BS EN 12596:2023



Bitumen and bituminous binders — Determination of dynamic viscosity by vacuum capillary



National foreword

This British Standard is the UK implementation of EN 12596:2023 supersedes BS EN 12596:2014, which is withdrawn.

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

A list of organizations represented on his sommittee can be obtained on request to its committee manager

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This European Standard was approved by CEN on 28 May 2023.

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Cont	ents ean foreword	7
Europ	ean foreword	
1	Scope	
2	Normative references	
3	Terms and definitions	
4	Principle	
5	Apparatus	
6	Preparation of test samples Q. 1	
7	Procedure	
8	Calculation	
9	Expression of results	
10 10.1 10.2	Precision	
11	Test report10	
Annex	A (normative) Specifications of viscometers11	
Annex	B (informative) Calibration of viscometers17	
Annex	C (informative) Example for calculation of results	
Biblio	graphy	

European foreword

This document (EN 12596:2023) has been prepared by Technical Committee CEN/TC 336 "Bitum inous binders", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either publication of an identical text or by endorsement, at the latest by January 2024, and conflicting itional standards shall be withdrawn at the latest by January 2024.

Attention is drawn to the possibility that some of the this document may be the subject of patent rights. CEN shall not be held responsible fonid on fying any or all such patent rights. This document supersedes EN 12596 20 M

ut dition, the main technical changes are: In comparison with the pres

- amended scope (clarification of applicability of test methods); deletion of notes from scope;
- "accuracy" is changed to "maximum permissible error" in several clauses (5.2, 5.4, 5.5 and 5.6);
- reference to mercury thermometer deleted (5.2);
- reference to total immersion thermometer deleted (5.2);
- maximum permissible error for temperature of bath changed from 0,5 °C to 0,3 °C (5.3);
- the required precision of the oven reduced to (135 ± 5) °C in (5.7, 7.2, 7.4 and 7.5);
- time to reach thermal equilibrium 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);
- new subclause 5.8 added on Calibration/Verification;
- information on validity of individual test data to calculate mean value added in Clause 8; including a new NOTE 2 and renumbering existing notes respectively;
- appropriate range of flow time readings added in Clause 9;
- Table B.1 updated with informative values for viscosity standards;
- all time measurements with a maximum permissible error of 0,1 s in B.3.1;
- Annex C deleted;
- new Annex C introduced with examples on calculation;
- ASTM E77-98 deleted from the Bibliography;
- reference to ASTM D2171-01 in Bibliography updated and reference (footnote) to Institute of Petroleum deleted.

BS EN 12596:2023 EN 12596:2023 (E)

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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 58 ON Pa·s. Other temperatures are possible if calibration constants are known. Bituminous emulsions and nonnewtonian 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, operation ns and equipment. This document does not purport to address all of the safety problem calculated 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 to the appropriate safety and health practices and determination of the applicability of regulatory limitations prior to use
2 Normative references O

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 terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <u>https://www.iso.org/obp</u>
- IEC Electropedia: available at <u>https://www.electropedia.org/</u>

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 **Counted** explicitly, for example kg/m³. Note 2 to entry: The SI unit of density is kg/m³. **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 standard stand

Note 1 to entry: Kinematic viscosity is a measure of the resistance to flow of a liquid under gravity.

the viscosity is m^2/s ; for practical use, a sub-multiple (mm^2/s) is more Note 2 to entry: The SI unit of kin 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 seconds by the viscometer calibration factor.

5 **Apparatus**

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

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 aper. Measuring tube section B, tube section C and tube section D, on the glass capillary are born 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 grameter, through a No. 11 rubber stopper. The centre-to-centre distance between holes shall be 13 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 visconder in place. For the MKVV the viscometer holder can be made by drilling a 28 mm hole through the entre 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 a maximum permissible error 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 0,3 °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 maximum permissible error 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 a maximum permissible error of 0,5 s over 1 000 s when tested over intervals of not less than 15 min. Verify the maximum permissible error frequently.

5.6 Electrical timing devices, used only on electrical circuits the frequencies of which are controlled with a maximum permissible error of 0,5 s over 1 000 s. Verify the maximum permissible error frequently.

Alternating currents, the frequencies of which are intermittently and not continuously controlled, as NOTE provided by some public power systems, can cause large errors, particularly over short timing intervals, when used to actuate electrical timing devices.

5.8 Calibration/Verification, all equipment shall be calibrated/verified at least on the system.
5.8 Calibration/Verification, all equipment shall be calibrated/verified at least on the system.
6 Preparation of test samples
The laboratory sample shall be taken in accordance with EN 18 the pare the test sample in accordance with EN 12594.
Heat the sample with care to prevent local dreatesting until it has become sufficiently fluid to pour, if possible, stir the sample occasionally to and heat transfer and to ensure uniformity.
If the sample contains air bubbles transfer a minimum of 20 ml into a suitable of air of air.

Procedure 7

7.1 Maintain the bath (5.3) at (60,0 ± 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 below 1 000 s, and preheat to 60 °C. If the sample contains air bubbles, preheat the viscometer to (135 ± 5) °C.

7.3 Charge the viscometer by pouring the prepared sample to within $\pm 2 \text{ mm}$ of fill line E (Figure A.1, Figure A.2 and Figure A.3).

Carry out the test within 4 h of pouring.

7.4 If the sample contains air bubbles, place the charged viscometer in an oven or bath maintained at (135 ± 5) °C for a period of 10 min, to allow large air bubbles to escape.

7.5 Remove the viscometer from the oven or bath at (135 ± 5) °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.

Establish a $(40\ 000 \pm 100)$ Pa vacuum below atmospheric pressure in the vacuum system and 7.6 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 1 h to ensure that the sample reaches thermal equilibrium, 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 completing the test, clean the viscometer thoroughly by rinsing several times 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

(1)

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 3696:1995, and residue-free acetone and dry with filtered dry air.

Use of alkaline glass cleaning solutions can result in a change of viscometer calibration, is not recommended. Other cleaning methods (like pyrolysis) may be appropriate. It is recommended to check the viscometer calibration frequently to note any changes as soon as possible

recommended. Other cleaning methods (like pyrolysis) may be appropriate. It is the viscometer calibration frequently to note any changes as soon as possible **8 Calculation** Calculate the viscosity η , in Pa·s, selecting the calibration factor that correspondent marks used for the determination, as described in S, using Formula (1): $\eta = K \times t$ where tor that corresponds to the set of timing

- *K* is the selected calibration factor, in Pa;
- is the flow time, in s. t

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

For tests carried out with time readings from 60 s to 1 000 s, calculate the coefficient of variance CV, which is sample standard deviation divided by the mean value in %, see Formula (2):

$$CV = \frac{s}{\overline{x}} \times 100 \left[\%\right]$$
⁽²⁾

where

- is the sample standard deviation; S
- \overline{x} is the mean value.

NOTE 2 Formula (2) is applicable to the verification of the results obtained from the different bulbs or tube sections respectively of the same viscometer. Formula (2) can also be used to verify the individual results obtained from two viscometers if only one of the individual viscometer's bulbs/tube sections is within the time range.

For the individual bulbs the maximum acceptable range for measurements for two or more bulbs or tube sections in the same viscometer is the results having a coefficient of variance of 2 %.

NOTE 3 Larger coefficient of variance than 2 % can indicate shear susceptibility of the sample.

The maximum acceptable range for the mean values between two viscometers is the mean values having a coefficient of variance of 2 %.

If the appropriate maximum coefficient of variance is exceeded, discard the results and repeat the test from Clause 6 on a second sample test container.

If the appropriate range is exceeded again, report all individual results and the coefficient of variance under Clause 11 e).

NOTE 4 Annex C provides examples on the calculations of sample standard deviation and coefficient of variance.

Expression of results 9

Express the viscosity as the mean value of the viscosities calculated from the readings for all bulbs or tube

Express the viscosity as the mean value of the viscosities calculated from the readings for all bulbs or tube sections used, having flow time reading from 60 s to 1 000 s, to three significant figures below 1 000 Pass or as a whole number above 1 000 Pass, 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 plant in only one case in twenty.

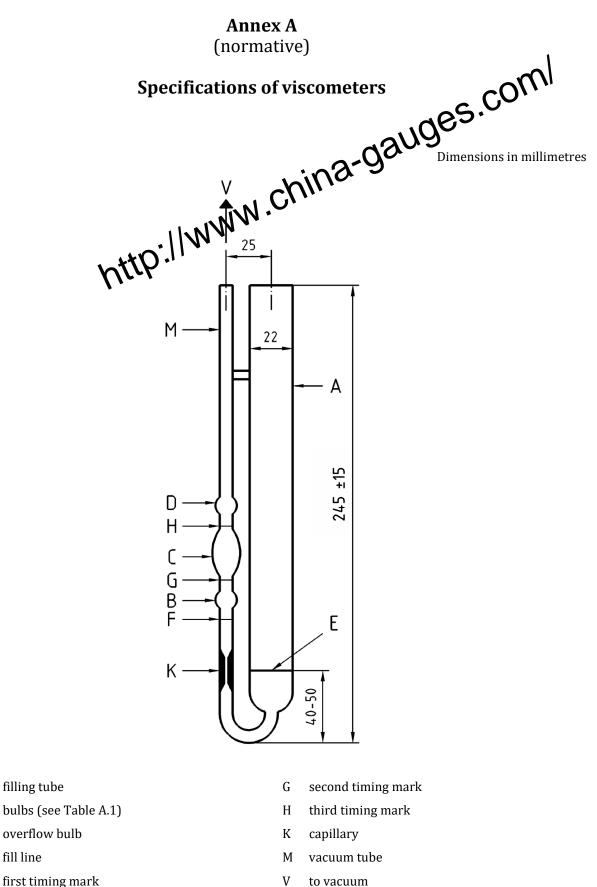
10.2 Reproducibility

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 $\eta \ge 2\,000$ Pa·s and 10 % of the mean for η < 2 000 Pa·s in only one case in twenty.

11 Test report

The test report shall contain at least the following information:

- type and complete identification of the sample under test; a)
- reference to this document including the year of publication; b)
- c) apparatus used;
- d) result of the test (see Clause 9);
- any deviation, by agreement or otherwise, from the procedure specified; e)
- any unusual features observed; f)
- g) date of the test.



F first timing mark

fill line

Key А

D

Е

B and C

Figure A.1 — Cannon-Manning vacuum capillary viscometer

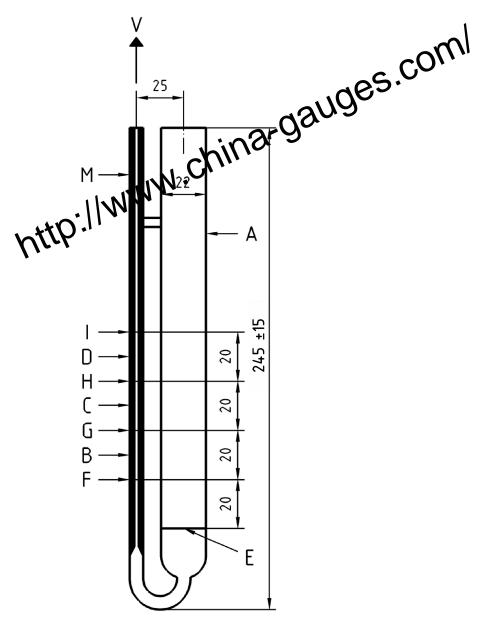
Table A.1 — Standard viscometer sizes, approximate calibration factors K, and viscosity rangesfor Cannon-Manning vacuum capillary viscometers

Viscometer size number	Approximate cal at 40 000	Viscosity range Viscosity range Viscosity range	
	Bulb B	Bulb C	Jana
4	0,000 2		0,003 6 to 0,08
5	0,000 6	0,000 2 0,000 6	0,012 to 0,24
6	^{0,002} IIN	0,000 6	0,036 to 0,8
7	400.	0,002	0,12 to 2,4
8	0,02	0,006	0,36 to 8
9	0,06	0,02	1,2 to 24
10	0,2	0,06	3,6 to 80
11	0,6	0,2	12 to 240
12	2,0	0,6	36 to 800
13	6,0	2,0	120 to 2 400
14	20,0	6,0	360 to 8 000

^a Exact calibration factors shall be determined with viscosity standards.

^b The viscosity ranges shown in this table correspond to a filling time of 60 s to 400 s. Longer flow times (up to 1 000 s) may be used.

Dimensions in millimetres



Кеу

А	filling tube
B, C and D	tube sections (see Table A.2)
Е	filling line
F	first timing mark
G	second timing mark
Н	third timing mark
Ι	fourth timing mark
М	vacuum tube
V	to vacuum

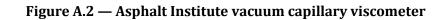


Table A.2 — Standard viscometer sizes, capillary radii, approximate calibration factors K, and viscosity ranges for Asphalt Institute vacuum capillary viscometers

1

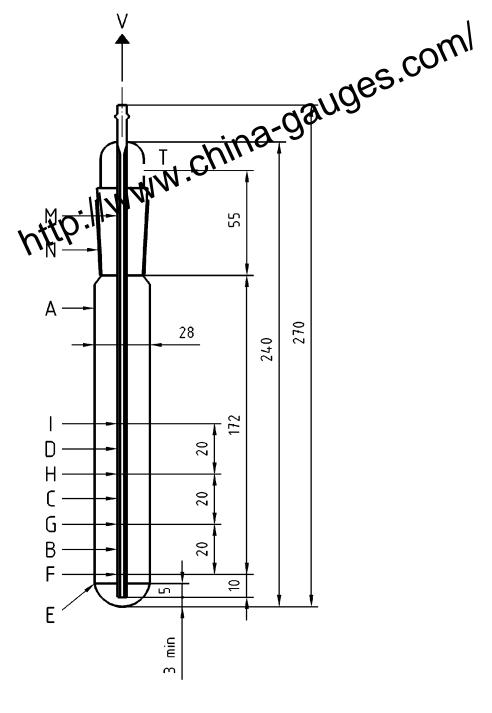
Viscometer size number	Capillary radius mm	Approximate calibration factor K ^a at 40 000 Pa vacuum Pa			Viscosip a seb Despars
		Tube section B	Tube section C	Tube section D	
25	0,125	0,2		0,07	4,2 to 80
50	0,25	0,8	. NYN .	0,3	18 to 320
100	0,50	3,2	N 1,6	1,0	60 to 1 280
200	1,0	whete P.I.	6,4	4,0	240 to 5 200
400	2,0	50,0	25,0	16,0	960 to 20 000
400 R ^c	2,0	50,0	25,0	16,0	960 to 140 000
800 R ^c	4,0	200,0	100,0	64,0	3 800 to 580 000

^a Exact calibration factors shall be determined with viscosity standards. Some manufacturers provide viscometers with 5 or 6 tube sections calibrated extending the viscosity range.

^b The viscosity ranges shown in this table correspond to a filling time of 60 s to 400 s. Longer flow times (up to 1 000 s) may be used.

^c Special design for roofing asphalts having additional marks at 5 mm and 10 mm above timing mark F (see Figure A.2). Thus, using these marks, the maximum viscosity range is increased from that using the tube section B calibration factor.

Dimensions in millimetres



Key

А	filling tube	Ι	fourth timing mark
B, C and D	tube sections (see Table A.3)	М	vacuum tube
Е	fill line	Ν	ground glass joint – standard taper: 24/4
F	first timing mark	Т	to atmosphere
G	second timing mark	V	to vacuum
Н	third timing mark		

Figure A.3 — Modified Koppers vacuum capillary viscometer

Table A.3 — Standard viscometer sizes, capillary radii, approximate calibration factors *K*, viscosity ranges and viscometer size number for Modified Koppers vacuum capillary viscometers

Viscometer size number	Capillary radius	Approximate calibration factor <i>K</i> ^a at 40 000 Pa vacuum Pa			Visesity range
Size number	mm		Pa·s		
		Tube section B	Tube section C	Tube section D	
25	0,125	0,2		0,07	4,2 to 80
50	0,25	0,8	N 10,4	0,3	18 to 320
100	0,50	32. N	1,6	1,0	60 to 1 280
200	1,0	ntre	6,4	4,0	240 to 5 200
400	2,0	50,0	25,0	16,0	960 to 20 000

^b The viscosity ranges shown in this table correspond to a filling time of 60 s to 400 s. Longer flow times (up to 1 000 s) may be used.

Annex B (informative)

B.1 General This annex describes the materials and procedures used by calibrating, or checking the calibration of, viscometers used in this document. Capillary viscometers shall preferably be calibrated at the intended operational temperature. B.2 Reference materials the state of the calibration of the calibration

	Approximate viscosity Pa·s				
Viscosity standards ^a					
	at 20 °C	at 37,78 °C (100 °F)	at 40 °C	at 60 °C	
N18000	92		16	3,8	
N190000	840		140	33	
N2700000				340	
S30000		23	20		

Table B.1 — Viscosity standards

The viscosity standards cited in this table are examples cited according to the reference system of the following firm:

CANNON Instrument Company

2139 High Tech Road

State College, PA 16803

USA

This information is given for the convenience of users of this document 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 vacuum viscometer by means of viscosity standards

Calibrate the vacuum viscometer as follows (see Figure A.1, Figure A.2 and Figure A.3).

Select from Table B.1 a viscosity standard with a minimum flow time of 60 s at the calibration temperature.

Charge a clean, dry viscometer by pouring the sample to within ± 2 mm of fill line E.

Place the charged viscometer in the viscometer bath, maintained at the calibration temperature \pm 0,1 °C.

Establish a $(40\ 000\ \pm\ 100)$ 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.

After the viscometer has been in the bath at least1 hr, start the flow of standard in the viscometer by opening the stopcock or toggle in the line leading to the vacuum system.

All time measurements (5.5 and 5.6) shall be made within 0,1 s according to the graduated divisions, the timer (5.5 or 5.6). Measure the time required for the leading edge of the meniscus to pass be timing marks F and G. Using a second timer, measure the time required for the leading edge the meniscus to pass between timing marks G and H (CMVV). If, as for the AIVV and MKVV to viscometer, the instrument contains additional timing marks (F, G, H and I), similarly determine the flow time for each successive tube section.

Calculate the viscometer bulb or tube section calibration factor Kuit rescal at 40 000 Pa, for each bulb or tube section using Formula (B.1) as follows: $K = \eta / t$ (B.1) where n is the viscosity of viscosity at a dard at with a time to be the section.

$$K = \eta / t$$

- is the viscosity of viscosity standard at calibration temperature, in Pa s; η
- is the flow time, in s. t

Repeat the calibration procedure using the same viscosity standard or another viscosity standard. Record the average calibration constant *K*, for each bulb or tube section.

The duplicate determination of calibration constant K, for each bulb or tube section shall agree with 2 % of their mean.

NOTE The bulb or tube section constants are independent of temperature.

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

Calibrate the vacuum viscometer as follows:

Select any bitumen having a flow time of at least 60 s. Select also a standard viscometer of known calibration factors.

Mount the standard viscometer together with the viscometer to be calibrated in the same bath at 60 °C and determine the flow times of the bitumen by the procedure described in Clause 7.

Calculate the factor *K*, for each bulb or tube section using Formula (B.2) as follows:

$$K_1 = \left(t_2 \times K_2\right) / t_1 \tag{B.2}$$

where

- K_1 is the factor of the viscometer bulb or tube section being calibrated;
- is the flow time of the viscometer bulb or tube section being calibrated; t_1
- is the calibration factor of the standard viscometer; K_2
- is the flow time of corresponding bulb or tube section in the standard viscometer. t_2

Annex C

(informative)

Sample standard deviation σ is calculated according to Formula (COAUGES, COM) $\sigma = \sqrt{\frac{\Sigma(x_i - \overline{x})^2}{(n-1)}}$ where x_i is the observed value (mean) \overline{x} is the observed value (mean) (C.1) \overline{x} is the mean value of results; is the number of values. n Coefficient of variance *CV* is calculated according to Formula (C.2): $CV = \frac{\sigma}{\frac{\sigma}{x}} \times 100 \%$ (C.2) EXAMPLE (e.g. Asphalt Institute vacuum capillary viscometer, size number 50) Tube section B: 114 Pa·s Tube section C: 114 Pa·s Tube section D: 117 Pa·s $x_1 = 114 \text{ Pa} \cdot \text{s}$ $x_2 = 114 \text{ Pa} \cdot \text{s}$ $x_3 = 117 \text{ Pa} \cdot \text{s}$

 $\overline{x} = 115 \text{ Pa} \cdot \text{s}$

n = 3

Sum of squared deviations = $\Sigma (x_i - \overline{x})^2 = (114 - 115)^2 + (114 - 115)^2 + (117 - 115)^2 = 1 + 1 + 4 = 6$.

$$\sigma = \sqrt{\frac{6}{(3-1)}} = \sqrt{3} \approx 1,73$$
$$CV = \frac{\sigma}{\overline{x}} = \frac{1,73}{115} \times 100 \approx 1,51\%$$

The results from tube sections B, C and D in the same viscometer are within the maximum acceptable range for results, since the coefficient of variance CV is below 2 %.

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