SLOVENSKI STANDARD
SIST EN 12596:2000

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Bitumen in bitumenska veziva - Določ evanje dinamič ne 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 viscosimetre capillaire sous vide

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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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EUROPEAN STANDARD

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English version

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

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of an identical text or by endorsement, at the latest by May 2000, and conflicting national standards shall be withdrawn at the latest by May 2000.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

This draft European Standard is based on ASTM D 2171-94.

This standard includes 3 annexes where annexes A and B are normative and annex C is informative.

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 the range from 0,0036 Pa·s to over 580 000 Pa·s.

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

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 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 following definitions apply :

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

NOTE 1 Dynamic viscosity is a measure of the resistance to flow of a liquid under gravity, and is commonly called the viscosity of the liquid. For the purposes of this standard, the word viscosity means the dynamic viscosity of a liquid.

NOTE 2 The SI unit of dynamic viscosity is Pa·s.

3.2
newtonian liquid
liquid having a viscosity that is independent of the rate of shear.

NOTE The constant ratio of the shear stress to the velocity gradient is the viscosity of the liquid. If the ratio is not constant, the liquid is non-newtonian.

4 Principle

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

5 Apparatus

5.1 Viscometer, capillary-type, made of borosilicate glass, as described in 5.1.1 to 5.1.3.

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

NOTE Tables A.1, A.2, A.3 and figures A.1, A.2, A.3 are given in annex A.

5.1.1 Cannon Manning vacuum capillary viscometer (CMVV).

- The CMVV is available in eleven sizes (table A.1), covering a range from 0,0036 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 bulb 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 the bulb B. Bulbs B, C and D are separated by timing marks F, G and H.

5.1.2 Asphalt Institute vacuum capillary viscometer (AIVV)

- The AIVV is available in seven sizes (table A.2) from a range from 4,2 Pa·s to 580 000 Pa·s. Sizes 50 through 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 bulb factors, *K*, and viscosity range for the series of AIVV are given in table A.2.

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

5.1.3 Modified Koppers vacuum capillary viscometer (MKVV)

- The MKVV is available in five sizes (table A.3) covering a range from 4,2 Pa·s to 20 000 Pa·s. Sizes 50 through 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 bulb 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, having a 24/40 standard taper. The measuring bulbs B, C and D, on the glass capillary are 20 mm long capillary segments, separated by timing marks F, G, H and I.

5.1.4 Holder, made by drilling two holes, 22 mm and 8 mm internal diameter, respectively, through a No. 11 rubber stopper. The center-to-center distance between holes shall be 25 mm. Make a slit of 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 n°11 rubber stopper and slitting the stopper between the hole and the edge.

Such holders are commercially available.

5.2 Thermometers, calibrated liquid-in-glass, of an accuracy of 0,2 °C, or any other thermometric device of equal accuracy.

5.2.1 The specified thermometers shall be standardized at total immersion, that is immersion to the top of the mercury column with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature.

NOTE The practice 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.

5.2.2 It is essential that liquid-in-glass thermometers be recalibrated periodically and that 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.

NOTE 1 Thermometers should be checked at regular intervals.

NOTE 2 A commonly used procedure given in Method ASTM E 77 applies a correction which is based on changes in the ice point calibration.

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

NOTE 3 When measuring and controlling nominally constant temperatures, as in this test method, alternative devices can indicate greater cyclic variations than mercury thermometers, to an extent depending on the cycle time of heating and the power of the controlled heat input.

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 upper 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 input shall be such that the temperature of the bath medium does not vary by more than 0,3 °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 ± 67 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 1000 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 1000 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.

6 Sampling

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

Transfer a minimum of 20 ml into a suitable container and heat to 135,0 °C ± 5,5 °C, stirring occasionally to prevent local overheating and taking care to avoid the entrapment of air.

Carry out the test within 4 h from pouring.

7 Procedure

7.1 Maintain the bath (5.3) at the test temperature to within ± 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 135,0 °C ± 5,5 °C.

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

7.4 Place the charged viscometer in an oven or bath maintained at 135,0 °C ± 5,5 °C for a period of 10 min ± 2 min, to allow large air bubbles to escape.

7.5 Remove the viscometer from the oven or bath at 60,0 °C ± 0,3°C and within 5 min, insert the viscometer into the holder (5.1.4) 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 Pa ± 67 Pa vacuum 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 ± 5 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 completion of the test, clean the viscometer thoroughly by several rinsings 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, and residue-free acetone and dry with filtered dry air.

NOTE Use of alkaline glass cleaning solutions can result in a change of viscometer calibration, and is not recommended.

8 Calculation

Calculate the viscosity, η, in pascal-seconds, selecting the calibration factor that corresponds to the set of timing marks used for the determination, as prescribed in 7.8, using the following equation :

$$\eta = K \times t$$

where :

K is the selected calibration factor, in pascals;

t is the flow time, in seconds.

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

9 Expression of results

Express the viscosity as the mean value of the two readings to three significant figures below 1 000 Pa·s or as the 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 $\eta > 2\,000$ Pa·s and 10 % of the mean for $\eta < 2\,000$ Pa·s in only one case in twenty.