

BS EN 12595:2014
BS 2000-319:2014



BSI Standards Publication

Bitumen and bituminous binders — Determination of kinematic viscosity

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum Testing and Terminology.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Published by BSI Standards Limited 2014

ISBN 978 0 580 77119 4

ICS 75.140; 91.100.50

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2014.

BS 2000 Series

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Amendments/corrigenda issued since publication

Date	Text affected
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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 16 August 2014.

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CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 12595:2014) has been prepared by Technical Committee CEN/TC 336 "Bituminous binders", the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by May 2015 and conflicting national standards shall be withdrawn at the latest by May 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 12595:2007.

In comparison with EN 12595:2007, the following significant changes have been made:

- changed/added wording of the Warning in the Scope;
- EN 12607-2, *Bitumen and bituminous binders — Determination of the resistance to hardening under the influence of heat and air — Part 2: TFOT Method* was added as a normative reference in Clause 2;
- the reference to mercury thermometer has been deleted (see subclause 5.2) and Annex C is informative;
- alternative cleaning procedure added as optional in subclause 7.2.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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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 recovered and/or stabilized binders obtained from emulsions.

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 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, 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 12607-2, *Bitumen and bituminous binders - Determination of the resistance to hardening under the influence of heat and air - Part 2: TFOT Method*

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

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 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/m^3 .

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

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.

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

5.2 Temperature measuring device

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

- have a range from at least $55\text{ }^{\circ}\text{C}$ to $140\text{ }^{\circ}\text{C}$,
- be readable to $0,05\text{ }^{\circ}\text{C}$ or less, and
- have an accuracy of $0,1\text{ }^{\circ}\text{C}$ 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 C). 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 C) is also allowed if national regulations permit its use.

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. 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 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 E77 [4] 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.

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.

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 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 and, dry containers having an approximate volume of 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 4/5th 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 1 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.

NOTE 2 Time intervals between 60 s and 400 s are practical, but intervals can be extended up till 1 000 s.

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. An alternative cleaning

procedure is to put the capillary viscometer upside down in oven and allow the bitumen to drop out. After this use solvent. For both methods 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.

Using alkaline glass cleaning solutions can result in a change of viscometer calibration and is not recommended. Other cleaning methods (like pyrolysis) 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):

$$\nu = C \times t \quad (1)$$

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.

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 values 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 ² /s	4	6
≥ 600 mm ² /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
Kinematic Viscosity ratio at 60 °C is the ratio of kinematic viscosity at 60 °C of bituminous binder conditioned according to EN 12607-2 over the original unconditioned binder.		

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

Annex A (normative)

Specifications of viscometers

Dimensions in millimetres

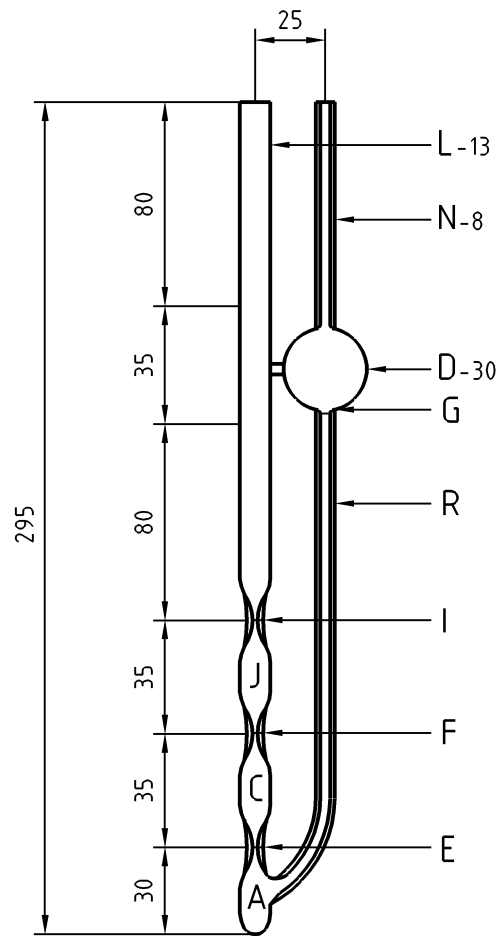


Figure A.1 — Cannon-Fenske opaque viscometer for opaque and transparent liquids

Table A.1 — Dimensions and kinematic viscosity ranges for the Cannon-Fenske viscometer

Size No.	Nominal viscometer constant	Kinematic viscosity range	Inside diameter of tube R, (± 2 %)	Inside diameter of tubes N and G, tubes E, F and I, (± 5 %)	Volume bulbs A, C, and J, (± 5 %)	Volume bulb D, (± 5 %)
	mm ² /s ²	mm ² /s	mm	mm	ml	ml
200	0,1	6 to 100	1,02	3,2	2,1	11
300	0,25	15 to 200	1,26	3,4	2,1	11
350	0,5	30 to 500	1,48	3,4	2,1	11
400	1,2	72 to 1 200	1,88	3,4	2,1	11
450	2,5	150 to 2 500	2,20	3,7	2,1	11
500	8	480 to 8 000	3,10	4,0	2,1	11
600	20	1 200 to 20 000	4,00	4,7	2,1	13

Dimensions in millimetres

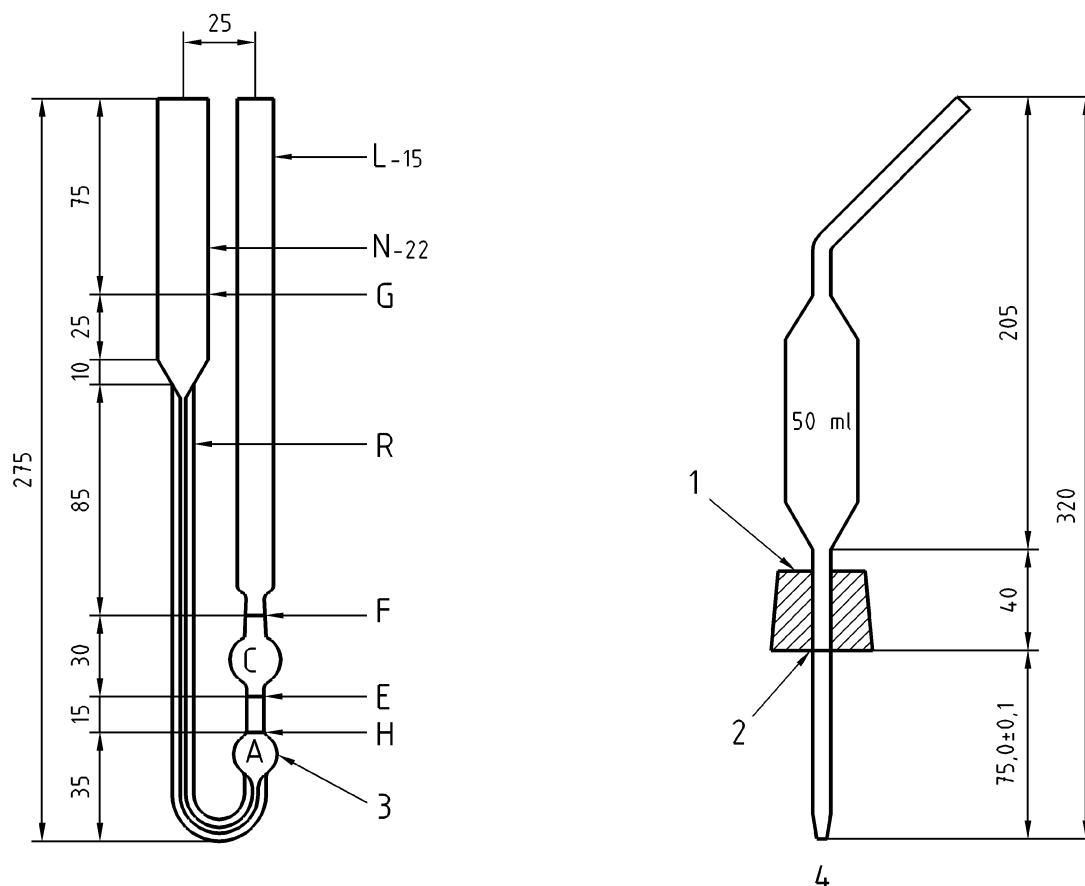


Figure A.2 — Example of BS/IP/RF U-tube reverse flow viscometer for opaque liquids

Table A.2 — Dimensions and kinematic viscosity ranges for the BS/IP/RF viscometer

Size No.	Nominal viscometer constant mm^2/s^2	Kinematic viscosity range mm^2/s	Inside diameter of tube R, ($\pm 2\%$) mm	Length of tube R mm	Inside diameter at E, F, and H mm	Volume bulb C, ($\pm 5\%$) ml
4	0,1	6 to 100	1,26	185	3,0 to 3,3	4,0
5	0,3	18 to 300	1,64	185	3,0 to 3,3	4,0
6	1,0	60 to 1 000	2,24	185	3,0 to 3,3	4,0
7	3,0	180 to 3 000	2,93	185	3,3 to 3,6	4,0
8	10	600 to 10 000	4,00	185	4,4 to 4,8	4,0
9	30	1 800 to 30 000	5,50	185	6,0 to 6,7	4,0
10	100	6 000 to 100 000	7,70	210	7,7	4,0
11	300	18 000 to 300 000	10,00	210	10,0	4,0

Dimensions in millimetres

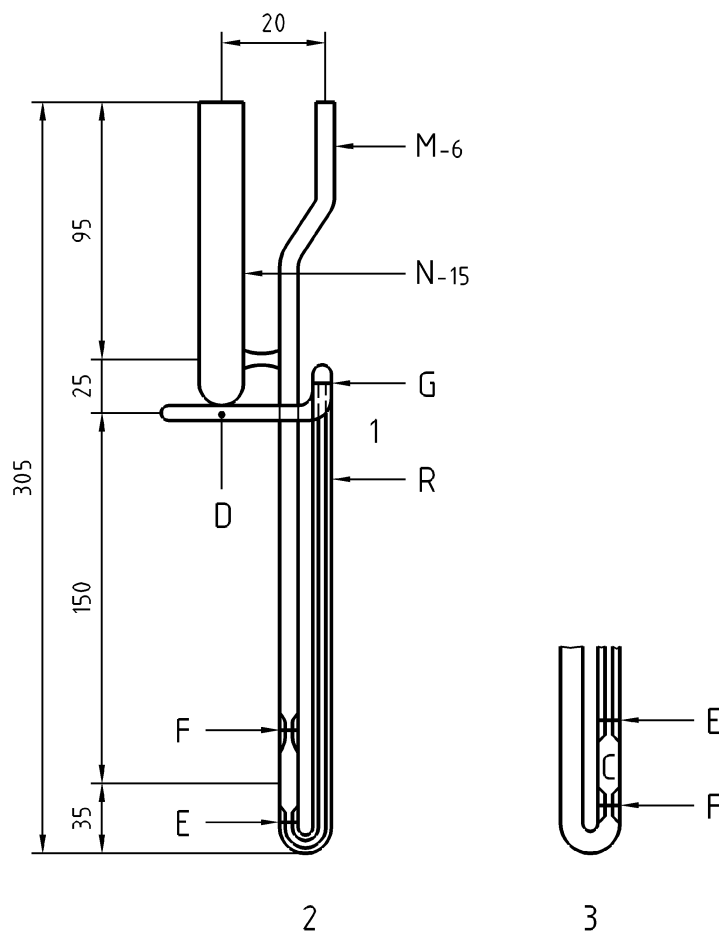


Figure A.3 — Zeifuchs cross-arm viscometer for transparent and opaque liquids

Table A.3 — Dimensions and kinematic viscosity ranges for the Zeifuchs cross-arm viscometer

Size	Nominal viscometer constant mm^2/s^2	Kinematic viscosity range mm^2/s	Inside diameter of tube R, ($\pm 2\%$) mm	Length of tube R from G to E mm	Lower bulb volume, ($\pm 5\%$) ml	Horizontal tube diameter, ($\pm 5\%$) mm
4	0,10	6 to 100	0,64	210	0,3	3,9
5	0,3	18 to 300	0,84	210	0,3	3,9
6	1,0	60 to 1 000	1,15	210	0,3	4,3
7	3,0	180 to 3 000	1,42	210	0,3	4,3
8	10,0	600 to 10 000	1,93	165	0,25	4,3
9	30,0	1 800 to 30 000	2,52	165	0,25	4,3
10	100,0	6 000 to 100 000	3,06	165	0,25	4,3

Annex B (informative)

Calibration of viscometers

B.1 General

Annex B describes the materials and procedures used for calibrating, or checking the calibration of viscometers used in this European Standard. Capillary viscometers shall preferably be calibrated at the intended operational temperature.

B.2 Reference materials

Viscosity oil standards conforming to ASTM viscosity oil standards having approximate kinematic viscosities as shown in Table B.1.

Certified kinematic viscosity values established are supplied with each sample.

Table B.1 — Viscosity oil standards

Viscosity oil standards^a	Approximate kinematic viscosity		
	mm²/s		
	At 37,8 °C	At 50 °C	At 99 °C
S 60	60
S 200	200
S 600	600	280	32
S 2 000	2 000
S 8 000	8 000
S 30 000	27 000	11 000	...

^a The viscosity standards cited in Table B.1 are cited according to the reference system of the following firm:

CANNON INSTRUMENTS Co
2139 High Tech Road
State College, PA 16803
USA

This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

B.3 Calibration

B.3.1 Calibration of routine viscometer by means of viscosity standards

B.3.1.1 Select from Table B.1 a liquid standard having a minimum efflux time of 200 s at the calibration temperature (preferably 37,8 °C). Determine the efflux time to the nearest 0,1 s using the procedure described in Clause 7 and calculate the viscometer constant, C , in square millimetres per square second, as follows:

$$C = \frac{\nu}{t} \quad (\text{B.1})$$

where

ν is the viscosity of standard liquid in square millimetres per second;

t is the efflux time, in seconds.

It is recommended to avoid the use of a silicone oil as a viscosity oil for calibration. It renders the viscometers unsuitable for further use.

NOTE The viscometer constant is independent of temperature for BS/IP/RF "U-tube viscometers".

B.3.1.2 The Cannon-Fenske opaque viscometer (see Figure A.1) has a fixed volume of sample charged at the fill temperature. If the test temperature differs from the fill temperature, the viscometer constant is calculated as follows:

$$C_T = C_0 \{1 + F(T_t - T_f)\} \quad (\text{B.2})$$

where

C_T is the viscometer constant at temperature T ;

C_0 is the viscometer constant when filled and tested at the same temperature;

F is the temperature dependence factor;

T_t is the test temperature, in degrees Celsius;

T_f is the fill temperature, in degrees Celsius.

B.3.1.3 Calculate the temperature dependence constant by means of Formula (B.3):

$$F = 4\alpha \left[\frac{V}{\pi d^2 h} \right] = \left[\frac{4V(p_f - p_t)}{\pi d^2 h p_t (T_t - T_f)} \right] \quad (\text{B.3})$$

where

V is the volume of charge, in cubic centimetres;

d is the average diameter of the meniscus in the upper reservoir, in centimetres;

h is the average driving head, in centimetres;

- α is the coefficient of thermal expansion of the test sample between the fill temperature and the test temperature;
- p_f is the density at the fill temperature, in grams per cubic centimetre;
- p_t is the density at the test temperature, in grams per cubic centimetre;
- T_t is the test temperature, in degrees Celsius;
- T_f is the fill temperature, in degrees Celsius.

B.3.1.4 If the viscometer is used at a location other than the calibration laboratory, the C constant should be corrected for the difference in the acceleration of gravity, g , at the two locations as follows:

$$C_2 = \left(\frac{g_2}{g_1} \right) C_1 \quad (\text{B.4})$$

where

- C_2 is the calibration constant in the testing laboratory;
- C_1 is the calibration constant in the calibrating laboratory;
- g_2 is the acceleration of gravity at the testing laboratory;
- g_1 is the acceleration of gravity at the calibrating laboratory.

Certificates for viscometers should state the value of g at the location of the calibrating laboratory. Failure to correct for gravity can result in errors of 0,2 %.

B.3.2 Calibration of routine viscometer by means of standard viscometer

Select any petroleum oil or bitumen having an efflux time of at least 200 s. Select also a standard viscometer of known C constant or a routine viscometer of the same type that has been calibrated by comparison with a reference viscometer. Calibrated viscometers are available from a number of commercial suppliers. Mount the standard viscometer together with the viscometer to be calibrated in the same bath and determine the efflux times of the oil by the procedure described.

Calculate the C constant as follows:

$$C_1 = \frac{(t_2 \times C_2)}{t_1} \quad (\text{B.5})$$

where

- C_1 is the C constant of routine viscometer;
- t_1 is the efflux time to nearest 0,1 s in routine viscometer;
- C_2 is the C constant of standard viscometer;
- t_2 is the efflux time to nearest 0,1 s in standard viscometer.

Annex C (informative)

Characteristics of thermometers

Table C.1 — Characteristics of thermometer

Characteristics	Units	Viscosity temperature	
		60 °C	135 °C
Temperature range	°C	58,6 to 61,4	133,6 to 136,4
Scale marks			
Subdivision	°C	0,05	0,05
Long lines at each	°C	0,1 and 0,5	0,1 and 0,5
Numbers at each	°C	1	1
Maximum scale error	°C	0,1 at 60	0,15 at 135
Immersion		total	total
Expansion chamber permits heating to	°C	105	170
Total length	mm	300 to 310	300 to 310
Stem outside diameter	mm	6,0 to 8,0	6,0 to 8,0
Bulb length	mm	45 to 55	45 to 55
Bulb outside diameter	mm	< stem	< stem
Scale location			
Between bottom of bulb to line at	°C	58,6	133,6
Distance	mm	145 to 165	160 to 180
Length of scale	mm	40 to 90	40 to 90
NOTE The following thermometers have been found suitable: ASTM 47C/IP 35C and ASTM 110C/IP93C respectively for viscosity temperatures of 60 °C and 135 °C.			

Bibliography

- [1] EN 13074-1, *Bitumen and bituminous binders - Recovery of binder from bituminous emulsion or cut-back or fluxed bituminous binders - Part 1: Recovery by evaporation*
- [2] EN 13074-2, *Bitumen and bituminous binders - Recovery of binder from bituminous emulsion or cut-back or fluxed bituminous binders - Part 2: Stabilisation after recovery by evaporation*
- [3] ASTM D2170-01a¹⁾, *Standard test method for kinematic viscosity of asphalts (bitumen)*
- [4] ASTM E77-98 (2003), *Test method for verification and calibration of thermometers*

1) This standard is a joint method with the Institute of Petroleum. It has the reference IP 319/84.

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