INTERNATIONAL **STANDARD**

ISO 75-1

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Plastics — Determination of temperature of Ander Aral test metho Plastiques — Détermination de la charge — Partie 1: Méthode d'essai générale Circle de la charge — Partie 1: Méthode d'essai générale deflection under load —

General test method

Plastiques — Détermination de la température de fléchissement sous



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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 second edition cancels and replaces the first edition (ISO 75-1:1993), 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

ISO 75-1:1993 and ISO 75-2:1993 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 3167). These "ISO bars" cannot be easily used in the edgewise position, because this would require both a reduction in span and an increase in test load by the same factor, and this may be impossible to achieve on existing instruments for edgewise testing. Specimens for testing in the edgewise position are less closely specified. Using the $80 \text{ mm} \times 10 \text{ mm} \times 4 \text{ mm}$ ISO bar in the flatwise mode has the following advantages:

- Thermal expansion of the test specimen has less influence on the test result.
- Draft angles do not influence the test result. The specimen does not stand "on edge".
- The moulding parameters and the specimen dimensions are specified more closely.

This increases the comparability of the test results. Therefore, it was decided that the possibility of testing in the edgewise position would be deleted from the standard. In order to provide a sufficient transition period, in this edition the flatwise position is described as the preferred and recommended one, while testing in the edgewise position is optional and has been moved to a normative annex (in ISO 75-2). This annex and all other references to edgewise testing will be deleted on occasion of the next revision of this document.

Earlier editions of this International Standard allowed methods other than using a heating bath for heating the test specimen, namely forced-circulation ovens or fluidized beds. None of these alternative methods is widely used and no proven instruments are commercially available. Furthermore, there is no general comparability between tests using different heating methods due to the differences in the heat transfer characteristics and the temperature control methods described in this standard.

Therefore only heating in heating baths is allowed in this edition.

In order to maintain consistency with ISO 10350-1:1998, $T_{\rm f}$ has been used as the symbol for temperature of deflection under load.

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Plastics — Determination of temperature of deflection under load —

Part 1:

General test method

1 Scope

- **1.1** ISO 75 specifies methods 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.
- **1.2** This part of ISO 75 gives a general test method, part 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 part 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.
- 1.3 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 may differ from the test conditions. True comparability of data can only be achieved for materials having the same room-temperature flexural modulus.
- **1.4** The methods specify preferred dimensions for the test specimens. 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 should be carefully controlled and recorded.
- **1.5** Data obtained using the test methods described may not be used to predict actual end-use performance. The data are not intended for design analysis or predicting the endurance of materials at elevated temperatures.
- **1.6** For part 2, two test specimen positions are allowed. However, the flatwise position is the preferred and recommended one, while testing in the edgewise position is described as optional only. It is intended to remove this specimen position altogether on occasion of the next revision of this standard. Part 3 only allows flatwise testing.
- **1.7** 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 referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 75-2:2004, Plastics — Determination of temperature of deflection under load — Part 2: Plastics and ebonite

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ISO 75-3:2004, 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

Terms and definitions

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

3.1

flexural strain

PDF of 180 15.7. nominal fractional change in length of an element of the outer surface of the test specimen at mid-spar

NOTE It is expressed as a dimensionless ratio or a percentage (%).

flexural strain increase

specified increase in flexural strain that takes place during heating

NOTE 1 It is expressed as a percentage (%).

This quantity has been introduced to highlight the fact that the initial deflection caused by application of the test load NOTE 2 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 also 3.4). This new quantity does not change the test or evaluation procedure compared to older editions of this International Standard, but serves only to clarify what is really measured.

3.3 deflection

distance over which the top or bottom surface of the test specimen at mid-span has deviated during flexure from its original position

NOTE It is expressed in millimetres (mm

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standard deflection

increase in deflection corresponding to the flexural strain increase $\Delta \varepsilon_{\rm f}$ at the surface of the test specimen, and which is specified in the relevant part of this International Standard

It is expressed in millimetres (mm) [see 8.3, equations (5) and (6)]. NOTE 1

NOTE 2 The standard deflection depends on the dimensions and position of the test specimen and the span between the supports.

3.5

flexural stress

nominal stress at the outer surface of the test specimen at mid-span

NOTE It is expressed in megapascals (MPa).

3.6

load

force, applied to the test specimen at mid-span, that results in a defined flexural stress

NOTE It is expressed in newtons (N) [see 8.1, equations (1) to (3)].

3.7

temperature of deflection under load

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

NOTE It is expressed in degrees Celsius (°C).

Principle

A standard test specimen is subjected to three-point bending under a constant load in the flatwise (preferred) or the edgewise position to produce one of the flexural stresses given in the relevant part of this International Standard. of 150 151.7. 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.

Apparatus

Means of producing a flexural stress

The apparatus shall be constructed essentially as shown in Figure 1. 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 the relevant part of this International Standard. 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 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.

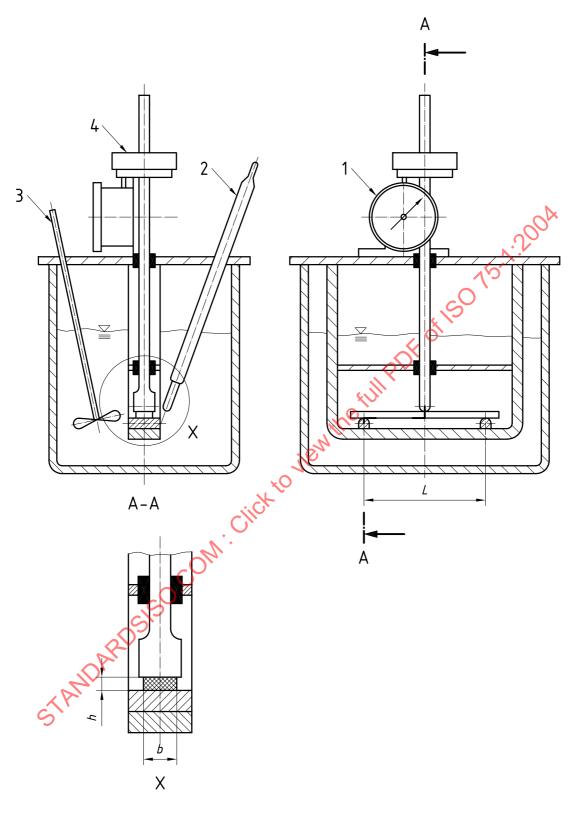
NOTE Invarand borosilicate glass have been found suitable as materials for the test specimen in the blank test.

Heating equipment

The heating equipment shall be a heating bath containing a suitable liquid in which the test specimen can be immersed to a depth of at least 50 mm. An efficient stirrer shall be provided. 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 heating equipment shall be provided with a control unit so that the temperature can be raised at a uniform rate of (120 \pm 10) °C/h.

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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 (shown with specimen in flatwise position)

The heating rate shall be verified periodically:

- either by checking the automatic temperature reading;
- or by 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 liquid in the heating bath between the ends and the centre of the test specimen shall not exceed \pm 1 °C.

- NOTE 1 The apparatus may 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.

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 may 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.

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

NOTE 1 It may 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 existed 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 $F_{\rm S}$ exerted by the dial gauge spring acts upwards and therefore reduces the downward force exerted by the weighted rod, while, in other types, $F_{\rm S}$ acts downwards and augments that exerted by the weighted rod. In such cases, it is necessary to determine the magnitude and direction of $F_{\rm S}$ so as to be able to compensate for it (see 8.1). Since, in certain dial gauges, $F_{\rm S}$ 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.

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6 Test specimens

6.1 General

All test specimens shall be free of warpage caused by any asymmetry with respect to the thickness. Due to e.g. differences in the cooling conditions for moulded specimens or any asymmetry in structure, the test specimens may 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 the relevant part of this International Standard.

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 the relevant part of this International Standard.

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.

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 repeat tests are necessary (see ISO 75-2:2004 and ISO 75-3:2004, subclause 8.3), an additional two test specimens will be required for each repeat test.

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.

Procedure

Calculation of force to be applied 8.1

In the three-point loading method employed in this International Standard, the force applied to the test specimen is given, in newtons, as a function of the flexural stress by one of the following equations:

For the preferred (flatwise) position:
$$F = \frac{2\sigma_{\rm f} \cdot b \cdot h^2}{3L} \tag{1}$$

For the optional (edgewise) position:
$$F = \frac{2\sigma_{\mathsf{f}} \cdot h \cdot b^2}{3L}$$
 where
$$F \quad \text{is the load, in newtons;}$$

$$\sigma_{\mathsf{f}} \quad \text{is the flexural stress, in megapascals, at the test-specimen surface;}$$

$$b \quad \text{is the width, in millimetres, of the test specimen;}$$

$$h \quad \text{is the thickness, in millimetres, of the test specimen;}$$

$$L \quad \text{is the span, in millimetres, between the supports.}$$

where

is the load, in newtons;

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

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

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

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 the relevant part of this International Standard.

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 $m_{\rm W}$, of any additional weights which need to be placed on the rod to produce the required total load F is given by the equation

$$F = 9.81(m_{W} + m_{\Gamma}) + F_{SC}$$
 (3)

from which

$$m_{\mathsf{W}} = \frac{F - F_{\mathsf{S}}}{9.81} m_{\mathsf{f}} \tag{4}$$

where

is the mass, in kilograms, of the rod assembly that applies the test force;

is the mass, in kilograms, of the additional weights;

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

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.

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

NOTE All equations referring to flexural properties hold exactly for linear stress/strain behaviour only; thus for most plastics they are accurate only at small deflections. The equations given may, 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

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 calculations 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 the relevant part of this International Standard. 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 the relevant part of this International Standard. The standard deflection is a function of the height (h or b, depending on the orientation of the test specimen, see 8.1), the span used and the flexural-strain increase given in the relevant part of this International Standard. It is calculated as follows:

For the preferred (flatwise) position:
$$\Delta s = \frac{L^2 \Delta \varepsilon_f}{600h}$$
 (5)

For the optional (edgewise) position:
$$\Delta s = \frac{L^2 \Delta \varepsilon_1}{600b}$$
 (6)

where

 Δs is the standard deflection, in millimetres;

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

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

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

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

NOTE 1 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 which occurs in the first 5 min is usually a significant fraction of that which occurs in the first 30 min. This waiting period may be omitted when testing materials that show no appreciable creep during the first 5 min at the initial temperature used.

NOTE 2 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.