



SLOVENSKI STANDARD
SIST EN 1427:2007

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Bitumen and bituminous binders - Determination of the softening point - Ring and Ball method

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Erweichungspunktes - Ring- und Kugel-Verfahren

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Bitumes et liants bitumineux - Détermination du point de ramollissement - Méthode Bille et Anneau

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ICS:

75.140	Voski, bitumni in drugi naftni proizvodi	Waxes, bituminous materials and other petroleum products
91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

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English Version

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This European Standard was approved by CEN on 3 February 2007.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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Foreword

This document (EN 1427:2007) has been prepared by Technical Committee CEN/TC 336 "Bituminous binders", the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by September 2007, and conflicting national standards shall be withdrawn at the latest by September 2007.

This document supersedes EN 1427:1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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1 Scope

This European Standard specifies a method for the determination of the softening point of bitumen and bituminous binders in the range of 28 °C to 150 °C.

NOTE The method described is also applicable to bituminous binders that have been recovered from bituminous mixes, e.g. by extraction.

WARNING — Use of this European Standard can involve hazardous materials, operations and equipment. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 58, *Bitumen and bituminous binders – Sampling bituminous binders*

EN 12594, *Bitumen and bituminous binders – Preparation of test samples*

EN 12597, *Bitumen and bituminous binders – Terminology*

EN ISO 3696:1995, *Water for analytical laboratory use – Specification and test methods (ISO 3696:1987)*

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3 Terms and definitions

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For the purposes of this document, the terms and definitions given in EN 12597 and the following apply.

3.1

softening point

temperature at which material under standardised test conditions attains a specific consistency

4 Principle

Two horizontal discs of bituminous binder, cast in shouldered brass rings shall be heated at a controlled rate in a liquid bath while each supports a steel ball. The softening point shall be reported as the mean of the temperatures at which the two discs soften enough to allow each ball, enveloped in bituminous binder, to fall a distance of $(25,0 \pm 0,4)$ mm.

5 Reagents and materials

5.1 General

Use only reagents of specified analytical grade and water conforming to grade 3 of EN ISO 3696:1995 unless otherwise specified.

5.2 Bath liquid

5.2.1 Freshly boiled distilled water, or deionized water

The use of freshly boiled distilled water or deionized water is essential to avoid trapping air bubbles on the surface of the test sample which can affect the results.

5.2.2 Glycerol, with a density $(1\,250 \pm 10)$ kg/m³ at 20 °C, with a 99 % mass fraction purity.

WARNING — Glycerol has a flash point of 160 °C, measured in accordance with EN ISO 2592. In temperatures above 100 °C, as glycerol may contain water due to hygroscopic properties, glycerol may splash on the heating elements and ignite.

5.3 Release agent, mixture of glycerol and dextrin or mineral talc, or another commercially available release agent.

To prevent the bituminous binder adhering to the pouring plate when casting discs, the surface of the metal pouring plate shall be thinly coated just before use with the release agent.

NOTE 1 Applying the release agent coating is easier when the plate is warmed to approximately 40 °C.

NOTE2 Instead of a release agent, other materials, e.g. baking paper, can be used.

6 Apparatus iTeh STANDARD PREVIEW (standards.iteh.ai)

6.1 Ring and Ball apparatus,

NOTE Either manual, semi-automatic or automatic, comprising the elements given in 6.1.1 to 6.1.9.
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6.1.1 Rings, two, square-shouldered, in brass and conforming to the dimensions shown in Figure 1.

6.1.2 Pouring plate, flat, smooth, metal, approximately 50 mm × 75 mm and 1,5 mm to 2,0 mm thick with the edges turned down (see Figure 2) or a ceramic tile 5 mm to 6 mm thick.

6.1.3 Balls, stainless steel, two, $(9,50 \pm 0,05)$ mm in diameter, each having a mass of $(3,50 \pm 0,05)$ g.

6.1.4 Ball centering guides, brass, two for centring the steel balls, one for each ring. An example of ball centring guide is given in Figure 3.

6.1.5 Ring holder and assembly, stainless steel or brass, with a holder (A) to support the two rings in a horizontal position, conforming to the shape and dimensions shown in Figure 4, supported in the assembly shown in Figure 5. The bottom of the shouldered rings in the ring holder shall be $(25,0 \pm 0,4)$ mm above the upper surface of the bottom plate (B) (see Figure 6); the upper edge of the rings shall be (50 ± 3) mm below the surface of the bath liquid.

6.1.6 Bath, glass beaker capable of being heated, with not less than an 85 mm inside diameter and not less than a 120 mm depth from the bottom of the beaker, as shown in Figure 5.

NOTE A squat form 600 ml beaker is suitable.

6.1.7 Thermometers

6.1.7.1 Thermometers, calibrated, as specified in Annex A.

6.1.7.2 The appropriate thermometer shall be suspended in the assembly as shown in Figure 5 so that the bottom of the bulb is level with the bottom of the rings and within 13 mm of the rings, but not touching the rings or the ring holder.

Other temperature measuring devices may be used instead of mercury stem thermometers, however, the mercury stem thermometer is the reference device and any alternative device employed shall be calibrated to provide the same readings as a mercury stem thermometer, recognising and allowing for changed thermal response times compared with the mercury thermometer.

NOTE For the method described in which increasing temperatures are read during the test procedure, documented corrections should be determined in advance and applied to the observed readings.

6.1.8 **Stirrer**, propeller stirrer which operates smoothly or a magnetic stirrer/hot plate with suitably coated stirring bar of length approximately 40 mm and diameter 8 mm to ensure uniform heat distribution throughout the bath and to avoid turbulent flow throughout the bath. The stirrer shall be placed so that it does not disturb the samples when the test is in operation.

The rotation speed of the stirring bar shall be approximately 100 r/min.

NOTE The stirrer blade of the propeller stirrer should preferably be placed at a level between the bottom plate of the the assembly and the bottom of the beaker (see Figure 5).

WARNING — If the propeller stirrer is electrically driven, ensure that it is safely earthed.

6.1.9 Semi-automatic or automatic equipment

Instead of the apparatus described in 6.1.1 to 6.1.8, a semi-automatic or automatic apparatus may be used (after it has been calibrated). The results obtained shall be the same as with the manual method and shall be within the precision of the manual method. If automatic equipment is used, it shall be calibrated as frequently as necessary, but at least once a year. In case of doubt, the referee method shall be the manual one with the apparatus as shown in Figure 5.

NOTE When using an automatic apparatus, it is possible for the ball to trigger the interruption of a ray of light at a distance of $(25,0 \pm 0,4)$ mm below the underside of the ring, rather than the ball touching the bottom plate.

7 Preparation and preservation of laboratory samples and test samples

Take the laboratory sample in accordance with EN 58 taking all necessary safety precautions and ensuring that the test sample is representative of the laboratory sample from which it is taken. Prepare the test sample in accordance with EN 12594.

For modified bitumen the quantity of sample shall be adjusted to fill four rings in case the test has to be repeated (see 8.6, a)).

Heat the two brass rings but not the pouring plate, to approximately 90 °C above the expected softening point and place them on the pouring plate treated with the release agent (see NOTE 1 in 5.3).

Do not use too much of the release agent to avoid particles in the bath liquid that can interfere with the ray of light if used.

Pour a slight excess of the heated bituminous binder into each of the rings, then allow the specimens to cool in ambient air for at least 30 min. For materials that are soft at room temperature, cool the test sample for at least 30 min at an air temperature at least 10 °C below the expected softening point. No more than 4 h shall elapse until completion of the test from the time the test samples are poured.

When the test samples have cooled, cleanly cut away the excess bituminous binder with a warmed knife or blade, so that each test sample is flush and level with the top of its ring.

8 Procedure and reporting

8.1 Select the appropriate bath liquid and thermometer for the expected softening point, as follows:

- a) softening points between 28 °C and 80 °C: use freshly boiled distilled or deionized water. Use a thermometer with subdivisions of 0,2 °C. The initial bath temperature shall be (5 ± 1) °C.
- b) softening points above 80 °C and up to 150 °C: use glycerol and a thermometer with subdivisions of 0,5 °C. The initial bath temperature shall be (30 ± 1) °C.

For reference purposes, all softening points of 80 °C or less shall be determined in a water bath. Softening points above 80 °C up to 150 °C, shall be determined in a glycerol bath.

8.2 Assemble the apparatus with the test sample rings, ball-centering guides and thermometer in position and fill the bath so that the surface of the bath liquid is (50 ± 3) mm above the upper edge of the rings. Using forceps, place the two steel balls in the bath or in a separate container at 5 °C or 30 °C, as appropriate. Ensure that the balls have the same temperature as the rest of the assembly. Take care not to contaminate the bath liquid with any material that could affect the results.

8.3 Place the bath in ice water or a thermostatic apparatus (device) to cool to (5 ± 1) °C (where water is the bath liquid), or gently heat to (30 ± 1) °C (where glycerol is the bath liquid) to establish the correct initial bath temperature. Maintain at this temperature for 15 min with the apparatus in place.

8.4 Using forceps, place a ball in each ball centering guide.

8.5 Stir the bath liquid and heat from below so that the temperature rises at a uniform rate of 5 °C/min (see above). Protect the bath from draughts using shields if necessary. Do not average the rate of temperature rise over the test period. The maximum permissible variation for any 1 min period after the first 3 min shall be $\pm 0,6$ °C. Over the whole range of the measurements the temperature deviation shall be less than ± 1 °C. Reject any test in which the rate of temperature rise does not fall within these limits.

Rigid adherence to the specified heating rate is essential for reproducibility of results. Either a gas burner or electric heater can be used. If a gas burner is used it should be protected from draughts using shields. To maintain the prescribed rate of heating, the electrical heater should be the low-lag, variable output type.

After the first 3 min, the temperature rise shall be between 4,4 °C and 5,6 °C in every individual minute measured.

After the first 3 min, the overall temperature rise at the end of the test shall be within 1 °C of the number of minutes, $\times 5$ °C.

8.6 For each ring and ball, record the temperature indicated by the thermometer the instant the bituminous binder surrounding the ball touches the bottom plate if the manual method is used, or interrupts the ray of light if the semi-automatic or automatic apparatus is used. Do not apply a correction for the emergent stem of the thermometer.

If the difference between the two temperatures exceeds 1 °C for softening points below 80 °C or exceeds 2 °C for softening points above 80 °C, repeat the test.

For modified bitumen repeat the test if:

- a) difference between the two temperatures exceeds 2 °C;