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ISO RECOMMENDATION
R 974

PLASTICS

METHOD OF DETERMINING THE BRITTLENESS TEMPERATURE BY IMPACT

1st EDITION

February 1969

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BRIEF HISTORY

The ISO Recommendation R 974, *Plastics – Method of determining the brittleness temperature by impact,* was drawn up by Technical Committee ISO/TC 61, *Plastics,* the Secretariat of which is held by the United States of America Standards Institute (USASI).

Work on this question led, in 1964, to the adoption of a Draft ISO Recommendation.

In July 1965, this Draft ISO Recommendation (No. 822) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Greece	Romania
Australia	Hungary	Spain
Austria	India	Sweden
Belgium	Ireland	Turkey
Canada	Israel	U.A.R.
Chile	Italy	United Kingdom
Colombia	Japan	U.S.A.
Czechoslovakia	Netherlands	
Finland	New Zealand	

Four Member Bodies opposed the approval of the Draft :

France	Switzerland
Germany	U.S.S.R.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 974 February 1969

PLASTICS

METHOD OF DETERMINING THE BRITTLENESS TEMPERATURE BY IMPACT

1. SCOPE

1.1 This ISO Recommendation describes a method for determining the temperature at which plastics which are not rigid at normal ambient temperature exhibit brittle failure under specified conditions of deformation. A supplementary technique uses notched specimens and these show brittle failure at a much higher temperature. The method takes account of the statistical nature of brittle failure and makes provision for the testing of sufficient specimens to permit calculation of the brittleness temperature on a statistical basis.

1.2 The "brittleness temperature" test was originally developed to measure the temperature at which a polymer ceased to be flexible and became "glasslike". Because of the statistical nature of these failures, "brittleness temperature" is now defined as given in clause 3.1. The method specified in this ISO Recommendation establishes the temperatures at which there is a 50 % chance of failure in unnotched or notched specimens. It has been found useful for specification purposes although it does not necessarily relate to the lowest temperature at which the material may be used, since the basic polymer brittleness will be modified by any orientation produced during fabrication, by thermal history, and by the stress system applied, especially by the rate of impact. The typical precision of ± 5 °C should be recognized in establishing values used in material specifications.

2. PRINCIPLE OF METHOD

Bending a cantilever specimen through 90° around a mandrel of specified radius, at a constant testing speed in an inert medium, the temperature of which is accurately known and precisely controlled.

3. DEFINITIONS

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 *Testing speed.* The relative velocity between the striking edge of the test apparatus and a test specimen held in the specimen clamp.

4. APPARATUS

4.1 *Testing machine.* The testing machine consists 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 testing speed relative to the test specimens.

NOTES

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.

2. Commercial apparatus is available meeting the requirements of this section, in which the striking edge is driven by a motor, a solenoid, by gravity or by a spring. In all cases it should satisfy the definition given in clause 3.2 under actual conditions of test.

Dimensions in millimetres

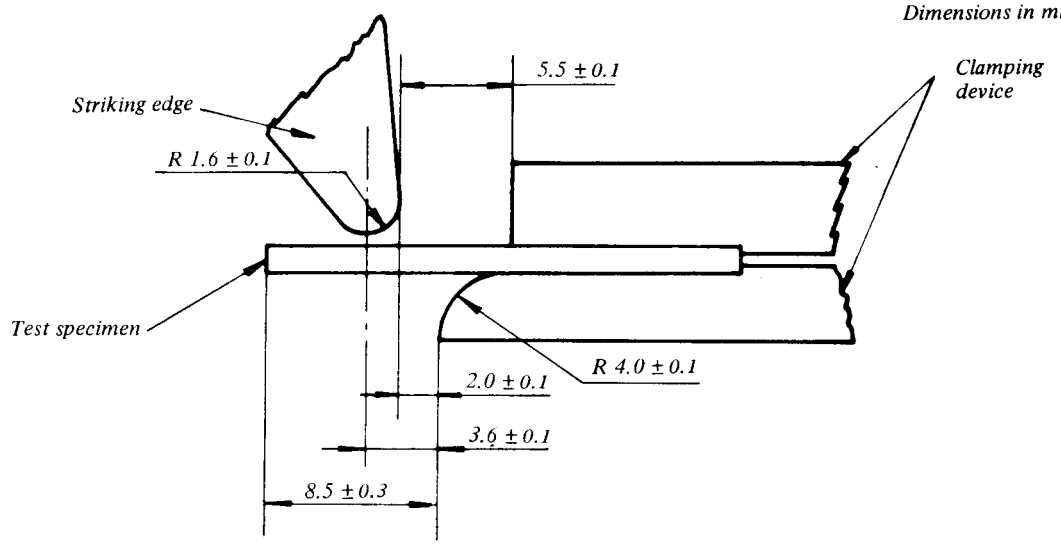

FIG. 1 – **Dimensional details of striking edge and clamping device (Positioning of unnotched test specimen)**

The principal dimensions of the apparatus should be as follows :

(*a*) radius of striking edge : 1.6 ± 0.1 mm;

(*b*) radius of lower jaw of clamping device : 4.0 ± 0.1 mm;

(*c*) separation between point of impact of striking edge and clamping device : 3.6 ± 0.1 mm;

(*d*) clearance between outside of striking edge and clamping device : 2.0 ± 0.1 mm.

The testing speed should be 200 ± 20 cm per second at impact and during at least the next 0.5 cm of travel.

4.2 *Temperature indicator.* A thermocouple constructed of copper and constantan wires of diameter in the range 0.2 to 0.5 mm, welded at their junction, (or equivalent thermometer) should be placed as near to the test specimens as possible. Any temperature indicator used with the thermocouple or the thermometer should be adequate to cover the range being tested and should be accurate to within ± 0.5 °C.

4.3 *Heat transfer medium.* A 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 should be used. The medium should be maintained at the test temperature to within ± 0.5 °C.

NOTE. – As the time of contact between the liquid and the plastics 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 region 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, selected physical properties should be measured before and after 15 minutes exposure at the highest temperature used (see ISO Recommendation R 175, *Determination of the resistance of plastics to chemical substances*). They should not differ significantly.

Dimensions in millimetres

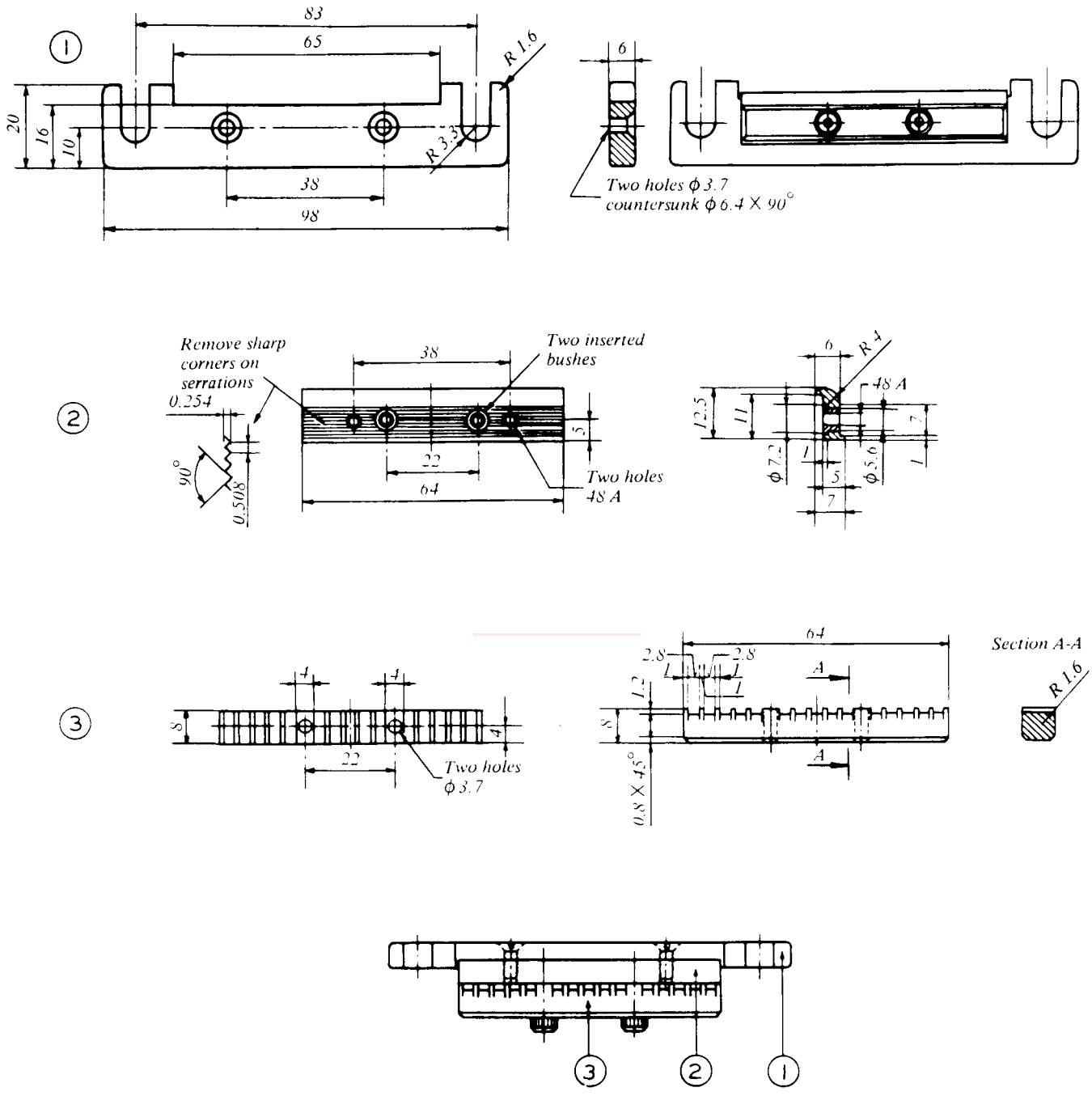

FIG. 2 – **Details of one form of clamp meeting the requirements of clause 4.1**

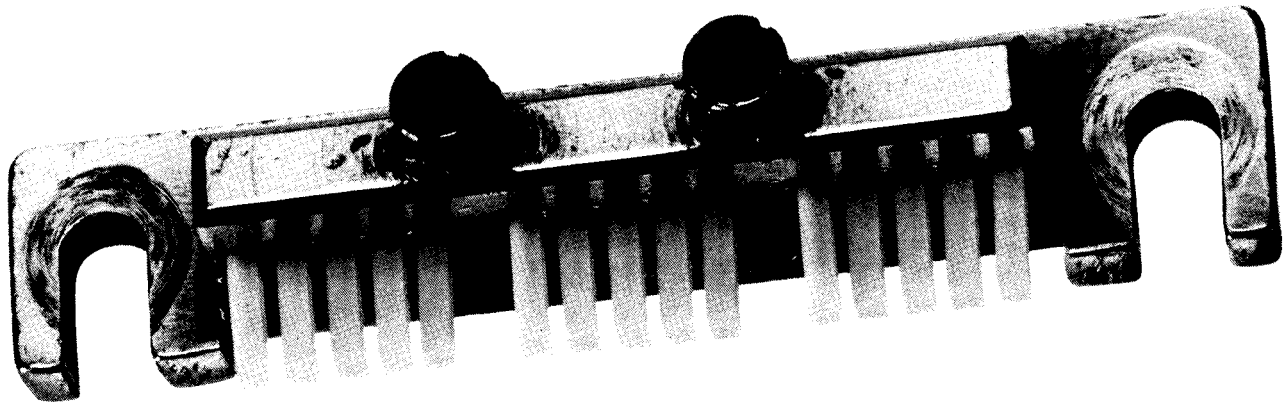

FIG. 3 – **Assembled clamp with test specimens**

5. TEST SPECIMENS

5.1 For many polymers, the results of this test depend to a large degree on the conditions used for sample moulding and on the mode of specimen preparation (three methods for moulding of polyolefins are described in the Appendix); the cleaner the edges and the freer the specimens from accidental notches, the lower will be the observed brittleness temperature.

It is therefore essential that the specimens be prepared in a reproducible way. A razor blade or other sharp tool should 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 the automatic method be used, since this enables accurately reproducible specimens to be prepared, even by semi-skilled operators, from laboratory to laboratory. Whatever method is used, it is essential that the cutter be inspected frequently and maintained in the sharpest possible condition.

NOTE. – The use of an automatic cutter for specimen preparation is discussed by P.N. Bestelink and S. Turner in a paper entitled *Low-temperature brittleness testing of polyethylene,* ASTM Bulletin No. 231, 68 (1958).

5.2 Test specimens 20.00 ± 0.25 mm long by 2.50 ± 0.05 mm wide and 1.6 ± 0.1 mm thick should be cut from a test sheet. They can be cut conveniently from a strip of the required thickness and 20.00 ± 0.25 mm wide by slicing off the required widths, preferably automatically.

5.3 Where a notch is desired, a clean cut should be made at about the middle of one of the 20 mm × 1.6 mm sides of the specimen at right angles to its long axis and 0.40 ± 0.02 mm deep using a sharp razor blade or, preferably, the automatic cutting machine already mentioned.

NOTE. – For some materials, particularly polyethylene, it may be desirable to use notched specimens, except when the test is being used to follow the effects of aging. The presence of the notch has the double effect of reducing the scatter of results and of raising the brittleness temperature in polyethylene from the vicinity of about −100 °C into a temperature region which is more easily reached experimentally, i.e. above −70 °C. (See *A study of the low-temperature brittleness testing of polyethylene,* by E.A.W. Hoff and S. Turner, ASTM Bulletin No. 225, 58 (1967)).

6. CONDITIONING

The test specimens should be conditioned prior to testing, but (if notched) after notching, in accordance with ISO Recommendation R 291, *Standard atmospheres for conditioning and testing.*

7. TEST PROCEDURE

7.1 The test specimens are mounted firmly in the clamping device and this is secured in the testing machine. When notched specimens are used, the notch is in the side of the specimen, not in the top or bottom faces, and is positioned on a line tangent with the bottom of the curve of the mandrel (the lower edge of the clamping device).

7.2 The specimens are brought to equilibrium at the test temperature. With liquid heat-transfer media, this requires 3 minutes; with gaseous media, allow 20 minutes.

7.3 The testing machine is actuated, bending the specimens around the mandrel.

7.4 The specimens are removed from the cold bath and the number which have failed noted. Failure is indicated by separation into two or more pieces.

7.5 Repeat tests are made 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 section 8.)

7.6 At least 100 specimens are tested. If four temperatures are used, test at least 25 specimens at each temperature. If more temperatures are used, fewer specimens may be tested at each temperature, but never less than 10 at any one temperature.

8. CALCULATIONS

Percentage failure at any temperature should be plotted against the test temperature on arithmetic probability paper and the best straight line drawn through the results. The brittleness temperature should be read from the graph where this line intersects that for 50 % probability.

NOTE. – The temperature T_{50} may also be calculated using the following formula :

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

where

T_b is the brittleness temperature in degrees Celsius;

T_h is the highest temperature at which failure of all the test specimens occurs (correct algebraic signs must be used);

ΔT is the uniform temperature increment, in degrees Celsius;

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).

9. TEST REPORT

The test report should include the following particulars :

(*a*) complete identification of the material tested, including type, source manufacturer's code designation, form in which supplied, and previous history;

(*b*) brittleness temperature to the nearest degree Celsius;

(*c*) whether notched or unnotched test specimens were used;

(*d*) the method of preparation used for the test sheets;

(*e*) conditioning procedure used, including time elapsed since moulding or annealing;

(*f*) the heat transfer medium used;

(*g*) date of the test.

APPENDIX

POLYOLEFIN SAMPLE PREPARATION

Z.1 GENERAL

Compression mould the test sheet using one of the three procedures listed below, the procedure chosen depending upon the material. The following choices will be found suitable :

Polymer	Procedure
Low-or medium-density polyethylene	A or C
High-density polyethylene Polypropylene	B or C

Z.2 PROCEDURE A (oven annealing)

The required sheets are formed in a simple three-part mould. This consists of a chase having an opening of suitable size for the desired test sheet dimensions and sufficient depth to produce sheets 1.6 ± 0.1 mm thick, plus *two* smooth metal backing plates at least 1 mm thick and large enough to cover the chase. Two sheets of clean aluminium foil 0.05 to 0.2 mm thick and of sufficient size to cover the chase are also required.

The backing plates and the aluminium foil are cleaned with solvent and thoroughly dried. Liquid release agents or waxes should not be used. One plate is then laid down and covered with a sheet of foil and the chase is placed on top of this assembly thus forming the cavity.

While pellets or granules may be charged directly into a cavity, milled crêpe is recommended since milling serves to erase pre-existent crystalline structure. To prepare the crêpe, the mill rolls should be hot enough to flux the plastic but not so hot as to cause it to drip. The crêpe should be slashed or turned frequently to promote mixing.

Subsequent compression moulding is facilitated if the rolls are adjusted to produce a crêpe which is as thin as possible. Ethylene plastics ordinarily should not be milled for more than 5 minutes to minimize oxidative and thermal changes.

The mould is loaded with enough material to form a sheet which fills the cavity completely and an excess of 2 to 10 % is added for flash. The charge is leveled and then covered, first with a sheet of clean dry foil and then with the second backing plate. The assembly is then placed in a press between platens preheated and maintained at a temperature high enough to flux the material and to ensure that it adheres well to the aluminium foil. Platen temperatures of 150 to 180 °C may be required depending upon the characteristics of the material being moulded.

Using low pressure, the press is closed enough to establish good contact between platens, plates and material and is held in that position for about 5 minutes to flux the material. Sufficient pressure is then applied to form a smooth, void-free sheet and this pressure is maintained for 5 minutes. After the moulding has been formed, the platens are cooled at any convenient rate.

When the mould has been cooled to 50 °C or less, it is taken out of the press and the backing plates are pried off without disturbing the aluminium foil, which should be adhering tightly to the chase and sheet and should appear smooth and free of dimples or sink marks.

If the sheet is not smooth and free of voids, it should be pressed over again. In that event, the sheet is cut into at least four pieces which are loaded into the mould as previously described together with enough additional material, preferably crêpe, to make up for flash lost in the first moulding. Pressing is then repeated as described in the preceding paragraph.