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Glass — Determination of coefficient of mean linear thermal expansion

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Foreword

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International Standard ISO 7991 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 — Determination of coefficient of mean linear thermal expansion

1 Scope and field of application

This International Standard specifies a method for determining the coefficient of mean linear thermal expansion of glass in the elastic solid-like state, well below the transformation temperature (see ISO 7884-8).

This International Standard applies to all glasses of normal bulk-production compositions. It does not apply to fused silica, glass ceramics or other glasses of similarly low linear thermal expansion coefficients.

2 References

ISO 7884-8, *Glass — Viscosity and viscometric fixed points — Part 8: Determination of (dilatometric) transformation temperature.*

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

3 Definition

For the purposes of this International Standard, the following definition applies.

coefficient of mean linear thermal expansion, $\alpha(t_o; t)$: The ratio of the change in length of a specimen within a temperature interval to that temperature interval, related to the initial specimen length.

It is given by the following equation:

$$\alpha(t_o; t) = \frac{1}{l_o} \times \frac{l - l_o}{t - t_o} \quad \ldots (1)$$

where

t_o is the initial or reference temperature;

t is the actual (constant or variable) specimen temperature;

l_o is the length at temperature t_o of the specimen of glass under test (usually a rod made from the glass);

l is the specimen length at temperature t.

For the purposes of this International Standard, the nominal reference temperature, t_o, is 20 °C; therefore the coefficient of mean linear thermal expansion is denoted by $\alpha(20\ °C; t)$.

4 Apparatus

4.1 Device for measuring the specimen length, to an accuracy of 0,1 %.

4.2 Push-rod dilatometer, capable of determining changes in length of the specimen of $2 \times 10^{-5}\ l_o$ (i.e. 2 µm per 100 mm).

The contact force of the extensometer should not exceed 1 N. That force shall act through contacts of planes with spherical faces the radii of curvature of which shall be not less than the rod diameter of the specimen. In some special assemblies (see figure 1) parallel planes are needed.

The specimen-holding assembly shall ensure that the specimen is held firmly in position and shall prevent even small changes in its alignment with respect to the push-rod axis throughout the test (see examples given in the annex).

If the specimen-holding assembly is made of vitreous silica, see the precautions given in 7.2.

From time to time, a performance test shall be carried out using a reference material (see clause 8).

4.3 Furnace, compatible with the dilatometer assembly, for temperatures up to 50 °C above the expected transformation temperature. The working position of the furnace relative to the dilatometer assembly shall be defined with a repeatability of 0,5 mm in both the axial and the radial directions.

Within the range of testing temperatures (i.e. up to temperatures about 150 °C below the highest expected transformation temperature, t_g, and at least up to 300 °C), the furnace shall be capable of maintaining a constant temperature to ± 2 °C over the whole specimen length.

4.4 Furnace control device, suitable for the desired rate of increase in temperature up to (5 ± 1) °C/min within the test range (see 6.1) and for a cooling rate of (2 ± 0,2) °C/min for the annealing procedure according to 5.2.

4.5 Temperature-measuring device (e.g. a thermocouple of type E, J or K in accordance with IEC 584-1), capable of determining the temperature of the specimen to ± 2 °C in the temperature range between t_0 and t.

5 Test specimen

5.1 Shape and size

The test specimen is usually in the form of a rod. Its shape depends on the type of dilatometer used. The length l_0 shall be at least 5×10^4 times the resolution of the dilatometer's measuring device for the change in length.

NOTE — The specimen may be, for example, a rod either with a circular cross-section having a diameter of 5 mm or with a square cross-section 5 mm × 5 mm, and between 25 and 100 mm in length. In certain cases, a cross-section of at least 100 mm^2 is more convenient (see the annex).

5.2 Preparation

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

5.3 Number

The test shall be carried out with two test specimens (see also 7.4).

6 Procedure

6.1 Choice of the test range

In accordance with clause 3, the nominal reference temperature is 20 °C. For practical reasons, however, the measurement may be started between 18 and 28 °C. The preferred final actual temperature is 290 °C < t < 310 °C. If this is not practical, then the alternative values 190 °C < t < 210 °C, or, in special cases, 95 °C < t < 105 °C or 390 °C < t < 410 °C may be chosen. The corresponding nominal values of t are 300 °C, 200 °C, 100 °C, and 400 °C, respectively.

All readings of temperatures and temperature differences shall be taken with an accuracy to 2 °C. Though these actual values are used in the calculations in accordance with clause 7, the test range shall be expressed in terms of the nominal temperatures (see 7.4). For a given coefficient $\alpha(20\ °C;\ t)$ expressed in terms of the nominal temperature, no influence on the value of the coefficient can be detected within the limits specified for the preferred actual temperatures.

6.2 Determination of the reference length

Determine the reference length l_0 of the annealed specimen (see 5.2) to an accuracy of 0,1 % at the reference temperature t_0. Subsequently insert the specimen into the dilatometer and wait for about 5 min before beginning the test as described in 6.3 or 6.4.

6.3 Test at increasing temperature

Determine the position of the dilatometer at the initial temperature t_0 and take this reading as zero for the uncorrected change in length, Δl_{meas}, which will be measured. Subsequently set the furnace control device (4.4) to the desired heating programme and start the programme. Record the temperature t and the related change in length Δl_{meas} until the desired final temperature has been reached.

NOTE — The rate of temperature increase should not exceed 5 °C/min.

As the dilatometer readings of Δl_{meas} are recorded during the increase in the temperature between t_0 and t (values chosen in accordance with 6.1), it should be borne in mind that a temperature difference will exist between the hot junction of the thermocouple and the test specimen; therefore a correction shall be applied to the apparent temperature of the test specimen.

NOTE — The magnitude of this correction depends on the rate of temperature change and the rate of heat exchange between the furnace and the test specimen. It is essential that the correction is determined experimentally by comparison with measurements at constant temperatures.

6.4 Test at constant temperature

Determine the position of the dilatometer at the initial temperature t_0 and take this reading as zero for the uncorrected change in length, Δl_{meas}, which will be measured. Subsequently heat the furnace to the selected final temperature t and hold it constant to ± 2 °C for 20 min. Then take from the dilatometer reading the value of Δl_{meas}.

NOTE — Although the test at increasing temperature (6.3) enables a set of coefficients $\alpha(t_0;\ t)$ with various values of t to be determined in one test run, the test at constant temperature (6.4) should be preferred if only one final value of t is required since this test affords the better precision.

7 Expression of results

7.1 Calculation of the final length

From the measured change in length, Δl_{meas}, the corrected length l at temperature t is calculated using the following equation:

$$l = l_0 + \Delta l_{meas} + \Delta l_Q - \Delta l_B \qquad \ldots (2)$$

where the correction terms Δl_Q and Δl_B are explained in 7.2 and 7.3 respectively.

7.2 Calculation of the expansion of the specimen-holding assembly

In the case of a simple push-rod dilatometer, the correction term Δl_Q in equation (2) is the thermal expansion of that part of

the specimen-holding assembly alongside the specimen, having the length l_o at temperature t_o.

In the case of a differential push-rod dilatometer, the correction term Δl_Q is the expansion of a reference rod with the specimen length l_o at temperature t_o.

In either case, the correction term Δl_Q is calculated using the following equation:

$$\Delta l_Q = l_o \cdot \alpha_Q \cdot (t - t_o) \quad \ldots (3)$$

where α_Q is (in the case of a simple push-rod dilatometer) the coefficient of mean linear thermal expansion of the material from which the specimen-holding asembly is made or (in the case of a differential push-rod dilatometer) the coefficient of mean linear thermal expansion of the material of the reference rod.

If specimen-holding assemblies, push-rods or reference rods are made from vitreous silica which is essentially hydroxyl-free, the values of α_Q given in the table may be used. Before these parts of the dilatometer are used for the first time, they shall be annealed for 7 h at 1 100 °C, and then cooled from 1 100 to 900 °C at a constant rate of 0,2 °C/min.

In order to avoid devitrification of vitreous silica the surfaces shall be kept clean. It is recommended that they are cleaned twice with analytical-grade alcohol, after which contact with bare fingers shall be avoided.

Table — Coefficient of mean linear thermal expansion α_Q for vitreous silica

Range of temperature, °C	Value of α_Q, K^{-1}
20 to 100	$0{,}54 \times 10^{-6}$
20 to 200	$0{,}57 \times 10^{-6}$
20 to 300	$0{,}58 \times 10^{-6}$
20 to 400	$0{,}57 \times 10^{-6}$

NOTE — The values of α_Q given in the table are altered if the system is heated to above 700 °C.

7.3 Determination of the dilatometer correction

The dilatometer correction term Δl_B is needed mainly because of irregularities in temperature distribution within the transient range between the specimen at temperature t and the extensometer at ambient temperatures. The dilatometer correction term should be determined by means of a blank test.

In the case of a simple push-rod dilatometer, the specimen for the blank test is made of the same material as the dilatometer. If that material is vitreous silica, the specimen for the blank test shall be annealed in accordance with 7.2.

In the case of a differential push-rod dilatometer, two identical specimens of any suitable material can be used.

The measurements on glass and the blank test shall be carried out under identical conditions. The blank test shall be repeated at least whenever a performance test in accordance with clause 8 is carried out.

7.4 Calculation of the coefficient of mean linear thermal expansion

In order to calculate the coefficient of mean linear thermal expansion, $\alpha(t_o; t)$, insert the measured values of l_o and Δl_{meas}, the corrections established in accordance with 7.2 and 7.3, and the actual values of t_o and t (with t corrected, if it is determined using the test at increasing temperature) into the following equation:

$$\alpha(t_o; t) = \frac{1}{l_o} \times \frac{\Delta l_{meas} + \Delta l_Q - \Delta l_B}{t - t_o} \quad \ldots (4)$$

Calculate α(20 °C; 300 °C), α(20 °C; 200 °C), α(20 °C; 100 °C) or α(20 °C; 400 °C) for the two test specimens (5.3) to two significant figures if $\alpha(20\ °C; t) < 10 \times 10^{-6}\ K^{-1}$ or to three significant figures if $\alpha(20\ °C; t) \geqslant 10 \times 10^{-6}\ K^{-1}$.

If the results for the two test specimens differ by not more than $0{,}2 \times 10^{-6}\ K^{-1}$, take the arithmetic mean. If the difference is larger, repeat the test with two other test specimens.

8 Performance test

In order to check that the whole test device is functioning correctly, the test procedure and calculation laid down in clauses 6 and 7 shall be carried out on a specimen of a reference material, the value of the coefficient of mean linear thermal expansion of which is certified.[1)]

Recommended reference materials are as follows :

— vitreous silica annealed according to 7.2;

— sapphire single crystal;

— chemically pure platinum.

NOTE — Sintered alumina (Al_2O_3) as a reference material is very insensitive to the thermal treatment applied in the test procedure laid down in this International Standard. However, the values of the mean linear thermal expansion coefficient differ from one rod to another.

The shape and dimensions of the reference specimen shall be similar to those of the specimens usually tested in the test device.

Care shall be taken to ensure that the thermal expansion behaviour of the reference material is not altered by the test. If the reference material is a glass, it shall be annealed (or reannealed) in accordance with 5.2, unless other procedures are specified by the certifier.

1) Enquiries about sources of certified reference materials (CRMs) may be addressed to the Secretariat of REMCO, International Organization for Standardization (ISO), 1, rue de Varembé, Case postale 56, CH-1211 Geneva 20, Switzerland.

9 Test report

The test report shall include the following information:

a) reference to this International Standard;

b) specification, type and state of delivery of the glass tested;

c) shape, size and number of test specimens;

d) type of push-rod dilatometer used;

e) type of test run (constant or increasing temperature, rate of increase);

f) coefficient of mean linear thermal expansion $\alpha(20\ °C; t)$ expressed in $10^{-6}\ K^{-1}$

— to two significant figures, if $\alpha(20\ °C; t) < 10 \times 10^{-6}\ K^{-1}$;

— to three significant figures, if $\alpha(20\ °C; t) \geqslant 10 \times 10^{-6}\ K^{-1}$.

For the temperatures t_0 and t, use the nominal values (see 7.4).

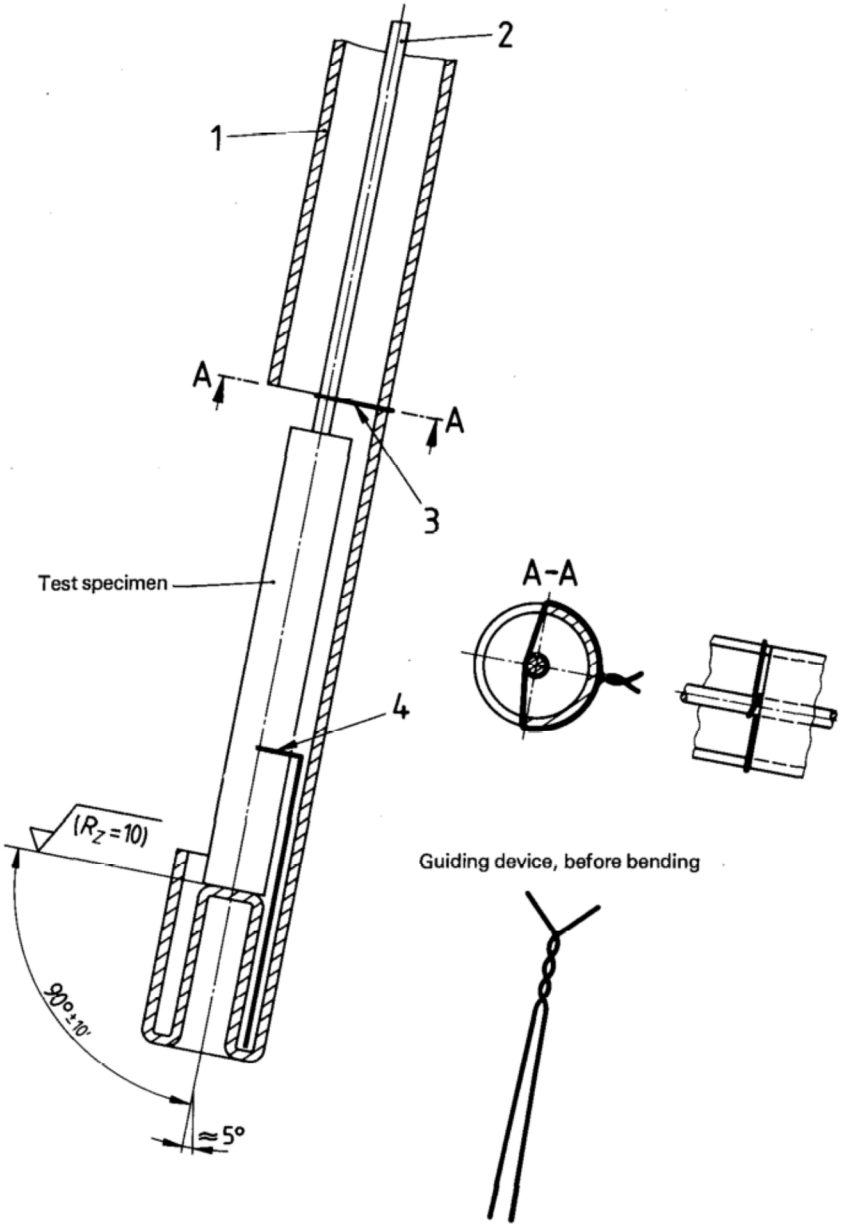
Annex

Devices for self-adjusting alignment of specimen and push-rod axis

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

Ideally the axes of the test specimen and push-rod coincide, and the length l_0 should lie in the same axis. In practice, small deviations between the axes of the test specimen and push-rod may occur. Such deviations are negligible only when that misalignment remains constant throughout the test. Similar considerations hold true for the push-rod direction and the working direction of the extensometer. Changes in alignment (e.g. caused by vibration of the apparatus) shall be avoided by appropriate devices as shown in the examples (figures 1 and 2).

An example for minimizing changes in alignment in a dilatometer assembly working almost vertically is illustrated in figure 1. The guiding devices made from platinum wire prevent further lateral changes in the position of specimen and push-rod once the stable position is achieved by slight shaking. The axial movements caused by thermal expansion, however, are not hindered. Exactly vertically mounted dilatometer assemblies have been found to be the most sensitive with respect to changes in alignment during the test.



1 Specimen-holding tube with dilatometer base and sealed end plug ground flat perpendicular to tube axis, made from fused silica

2 Push-rod made from fused silica

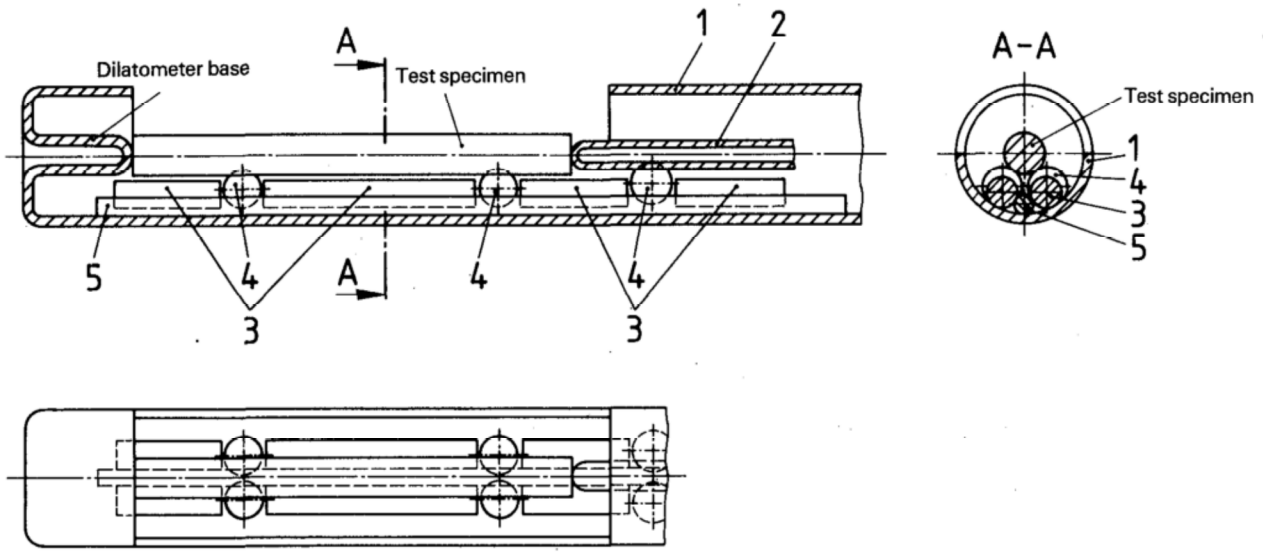
3 Guiding device for the push-rod, made from platinum wire, 0,5 to 1 mm in diameter

4 Guiding device for the specimen, made from platinum wire, 0,5 to 1 mm in diameter

NOTE — Between the base and the push-rod, half the tube is cut away so that the test specimen can be easily changed.

Figure 1 — Example of a specimen-holding and push-rod assembly of a dilatometer working almost vertically

An example for minimizing changes in alignment in a dilatometer working horizontally is illustrated in figure 2. The support for the specimen consists of four spheres (e.g. made from ruby or fused silica), a cylindrical guide-rod, and suitable distance holders. The push-rod is also supported by two spheres of suitable diameter guided on the same guide-rod. After the apparatus has been shaken gently the test specimen and push-rod achieve a stable position.



1 Specimen-holding tube with dilatometer base made from fused silica

2 Push-rod made from fused silica

3 Distance holders made from fused silica

4 Supporting spheres made from fused silica or ruby

5 Guide-rod made from fused silica

NOTE — Between the base and the push-rod, half the tube is cut away so that the test specimen can be easily changed.

Figure 2 — Example of a specimen-holding and push-rod assembly of a dilatometer working horizontally

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