EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 12596

December 2021

ICS 75.140; 91.100.50

Will supersede EN 12596:2014

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

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

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Ref. No. prEN 12596:2021 E

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European foreword

This document (prEN 12596:2021) 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 12596:2014.

In comparison with the previous edition, the following technical modifications have been made:

— amended scope (clarification of applicability of test methods); deletion of notes from scope;

— removal of dated references;

— reference to mercury thermometer has been deleted (5.2);

— reference to total immersion thermometer has been deleted (5.2);

— time to reach thermal equilibrium has been prolonged to 1 hour;

— “bulb” changed into “tube section” in 5.1.2, 5.1.3, Figures A.2 and A.3, and added “tube section” to Clause 9 (consistency of wording);

— information on validity of individual test data to calculate mean value added in Clause 8;

— appropriate range of flow time readings added in Clause 9;

— Annex C has been deleted;

— new Annex C introduced with examples on calculation;

— ASTM E77-98 deleted from the Bibliography.

1 Scope

This document 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 and 580 000 Pa·s. Other temperatures are possible if calibration constants are known. Bituminous emulsions and non-newtonian binders (e.g. some polymer modified bitumen) are not within the scope of this method.

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to identify the hazards and assess the risks involved in performing this test method and to implement sufficient control measures to protect individual operators (and the environment). This includes appropriate safety and health practices and determination of the applicability of regulatory limitations prior to use.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at https://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

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 document, 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/m^3.

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 Sl unit of kinematic viscosity is m^2/s; for practical use, a sub-multiple (mm^2/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 5.1.1 to 5.1.3.

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

5.1.1 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.

5.1.2 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 tube section B, tube section C and tube section D, located on the viscometer arm M, which is a precision bore glass capillary. The measuring bulbs/test sections are 20 mm long capillary segments defined by timing marks F, G, H and I.

5.1.3 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 tube section B, tube section C and tube section D, on the glass capillary are 20 mm long capillary segments, defined by timing marks F, G, H and I.

5.1.4 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 temperature measuring device shall be calibrated regularly.

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.