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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: **EN 12595:1999**

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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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**en**

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ICS 75.140; 91.100.50

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 European Standard was approved by CEN on 19 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.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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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 2170-95.

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

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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 the range from 6 mm<sup>2</sup>/s to 300 000 mm<sup>2</sup>/s.

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

**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*

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 following 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 Kinematic viscosity is a measure of the resistance to flow of a liquid under gravity.

NOTE 2 The SI unit of kinematic viscosity is m<sup>2</sup>/s; for practical use, a submultiple (mm<sup>2</sup>/s) is more convenient.

### 3.2

#### density

mass of a liquid divided by its volume.

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NOTE When reporting density, the unit of density used, together with the temperature, is stated explicitly, for example kilogram per cubic metre.

### 3.3

#### 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.

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

### 3.4 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 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. The kinematic viscosity is calculated by multiplying the efflux time in seconds by the viscometer calibration factor.

## 5 Apparatus

Usual laboratory apparatus and glassware, together with the following :

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

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

**5.2 Thermometers**, calibrated liquid-in-glass, of an accuracy of 0,2 °C can be used or another thermometric device of equal accuracy as described in Annex B.

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.

**NOTE 1** 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 standardisation, and can cause high or low reading on the thermometer.

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 2** Thermometers should be checked at regular time intervals.

**NOTE 3** A commonly used procedure given in Method ASTM E 77 applies 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 4** 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 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, 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 22592.

**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 1000 s when tested over intervals of not less than 15 min.

**5.5 Electrical timing devices**, for use only on electrical circuits the frequencies of which are accurate to 0,5 s over 1000 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.

**5.6 Automatic or semi-automatic equipment**, are allowed providing that they meet the specifications for temperature regulation and time accuracy described in this clause 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  $\pm 30$  °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 clean, dry, containers having a capacity of approximately 30 ml and immediately seal with an airtight closure.

## 7 Procedure

### 7.1 Test conditions

Maintain the bath (5.3) at the test temperature to within  $\pm 0,3$  °C (measurements at 60 °C) or  $\pm 0,5$  °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 4/5 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 pipet until its cork rests on top of tube N; apply gently suction until air is drawn through. The upper meniscus shall coincide with mark G.

Mount the Zeiffuchs 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. Test shall be performed within 4 h.

NOTE 1 Temperature equilibrium is not normally achieved for at least 30 min.

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.

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, and residue-free acetone and dry with filtered dry air.

NOTE 2 Use of alkaline glass cleaning solutions can result in a change of viscometer calibration, and is not recommended.

## 8 Calculation

Calculate the kinematic viscosity,  $\nu$ , in millimetres squared per second, using results from different timing marks, using the following equation :

$$\nu = C \times t$$

where :

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

$t$  is the efflux time, in seconds.

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## 9 Expression of results

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Express the kinematic viscosity as the mean value, to three significant figures below 1000 mm<sup>2</sup>/s or as the whole number above this value, together with the test temperature.

## 10 Precision

### 10.1 Repeatability

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 value given in table 1 in only one case in twenty.

**Table 1 - Precision values**

	Repeatability <i>r</i> % of mean	Reproducibility <i>R</i> % of mean
at 135 °C		
< 600 mm <sup>2</sup> /s	4	6
≥ 600 mm <sup>2</sup> /s	4	9
at 60 °C		
soft bitumen	7	9
soft bitumen after hardening (TFOT)	9	20
ratio of kinematic viscosity at 60 °C (KV - ratio < 1,5)	6	16

NOTE These precision data are not automatically applicable to modified bitumen and for modified bitumen they should only be used for guidance, until criteria data are available.

## 11 Test report

The test report shall contain at least the following information :

- a) the type and full identification of the sample under test;
- b) a reference to this European Standard;
- c) the apparatus used;
- d) the result of the test (see clause 9);
- e) any deviation, by agreement or otherwise, from the procedure specified;
- f) the date of the test.

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