



SLOVENSKI STANDARD
SIST EN 1427:2000

01-julij-2000

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

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

Bitumes et liants bitumineux - Détermination de la température de ramolissement - Méthode Bille et Anneau

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Ta slovenski standard je istoveten z: EN 1427:1999

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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en

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ICS 75.140; 91.100.50

English version

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

Bitumes et liants bitumineux - Détermination de la température de ramollissement - Méthode Bille et Anneau

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

This European Standard was approved by CEN on 5 September 1999.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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 Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NNI.

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of an identical text or by endorsement, at the latest by April 2000, and conflicting national standards shall be withdrawn at the latest by April 2000.

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

Annex A is normative.

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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 30 °C to 150 °C.

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

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

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 58, *Sampling bituminous binders.*

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

prEN 12597:1996, *Bitumen and bituminous binders- Terminology*

EN 22592, *Petroleum products – Determination of flash and fire points - Cleveland open cup method (ISO 2592:1973)*

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

3 Terms and definitions

For the purposes of this European Standard, the definitions given in prEN 12597:1996 and the following apply:

3.1 softening point

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

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4 Principle

Two horizontal discs of bituminous binder, cast in shouldered brass rings, are heated at a controlled rate in a liquid bath while each supports a steel ball. The softening point is 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 $25,0 \text{ mm} \pm 0,4 \text{ mm}$.

5 Reagents and materials

5.1 General

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

5.2 Bath liquid

5.2.1 Freshly boiled distilled water, or deionized water.

NOTE 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 affects the results.

5.2.2 Glycerol, density $1\,250 \text{ kg/m}^3 \pm 10 \text{ kg/m}^3$ at $20 \text{ }^\circ\text{C}$, 99 % (*m/m*) purity.

WARNING Glycerol has a flash point of $160 \text{ }^\circ\text{C}$, measured in accordance with EN 22592.

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

NOTE To prevent adhesion of bituminous binder to the pouring plate when casting discs, the surface of the metal pouring plate should be thinly coated just before use with the release agent. Applying the release agent coating is easier when the plate is warmed to approximately $40 \text{ }^\circ\text{C}$.

6 Apparatus

6.1 Ring and ball apparatus, 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 $50 \text{ mm} \times 75 \text{ mm}$ and $1,5 \text{ mm}$ to $2,0 \text{ mm}$ thick with edges turned down (see figure 2).

6.1.3 Balls, two, $9,50 \text{ mm} \pm 0,05 \text{ mm}$ in diameter, each having a mass of $3,50 \text{ g} \pm 0,05 \text{ g}$.

6.1.4 Ball centring guides, brass, two for centring the steel balls, one for each ring.

NOTE As an example, figure 3 shows an apparatus of which general shape and dimensions have been found suitable.

6.1.5 Ring holder and assembly, brass 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 $25,0 \text{ mm} \pm 0,4 \text{ mm}$ above the upper surface of the bottom plate (B) (see figure 6) ; the upper edge of the rings shall be $50 \text{ mm} \pm 3 \text{ mm}$ below the surface of the bath liquid.

NOTE In a laboratory where many samples are tested, it may be found advantageous to use a circular ring holder carrying up to six test sample rings. The distance of the rings from the centre is not necessarily the same as in the standard two ring holder, but extended tests with such an instrument have shown that it gives results in sufficient practical agreement with those using the standard two ring instrument to warrant its use but for routine operations only.

6.1.6 Bath, glass beaker capable of being heated, not less than 85 mm inside diameter and not less than 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, 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 so as to provide the same readings as a mercury stem thermometer, recognising and allowing for of 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 a suitably coated stirring bar of length approximately 40 mm and diameter 8 mm to ensure uniform heat distribution throughout the bath. The stirrer shall be placed such that it does not disturb the samples when the test is in operation. The rotation speed of the stirring bar shall be approximately 100 min⁻¹.

NOTE The stirrer blade of the propeller stirrer should preferably be placed at a level between the bottom plate of the ring holder 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 dispute, the referee method shall be the manual one with the apparatus as shown in figure 5.

NOTE When using automatic apparatus, it is possible for the ball to trigger the interruption of a ray of light at a distance of $25,0 \text{ mm} \pm 0,4 \text{ mm}$ 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.

NOTE For modified bitumen the quantity of sample should be adjusted to fill four rings in case the test has to be repeated (see note to 8.6).

Heat the two brass rings, but not the pouring plate, to approximately $90 \text{ }^\circ\text{C}$ above the expected softening point and place them on the pouring plate treated with the release agent (see note to 5.3).

Pour a slight excess of the heated bituminous binder into each of the rings, and 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 $10 \text{ }^\circ\text{C}$ below the expected softening point. Not 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, cut away the excess bituminous binder cleanly 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 For routine tests, select the bath liquid and thermometer appropriate for the expected softening point, as follows.

a) For softening points between $30 \text{ }^\circ\text{C}$ and $80 \text{ }^\circ\text{C}$, use freshly boiled distilled or deionized water. Use a thermometer with subdivisions of $0,2 \text{ }^\circ\text{C}$. The initial bath temperature shall be $5 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$.

b) For softening points above $80 \text{ }^\circ\text{C}$ and up to $150 \text{ }^\circ\text{C}$, use glycerol. Use a thermometer with subdivisions of $0,5 \text{ }^\circ\text{C}$. The initial bath temperature shall be $30 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$.

For referee purposes, all softening points of $80 \text{ }^\circ\text{C}$ or less shall be determined in a water bath. Softening points above $80 \text{ }^\circ\text{C}$ up to $150 \text{ }^\circ\text{C}$ 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 $50 \text{ mm} \pm 3 \text{ mm}$ above the upper edge of the rings. Using forceps, place the two steel balls in the bath or in a separate container at $5 \text{ }^\circ\text{C}$. Ensure that the balls have the same temperature as the rest of the assembly.

8.3 Place the bath in ice water to cool to $5 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ (where water is the bath liquid), or gently heat to $30 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ (where glycerol is the bath liquid) to establish the correct initial bath temperature and maintain at this temperature for 15 min with the apparatus in place. Take care not to contaminate the bath liquid with any material which could affect the results.

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 note). 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 $\pm 0,5$ °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.

NOTE Rigid adherence to the specified heating rate is essential to 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. The electrical heater should be the low-lag, variable output type to maintain the prescribed rate of heating.

8.6 Record for each ring and ball the temperature indicated by the thermometer at 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) the difference between the two temperatures exceeds 2 °C;
- b) the 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.

8.7 Report the results as follows.

NOTE The softening point of a 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 a discontinuity leading to an ambiguity. For example, a bitumen having a softening point of 78 °C determined in water can be expected to give value of 82 °C if determined in glycerol. It is therefore highly recommended to define arbitrary protocols for tests giving results around 80 °C. Such protocols are defined in 8.7.1 and 8.7.4. In these clauses 'softening points results' refers to the mean or corrected mean of the two test temperatures determined according to 8.6.

8.7.1 Softening points results from 30 °C to 80 °C determined in a water bath. Report the result obtained.

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

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

8.7.4 Softening points 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 8.6 as the softening point.

For softening points above 80 °C, express to the nearest 0,5 °C the mean of the temperatures recorded in 8.6 as the softening point.