SLOVENSKI STANDARD
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Bitumen in bitumenska veziva - Določanje dinamične viskoznosti z metodo kapilare z vakuumom

Bitumen and bituminous binders - Determination of dynamic viscosity by vacuum capillary

Bitumen und bitumenhaltige Bindemittel - Bestimmung der dynamischen Viskosität mit Vakuum-Kapillaren

Bitumes et liants bitumineux - Détermination de la viscosité dynamique par viscosimètre capillaire sous vide

Ta slovenski standard je istoveten z: **prEN 12596**

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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Bitumen and bituminous binders - Determination of dynamic viscosity by vacuum capillary

Bitumes et liants bitumineux - Détermination de la viscosité dynamique par viscosimètre capillaire sous vide

Bitumen und bitumenhaltige Bindemittel - Bestimmung der dynamischen Viskosität mit Vakuum-Kapillaren

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

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Foreword

This document (prEN 12596:2013) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 12596:2007.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

1 Scope

This European Standard specifies a method for the determination of the dynamic viscosity of bituminous binders by means of a vacuum capillary viscometer at 60 °C in a range between 0,003 6 Pa · s to over 580 000 Pa · s. Bituminous emulsions are not within the scope of this method.

NOTE 1 Emulsions containing bituminous binders are not considered to be covered by this method. This method can be used for anhydrous binders obtained from emulsions (stabilised and/or recovered binders).

NOTE 2 The viscosity behaviour of some polymer modified bitumens (PMB) is not demonstrated in a vacuum capillary viscometer. Other methods are more relevant.

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 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 following terms and definitions apply.

3.1
dynamic viscosity
ratio between the applied shear stress and the velocity gradient

Note 1 to entry: Dynamic viscosity is a measure of the resistance to the flow of a liquid and is commonly called the viscosity of the liquid. For the purposes of this European Standard, the word viscosity means the dynamic viscosity of a liquid.

Note 2 to entry: The SI unit of dynamic viscosity is Pa · s.

3.2
Newtonian liquid
liquid with a viscosity that is independent of the rate of shear

Note 1 to entry: The constant ratio of the shear stress to the velocity gradient is the dynamic viscosity of the liquid. If this ratio is not constant, the liquid is non-Newtonian.

3.3
density
mass of a liquid divided by its volume

Note 1 to entry: When reporting density, the unit of density used, together with the temperature, is stated explicitly, for example kilogram per cubic metre.

Note 2 to entry: The SI unit of density is kg/m³.

3.4
kinematic viscosity
ratio between the dynamic viscosity and the density of a liquid at the temperature of viscosity measured

Note 1 to entry: Kinematic viscosity is a measure of the resistance to flow of a liquid under gravity.

Note 2 to entry: The SI unit of kinematic viscosity is m²/s; for practical use, a sub-multiple (mm²/s) is more convenient.

4 Principle

To determine the time for a fixed volume of the liquid to be drawn up through a capillary tube by means of a vacuum, under closely controlled conditions of vacuum and temperature. The viscosity is calculated by multiplying the flow time in s by the viscometer calibration factor.

5 Apparatus

5.1 Viscometer, capillary-type and made of borosilicate glass as described in 5.1.2 to 5.1.4.

5.1.1 General

Calibrated viscometers are available from commercial suppliers. Details regarding the calibration of viscometers are given in Annex C.

5.1.2 Cannon-Manning vacuum capillary viscometer (CMVV)

The CMVV is available in eleven sizes (see Table A.1), covering a range between 0,003 6 Pa · s to 8 000 Pa · s.

Details of the design and construction of CMVV are shown in Figure A.1. The size numbers, approximate calibration factors, K, and viscosity ranges for the series of CMVV are given in Table A.1.

For all viscometer sizes, the volume of measuring bulb C is approximately three times that of bulb B. Bulb B, bulb C and bulb D are defined by timing marks F, G and H.

5.1.3 Asphalt Institute vacuum capillary viscometer (AIVV)

The AIVV is available in seven sizes (see Table A.2) from a range between 4,2 Pa · s to 580 000 Pa · s. Sizes 50 to 200 are best suited to viscosity measurements of bituminous binders at 60 °C.

Details of design and construction of the AIVV are shown in Figure A.2. The size numbers, approximate capillary radii, approximate calibration factors, K, and viscosity range for the series of AIVV are given in Table A.2.

This viscometer has measuring bulb, B, bulb C and bulb D, located on the viscometer arm, M, which is a precision bore glass capillary. The measuring bulbs are 20 mm long capillary segments defined by timing marks F, G, H and I.

5.1.4 Modified Koppers vacuum capillary viscometer (MKVV)

The MKVV is available in five sizes (see Table A.3) covering a range between 4,2 Pa·s to 20 000 Pa·s. Sizes 50 to 200 are best suited to viscosity measurements of bituminous binders at 60 °C.

Details of design and construction of the MKVV are shown in Figure A.3. The size numbers, approximate capillary radii, approximate calibration factors, K, and viscosity ranges for the series of MKVV are given in Table A.3.

This viscometer consists of a separate filling tube, A, and precision-bore glass capillary vacuum tube, M. These two parts are joined by a borosilicate ground glass joint, N, with a 24/40 standard taper. Measuring bulb B, bulb C and bulb D, on the glass capillary are 20 mm long capillary segments, defined by timing marks F, G, H and I.

5.1.5 Holder, made by drilling two holes, 22 mm and 8 mm internal diameter, through a No. 11 rubber stopper. The centre-to-centre distance between holes shall be 25 mm. Slit the rubber stopper between the holes and between the 8 mm hole and edge of the stopper. When placed in a 51 mm diameter hole in the bath cover, the stopper shall hold the viscometer in place. For the MKVV the viscometer holder can be made by drilling a 28 mm hole through the centre of a No. 11 rubber stopper and slitting the stopper between the hole and the edge.

Such holders are commercially available.

5.2 Temperature measuring device

A temperature measuring device (combining sensor and reading unit) shall

— have a range from at least 55 °C to 65 °C,

— be readable to 0,05 °C or less,

— have an accuracy of 0,2 °C.

Sensors based on platinum resistance thermometers 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 B). 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 B) is also allowed if national regulations permit its use.

The specified thermometers shall be standardised at total immersion; that is immersion to the top of the mercury column with the reminder of the stem and the expansion chamber at the top exposed to room temperature. The practise of completely submerging the thermometer is not recommended. When thermometers are completely submerged, corrections for each individual thermometer based on calibration under conditions of complete submergence are determined and applied. If the thermometer is completely submerged in the bath during use, the pressure of the gas in the expansion chamber will be higher or lower than during standardization, and can cause a high or low reading on the thermometer. It is essential that liquid-in-glass thermometers are calibrated periodically and those official corrections be adjusted as necessary to conform to any changes in temperature readings. The thermometer shall be read, estimating the reading to 0,1 °C. Thermometers should be checked at regular intervals. A commonly used procedure given in method ASTM E 77 [2] applies a correction that is based on changes in the ice point calibration.

When measuring and controlling nominally constant temperatures, as in this test method, the thermal response time can be rather high (e.g. slow response to a change in temperature). Care shall be taken to consider this aspect since low thermal response time of the sensor can indicate greater cyclic variations than the bituminous material in practise experiences.

5.3 **Bath**, suitable for immersion of the viscometer so that the liquid reservoir or the top of the capillary whichever is uppermost, is at least 20 mm below the top of the bath level, and with provisions for visibility of the viscometer and the thermometer. Firm supports for the viscometer shall be provided, or the viscometer shall be an integral part of the bath. The efficiency of the stirring and the balance between heat losses and heat input shall be such that the temperature of the bath medium does not vary by more than 0,5 °C over the length of the viscometer, or from viscometer to viscometer in the various bath positions.

5.4 **Vacuum system**, capable of maintaining a vacuum with a reading accuracy of ± 100 Pa of the desired level up to and including 40 000 Pa. A vacuum or aspirator pump is suitable for the vacuum source.

5.5 **Timer**, or stop watch (spring or battery driven) graduated in divisions of 0,1 s or less and accurate to 0,5 s over 1 000 s when tested over intervals of not less than 15 min.

5.6 **Electrical timing devices**, used only on electrical circuits the frequencies of which are controlled to an accuracy of 0,5 s over 1 000 s.

NOTE Alternating currents, the frequencies of which are intermittently and not continuously controlled, as provided by some public power systems, can cause large errors, particularly over short timing intervals, when used to actuate electrical timing devices.

5.7 **Oven**, capable of maintaining (135,0 ± 5,0) °C.

6 Preparation of test samples

The laboratory sample shall be taken in accordance with EN 58. Prepare the test sample in accordance with EN 12594.

Heat the sample with care to prevent local overheating until it has become sufficiently fluid to pour, if possible, stir the sample occasionally to aid heat transfer and to ensure uniformity.

If the sample contains air bubbles, transfer a minimum of 20 ml into a suitable container and heat to (135 ± 5) °C, stirring occasionally to prevent local overheating and taking care to avoid the entrapment of air.

7 Procedure

7.1 Maintain the bath (5.3) at (60,0 ± 0,3) °C. Apply the necessary corrections, if any, to all thermometer readings.

7.2 Select a clean, dry viscometer that will give a flow time greater than 60 s, and preheat to 60 °C. If the sample contains air bubbles, preheat the viscometer to (135,0 ± 5,0) °C.

7.3 Charge the viscometer by pouring the prepared sample to within ± 2 mm of fill line E (Figure A.1, Figure A.2 and Figure A.3).

Carry out the test within 4 h of pouring.

7.4 If the sample contains air bubbles, place the charged viscometer in an oven or bath maintained at (135,0 ± 5,0) °C for a period of 10 min, to allow large air bubbles to escape.

7.5 Remove the viscometer from the oven or bath at (135,0 ± 5,0) °C and within 5 min, insert the viscometer into the holder (5.1.5) and position the viscometer vertically in the bath (5.3) so that the upper most timing mark is at least 20 mm below the surface of the bath liquid.

7.6 Establish a (40 000 ± 100) Pa vacuum below atmospheric pressure in the vacuum system and connect the vacuum system to the viscometer with the toggle valve or stopcock closed in the line leading to the viscometer.

7.7 After the viscometer has been in the bath for at least 30 min, start the flow of binder in the viscometer by opening the toggle valve or stopcock in the line leading to the vacuum system.

7.8 Read to within 0,1 s, the time required for the leading edge of the meniscus to pass between all successive pairs of timing marks. Report flow times between 60 s and 1 000 s, noting the identification of the pair of timing marks.

7.9 Upon completing the test, clean the viscometer thoroughly by rinsing several times with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. Dry the tube by passing a slow steam of filtered dry air through the capillary for 2 min, or until the last trace of solvent is removed. Periodically clean the instrument with a suitable non-caustic cleaning solution to remove organic deposits, rinse thoroughly with water, conforming to grade 3 of EN ISO 3696:1995, and residue-free acetone and dry with filtered dry air.

Use of alkaline glass cleaning solutions can result in a change of viscometer calibration, and is not recommended. Other cleaning methods (like pyrolisis) may be appropriate. It is recommended to check the viscometer calibration frequently to note any changes as soon as possible.

8 Calculation

Calculate the viscosity, η, in Pa · s, selecting the calibration factor that corresponds to the set of timing marks used for the determination, as prescribed in 7.8, using Formula (1):

$$\eta = K \times t \tag{1}$$

where

K is the selected calibration factor, in Pascal;

t is the flow time, in seconds.

NOTE If the calibration factor is given in poise, it can be converted to Pascal by multiplying by 0,1.

9 Expression of results

Express the viscosity as the mean value of the viscosities calculated from the readings for all bulbs used, to three significant figures below 1 000 Pa · s or as a whole number above 1 000 Pa · s, together with the test temperature.

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 6 % of the mean in only one case in twenty.

10.2 Reproducibility

The difference between two single and independent test 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 12 % of the mean for $\eta \geq 2\,000$ Pa · s and 10 % of the mean for $\eta < 2\,000$ Pa · s in only one case in twenty.

11 Test report

The test report shall contain at least the following information:

a) type and complete identification of the sample under test;

b) reference to this European Standard;

c) apparatus used;

d) result of the test (see Clause 9);

e) any deviation, by agreement or otherwise, from the procedure specified;

f) date of the test.