,}5}$ to 10^8 dPa·s.

The softening point can also be determined by the method described in ISO 7884-2.

3.3.3 annealing point ϑ_{f3}: The temperature determined in accordance with the method described in ISO 7884-7.

NOTE — From the beam bending method a non-equilibrium viscosity of $10^{13{,}2}$ dPa·s is assigned to the annealing point.

From various measurement techniques, a non-equilibrium viscosity of 10^{13} to $10^{13{,}2}$ dPa·s has been found to approximate to the viscosity at the annealing point.

3.3.4 strain point ϑ_{f4}: The temperature determined in accordance with the method described in ISO 7884-7.

NOTE — From the beam bending method a non-equilibrium viscosity of $10^{14{,}7}$ dPa·s is assigned to the strain point.

From various measurement techniques, a non-equilibrium viscosity of $10^{14{,}5}$ to $10^{14{,}7}$ dPa·s has been found to approximate to the viscosity at the strain point.

3.3.5 transformation temperature t_g: The temperature determined in accordance with the method described in ISO 7884-8.

NOTE — From the dilatometric method a non-equilibrium viscosity of about $10^{13{,}3}$ dPa·s is assigned to the transformation temperature.

An exact relation to the annealing point ϑ_{f3} does not exist.

4 Viscosity-temperature relationship

4.1 The Vogel, Fulcher and Tammann equation

For the purpose of interpolation the viscosity-temperature relationship is conveniently described by the equation of Vogel, Fulcher and Tammann (the VFT-equation):

$$\lg \eta = A + \frac{B}{\vartheta - C} \qquad \ldots (2)$$

The numerical value of the dynamic viscosity η shall be inserted in decipascal seconds (dPa·s), and the temperature ϑ in degrees Celsius (°C).

The constants A, B and C are characteristic for the glass under test (see 4.2).

NOTE — The order of magnitude of the constants for technical glasses is as follows:

$A = -10^0$ (decadic logarithm of viscosity in decipascal seconds)

$B = 10^3$ °C

$C = 10^2$ °C

4.2 Determination of the constants

Three pairs of measured temperatures and equilibrium viscosities $i = 1, 2, 3$, covering the range of the measurement and separated sufficiently from each other, are chosen to calculate the constants of the VFT-equation by means of equations (3) to (5):

$$C = \vartheta_1 + \frac{(\vartheta_2 - \vartheta_1)\cdot(\vartheta_3 - \vartheta_1)\cdot(\lg \eta_3 - \lg \eta_2)}{(\vartheta_2 - \vartheta_1)\cdot(\lg \eta_3 - \lg \eta_1) - (\vartheta_3 - \vartheta_1)\cdot(\lg \eta_2 - \lg \eta_1)} \qquad \ldots (3)$$

$$A = \frac{\lg \eta_2 \cdot (\vartheta_2 - C) - \lg \eta_1 \cdot (\vartheta_1 - C)}{\vartheta_2 - \vartheta_1} \qquad \ldots (4)$$

$$B = (\vartheta_1 - C)\cdot(\lg \eta_1 - A) \qquad \ldots (5)$$

As a proof of the evaluation, calculate the constant B once more using $i = 2$ or 3.

4.3 Temperature coefficient of viscosity

The temperature coefficient of the viscosity of a glass is defined by equation (6):

$$U_\eta = -\frac{1}{\eta}\frac{d\eta}{d\vartheta} \qquad \ldots (6)$$

Using the VFT-equation, the temperature coefficient U_η is given by equation (7):

$$U_\eta = \frac{2{,}303\,B}{(\vartheta - C)^2} \qquad \ldots (7)$$

4.4 Error characterization

The deviation of a measured point from a fitted temperature-viscosity curve (e.g. found by a regression analysis) can be expressed either by a viscosity difference $\Delta \lg \eta$ or by a temperature difference $\Delta\vartheta$. These differences are related to one another by equation (8):

$$\Delta \lg \eta \approx -0{,}4343\, U_\eta\, \Delta\vartheta \qquad \ldots (8)$$

where U_η is the mean temperature coefficient of viscosity in the corresponding range of temperature.

Annex A contains values for estimating the influence of errors in viscosity or temperature determination.

4.5 Viscosity-temperature plot

The graphical representation of the viscosity-temperature relationship is usually performed by plotting the logarithm of viscosity as ordinate against the temperature as abscissa (linear).

5 Principles of measurement and calibration

5.1 Measurement of the viscosity

The performance of the apparatus, the measurement procedure and the evaluation of viscosity depend heavily on the type of method used. The methods are described in ISO 7884-2 to ISO 7884-8.

The different ranges of viscosity should preferably be determined using the following methods of measurement:

a) rotation (see ISO 7884-2): 10 to 10^8 dPa·s

b) fibre elongation (see ISO 7884-3): 10^8 to 10^{13} dPa·s

c) beam bending (see ISO 7884-4): 10^9 to 10^{15} dPa·s

5.2 Viscometric calibration

In some cases the calculation of the viscosity from the shape and dimension of the flow field and from the measured forces and rates of deformation is possible. In practice, however, it is convenient to calibrate or to check the test devices by means of appropriate viscometric certified reference glasses (see annex B for further information).

5.3 Measurement of temperature

The temperature is determined mainly by appropriate thermocouples according to IEC 584-1. For temperatures above 1 200 °C thermocouples such as platinum-30 % rhodium/platinum-6 % rhodium (type B) according to IEC 584-1 should preferably be used.

For minimizing systematic errors in temperature measurement, the general rules for the use of thermocouples shall be obeyed. The leads of the thermocouple within the hot region of which the temperature is measured shall be sufficiently long.

The electromotive force shall be measured by a potentiometer or by an electronic voltmeter having a sufficiently high input resistance.

5.4 Calibration of thermocouples

The thermocouples shall be calibrated with the aid of the thermometric fixed points of the International Practical Temperature Scale of 1968. The instruments used for the measurement of the electrical output of the thermocouples shall also be calibrated.

The constancy of the relationships between electromotive force and temperature of frequently used thermocouples shall be checked from time to time by a thermocouple which is not otherwise used.

6 Apparatus

6.1 Viscometric devices

The viscometric devices are specified in ISO 7884-2 to ISO 7884-8.

6.2 Furnaces

In some cases (see ISO 7884-6), special furnace devices are prescribed in detail.

Generally, a well-insulated furnace, electrically heated, shall have a constancy of temperature with respect to time of ± 1 °C within that part of its hot region which is relevant for the measurement of viscosity.

In some cases the furnace and its control shall be capable of achieving required rates of increase or decrease in temperature.

6.3 Materials of the apparatus

The materials in the hot region of the furnace, the heaters, the crucibles, sockets, and measuring devices shall be able to withstand the relevant temperatures and mechanical forces without fracture or essential deformation. The materials in contact with the glass melt shall be sufficiently resistant to chemical attack by this melt. This property should be checked before the measurement is performed, if the type of glass is unknown.

For temperatures above 800 °C, platinum-rhodium alloys and Al_2O_3 ceramics are appropriate materials for contact with the glass sample. Some other special high-temperature ceramics[1)] may be used, if no contact with the glass sample is provided.

7 Glass under test

7.1 Sample

Unless other procedures are specified, the viscosity of a typical sample has to be determined in the state of delivery; therefore, any change in composition of the glass should be avoided as far as possible, in particular by taking the following precautions:

— do not grind to powder (to avoid water adsorption);

— do not heat above the measuring temperatures if possible, or heat only for as short a time as possible (to avoid loss of volatile components);

— do not heat for a long time within the temperature region of maximum rate of crystallization;

— select pieces without inhomogeneities, inclusions or bubbles.

1) Pythagoras® is an example of a suitable product supplied by W. Haldenwanger Technische Keramik GmbH & Co. KG, Pichelswerder Straße 12, D-1000 Berlin 20.

This information is given for the convenience of users of this part of ISO 7884 and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

7.2 Preparation of test specimens

The preparation of the test specimens depends on the testing method to be chosen and is specified in ISO 7884-2 to ISO 7884-8.

7.3 Treatment of melts

Unless special procedures are prescribed, the following principles should be observed.

a) A melted sample should be heated to, but not higher than, the temperature at which homogenization and removal of bubbles occurs, and should be measured at that temperature and at the lower temperatures desired for the test. Viscosity measurements at higher temperatures should follow, if needed, as the final procedure in the test series.

b) Check the glass for devitrification or demixing effects by repeated measurements using correspondingly variable temperature-time programmes.

NOTE — The protection of the melt by an appropriate inert gas atmosphere may be useful in some special cases.

8 Test report

ISO 7884-2 to ISO 7884-8 specify what particulars the test report shall include.

Generally, all facts and circumstances which could be essential for the judgement or a possible later re-examination shall be registered. The results should be expressed according to clause 4 of this part of ISO 7884.

Annex A

Tables for estimating the influence of errors in viscosity or temperature determination

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

During the measurement, it is not possible to calculate the viscosity-temperature behaviour, the related temperature coefficient and the error characterization following 4.2 to 4.4.

As a survey and preliminary estimation of the relationship between the differences of temperature, $\Delta\vartheta$, and of viscosity, $\Delta\eta/\eta$, table 1 may be used if it is known whether the glass is "long", "medium", or "short".

Table 1 — Relative change $\Delta\eta/\eta$ and $\Delta \lg \eta$ in the viscosity corresponding to a change in temperature of -1 °C

$\vartheta_{f1} - \vartheta_{f2}$ °C	Temperature °C	$\lg \eta$	$\frac{\Delta\eta}{\eta} \times 100$ %	$\Delta \lg \eta = 0{,}4343 \times \frac{\Delta\eta}{\eta}$
450 "long"	1 400	3,37	1,0	0,004 4
	1 200	4,33	1,3	0,005 7
	1 000	5,10	1,8	0,008 0
	800	7,82	3,7	0,016 0
	700	9,68	5,1	0,022 0
	600	11,96	6,0	0,026 0
286 "medium"	1 400	2,02	0,9	0,004 0
	1 200	2,84	1,1	0,004 8
	1 000	3,97	1,6	0,007 0
	800	6,06	3,5	0,015 0
	700	7,80	4,6	0,020 0
	600	10,74	8,3	0,036 0
103 "short"	950	1,86	2,6	0,011 2
	850	3,38	4,1	0,017 6
	750	5,55	7,4	0,032 0
	700	7,78	12,6	0,054 8
	650	10,66	13,4	0,058 0
	600	13,54	13,4	0,058 0

As an example for a typical technical glass (reference glass DGG 1, see table 3), table 2 gives a survey on the influence of the error in viscosity introduced by rounding the constants of the VFT-equation.

Table 2 — Influence on error introduced by rounding constants A, B and C

Rounding of constants A, B, C		Temperature	Relative error $(\Delta\eta/\eta) \times 100$ % in the case of constant		
to . . . significant figures	to the nearest . . . unit(s) in the last digit	ϑ °C	A maximum	B maximum	C maximum
5	2	1 400 800 550	0,02	0,02 0,04 0,08	0,01 0,03 0,11
5	5	1 400 800 550	0,06	0,05 0,10 0,19	0,02 0,08 0,28
4	1	1 400 800 550	0,1	0,1 0,2 0,4	0,04 0,2 0,6
4	2	1 400 800 550	0,2	0,2 0,4 0,8	0,1 0,3 1,1
4	5	1 400 800 550	0,6	0,5 1,0 1,9	0,2 0,8 2,8
3	1	1 400 800 550	1,2	1,0 2,1 3,9	0,4 1,6 5,5

Annex B

Examples of certified reference glasses for viscometric calibration

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

The tabulated certified reference glasses (CRG) NBS 709 to NBS 717 are available from the National Bureau of Standards, Washington (see NBS Special Publication 260) and the certified reference glass (CRG) DGG I from the Deutsche Glastechnische Gesellschaft (DGG), Mendelssohnstraß 75-77, D-6000 Frankfurt/M. 1, Germany, F.R.

The certified reference glasses (CRG) NBS 710, 711 and 717 are supplied as rectangular-shaped bars, and are certified for viscosity values between 10^2 and 10^{12} dPa·s. They are supplied to check the performance of high-temperature viscosity equipment (rotating cylinders) and low-temperature viscosity equipment (fibre elongation, beam bending, parallel-plates, etc.).

The reference glasses are based on the International Practical Temperature Scale (UPTS) as follows:

NBS 709 on IPTS-68;

NBS 710 to NBS 717 on IPTS-48;

DGG 1 on IPTS-68.

Table 3 — Certified reference glasses for glass viscosity

Temperatures in degrees Celsius

Certified reference glass (CRG)	Type of glass	Temperatures (best-fit values) at viscosities, in decipascal seconds, of										
		10^2	10^3	10^4	10^5	10^6	10^7	10^8	10^9	10^{10}	10^{11}	10^{12}
NBS 710	Soda-lime-silicate	1 434,3	1 181,7	1 019,0	905,3	821,5	757,1	706,1	664,7	630,4	601,5	576,9
NBS 711	Lead-silicate	1 327,1	1 072,8	909,0	794,7	710,4	645,6	594,3	552,7	518,2	489,2	464,5
NBS 717	Borosilicate	1 545,1	1 248,8	1 059,4	927,9	831,2	757,1	698,6	651,1	611,9	579,0	550,9
DGG 1	Soda-lime-silicate	1 453,4	1 193,5	1 023,7	904,8	817,8	751,7	699,8	657,8	622,9	593,0	566,9

Table 4 — Certified reference glasses for glass viscometric fixed points

Temperatures in degrees Celsius

Certified reference glass (CRG)	Type of glass	Softening point	Annealing point	Strain point
NBS 709	Extra dense lead	384	328	311
NBS 710	Soda-lime-silicate	724	546	504
NBS 711	Lead-silicate	602	432	392
NBS 712	Alkali lead silicate	528	386	352
NBS 713	Dense barium crown	738	631	599
NBS 714	Alkaline earth alumina silicate	908	710	662
NBS 715	Alkali-free aluminium silicate	961	764	714
NBS 716	Neutral (borosilicate) glass	794	574	530
NBS 717	Borosilicate glass	720	516	471