INTERNATIONAL STANDARD

ISO 974

Second edition
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Plastics — Determination of the brittleness temperature by impact

Plastiques — Détermination de la température de fragilité au choc

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 974 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

This second edition cancels and replaces the first edition (ISO 974:1980), which has been technically revised.

Introduction

Plastics are used in many applications requiring low-temperature flexing with or without impact. Polymer brittleness is affected by any orientation produced during fabrication, by thermal history and by the application of stress to the material, especially the rate of applied stress as in impact. Brittleness temperature data may be used to predict the behaviour of plastic materials at low temperature only in applications in which the conditions of deformation are similar. The brittleness temperature test was originally developed to measure the temperature at which a polymer ceases to be flexible and becomes "glasslike".

Plastics — Determination of the brittleness temperature by impact

1 Scope

This International Standard specifies a method for the determination of the temperature at which plastics that are not rigid at normal ambient temperature exhibit brittle failure under specified impact conditions. A supplementary technique using notched specimens develops brittleness values at a much higher temperature than are observed for unnotched specimens of the same plastic material. The method utilizes a statistical technique to quantify the brittleness failure temperature. Provisions are made for the testing of sufficient specimens to permit the calculation of the brittleness temperature on a statistical basis. Statistical techniques have been developed to quantify the brittleness temperature as is defined in 3.1.

The method establishes the temperature at which there is a 50 % chance of failure in either unnotched or notched specimens. This method has been found useful for specification purposes, although it does not necessarily measure the lowest temperature at which the material may be used. In the measurement of the brittleness temperature, the precision of the measurement should preferably be ± 5 °C at the worst when establishing values used in material specifications.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 175:1999, *Plastics — Methods of test for the determination of the effects of immersion in liquid chemicals.*

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing.*

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply:

3.1
brittleness temperature
the temperature at which there is a 50 % probability of failure in a specimen when tested by the method specified

It may be designated T_{50}.

3.2
test speed
the relative velocity between the striking edge of the test apparatus and a test specimen held in the specimen clamp

4 Principle

Test specimens, supported as cantilever beams, are immersed in a heat-transfer medium whose temperature is accurately known and precisely controlled. The specimens are conditioned for a specified period of time and then impacted by a single swing of the striking edge of the apparatus at a specified constant speed. A sufficient number of specimens are tested to permit the calculation of the brittleness temperature on a statistical basis. The temperature at which 50 % of the specimens fail is defined as the brittleness temperature.

5 Apparatus

5.1 Test machine, consisting of a clamping device to hold the test specimens, a striking edge and a mechanical arrangement appropriate to ensure that these are maintained in proper relation to each other and that the striking edge moves at a constant speed relative to the test specimens.

NOTE 1 Details of the striking edge and clamping device are shown in Figures 1 and 2, and a photograph of the clamp with mounted specimens is shown in Figure 3.

The principal dimensions of the apparatus shall be as follows:

a) radius of striking edge: 1,6 mm ± 0,1 mm;

b) radius of lower jaw of clamping device: 4,0 mm ± 0,1 mm;

c) distance between point of impact of striking edge and clamping device: 3,6 mm ± 0,1 mm;

d) clearance between outside of striking edge and clamping device: 2,0 mm ± 0,1 mm;

The test speed shall be 200 cm/s ± 20 cm/s at impact and during at least the next 0,5 cm of travel.

NOTE 2 Commercial apparatus is available meeting the requirements of this subclause, in which the striking edge is driven by a motor, by a solenoid, by gravity or by a spring.

5.2 Temperature-measuring system: Any suitable device may be used. It shall be calibrated over the required range and accurate to within ± 0,5 °C. The temperature-measuring device shall be placed as near to the specimen as possible.

5.3 Liquid or gaseous heat-transfer medium, preferably liquid, which remains fluid at the test temperature and which does not appreciably affect the material being tested. The medium shall be maintained at the test temperature to within ± 0,5 °C.

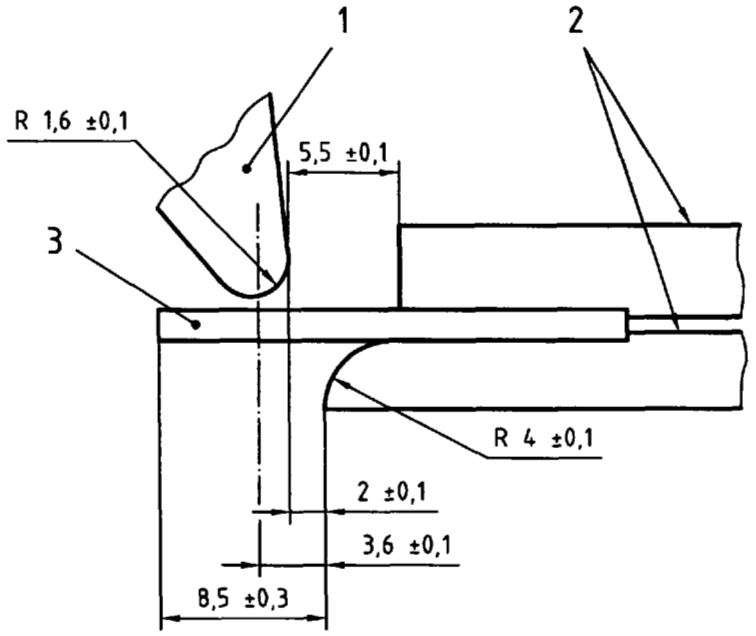
NOTE Given that the time of contact between the liquid and the plastic specimens is short and the temperature low, the use of a methanol/solid-CO_2 mixture has been found suitable for most plastics. This mixture can be used successfully down to − 76 °C. Below this temperature, other heat-transfer media are needed, for example silicone oils, dichlorodifluoromethane/liquid nitrogen, or an air bath.

Should any doubt exist regarding the inertness of the plastics to the mixture used, measure selected physical properties before and after 15 min exposure at the highest temperature used (see ISO 175). The values obtained should not differ significantly.

5.4 Tank, insulated.

5.5 Stirrer, to provide thorough circulation of the heat-transfer medium.

Dimensions in millimetres



Key

1 Striking edge
2 Clamping device
3 Test specimen

Figure 1 — Dimensional details of striking edge and clamping device
(positioning of unnotched test specimen)

6 Test specimens

6.1 For many polymers, the results of this test depend to a large degree on the conditions used for sample preparation and on the method of specimen preparation. Unless otherwise specified, the relevant ISO material specification shall be used for the preparation of the test sample from which the specimens are cut. Cleaner edge cuts and the reduction or elimination of accidental notches will result in lower observed brittleness temperatures.

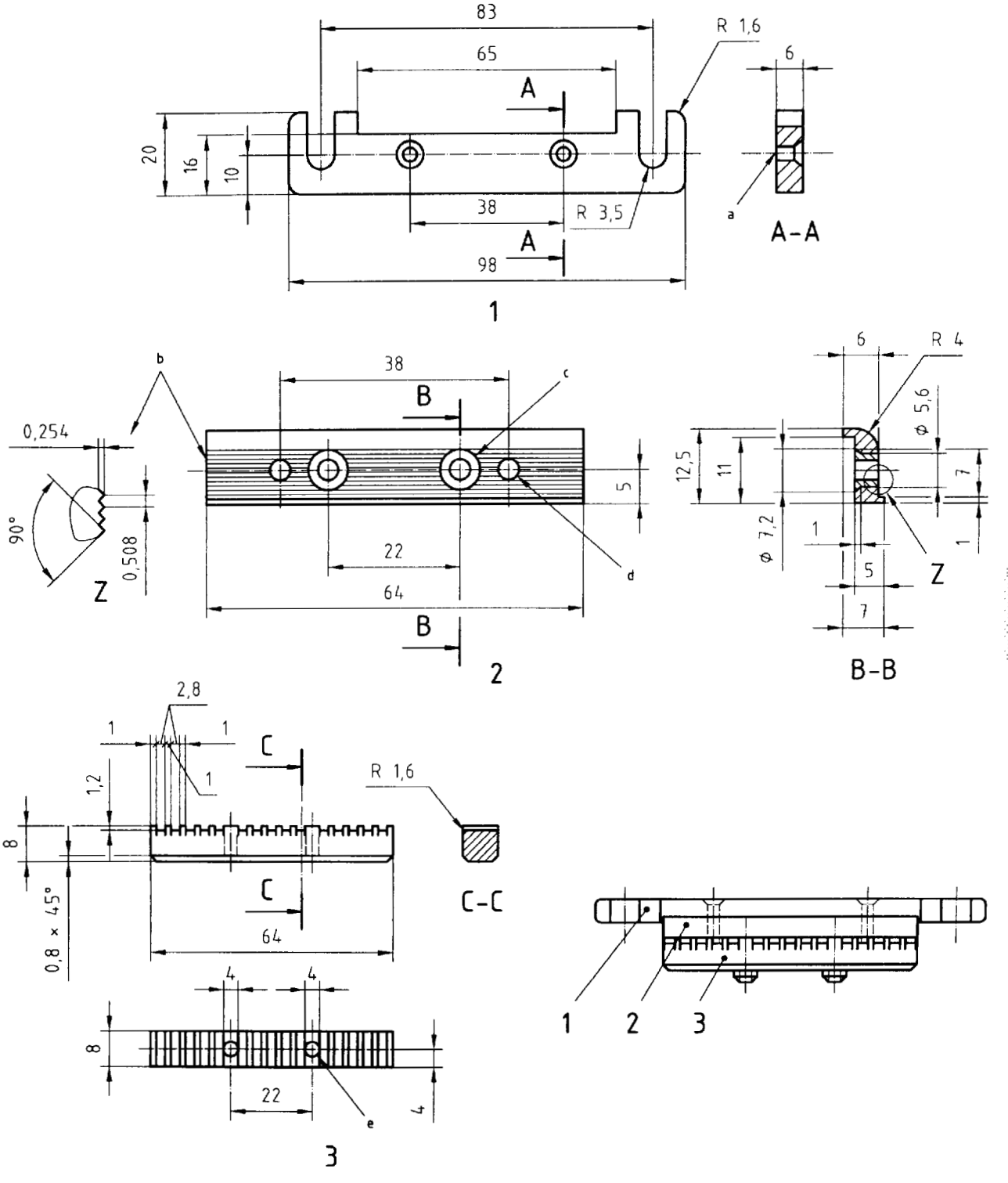
It is essential that the specimens be prepared in a reproducible way. A razor blade or other sharp tool shall be used to cut the specimens, preferably in a single smooth stroke. Die-cut specimens are not recommended. Although it is possible to prepare satisfactory specimens by hand, it is strongly recommended that an automatic method be used. Whatever method is used, it is essential that the cutter be inspected frequently and maintained. Sharp cutters must be used in the preparation of specimens for this test if reliable results are to be obtained.

The condition of the die may be judged by investigating the rupture point on any series of broken specimens. When broken specimens are removed from the clamps of the test machine, it is convenient to make a pile of these specimens and note if there is any tendency to break at or near the same point on each specimen. Rupture points consistently at the same place may be an indication that the die is dull, nicked or bent at that particular position.

NOTE For the use of an automatic cutter for specimen preparation, see Bestelink, P.N., and Turner, S.: *Low-temperature brittleness testing of polyethylene*, ASTM Bulletin No. 231, **68** (1958).

6.2 Test specimens 20,00 mm ± 0,25 mm long by 2,50 mm ± 0,05 mm wide and 2,0 mm ± 0,1 mm thick (see Figure 4) shall be cut from a test sheet. Specimens can be conveniently cut to the specified dimensions from a strip of material 20,00 mm ± 0,25 mm in width and of the required thickness. This is preferably accomplished using an automatic machine (see note to 6.1).

Dimensions in millimetres



Key

a Two holes, ϕ 3,7 mm, countersunk ϕ 6,4 mm × 90°

b Remove sharp corners on serrations

c Two inserted bushes

d Two holes, 48 A

e Two holes, ϕ 3,7 mm

Figure 2 — Details of one form of clamp meeting the requirements of 5.1

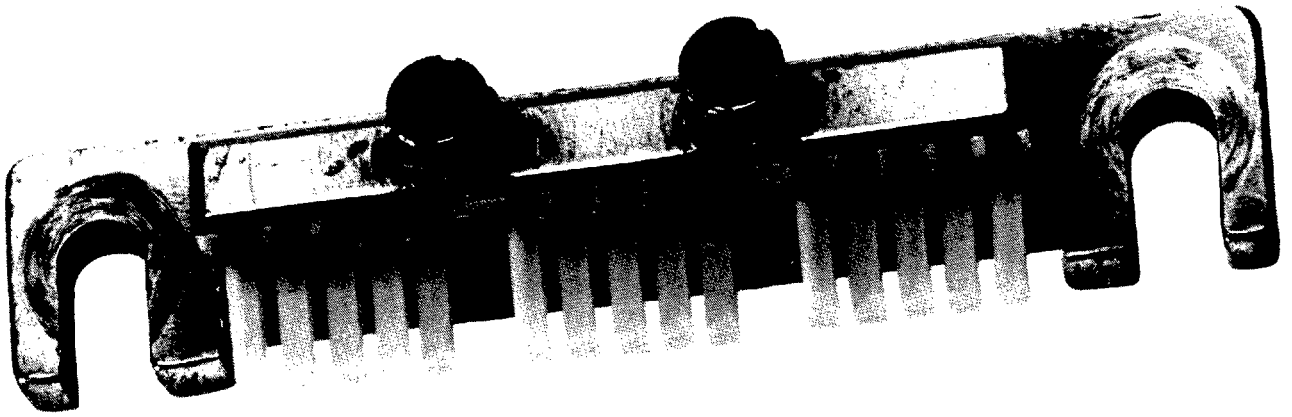


Figure 3 — Assembled clamp with test specimens

Dimensions in millimetres

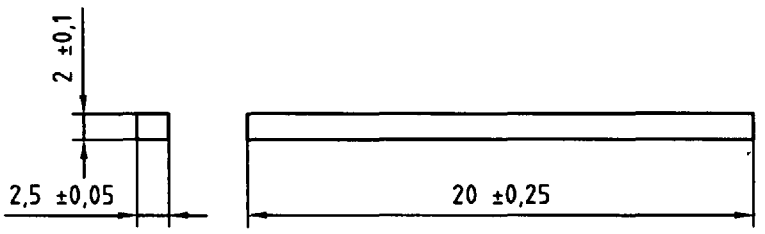


Figure 4 — Specimen size

7 Conditioning

The test specimens shall be conditioned as specified in the standard for the material tested. In the absence of this information, select the most appropriate conditions from ISO 291, unless otherwise agreed upon by the interested parties, e.g. for testing at high or low temperatures.

8 Procedure

8.1 In establishing the brittleness temperature of a material, it is recommended that the test be started at a temperature at which 50 % failure is expected. Test a minimum of ten specimens at this temperature. If all of the specimens fail, increase the temperature of the bath by 10 °C and repeat the test using new specimens. If none of the specimens fail, decrease the bath temperature by 10 °C and repeat the test using new specimens. If the approximate brittleness temperature is not known, choose the starting temperature arbitrarily.

8.2 Prior to beginning a test, prepare a bath and bring the apparatus to the desired starting temperature. If the bath is cooled using dry ice, place a suitable amount of powdered dry ice in the insulated tank and slowly add the heat-transfer medium until the tank is filled to a level 30 mm to 50 mm from the top. If the apparatus is equipped with a liquid-nitrogen or CO_2 cooling system and automatic temperature control, follow the instructions provided by the manufacturer of the instrument for preparing and operating the bath.

8.3 Mount the test specimens firmly in the clamping device and secure the latter in the test machine (see Figure 3).

NOTE Excessive clamping forces may pre-stress some materials, causing premature failure during the test. A torque wrench may be used so that the force necessary to hold the specimens in the clamps can be monitored and the minimum force applied uniformly to each specimen. It is recommended that a torque of 2,8 N·m be used to secure the specimens in the clamps.

8.4 Lower the clamping device into the heat-transfer medium. If dry ice is being used as a coolant, maintain a constant temperature by the judicious addition of small quantities of dry ice. If the apparatus is equipped with a liquid-nitrogen or CO_2 cooling system and automatic temperature control, follow the manufacturer's instructions for setting and maintaining the temperature.

8.5 After waiting for 3 min ± 0,5 min when using a liquid medium, or 20 min ± 0,5 min when using a gaseous medium, record the temperature and deliver a single impact on the specimens.

8.6 Remove the clamping device from the test apparatus and remove the individual specimens from the clamping device. Examine each specimen to determine whether or not it has failed. Failure is defined as the division of a specimen into two or more completely separated pieces or as any crack in the specimen which is visible to the unaided eye. Where a specimen has not completely separated, it shall be bent to an angle of 90° in the same direction as the bend caused by the impact and then be examined for cracks at the bend. Record the number of failures and the temperature at which the specimens were tested.

NOTE The temperature of a specimen when it is bent will be substantially above the temperature at which the specimen was impacted.

Increase or decrease the temperature of the bath in uniform increments of 2 °C or 5 °C and repeat the procedure until the lowest temperature at which none of the specimens fail and the highest temperature at which all of the specimens fail is determined. Use new test specimens for each test.

8.7 Carry out tests at four or more temperatures in the range including 10 % to 90 % failure (0 % and 100 % failure are not useful in determining T_{50} by the graphical method given in 9.1).

9 Expression of results

The temperature T_{50} may be determined by either of the following methods:

9.1 Graphical method

Plot the percentage failure at any temperature against the test temperature on arithmetic probability paper and draw the best straight line through the results. Read the brittleness temperature from the graph where this line intersects that for 50 % probability.

9.2 Calculation method

Calculate T_{50} using following equation:

$$T_{50} = T_h + \Delta T\left(\frac{S}{100} - \frac{1}{2}\right)$$

where

T_{50} is the brittleness temperature, in degrees Celsius;

T_h is the highest temperature, in degrees Celsius, at which failure of all the test specimens occurred (use the correct algebraic sign);

ΔT is the uniform temperature increment, in kelvins, between successive tests;

S is the sum of the percentage of failures at each temperature (from a temperature corresponding to no failures down to and including the temperature T_h).

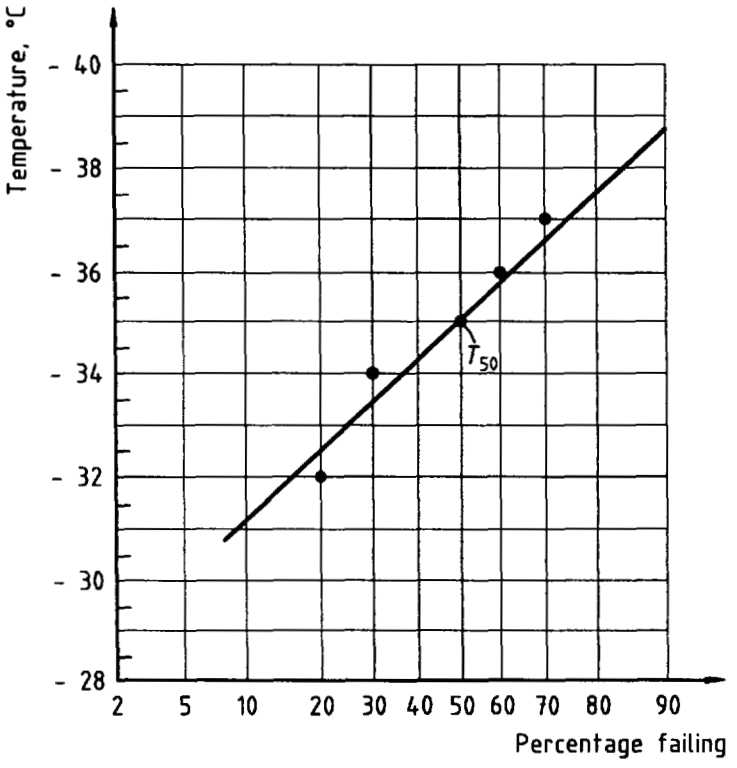


Figure 5 — Graphical method for determining the brittleness temperature T_{50}

10 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for complete identification of the material tested, including type, source, manufacturer's designation, form in which supplied and previous history;

c) the brittleness temperature, to the nearest 1 °C;

d) the method of preparation used for the test sheets and test specimens;

e) the conditioning procedure used, including the time which elapsed after moulding or annealing;

f) the heat-transfer medium used;

g) the date of the test.

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