INTERNATIONAL STANDARD 306

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Plastics — Determination of the Vicat softening temperature of thermoplastics

Matières plastiques — Détermination de la température de ramollissement Vicat des thermoplastiques

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 306 was drawn up by Technical Committee ISO/TC 61, *Plastics*, and circulated to the Member Bodies in December 1971.

It has been approved by the Member Bodies of the following countries :

Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
Brazil	Israel	Sweden
Canada	Italy	Switzerland
Czechoslovakia	Japan	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
Finland	New Zealand	U.S.A.
France	Portugal	U.S.S.R.
Germany	Romania	

No Member Body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 306-1968.

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Plastics — Determination of the Vicat softening temperature of thermoplastics

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies two methods for the determination of the Vicat softening temperature of thermoplastics materials :

- method A using a load of 9,81 N (1 kgf);
- method B using a load of 49,05 N (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 REFERENCES

ISO/R 293, *Plastics — Compression moulding test specimens of thermoplastic materials.*

ISO/R 294, *Plastics — Injection moulding test specimens of thermoplastic materials.*

3 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 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.

4 APPARATUS

The apparatus consists essentially of :

4.1 A **rod** provided with a load-carrying plate, held in a rigid metal frame so that it can move freely in the vertical direction, the base of the frame serving to support the test specimen under the indenting tip at the end of the rod (see figure).

4.2 An **indenting tip**, preferably of hardened steel, 3 mm long, of circular cross-section, and area 1,000 ± 0,015 mm², fixed at the bottom of the rod. The lower surface of the indenting tip shall be plane and perpendicular to the axis of the rod and free from burs.

4.3 A **micrometer dial gauge** (or other suitable measuring instrument), graduated in divisions of 0,01 mm, to measure the penetration of the indenting tip into the test specimen. The thrust of the dial gauge, which contributes to the thrust on the test specimen, must be known and shall comply with the requirements of 4.4.

4.4 A **load-carrying plate,** fitted to the rod (4.1), and suitable weights adjusted centrally so that the total thrust applied to the test specimen can be made up to between 9,81 N (1 000 gf) and 10,30 N (1 050 gf) for method A, and to between 49,05 N (5 000 gf) and 49,54 N (5 050 gf) for method B. The combined masses of the rod, indenting tip and load-carrying plate shall not exceed 100 g.

NOTE — The construction of the apparatus shall 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.

4.5 A **heating bath,** containing a suitable liquid (see notes 1 and 2 below), in which the apparatus is placed so that the specimen is at least 35 mm below the surface of the liquid. An efficient stirrer shall be provided. The heating bath shall be equipped with a means of control so that the temperature can be raised at a uniform rate either of 50 ± 5 °C/h or of 120 ± 5 °C/h (see note 3, below). These heating rates shall be considered to be met if, over every 5 min interval during the test, the temperature change is within the specified limits.

4.6 A **mercury-in-glass thermometer** (or other accurate temperature-measuring device), of appropriate range, and with graduations at least at each 0,5 °C. The scale error at any reading shall 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 shall 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, shall 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 between determinations. This must be removed or drained before starting a test, as boiling of coolant can affect the rate of temperature rise.

5 TEST SPECIMENS

5.1 Two test specimens are to be used to test each sample.

The test specimens shall be between 3 and 6 mm thick and at least 10 mm square. Their surfaces shall be reasonably flat and parallel and free from flash. They shall 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.

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

5.3 For sheet materials, the thickness of the test specimens shall be equal to the thickness of the sheet. However,

a) if the thickness exceeds 6 mm, the test specimens shall be reduced in thickness to approximately 3 mm by machining one surface, the other surface being left intact;

b) if the thickness of the sheet is less than 3 mm, not more than three pieces shall be stacked together to give a total thickness of between 3 and 6 mm.

5.4 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.

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

6 PROCEDURE

6.1 Mount the test specimen horizontally under the indenting tip (4.2) of the unloaded rod (4.1). The indenting tip shall at no point be nearer to the edge of the test specimen than 3 mm. The surface of the test specimen in contact with the base of the apparatus shall be flat.

6.2 Immerse the assembly in the heating bath (4.5), the temperature of which shall be constant and at least 50 °C below the expected softening temperature of the material (see note 4, clause 4). The bulb of the thermometer (4.6) shall be at the same level as, and as close as possible to, the test specimen.

6.3 After 5 min, with the indenting tip still in position, note the reading of the micrometer dial gauge or set the micrometer (4.3) to zero. Then add the weight to the load-carrying plate (4.4) so that the total thrust on the test specimen is between 9,81 N (1 000 gf) and 10,30 N (1 050 gf) for method A, and between 49,05 N (5 000 gf) and 49,54 N (5 050 gf) for method B.

6.4 Increase the temperature of the bath at a uniform rate of 50 ± 5 °C/h or, alternatively, of 120 ± 5 °C/h; stir the liquid well during the test. For referee tests a rate of 50 °C/h shall be used.

NOTE — For some materials, at the higher rate (120 °C/h), Vicat softening temperature of up to 4 °C higher can be observed.

6.5 Note the temperature of the bath at which the indenting tip has penetrated into the test specimen by 1,00 mm beyond its starting position defined in 6.3 and record it as the Vicat softening temperature (VST) of the test specimen (see note 2, clause 4).

6.6 Express the VST of the material under test 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.

7 TEST REPORT

The test report shall include the following particulars :

a) the method employed (A or B), and the rate of temperature increase;

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

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

d) the thickness and number of layers of composite test specimens (i.e. specimens consisting of more than one layer) if these are used;

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.

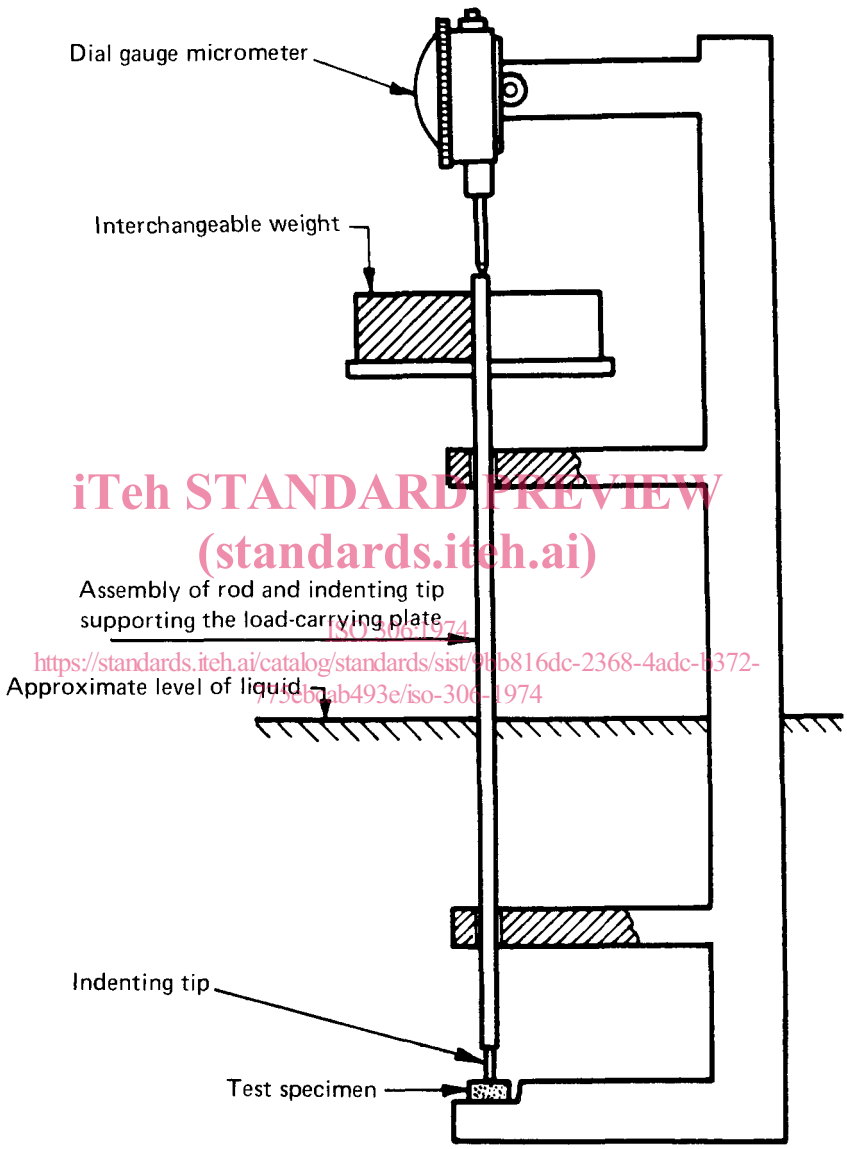

FIGURE – **Schematic diagram of apparatus for the determination of the Vicat softening temperature**