UDC 678.073 : 620.192.2

Ref. No. : ISO/R 306-1968 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 306

PLASTICS

DETERMINATION OF THE VICAT SOFTENING TEMPERATURE OF THERMOPLASTICS

2nd EDITION

October 1968

This second edition supersedes the first edition

For each individual country the only valid standard is the national standard of that country.

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 306, *Determination of the Vicat softening point of thermoplastics,* 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 by the Technical Committee began in 1956 and led, in 1959, to the adoption of a Draft ISO Recommendation.

In October 1960, this Draft ISO Recommendation (No. 380) was circulated to all the ISO Member Bodies for enquiry. It was approved by 22 Member Bodies. Two Member Bodies opposed the approval of the Draft : Italy and U.S.S.R.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in May 1963, to accept it as an ISO RECOMMENDATION.

BRIEF HISTORY RELATING TO THE 2nd EDITION

In September 1964, a Draft Revision (No. 750) 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	Germany	Romania
Australia	Hungary	South Africa, Rep. of
Austria	India	Spain
Belgium	Israel	Sweden
Bulgaria	Italy	Switzerland
Canada	Japan	Turkey
Czechoslovakia	Korea, Rep. of	U.A.R.
Denmark	Netherlands	United Kingdom
Finland	New Zealand	U.S.A.
France	Poland	Yugoslavia

One Member Body opposed the approval of the Draft :

U.S.S.R.

The Draft Revision was then submitted by correspondence to the ISO Council, which decided, in October 1968, to accept it.

The title of ISO Recommendation R 306-1963 has been modified as follows : *Plastics – Determination of the Vicat softening temperature of thermoplastics.*

This edition (2nd edition) supersedes the first edition of ISO Recommendation R 306-1963.

ISO Recommendation R 306 October 1968

PLASTICS

DETERMINATION OF THE VICAT SOFTENING TEMPERATURE OF THERMOPLASTICS

1. SCOPE

1.1 This ISO Recommendation describes two methods for the determination of the Vicat softening temperature of thermoplastics materials under compression:

- method A using a load of 1 kgf;
- method B using a load of 5 kgf.

1.2 These two methods are only applicable to thermoplastics, for which they give a measure of the temperature at which they start to soften rapidly.

2. PRINCIPLE

Determination of the temperature at which a standard indenter penetrates 1 mm into the surface of a plastics test specimen under one of the loads given in clause 1.1. During the test the temperature is raised at a uniform rate.

The temperature at 1 mm penetration is quoted as the *Vicat softening temperature* (VST) in Celsius degrees.

3. APPARATUS

The apparatus consists essentially of :

3.1 *A rod provided with a load carrying plate,* held in a rigid metal frame so that it can move freely and vertically, the base of the frame serving to support the test specimen under the indenter at the end of the rod (see Figure, page 7).

3.2 *An indenting tip,* preferably of hardened steel, 3 mm (1/8 in) long, of circular cross-section, and area 1.000 ± 0.015 mm^2 is fixed at the bottom of the rod. The lower surface of the indenting tip should be square to the axis of the rod and free from burs.

3.3 *A micrometer dial gauge* (or other suitable measuring instrument), graduated in divisions of 0.01 mm, should be used to measure the penetration of the indenter into the test specimen. The thrust of the dial gauge, which contributes to the thrust on the test specimen, should be known and should comply with clause 3.4 below.

3.4 *A load carrying plate* should be fitted to the rod (3.1) and a slotted weight adjusted centrally such that the total thrust applied to the test specimen can be made up to between 1000 gf and 1050 gf for Method A, and to between 5000 gf and 5050 gf for Method B. The combined masses of the rod, indenter and load carrying plate should not exceed 100 g.

NOTE. – The construction of the apparatus should be such that the micrometer dial gauge reading caused by differential thermal expansion over the intended temperature range does not exceed 0.02 mm when the test specimen is replaced by a piece of borosilicate glass or low thermal expansion alloy steel.
It is recommended that the apparatus be constructed of low thermal expansion alloy.

3.5 *A heating bath,* containing a suitable liquid (see Notes 1 and 2 below), should be provided in which the apparatus is placed so that the specimen is at least 35 mm (1.5 in) below the surface of the liquid. An efficient stirrer should be provided. The heating bath should be equipped with a means of control so that the temperature can be raised at a uniform rate of 50 ± 5 °C per hour (see Note 3, below).

3.6 *A mercury in glass thermometer* (or other accurate temperature measuring device), of appropriate range, and with graduations at least at each 0.5 °C should be used to measure temperature. The scale error at any reading should not exceed 0.5 °C.

NOTES

1. Liquid paraffin, transformer oil, glycerol and silicone oils may be suitable liquid heat-transfer media, but other liquids may be used. In all cases, it should be established that the liquid chosen is stable at the temperature used and does not affect the material under test.

2. If no suitable liquid can be found for use as a heat-transfer medium, as defined in Note 1, some different heating arrangement, for which air may be found to be a suitable heat-transfer medium, should be used. If air is used as the heat-transfer medium, it should be noted that errors in the quoted softening point may arise, unless care is taken to correct for possible differences in temperature between the air and the specimen.

3. A uniform rate of temperature rise can be obtained by controlling the heat input either manually or automatically, although the latter is strongly recommended. One method of operation found to be satisfactory is to provide an immersion heater adjusted to give the correct rate of temperature rise at the starting temperature of the test, and then to increase the power input (either in the same heater or in a subsidiary heater) by adjustment of a rheostat or variable transformer.

4. It is desirable to have a cooling coil in the liquid bath in order to reduce the time required to lower the temperature after previous determinations. This must be removed or drained before starting a test, as boiling of coolant can affect the rate of temperature rise.

4. TEST SPECIMENS

4.1 Two test specimens are used to test each sample.

The test specimens should be between 3 and 6.4 mm (1/8 and 1/4 in) in thickness and at least 10 mm (3/8 in) square. Their surfaces should be reasonably flat and parallel. They should be made in accordance with specifications, if any, relating to the material under test or to the preparation of test specimens. In the absence of such specifications, any other procedure may be used for the preparation of test specimens.

4.2 If the samples submitted for test are in the form of moulding materials (for example, powder or granulated materials), these should be moulded into specimens 3 mm (1/8 in) thick, in accordance with specifications, if any, relating to the material under test or ISO Recommendation R 293, *Compression moulding test specimens of thermoplastic materials* or ISO Recommendation R 294, *Injection moulding test specimens of thermoplastic materials.* If these are not available any other reproducible procedure may be followed which modifies the properties of the material as little as possible.

4.3 For sheet materials, the thickness of the test specimens should equal the thickness of the sheet. However,

(*a*) if the thickness exceeds 6.4 mm (1/4 in), the test specimens should be reduced in thickness to approximately 3 mm (1/8 in) by machining one surface, the other surface being left intact;

(*b*) if the thickness of the sheet is less than 2.5 mm (1/10 in), not more than three pieces should be stacked together to give a total thickness of at least 3 mm (1/8 in).

4.4 The test results obtained may depend on the moulding conditions used in the preparation of the specimen, although such a dependence is not common. When testing materials for which the results do depend on moulding conditions, special annealing or conditioning procedures are only used before testing if agreed to by all the parties concerned.

4.5 **Conditioning**

The test specimen should be conditioned in accordance with the procedure specified for each material, or with procedures agreed to by all the parties concerned.

5. PROCEDURE

5.1 The test specimen is mounted horizontally under the indenting tip (3.2) of the unloaded rod (3.1) shown in the Figure, page 7. The tip of the indenter should at no point be nearer to the edge of the test specimen than 3 mm (1/8 in). The surface of the test specimen in contact with the base of the apparatus should be flat.

5.2 The assembly is immersed in the heating bath (3.5), the temperature of which should be constant and at least 50 °C below the expected softening temperature of the material (see Note 4, section 3). The bulb of the thermometer (3.6) should be at the same level as, and as close as is practical to, the test specimen.

5.3 After 5 minutes, with the indenter still in position, the reading of the micrometer dial gauge is noted or the micrometer (3.3) is set to zero. The slotted weight is then added to the load carrying plate (3.4) such that the total thrust on the test specimen is between 1000 gf and 1050 gf for Method A, and between 5000 gf and 5050 gf for Method B.

5.4 The temperature of the bath is then raised at a uniform rate of 50 ± 5 °C per hour. The liquid is well stirred during the test.

5.5 The temperature of the bath at which the indenting tip has penetrated into the test specimen by 1 mm beyond its starting position defined in clause 5.3 is noted and recorded as the Vicat softening temperature (VST) of the test specimen (see Note 2, section 3).

5.6 The VST of the material under test is expressed as the arithmetic mean of the VST s of two test specimens. If the individual results differ by more than 2 °C the test is invalid and a repeat test must be carried out.

6. TEST REPORT

The test report should include the following :

(*a*) the method employed (A or B);

(*b*) Vicat softening temperature (VST) of the material;

(*c*) the method of specimen preparation (moulding conditions, etc);

(*d*) if composite test specimens are used, i.e. more than one layer, then the thickness and number of layers should be stated;

(*e*) the conditioning and annealing procedures used, if any;

(*f*) the nature of the immersion medium;

(*g*) any peculiar characteristics of the test specimen noted during the test or after removal from the apparatus.