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Plastics — Determination of temperature of deflection under load —

Part 1: **General test method**

Plastiques — Détermination de la température de fléchissement sous charge —

Partie 1: Méthode d'essai générale



Reference number ISO 75-1:2013(E)



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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 75-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

This third edition cancels and replaces the second edition (ISO 75-1:2004), which has been technically revised.

ISO 75 consists of the following parts, under the general title *Plastics* — *Determination of temperature of deflection under load*:

- Part 1: General test method
- Part 2: Plastics and ebonite
- Part 3: High-strength thermosetting laminates and long-fibre-reinforced plastics

Introduction

The first editions of this part of ISO 75 and ISO 75-2 described three methods (A, B and C) using different test loads and two specimen positions, edgewise and flatwise. For testing in the flatwise position, test specimens with dimensions $80 \text{ mm} \times 10 \text{ mm} \times 4 \text{ mm}$ were required. These can be moulded directly or machined from the central section of the multipurpose test specimen (see ISO 20753).

The previous (i.e. second) editions of this part of ISO 75 and ISO 75-2 specified the flatwise test position as preferred, while still allowing testing in the edgewise position with the test conditions given in Annex A until the next revision of this part of ISO 75 and ISO 75-2, as agreed in ISO/TC 61/SC 2/WG 5. Therefore, with this revision, the edgewise test position will be removed.

At the time of publication, technical development of testing instruments made instruments based on a fluidized bed or air ovens available. These are especially advantageous for use at temperatures at which the common silicone oil-based heat transfer fluids reach their limit of thermal stability. The fluidized bed and air oven methods of heat transfer are introduced in this part of ISO 75.

An additional precision statement covering the new heating methods is introduced in ISO 75-2.

Plastics — Determination of temperature of deflection under load —

Part 1:

General test method

1 Scope

This part of ISO 75 gives a general test method for the determination of the temperature of deflection under load (flexural stress under three-point loading) of plastics. Different types of test specimen and different constant loads are defined to suit different types of material.

ISO 75-2 gives specific requirements for plastics (including filled plastics and fibre-reinforced plastics in which the fibre length, prior to processing, is up to 7,5 mm) and ebonite, while ISO 75-3 gives specific requirements for high-strength thermosetting laminates and long-fibre-reinforced plastics in which the fibre length is greater than 7,5 mm.

The methods specified are suitable for assessing the relative behaviour of different types of material at elevated temperature under load at a specified rate of temperature increase. The results obtained do not necessarily represent maximum applicable temperatures because in practice essential factors, such as time, loading conditions and nominal surface stress, can differ from the test conditions. True comparability of data can only be achieved for materials having the same room-temperature flexural modulus.

The methods specify preferred dimensions for the test specimens.

Data obtained using the test methods described are not intended to be used to predict actual end-use performance. The data are not intended for design analysis or prediction of the endurance of materials at elevated temperatures.

This method is commonly known as the HDT test (heat deflection test or heat distortion test), although there is no official document using this designation.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 75-2, Plastics — Determination of temperature of deflection under load — Part 2: Plastics and ebonite

ISO 75-3, Plastics — Determination of temperature of deflection under load — Part 3: High-strength thermosetting laminates and long-fibre-reinforced plastics

ISO 291, Plastics — Standard atmospheres for conditioning and testing

ISO 20753, Plastics — Test specimens

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

flexural strain

εf

nominal fractional change in length of an element of the outer surface of the test specimen at midspan

Note 1 to entry: It is expressed as a dimensionless ratio or a percentage (%).

3.2

flexural strain increase

 $\Delta \varepsilon_{\mathsf{f}}$

specified increase in flexural strain that takes place during heating

Note 1 to entry: It is expressed as a percentage (%).

Note 2 to entry: This quantity is introduced to highlight the fact that the initial deflection caused by application of the test load is not measured and that therefore the criterion for the end of the test does not constitute an absolute strain value. Only the deflection increase is monitored (see 3.4). This new quantity does not change the test or evaluation procedure compared to previous editions of this part of ISO 75, but serves only to clarify what is really measured.

3.3

deflection

S

distance over which the top or bottom surface of the test specimen at midspan deviates during flexure from its original position

Note 1 to entry: It is expressed in millimetres (mm).

3.4

standard deflection

 Δs

increase in deflection corresponding to the flexural strain increase, $\Delta \varepsilon_f$, at the surface of the test specimen, and which is specified in ISO 75-2 or ISO 75-3

Note 1 to entry: It is expressed in millimetres (mm) [see 8.3, Formula (4)].

3.5

flexural stress

 σ_{f}

nominal stress at the outer surface of the test specimen at midspan

Note 1 to entry: It is expressed in megapascals (MPa).

3.6

load

F

force, applied to the test specimen at midspan, which results in a defined flexural stress

Note 1 to entry: It is expressed in newtons (N) [see 8.1, Formulae (1) to (3)].

3.7

temperature of deflection under load

 $T_{\rm f}$

temperature at which the deflection of the test specimen reaches the standard deflection as the temperature is increased

Note 1 to entry: It is expressed in degrees Celsius (°C).

4 Principle

A standard test specimen is subjected to three-point bending under a constant load in the flatwise position to produce one of the flexural stresses given in the relevant part of ISO 75 (all parts). The temperature

is raised at a uniform rate, and the temperature at which the standard deflection, corresponding to the specified increase in flexural strain, occurs is measured.

5 Apparatus

5.1 Means of producing a flexural stress

The apparatus shall be constructed essentially as shown in <u>Figure 1</u>. It consists of a rigid metal frame in which a rod can move freely in the vertical direction. The rod is fitted with a weight-carrying plate and a loading edge. The base of the frame is fitted with test-specimen supports; these and the vertical members of the frame are made of a material having the same coefficient of linear expansion as the rod.

The test-specimen supports consist of metal pieces that are cylindrical in the contact area and with their lines of contact with the specimen in a horizontal plane. The size of the span, i.e. of the distance between the contact lines, is given in ISO 75-2 or ISO 75-3. The supports are fitted to the base of the frame in such a way that the vertical force applied to the test specimen by the loading edge is midway (± 1 mm) between them. The contact edges of the supports are parallel to the loading edge and at right angles to the length direction of the test specimen placed symmetrically across them. The contact edges of the supports and loading edge have a radius of (3.0 ± 0.2) mm and shall be longer than the width of the test specimen.

Unless vertical parts of the apparatus have the same coefficient of linear thermal expansion, the difference in change of length of these parts introduces an error in the reading of the apparent deflection of the test specimen. A blank test shall be made on each apparatus using a test specimen made of rigid material having a low coefficient of expansion and a thickness comparable to that of the specimen under test. The blank test shall cover the temperature ranges to be used in the actual determination, and a correction term shall be determined for each temperature. If the correction term is 0,01 mm or greater, its value and algebraic sign shall be recorded and the term applied to each test result by adding it algebraically to the reading of the apparent deflection of the test specimen.

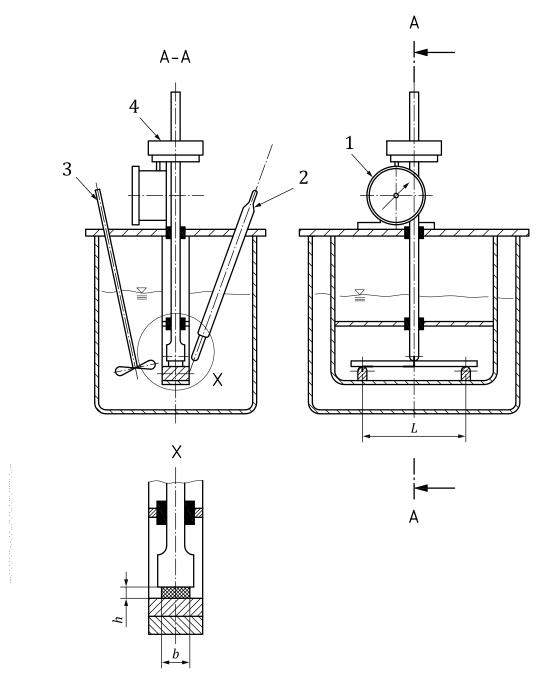
NOTE Invar and borosilicate glass have been found suitable as materials for the test specimen in the blank test.

5.2 Heating equipment

The heating equipment shall be a heating bath containing a suitable liquid, a fluidized bed or an air oven. For heat transfer media other than gas (air) the test specimen shall be immersed to a depth of at least 50 mm. An efficient stirrer or means to fluidize the solid heat transfer medium shall be provided. If liquids are used for heat transfer, it shall be established that the liquid chosen is stable over the temperature range used and does not affect the material under test, for example causing it to swell or crack.

The method using a liquid heat transfer medium shall be considered a reference method in case of doubts or conflicts, if possible in the temperature range under consideration.

The heating equipment shall be provided with a control unit so that the temperature can be raised at a uniform rate of (120 ± 10) °C/h.



Key

- 1 dial gauge
- 2 thermometer
- 3 stirrer
- 4 load
- b width of test specimen
- h thickness of test specimen
- L span between supports

Figure 1 — Typical apparatus for determination of temperature of deflection under load

The heating rate shall be verified periodically either by

checking the automatic temperature reading, or

— manually checking the temperature at least every 6 min.

The requirement for the heating rate shall be considered satisfied if, over every 6 min interval during the test, the temperature change is (12 ± 1) °C.

The difference in the temperature of the heat transfer medium in the heating bath between the ends and the centre of the test specimen shall not exceed \pm 1 °C.

NOTE 1 The apparatus can be designed to stop heating automatically when the standard deflection has been reached.

NOTE 2 Liquid paraffin, transformer oil, glycerol and silicone oils are suitable liquids, but others may be used. For fluidized beds, aluminium oxide powder has been found suitable.

5.3 Weights

A set of weights shall be provided so that the test specimen can be loaded to the required flexural stress, calculated as specified in 8.1.

NOTE It can be necessary to adjust these weights in 1 g increments.

5.4 Temperature-measuring instrument

This may be any suitably calibrated temperature-measuring device with an appropriate range and reading to 0,5 °C or less.

Temperature-measuring instruments shall be calibrated at the depth of immersion particular to the apparatus in use. The temperature-sensing part of the instrument shall be within (2 ± 0.5) mm of the centre of the test specimen, but not touching the specimen.

For calibration of temperature-measuring instruments, follow the manufacturer's instructions (see note 2).

NOTE 1 It can be helpful if the heating bath is equipped with a separate temperature-measuring instrument at each test station, if there are several.

NOTE 2 At the time of publication of this part of ISO 75, no International Standard exists for the calibration of temperature-measuring instruments.

5.5 Deflection-measuring instrument

This may be a calibrated micrometer dial gauge, or any other suitable instrument, capable of measuring to within 0,01 mm the deflection at the midpoint between the test-specimen supports.

In certain types of apparatus, the force, F_s , exerted by the dial gauge spring acts upwards and therefore reduces the downward force exerted by the weighted rod, while, in other types, F_s acts downwards and augments that exerted by the weighted rod. In such cases, it is necessary to determine the magnitude and direction of F_s so as to be able to compensate for it (see 8.1). Since, in certain dial gauges, F_s varies considerably over the measurement range of the instrument, it shall be measured in that part of the range in which the instrument is to be used.

5.6 Micrometers and gauges

These are used to measure the width and thickness of the test specimens. They shall be accurate to 0,01 mm.

6 Test specimens

6.1 General

All test specimens shall be free of warpage. Due to differences in the cooling conditions for moulded specimens or any asymmetry in structure, for instance, the test specimens can warp during heating, i.e. bend without a load. This shall be checked by loading two test specimens on opposite surfaces.

6.2 Shape and dimensions

Each test specimen shall be a bar of rectangular cross-section (length l > width b > thickness h). The dimensions of the test specimens shall be as specified in ISO 75-2 or ISO 75-3.

In each test specimen, the thickness and width over the central one third of the length shall nowhere deviate by more than 2 % from the mean value.

Test specimens shall be produced as described in ISO 75-2 or ISO 75-3.

6.3 Specimen inspection

Specimens shall be free of twist and shall have mutually perpendicular adjacent surfaces. All surfaces and edges shall be free from scratches, pits, sink marks and flash.

NOTE Injection moulded test specimens usually have draft angles of between 1° to 2° to facilitate demoulding. Therefore side faces in moulded test specimens will generally not be parallel. Injection moulded test specimens are never absolutely free of sink marks. Due to differences in the cooling history, generally, the thickness in the centre of the specimen is smaller than at the edge.

Ensure that all cut surfaces are as smooth as possible, and that any unavoidable machining marks are in the lengthwise direction.

The specimens shall be checked for conformity with these requirements by visual observation against straight edges, squares and flat plates, and by measuring with micrometer calipers.

Specimens showing measurable or observable departure from one or more of these requirements shall be rejected or machined to proper size and shape before testing.

6.4 Number of test specimens

Test at least two test specimens. To compensate for warpage effects, the specimens shall be tested with different sides facing the loading edge. If repeated tests are necessary (specified in ISO 75-2 and ISO 75-3:2004, 8.3), an additional two test specimens will be required for each repeat test.

For quality control purposes or by mutual agreement between the interested parties, testing on only one side of the specimen is acceptable. In that case, indicate the side which was loaded in the test report.

7 Conditioning

Unless otherwise required by the specification for the material being tested, the atmosphere used for conditioning shall be in accordance with ISO 291.

8 Procedure

8.1 Calculation of force to be applied

In the three-point loading method employed in this part of ISO 75, the force applied to the test specimen is given, in newtons, as a function of the flexural stress by Formula (1):

$$F = \frac{2\sigma_{\rm f} \cdot b \cdot h^2}{3L} \tag{1}$$

where

F is the load, in newtons;

 σ_f is the flexural stress, in megapascals, at the test-specimen surface;

b is the width, in millimetres, of the test specimen;

h is the thickness, in millimetres, of the test specimen;

L is the span, in millimetres, between the supports.

Measure dimensions *b* and *h* to the nearest 0,1 mm and dimension *L* to the nearest 0,5 mm.

The span and the flexural stress shall be as specified in ISO 75-2 or ISO 75-3.

The effect of the mass, m_r , of the rod that applies the load, F, shall be taken into account as contributing to the test force. If a spring-loaded instrument, such as a dial gauge, is used, the magnitude and direction of the force, F_s , exerted by the spring shall also be taken into account as a positive or negative contribution to the load F (see 5.5).

The mass m_w , of any additional weights which need to be placed on the rod to produce the required total load F is given by Formula (2):

$$F = 9.81 (m_{\rm w} + m_{\rm r}) + F_{\rm s} \tag{2}$$

from which

$$m_{\rm W} = \frac{F - F_{\rm S}}{9,81} - m_{\rm r} \tag{3}$$

where

 $m_{\rm r}$ is the mass, in kilograms, of the rod assembly that applies the test force;

 $m_{\rm w}$ is the mass, in kilograms, of the additional weights;

F is the total load, in newtons, applied to the test specimen;

 $F_{\rm s}$ is the force, in newtons, exerted by any spring-loaded instrument used.

The value of the force, F_s , is positive if the thrust of the spring is directed towards the test specimen (i.e. downwards), negative if the thrust of the spring is in the opposite direction (i.e. opposing the descent of the rod) or zero if no such instrument is used.

The actual load applied shall be the calculated load $F \pm 2.5 \%$.

All formulae referring to flexural properties hold exactly for linear stress/strain behaviour only; thus, for most plastics, they are accurate only at small deflections. The formulae given can, however, be used for comparison purposes.

8.2 Initial temperature of the heating equipment

The temperature of the heating equipment (5.2) shall be below 27 °C at the start of each test, unless previous tests have shown that, for the particular material under test, no error is introduced by starting at a higher temperature.

8.3 Measurement

Tests which are carried out on specimens of different dimensions, or on specimens which are prepared under different conditions, may produce different results. Consequently, when repeatable data are required, sample preparation conditions and test variables shall be the same.

Check the span between the test-specimen supports (see 5.1) and, if necessary, adjust to the appropriate value. Measure this distance to the nearest 0,5 mm and record for use in the calculation in 8.1.

Place a test specimen on the supports so that the longitudinal axis of the specimen is perpendicular to the supports. Place the loading assembly (5.1) in the heating bath. Apply the force calculated in 8.1 to give a flexural stress in the test specimen as specified in ISO 75-2 or ISO 75-3. Five minutes after first applying the force, record the reading of the deflection-measuring instrument (5.5) or set it to zero (see note 1).

Raise the temperature of the bath at a uniform rate of (120 ± 10) °C/h. Record the temperature at which the initial deflection of the bar has increased by the standard deflection, i.e. the temperature of deflection under load at the flexural stress specified in ISO 75-2 or ISO 75-3. The standard deflection is a function of the height, h, the span used and the flexural-strain increase given in ISO 75-2 or ISO 75-3. It is calculated as follows by Formula (4):

$$\Delta s = \frac{L^2 \Delta \varepsilon_{\rm f}}{600 \ h} \tag{4}$$

where

is the standard deflection, in millimetres; Δs

is the span, in millimetres, between the lines of contact of the test specimen and the specimen L supports;

 $\Delta \varepsilon_{\rm f}$ is the flexural-strain increase, in per cent;

h is the thickness, in millimetres, of the test specimen;

The 5 min waiting period is provided to compensate partially for the creep exhibited by some materials at room temperature when subjected to the specified flexural stress. The creep that occurs in the first 5 min is usually a significant fraction of that which occurs in the first 30 min. This waiting period can be omitted when testing materials that show no appreciable creep during the first 5 min at the initial temperature used.

It is frequently helpful in the interpretation of test results if the specimen deflection is known as a function of specimen temperature. It is thus recommended that, where possible, the specimen deflection be monitored continuously during the waiting and heating periods.

The test shall be carried out at least in duplicate. Each test specimen shall be used only once. To compensate for assymmetry effects, e.g. warpage of the specimen, test specimens shall be tested pairwise with opposite sides facing the loading edge, unless it is agreed to test specimens only on one side. see 6.4.

9 Expression of results

Unless otherwise specified in ISO 75-2 or ISO 75-3, express the flexural temperature of the material under test as the arithmetic mean of the temperatures of deflection under load of the specimens tested. Express the test results to the nearest degree Celsius.

10 Precision

For plastics and ebonite, precision data are available (given in ISO 75-2). For high-strength thermosetting laminates and long-fibre-reinforced plastics (given in ISO 75-3), no such data are available at the time of publication.

11 Test report

The test report shall include the following information:

- a) a reference to the relevant part of ISO 75 (all parts), i.e. ISO 75-2 or ISO 75-3;
- b) all details necessary for identification of the material tested;
- c) the method of preparation of the test specimens;
- d) type of heat transfer medium used;
- e) the conditioning and annealing procedures used, if any;
- f) the temperature of deflection under load, in degrees Celsius (if the individual results of the two measurements at different loading directions differ by more than the limit given in ISO 75-2 or ISO 75-3, all individual results shall be reported, separately for the two directions);
- g) the dimensions of the test specimens used;
- h) the flexural stress used;
- i) the span used;
- j) the side on which the specimen was loaded, if it was loaded only on one side
- k) any unusual behaviour of the test specimen noted during the test or after removal from the apparatus.

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