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Plastics - Thermoplastic materials - Determination of Vicat softening temperature (VST) (ISO 306:2013)

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Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 20.11.2013.	Date of Availability of the European standard is 20.11.2013.
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Plastics - Thermoplastic materials - Determination of Vicat softening temperature (VST) (ISO 306:2013)

Plastiques - Matières thermoplastiques - Détermination de la température de ramollissement Vicat (VST) (ISO 306:2013)

Kunststoffe - Thermoplaste - Bestimmung der Vicat-Erweichungstemperatur (VST) (ISO 306:2013)

This European Standard was approved by CEN on 2 November 2013.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN ISO 306:2013) has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 249 “Plastics” the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2014, and conflicting national standards shall be withdrawn at the latest by May 2014.

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Endorsement notice

The text of ISO 306:2013 has been approved by CEN as EN ISO 306:2013 without any modification.

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Introduction

This revision introduces heating equipment, consisting of a fluidized bed, as a new apparatus; this is as an alternative to liquid-filled heating baths and direct-contact heating units. Fluidized beds can reach higher temperatures than traditional liquid-filled heating baths; therefore, they represent a suitable way to measure the Vicat softening temperature (VST) of thermoplastic materials having improved thermo-mechanical properties.

It was also felt necessary to add

— precision data based on round robin testing performed in 2009, and

— comparison data for tests with liquid-filled and fluidized bed.

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Plastics — Thermoplastic materials — Determination of Vicat softening temperature (VST)

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1 Scope

This International Standard specifies four methods for the determination of the Vicat softening temperature (VST) of thermoplastic materials:

— method A50 using a force of 10 N and a heating rate of 50 K/h;

— method B50 using a force of 50 N and a heating rate of 50 K/h;

— method A120 using a force of 10 N and a heating rate of 120 K/h;

— method B120 using a force of 50 N and a heating rate of 120 K/h.

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

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable to its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

ISO 294-1, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles, and moulding of multipurpose and bar test specimens*

ISO 294-2, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 2: Small tensile bars*

ISO 294-3, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates*

ISO 472, *Plastics — Vocabulary*

ISO 2818, *Plastics — Preparation of test specimens by machining*

ISO 20753, *Plastics — Test specimens*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following apply.

3.1
penetration
distance over which the indenting tip has to penetrate into the specimen under test

Note 1 to entry: It is expressed in millimetres (mm).

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3.2
load
force applied to test specimen by means of the indenting tip

Note 1 to entry: It is expressed in Newtons (N).

3.3
Vicat softening temperature
VST
temperature at which a flat-ended needle penetrates the specimen to a depth of 1 mm under a specified load using a selected uniform rate of temperature rise

Note 1 to entry: It is expressed in degrees Celsius (°C).

4 Principle

The temperature at which a standard indenting tip with a flat point penetrates 1 mm into the surface of a plastic test specimen is determined. The indenting tip exerts a specified force perpendicular to the test specimen, while the specimen is heated at a specified and uniform rate.

The temperature, in degrees Celsius, of the specimen, measured as close as possible to the indented area at 1 mm penetration, is quoted as the VST.

5 Apparatus

5.1 Heating equipment, consisting of one of the following (5.1.1, 5.1.2 or 5.1.3) that will accept a minimum of two test frame assemblies and a cooling device (5.1.4).

The heating equipment shall be provided with means of temperature control, enabling the temperature to be raised at a uniform rate of (50 ± 5) K/h or (120 ± 10) K/h.

The heating rate shall be verified

— either by checking and recording automatically over the whole temperature range, or

— by manually checking and recording the temperature change at 6-min intervals over the temperature range being verified.

The requirement for the heating rate shall be considered satisfied if, over every 6-min interval during the test, the temperature change is (5 ± 0,5) °C or (12 ± 1) °C, respectively. For multiposition heating equipment, the heating rate shall be verified at each test station. The apparatus may be designed to shut off the heat automatically and sound an alarm when the specified indentation has been reached.

5.1.1 Liquid-filled heating bath, containing a liquid in which the test specimen can be immersed to a depth of at least 35 mm. Liquid paraffin, transformer oil, glycerol and silicone oil are suitable liquid heat-transfer media, but other liquids may be used. An efficient stirrer shall be provided. It shall be established that the liquid chosen is stable at the temperature used and does not affect the material under test, for example by swelling or cracking. Do not heat the liquid filled heating bath in excess of the flash point specified by the heat transfer media manufacturer.

5.1.2 Direct-contact heating unit, containing heaters and blocks, which through conductive heating, raise the temperature of the specimen at a controlled rate until the VST is reached.

5.1.3 Fluidized bed, containing a powder bed (e.g. aluminium oxide powder), in which the test specimen can be immersed to a depth of at least 35 mm. This type of apparatus uses a micrometric aluminium oxide powder, which when mixed with a suitable flow of heated air, creates a liquid-like heating medium. The maximum working temperatures (and measurable VSTs) are therefore much higher than those attainable

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with liquids according to 5.1.1. An efficient stirring mechanism shall be provided, in order to achieve a temperature homogeneity in the specimen area analogous to the case of a liquid-filled heating bath.

5.1.4 Cooling device, as an optional means to reduce the temperature of the heating device; it may be used to reduce the time between tests.

5.2 Test frame assemblies (see Figures 1 and 2), consisting of the following.

5.2.1 Rod and frame, provided with a support plate or other suitable load-application device, held in a rigid metal frame. The rod shall be able to move freely, with minimum friction, in a vertical direction. The rod shall be designed to accept weights that will apply the test load. The base of the frame supports the test specimen under the indenting tip at the end of the rod (see Figures 1 and 2). It is recommended that the rod and the frame(s) be constructed of low thermal expansion material.

5.2.2 Indenting tip, preferably of hardened steel, 1,5 mm to 3 mm long, of circular cross-section and of area (1,000 ± 0,015) mm^2 (corresponding to an indenting-tip diameter of (1,128 ± 0,008) mm), fixed at the bottom of the rod (5.2.1). The surface of the indenting tip in contact with the specimen shall be flat and perpendicular to the axis of the rod, and free from burrs.

5.2.3 Weights, applied to the rod (5.2.1) centrally, so that the total load applied to the test specimen is (10 ± 0,2) N for methods A50 and A120 and (50 ± 1) N for methods B50 and B120.

5.2.4 Penetration-measuring device, calibrated micrometer dial gauge, LVDT (linear variable differential transformer) or other suitable measuring instrument to measure the penetration of the indenting tip into the test specimen to an accuracy of ±0,01 mm.

5.2.5 Temperature-measuring device.

5.2.5.1 For a liquid-filled bath and a fluidized bed, use a suitable temperature-measuring instrument of appropriate range and accurate to within ±0,5 K. Thermometers shall be calibrated at the depth of immersion required by 5.1.1 and 5.1.3. The temperature-measuring device shall be positioned as close as possible to both the indenting tip and the specimen, but avoiding direct contact between the sensor and specimen.

5.2.5.2 For a direct-contact heating unit, use a suitable temperature-measuring instrument of appropriate range and accurate to within ±0,5 K. The sensor shall be positioned as close as possible to both the indenting tip and the specimen, but avoiding direct contact between the sensor and specimen.

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