SLOVENSKI STANDARD
SIST EN 1427:2015

01-oktober-2015

Nadomešča:
SIST EN 1427:2007

Bitumen in bitumenska veziva - Določanje zmehčišča - Metoda prstana in kroglice

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

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

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

Ta slovenski standard je istoveten z: **EN 1427:2015**

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

SIST EN 1427:2015 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1427

July 2015

ICS 91.100.50; 75.140

Supersedes EN 1427:2007

English Version

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

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

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

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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 1427:2015) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

This document supersedes EN 1427:2007.

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of an identical text or by endorsement, at the latest by January 2016, and conflicting national standards shall be withdrawn at the latest by January 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

The major changes in comparison to EN 1427:2007 are:

- mercury thermometer is no longer the normative reference thermometer (see 6.1.7);
- the description of establishing the temperature gradient has been improved (see 8.6);
- it is recognised that it is difficult to establish the temperature gradient homogeneously in the bath (6.1.6) in the temperature range from 30 °C till 60 °C when glycerol is used as bath liquid. A new procedure requires the gradient to be met from 60 °C. An informative Annex B with examples of valid and invalid temperature gradients has been added for clarification.

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

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.

Technical warning - The change from mercury thermometers to electronic temperature devices has revealed that the temperature definition in the mercury thermometer has not been precise enough to make a correct, unbiased transfer to electronic devices. Care should be taken for softening points ring and ball above 100 °C as the condition may have changed from previous practise to present days testing equipment. Below approx. 100 °C the difference in temperature readings between electronic and mercury stem thermometer is acceptable compared to the repeatability of this test methods. [Reference: ASTM E20 Group]

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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)*

3 Terms and definitions

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 ± 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 Distilled or deionized water

The use of freshly boiled, cooled, distilled 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 ± 10) kg/m^3 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.

NOTE 2 Instead of a release agent, other materials, e.g. baking paper, can be used.

6 Apparatus

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.

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**).

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

NOTE For automatic apparatus, see 6.1.9.

6.1.6 Bath, glass beaker capable of being heated, with not less than an 85 mm outside 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 Temperature measuring device

6.1.7.1 General

A temperature measuring device (combining sensor and reading unit) shall for determination in water:

— have a range from at least 0 °C to 90 °C;

— be readable to 0,2 °C or less and

— have an accuracy of 0,3 °C or better.

A temperature measuring device (combining sensor and reading unit) shall for determination in glycerol:

— have a range from at least 30 °C to 155 °C;

— be readable to 0,5 °C or less and

— have an accuracy of 0,3 °C or better.

Sensors based on platinum resistance thermometers cast in glass of the approximate dimension mentioned in Annex A have been found suitable but other principles are also allowed. The thermal response time of the sensor shall be comparable with the former used reference (see informative Annex A). The temperature-measuring device shall be calibrated regularly.

A solid stem mercury thermometer (which used to be the former reference thermometer as described in Annex A) is also allowed if national regulations permit its use.

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

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.

The stirrer blade of the propeller stirrer should preferably be placed at a level between the bottom plate of 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. 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 ± 0,4) mm below the underside of the ring, rather than the ball touching the bottom plate.

6.2 Calibration/Verification

All equipment shall be calibrated/verified at least once per year.

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 8.7, a)).

Heat the two brass rings but not the pouring plate, to a temperature of not more than 100 °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. Cut the excess of bituminous binder immediately before placing the rings in the assembly. In this way any surface contamination is avoided.

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, cooled, distilled or deionized water (5.2.1). Use a temperature measuring device as described in 6.1.7.1. readable to 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 temperature measuring device as described in 6.1.7.1. readable to 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 temperature sensor 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. Ensure that the bath liquid is clean and without contamination, e.g. by covering with a lid.

Before use, check the bath level.

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 the assembly at the temperature for at least 15 min but not more than 20 min.

8.4 Take the bath containing the assembled apparatus out of the ice water or thermostatic device, dry the outer surface gently to remove any liquid that might form mist, and place it into the testing apparatus as quickly as possible. The effect on the starting temperature shall be minimized.

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

8.6 When starting to heat and to stir, check the starting temperature. If it is not within the ranges given in 8.3, stop the test.

Stir the bath liquid and heat from below so that the temperature rises can settle at a uniform rate of 5 °C/min.

Protect the bath from draughts using shields if necessary. 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 can be the low-lag, variable output type.

When water is used as bath liquid the first three minutes are for settling the heating rate of 5 °C/min only. When glycerol is used as bath liquid the temperature range from 30 °C to 60 °C is intended for settling the heating rate of 5 °C/min and the temperature should have reached (60 ± 1) °C after 6 min. Check the correct temperature range at least once after the first three minutes, when using water as bath liquid, or after 60 °C, when using glycerol.

After the first 3 min (water) or when 60 °C is reached (glycerol), the temperature rise shall be between 4,4 °C and 5,6 °C in every individual minute measured. If not stop the test.

After the first 3 min (water) or from when 60 °C is reached, the overall temperature rise at the end of the test shall be within ± 1 °C of the number of minutes (in decimals) × 5°C.

Reject any test in which the rate of temperature rise does not fall within these limits

8.7 For each ring and ball, record the temperature indicated by the temperature device, 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.

If a total immersion thermometer is used as specified in Annex A 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;

b) ball breaks the surrounding film before touching the bottom plate (or interrupting the ray of light) or if partial detachment of bitumen from the ball is observed.

8.8 Results (mean of results) as follows:

8.8.1 General

The softening point of given bitumen determined in a water bath is approximately 4 °C lower than the softening point of the same bitumen determined in a glycerol bath. Consequently, changing from water to glycerol for determinations above 80 °C can create discontinuity and lead to ambiguity. For example, a bitumen with a softening point of 78 °C determined in water can be expected to give a value of 82 °C if determined in glycerol. It is therefore highly recommended to define arbitrary protocols for tests displaying results around 80 °C. Such protocols are defined in 8.8.2 to 8.8.5. In these clauses 'softening points results' refers to the mean or corrected mean of the two test temperatures determined according to 8.7.

8.8.2 Softening point results from 28 °C to 80 °C determined in a water bath. Report the result obtained.

8.8.3 Softening point results above 80 °C determined in a water bath. Reject the result as invalid and repeat the determination in a glycerol bath.

8.8.4 Softening point results of 84 °C and below determined in a glycerol bath. Repeat the determination in a water bath. If the result determined in a water bath is 80 °C or lower, report this result, otherwise report the result obtained in the glycerol bath.

8.8.5 Softening point results above 84 °C determined in a glycerol bath. Report the result obtained.

9 Expression of results

For softening points below or equal to 80 °C, express to the nearest 0,2 °C the mean of the temperatures recorded in 8.7 as the softening point.

For softening points above 80 °C, express to the nearest 0,5 °C the mean of the temperatures recorded in 8.7 as the softening point.

The mean values of determinations are arithmetic means rounded up to the nearest 0,2 °C or 0,5 °C whichever applicable.

It should be noted that the results obtained using this standard were up to 1,5 °C lower than those obtained using a comparable method that does not use a stirrer, such as ASTM D 36 version valid in 1999.

10 Precision

10.1 Repeatability

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values given in Table 1 in only one case in 20.

10.2 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values given in Table 1, in only one case in 20.

Table 1 — Precision

Bath liquid	Type of bitumen	Repeatability *r*, °C	Reproducibility *R*, °C
Water	Unmodified	1,0	2,0
Water	Polymer modified	1,5	3,5
Glycerol	Oxidized	1,5	5,5

These precision data estimates for unmodified and modified bitumens were the outcome of round robin programmes conducted by CEN Working Groups. Those for oxidized bitumens were the outcome of a round robin test conducted by the UK's Institute of Petroleum, and were adopted by CEN/TC 19/SC 1 WG 1. Specific precision data are not available for unmodified and modified bitumens with softening points greater than 80 °C, or for oxidized bitumens with softening points below 80 °C. It would be impracticable to carry out round robins on such materials as they are rarely encountered. For any such materials, the precision values relevant to the bath medium employed should be used as a guide.

NOTE Danish Round Robin found, as a mean for five polymer modified bitumens with softening point above 80 °C, a repeatability of 2,2 °C and a reproducibility of 6,3 °C.