SLOVENSKI STANDARD
oSIST prEN 12595:2013

01-april-2013

Bitumen in bitumenska veziva - Določanje kinematične viskoznosti

Bitumen and bituminous binders - Determination of kinematic viscosity

Bitumen und bitumenhaltige Bindemittel - Bestimmung der kinematischen Viskosität

Bitumes et liants bitumineux - Détermination de la viscosité cinématique

Ta slovenski standard je istoveten z: prEN 12595

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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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2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

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ICS 75.140; 91.100.50

Will supersede EN 12595:2007

English Version

Bitumen and bituminous binders - Determination of kinematic viscosity

Bitumes et liants bitumineux - Détermination de la viscosité cinématique

Bitumen und bitumenhaltige Bindemittel - Bestimmung der kinematischen Viskosität

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 336.

If this draft becomes a European Standard, 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.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (prEN 12595:2013) 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 12595:2007.

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).

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1 Scope

This European Standard specifies a method for the determination of the kinematic viscosity of bituminous binders at 60 °C and 135 °C, in a range from 6 mm²/s to 300 000 mm²/s. Other temperatures are possible if calibration constants are known. Bituminous emulsions are not covered within the scope of this method.

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

Results for this method can be used to calculate dynamic viscosity when the density of the test material is known or can be determined.

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.

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

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

EN ISO 2592, *Determination of flash and fire points — Cleveland open cup method (ISO 2592)*

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

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3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
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 a liquid's resistance to flow 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.

3.2
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 explicitly stated, for example kilogram per cubic metre.

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

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3.3
dynamic viscosity
ratio between the applied shear stress and the velocity gradient

Note 1 to entry: Dynamic viscosity is a measure of a liquid's resistance to flow, and is commonly called the viscosity of the liquid.

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

3.4
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.

4 Principle

The time for a fixed volume of the liquid to flow through the capillary of a calibrated glass capillary viscometer under an accurately reproducible head and at a closely controlled temperature is determined (efflux time). The kinematic viscosity is calculated by multiplying the efflux time in seconds by the viscometer calibration factor.

5 Apparatus

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Usual laboratory apparatus and glassware, together with the following:

5.1 Viscometer, CANNON-FENSKE, BS/IP/RF and the Zeitfuchs Cross-Arm viscometers, capillary-type, made of borosilicate glass, suitable for this method are described in Figure A.1, Figure A.2 and Figure A.3, and Table A.1, Table A.2 and Table A.3. Other viscometers are allowed if test results obtained are comparable.

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

5.2 Temperature measuring device

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

— have a range from at least 55 °C to 140 °C,

— be readable to 0,05 °C or less and,

— have an accuracy of 0,1 °C or better.

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 remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature. 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

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completely submerged in the bath during use, the pressure of the gas in the expansion chamber will be higher or lower than during standardisation, and can cause high or low reading on the thermometer. It is essential that liquid-in-glass thermometers are recalibrated 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 times 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 0,3 °C (measurement at 60 °C) or 0,5 °C (measurement at 135 °C) over the length of the viscometer, or from viscometer to viscometer in the various bath positions.

Water, conforming to the grade 3 of EN ISO 3696:1995, is a suitable bath liquid for determinations at 60 °C. USP white oil or any paraffinic or silicone oil with a flash point above 215 °C has been found suitable for determination at 135 °C. The flash point is determined in accordance with EN ISO 2592.

5.4 Timer, 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.

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5.5 Electrical timing devices, for use only on electrical circuits the frequencies of which are accurate to 0,5 s over 1 000 s or better.

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.

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5.6 Automatic or semi-automatic equipment, are allowed providing that they meet the specifications for temperature regulation and time accuracy described in Clause 5 and have been shown to achieve the same precision as given in Clause 10 and are fully calibrated.

6 Preparation of test samples

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

Bring the viscometer and the sample to within ± 30 °C of the test temperature (in order to avoid correction of constants of the viscometer). Stir the sample thoroughly without entrapment of air. If the temperature has dropped to 30 °C or more below the test temperature, reheat the sample.

Immediately charge the viscometer, or, if the test is to be made at a later time, pour approximately 20 ml into one or more 30 ml clean, dry, containers having an approximate volume of 30 ml and immediately seal with an airtight closure.

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7 Procedure

7.1 Test conditions

Maintain the bath (5.3) at the test temperature to within ± 0,3 °C (measurements at 60 °C) or ± 0,5 °C (measurements at 135 °C). Apply the necessary corrections, if any, to all thermometer readings.

Select a clean, dry viscometer giving an efflux time greater than 60 s and preheat it to the test temperature.

Charge the viscometer in the manner dictated by the design of the instrument, as prescribed in the following.

To charge the CANNON-FENSKE opaque viscometer, invert the viscometer and apply vacuum to the tube L, immersing tube N in the liquid sample. Draw liquid through tube N, filling bulb D to fill mark G. Wipe excess sample off tube N and invert the viscometer to its normal position. Mount the viscometer in the constant-temperature bath keeping tube L vertical.

Apply a stopper to the top of the apertures of tube L when bulb A is nearly 4/5th filled.

Mount the BS/IP/RF viscometer in the constant temperature bath keeping tube L vertical. Pour sample through tube N to a point just above filling mark G; allow the sample to flow freely through capillary R, taking care that the liquid column remains unbroken, until the lower meniscus is about 5 mm below the filling mark H and then arrest its flow by closing the timing tube with a cork or rubber stopper in tube L.

Add more liquid if necessary to bring the upper meniscus slightly above mark G.

After allowing the sample to attain bath temperature and any air bubbles to rise the surface, gently loosen the stopper allowing the sample to flow to the lower filling mark H and again arrest flow. Remove the excess sample above filling mark G by inserting the special pipette until its cork rests on top of tube N; apply gentle suction until air is drawn through. The upper meniscus shall coincide with mark G.

Mount the Zeitfuchs Cross-Arm viscometer in the constant temperature bath, keeping tube N vertical. Introduce sample through tube N taking care not to wet the sides of tube N, into the cross-arm D until the leading edge stands within 0,5 mm of fill mark G on the siphon tube.

7.2 Determination and measurement

Allow the viscometer to remain in the constant-temperature bath for sufficient time to ensure that the sample reaches temperature equilibrium. The test shall be performed within 4 h.

NOTE Temperature equilibrium is not normally achieved for at least 30 min.

For the Cannon-Fenske and BS/IP/RF viscometers, remove the stopper in tube L and allow the sample to flow by gravity until the lower meniscus is opposite the lower timing mark E.

For the Zeitfuchs Cross-Arm viscometer, apply slight vacuum to tube M-6 (or pressure to tube N-15, see Figure A.3) to cause the meniscus to move over the siphon tube and about 30 mm below the level of tube D in capillary R. Gravity flow is thus initiated.

Measure to the nearest 0,1 s the time required for the leading edge of the meniscus to pass from timing mark E to timing mark F and from F to I (Cannon-Fenske). If this efflux time is less than 60 s, select a viscometer of smaller capillary diameter and repeat the operation.

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 stream 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 cleaner 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.

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Using alkaline glass cleaning solutions can result in a change of viscometer calibration and is not recommended. Other cleaning methods (like pyrolisis) may be appropriate. In this case, it is recommended to verify the viscometer frequently to note changes as soon as possible.

8 Calculation

Calculate the kinematic viscosity, ν, in millimetres squared per second, using results from different timing marks, using Formula (1):

$$\nu = C \times t \tag{1}$$

where

C is the calibration constant of the viscometer, in millimetres squared per square second;

t is the efflux time, in seconds.

9 Expression of results

Together with the test temperature, express the kinematic viscosity as the mean value, to three significant figures below 1 000 mm²/s or as the whole number above this value.

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10 Precision

10.1 Repeatability

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The difference between two successive 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 value given in Table 1 in only one case in twenty.

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 twenty.

Table 1 — Precision values

	Repeatability, *r* % of mean	**Reproducibility, *R*** % of mean
at 135 °C		
< 600 mm²/s	4	6
≥ 600 mm²/s	4	9
at 60 °C		
soft bitumen	7	9
soft bitumen after hardening (TFOT)	9	20
kinematic viscosity (KV) ratio at 60 °C (only for KV ratio < 1,5)	6	16