BS EN 14770:2023

Bitumen and bituminous binders — Determination of complex shear modulus and phase angle — Dynamic Shear Rheometer (DSR)

bsi.

National foreword

This British Standard is the UK implementation of EN 14770:2023. It supersedes BS EN 14770:2012, which is withdrawn.

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 committee manager.

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English Version

Bitumen and bituminous binders - Determination of complex shear modulus and phase angle - Dynamic Shear Rheometer (DSR)

Bitumes et liants bitumineux - Détermination du module complexe en cisaillement et de l'angle de phase à l'aide d'un rhéomètre à cisaillement dynamique (DSR)

Bitumen und bitumenhaltige Bindemittel - Bestimmung des komplexen Schermoduls und des Phasenwinkels - Dynamisches Scherrheometer (DSR)

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

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Contents

European foreword

This document (EN 14770:2023) 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of an identical text or by endorsement, at the latest by January 2024, and conflicting national standards shall be withdrawn at the latest by January 2024.

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

This document supersedes EN 14770:2012.

In comparison with the previous edition, the main technical changes are:

a)   restriction of particle size added in the scope;

b)   reference to outdated standards IP PM CM-02 and XPT 66-065 removed;

c)   integration of "complex compliance" removed;

d)   use of the terms "shear strain" and "shear stress" unified;

e)   use of the term "bituminous binder" unified;

f)   reference to EN 1427 moved from Clause 2 to Bibliography; references to EN 12607-1, EN 14023 and EN 14769 added to Bibliography;

g)   definitions "shear strain controlled mode" and "shear stress controlled mode" added;

h)   use of the term "range of linear viscoelastic behaviour" unified;

i)   use of the term "complex shear modulus" together with the corresponding symbol $|G^*|$ unified; description of the complex shear modulus revised;

j)   6.1, 7.1 and 8.1 added with reference to Annex E;

k)   information on different plate diameters relocated from 5.1 to new 6.2; information about different plate diameters in 6.2 updated and plate diameter of 4 mm added;

l)   deviation for rheometer specification removed in 5.1;

m)   suitable dimensions for silicone moulds added in 5.2;

n)   vials for preparation of test specimen removed in 5.2, 7.2, 7.3 and 8.2;

o)   use of the term "specimen" unified;

p)   6.4 "Zero gap setting" revised and clarified;

q)   heating procedure in 7.2 simplified with reference to EN 12594;

r)   paring of specimen at room temperature removed in 7.3;

s) storage conditions and storage duration of specimens revised in 7.3;

t) 8.2 "Specimen placing into the rheometer" and 8.3 "Gap setting" revised;

u) gap compensation added in 8.4;

v) explanation of different testing procedures added in 8.5;

w) isochrones added Clause 9;

x) calculation of TX and δ_{TX} added in Clause 9 and new Annex D;

y) Clause 10 revised and complemented with new precision data, instead of coefficient of variation repeatability r and reproducibility R are now used;

z) terms c) and d) added in Clause 11;

aa) revision of Annex C "Determination of the linear viscoelastic (LVE) range";

bb) Annex E "Flow Chart" added.

Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

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1 Scope

This document specifies a general method of using a dynamic shear rheometer (DSR) for measuring the rheological properties of bituminous binders. The procedure involves determining the complex shear modulus and phase angle of binders over a range of test frequencies and test temperatures when tested in oscillatory shear.

From the test, the complex shear modulus, $|G^*|$, and its phase angle, δ, at a given temperature and frequency are calculated, as well as the components G' and G'' of the complex shear modulus.

This method is applicable to un-aged, aged, stabilized and recovered bituminous binders. The test procedure in accordance with this document is not applicable for bituminous binders with particles larger than 250 μm (e.g. filler material, granulated rubber).

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 establish appropriate safety and health practices and to determine 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 12594, *Bitumen and bituminous binders - Preparation of test samples*

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 https://www.iso.org/obp

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

3.1
shear strain controlled mode
rheometer control mode where a demand angular displacement is applied to the specimen and the corresponding torque is measured

Note 1 to entry: Using the shear strain factor of the measuring geometry, a specimen shear strain can be calculated from the applied angular displacement. Using the shear stress factor of the measuring geometry, a specimen shear stress can be calculated from the measured torque. Additional corrections can be applied to calculate true specimen shear strain and true specimen shear stress. These corrections are automatically carried out by the instrument software and are not the responsibility of the operator.

3.2
shear stress controlled mode
rheometer control mode where a demand torque is applied to the specimen and the corresponding angular displacement is measured

Note 1 to entry: Using the shear stress factor of the measuring geometry, a specimen shear stress can be calculated from the applied torque. Using the shear strain factor of the measuring geometry, a specimen shear strain can be calculated from the measured angular displacement. Additional corrections can be applied to calculate true

specimen shear stress and true specimen shear strain. These corrections are automatically carried out by the instrument software and are not the responsibility of the operator.

3.3
complex shear modulus
$|G^*|$
ratio of the amplitude of the shear stress to the amplitude of the shear strain in harmonic sinusoidal oscillation, in Pa

Note 1 to entry: The (mathematical) real part of the complex shear modulus $|G^*|$ is G´. It is associated with the elastic part of material behaviour which represents energy stored during a shear cycle. The real part is the complex shear modulus multiplied with cosine of phase angle expressed in degrees.

Note 2 to entry: The (mathematical) imaginary part of the complex shear modulus is G´´. It is associated with the viscous part of material behaviour which represents energy dissipated during a shear cycle. The imaginary part is the complex shear modulus multiplied with sine of phase angle expressed in degrees.

3.4
phase angle
δ
phase difference between shear stress and shear strain in harmonic oscillation, in °

3.5
isotherm
curve on a graph representing the behaviour of a material at a constant temperature

3.6
isochrone
curve on a graph representing the behaviour of a material at a constant frequency

3.7
range of linear viscoelastic behaviour
range in which complex shear modulus is independent of shear stress or shear strain

4   Principle

A known oscillatory shear stress is applied to the temperature controlled test geometry in which the bituminous test specimen is held. The binder's shear strain response to the shear stress is measured. Alternatively, a known oscillatory shear strain is applied to the test specimen and the resulting shear stress is measured.

Except for specific purposes, the test is performed in the region of linear viscoelastic behaviour.

5   Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1   Dynamic shear rheometer (DSR), with either an integral temperature control system or temperature control attachments, capable of controlling the temperature over a minimum range of 5 °C to 85 °C with a maximum permissible error of ± 0,1 °C throughout the test period. The rheometer shall be fitted with parallel plates, with a constant gap across the area of the plates. Depending on the expected complex shear modulus range different plate diameters (for example 25 mm, 8 mm or 4 mm) are used (see 6.2). The temperature control system shall encompass both plates to avoid temperature gradients across the plates. When the test specimen is immersed in liquid other than water, ensure that the liquid

does not affect the properties of the material being analysed. The rheometer shall be capable to determine $|G^*|$, at least in the range of 1 kPa to 10 MPa and the phase angle δ, in the range 0° to 90°.

NOTE 1   When liquid is used to immerse the test specimen, a water/glycol mixture has been found to be suitable. The proportions used depend on how low the temperature intended for testing is. Rheometers using radio frequency (RF) heating and/or liquid gas cooling or other heating/cooling systems can be used in accordance with the manufacturer's instructions.

Where the bottom plate is nominally the same diameter as the top plate, a visual check should be made to ensure the two plates are vertically aligned. If there is any doubt as to the alignment of the top and bottom plates, the manufacturer, or a qualified technician should re-align the plate geometry.

NOTE 2   The fact that the temperature control range is 5 °C to 85 °C does not imply that accurate results will necessarily be obtained for all binders over this range (see 6.2 and 6.3, NOTE 1). Furthermore, temperatures outside this range can also be used, provided the results are not affected by material or instrument limitations (see 6.2).

5.2   Moulds or sheet materials, for the preparation of the test specimens. The moulds or sheet material, where used, shall be of silicone or similar material, which does not adhere to the test specimen.

For a testing geometry with a diameter of 25 mm and a gap setting of 1 mm, a mould with a cavity of approximately 18 mm in diameter and 2 mm deep may be used. For a testing geometry with a diameter of 8 mm and a gap setting of 2 mm, a mould with a cavity of approximately 8 mm in diameter and 2,5 mm deep may be used. For a testing geometry with a diameter of 4 mm with different gap settings, a mould with a cavity of approximately 4 mm in diameter and 3 mm deep may be used. In any case, the operator shall ensure adequate filling of the gap according to 8.3.

The use of grease or other anti-stick products should be avoided because they can affect the adherence of the specimen to the rheometer plates.

5.3   Oven, ventilated laboratory model, capable of being controlled at temperatures between 50 °C and 200 °C with a maximum permissible error of ± 5 °C.

6   Preparation of rheometers

6.1 General

An informative flow chart for preparation of rheometers is given in Annex E, Figure E.1.

6.2 Selection of geometry

For different ranges of complex shear modulus plates of different diameters and gap settings shall be used to respect the instruments limitations.

For determining complex shear modulus of bituminous binders in the range 1 kPa to 100 kPa, the geometry with a diameter of 25 mm and a gap setting of 1,0 mm is suitable for most instruments. For determining complex shear modulus of bituminous binders in the range 100 kPa to 10 MPa, the geometry with a diameter of 8 mm and a gap setting of 2,0 mm is suitable for most instruments. Overlapping of test results from both geometries is recommended (see 8.5).

For determining complex shear modulus of bituminous binders below 1 kPa, a geometry with a diameter larger than 25 mm is recommended. Alternatively, the geometry with a diameter of 25 mm may be used provided that test results in the expected range of the complex shear modulus are verified with a calibrated fluid.

Plates of other diameters and other gap settings with different ranges of complex shear modulus may also be used, ensuring compliance effects of the instrument do not affect the results (see 6.3, NOTE 1), the minimum torque specification of the rheometer is respected and the testing is done in the linear viscoelastic range (see Clause 8).

NOTE   Recent research results demonstrate the suitability of a plate diameter of 4 mm for testing complex shear modulus in a range 10 MPa to 1 GPa. Depending on the specimen installation procedure, a gap setting between 1,0 mm and 3,0 mm is generally suitable.

6.3 Set up

Set up the rheometer in the sequence given in the manufacturer's instructions, including the procedure for selecting and setting the correct geometry and gap.

NOTE 1   The selection of system geometry can affect the accuracy of results. The manufacturer can have determined the operational limits and this information can be available, but if not, it can be determined by running a test specimen over a range of test temperatures using all the test geometries likely to be used in practice, and plotting $|G^*|$ against either frequency or phase angle δ. When the divergence between the plots for each geometry exceeds 15 %, this is an indication that compliance effects are affecting one or more of the geometries. The chosen geometry(ies) which shows the more rapid fall in $|G^*|$, or the lower phase angle, indicates that its accuracy limit has been reached. Also, for most rheometers generally referred to in this document, irrespective of the geometry chosen, values of $|G^*|$ in excess of 10^9 Pa are likely to be suspect. Software corrections to the stiffness can be acceptable provided appropriate validation is supplied by the manufacturer.

The rheometer and temperature control system should be calibrated at regular intervals in accordance with the quality assurance procedure of the laboratory. The rheometer and temperature control system should be calibrated by a means traceable to a national standard. Also, it is advisable to verify the accuracy of the temperature control system by means of a certified temperature-measuring device at regular intervals. Take note that external devices read the accurate temperature value only if they are calibrated correctly. A temperature verification procedure is described in Annex A.

NOTE 2   The temperature in the test specimen can differ from the temperature read by the device if insufficient equilibration time is used. A procedure for determining equilibration time is described in Annex B.

6.4 Zero gap setting

For initialization, the gap between the plates shall be set to zero to give a reference for the gap change for the thermal expansion of the geometry. Prior to loading the first test specimen, the zero gap is set with both clean plates at ambient temperature.

NOTE   For temperature control systems with minimized thermal gradients within the gap, the zero gap can be set at any temperature assuring thermal equilibrium of the geometry.

If the DSR has no gap compensation feature, the zero gap can be set at the mid-point of the temperature range to be tested.

7   Specimen preparation

7.1 General

WARNING — This document involves handling of apparatus and binders at very high temperatures. Always wear protective gloves and eyeglasses when handling hot binder, and avoid contact with any exposed skin.

An informative flow chart for specimen preparation is given in Annex E, Figure E.1.

7.2 Heating procedure for the preparation of the binder

Prepare the bituminous binder in accordance with EN 12594.

7.3 Specimen manufacturing and storage conditions

Moulds or sheet materials may be used for all types of binders.

When the binder reaches temperature after the heating period, stir and mix with a spatula to ensure homogeneity (especially for polymer modified binders); or after the heating period, remove a sub-sample of convenient size for handling safely and of sufficient volume, to prepare the required number of test specimens plus approximately 50 %.

Pour into moulds or directly on to sheets. Care shall be taken to avoid air bubbles in the specimen. Choose one or more test shapes that will give reliable measurements with the selected test apparatus. The moulds shall be stored at ambient temperature. If the ambient temperature is higher than 30 °C or the binder is very soft, specimens may be cooled down for storage, but not below 5 °C. All specimens shall be covered with an opaque lid.

A minimum storage duration before the de-moulding and testing procedure of 30 min shall be maintained for all bituminous binders. For modified binders that do exhibit phenomena such as crystallization (e.g. EVA modified binders) the minimum storage duration shall be increased to 12 h. A maximum delay of two weeks shall not be exceeded for all bituminous binders. The storage time shall be stated in the test report.

8 Procedure

8.1 General

An informative flow chart for the test procedure is given in Annex E, Figure E.2.

8.2 Specimen placing into the rheometer

Carefully prepare the rheometer plates for receipt of the test specimen by cleaning them with a suitable solvent and soft cleaning cloth or paper. Do not use metal or any other materials, which may damage the surfaces of the plates, and take care not to bend the shaft of the upper plate.

The specimens may be placed in the refrigerator prior to de-moulding. The temperature of the refrigerator shall not be below 5 °C. De-moulding and loading into the rheometer shall take place just after removal from the refrigerator.

Specimen installation shall be performed in specific temperature conditions which ensure satisfactory adhesion of the test specimen to the rheometer plate, which resets rheologic history of the specimen and which allows sufficient molecular mobility and ensures adequate filling of the gap (see 8.3). If the sample flows out of the gap before reaching gap setting and there is no adequate filling, the installation temperature shall be reduced.

Set the temperature of both plates to the expected softening point of the binder plus 5 °C to 20 °C. The temperature shall be within a tolerance of ± 1,0 °C for a period of at least 5 min. If the upper plate has no heating, it can be warmed by contact with lower plate and/or using a water bath. Load the specimen and set the trimming gap according 8.3.

Alternative temperatures may be used for the temperature for both plates provided that adhesion takes place between the binder and the plate, and that the binder is sufficiently fluid to allow the gap to be achieved. Any deviation from the recommended temperature tolerance (expected softening point plus 5 °C to 20 °C) shall be clearly stated in the report.

8.3 Gap setting

Values of 1,0 mm for 25 mm plate diameters and 2,0 mm for 8 mm plate diameters are recommended gap settings.

Bring the test specimen to the selected gap setting plus 0,05 mm for 25 mm plate diameter and plus 0,10 mm for 8 mm plate diameter. The normal force should be monitored during gap setting and reach the key value of 1,0 N or below to continue the procedure. If the normal force cannot be monitored by the instrument a waiting time of at least 5 min need to be maintained after gap setting. Then trim any excess

binder with a knife, a spatula or a special trimming tool. It has been found helpful to heat the trimming tool before trimming. After trimming, raise or lower the opposing plate to the set testing gap (±0,01 mm). Do not trim at this stage. The entire process of placing and trimming the specimen shall not take more than 10 min.

For the testing gap to be adequately filled, the test specimen shall cover the whole measuring plate (indicated by a slight bulging at the periphery of the test specimen). If the testing gap is not adequately filled, remove, re-prepare the rheometer plates, and prepare a fresh test specimen.

8.4 Temperature and frequency conditions selecting

Set up the rheometer to test in the oscillatory mode to ensure a dynamic response from the specimen under test.

Select the test temperatures appropriate to the binder being tested. Temperatures should be selected so that the binder can be adequately characterized with no temperature increment being greater than (10,0 ± 0,1) °C. Allow the test specimen to equilibrate within the test geometry at each test temperature before testing (see Annex B). The equilibration time between test temperatures shall be stated in the report.

NOTE 1   The procedure described in Annex B is useful to determine the necessary equilibrium time to homogenize the temperature in the test specimen. For most rheometers and most purposes, 10 min to 20 min equilibration time has been found to be satisfactory.

Caution shall be taken when testing at low test temperatures that the measured complex shear modulus values are not being affected by possible machine/geometry shear compliance, or by the test specimen de-bonding from the plates. Due to shrinkage of the specimen, the normal force can decrease during conditioning which might affect the test results. If available, controlled normal force mode shall be used for gap compensation at low test temperatures with a normal force in the range of (0,0 ± 0,2) N.

For binders that present crystallization phenomena in the selected range of temperatures, results are affected by the thermal history undergone by the specimen once mounted into the rheometer. In these cases, the procedure should be to slowly cool down the specimen (no more than 2 °C/min) to a low "reference" temperature (for instance 10 °C for a bitumen modified with a copolymer including ethylene) and stabilize at that temperature. All test temperatures above that temperature would then be performed from low to high whereas all test temperatures below would be performed from that temperature downwards. The given rate for moving from one temperature to another could be 1 °C/min.

Select the frequency range and number of frequencies to be tested. Select either a discrete test frequency (within the range 0,1 Hz to 10,0 Hz) or a frequency range for testing. For the frequency range, a minimum of two series of ten frequencies shall be specified with any individual frequency being at ± 10 % of the set value.

NOTE 2   For most purposes, ten equally spaced logarithmic steps including 0,1 Hz and 10,0 Hz, within the frequency range 0,1 Hz to 10,0 Hz, have been found to be suitable.

Select the shear strain (or shear stress) in order to ensure that the test specimen will be tested in the linear viscoelastic range over the temperature and frequency range chosen (see Annex C). If several test geometries are used, check for each of them that the chosen range of shear strain (or shear stress) remains in the adequate range of measurement of the apparatus.

8.5 Testing procedure

Three different basic testing procedures can be differentiated:

a)   For a temperature sweep (T-sweep) the test is carried out with a fixed oscillation frequency and gradual increase or decrease of test temperature. Between different test temperatures the temperature change should not exceed the rate of 5 °C/min.

b) For a frequency sweep (f-sweep) the test is carried out at a fixed test temperature and gradual increase of oscillation frequency.

c) A temperature frequency sweep (T-f-sweep) is a combination of a temperature sweep and a frequency sweep, where a frequency sweep is performed for each chosen test temperature.

For any of the testing procedures above, testing can continue until the complex shear modulus measured is outside the range of the particular test geometry chosen. Also, once the compliance limits have been reached (see 6.3, NOTE 1), the testing shall be stopped and a new test geometry will be needed to continue testing over further temperatures. For this, a new specimen of the same material is required. The preparation of the new test geometry shall be as described for the first test geometry and the first test temperature shall be at the temperature where the complex shear modulus measurements were not affected by machine compliance. The testing shall be continued with this new test geometry for the remainder of the test temperatures or until the measurements of complex shear modulus are outside the range of the test geometry.

NOTE   It can be convenient to test over the whole temperature range with one test geometry, and then to review the results to determine the temperature at which a second, or further, test geometry is necessary.

The temperature at which both test geometries have been tested is known as the overlap temperature and is used in the determination of the acceptability criteria. Examine the values of complex shear modulus $|G^*|$ and phase angle δ obtained at the temperature overlap using the two different test geometries, and apply acceptability criteria as follows:

a) the values of $|G^*|$ per test geometry at the test frequency should not differ from the mean of $|G^*|$ from the two geometries by more than 15 %;

b) the values of δ per test geometry at the test frequency should not differ from the mean of δ from the two geometries by more than 3°.

If either of the above are not met, repeat the whole test. If more than one overlap temperature occurs with a single test specimen, the criteria given above shall be met at each overlap temperature.

If no change of geometry has been made, repeat the test at one of the test temperatures using the same geometry on a new test specimen. If the acceptability criteria given above are not met, repeat the whole test.

Construct isotherms of $|G^*|$ (Pa) and δ (°) against frequency (Hz), or isochrones of $|G^*|$ (Pa) and δ (°) against temperature (°C).

All results where the measured shear strain is outside of the linear viscoelastic range of the tested binder, or where the results have been affected by machine compliance, should be omitted.

9   Expression of results

Report the test geometries, and the shear strain or shear stress conditions used in the test.

Report the test frequency(ies) and temperature(s), the values of complex shear modulus $|G^*|$ in Pa, to three significant figures, and values of phase angle (°), in degrees, to the nearest 0,1°.

If possible (and in case that a temperature sweep was performed), construct isochrones of $|G^*|$ (Pa) and δ (°) against temperature (°C) and attach them to the test report.

If possible (and in case that a frequency sweep was performed), construct isotherms of $|G^*|$ (Pa) and δ (°) against frequency (Hz) and attach them to the test report.

Temperatures TX at which $|G^*|$ has a specific value $|G^*|_{TX}$ and corresponding phase angles δ_{TX} may be calculated from the test results according to Annex D.

NOTE   Other parameters can be reported in addition to those stated.

10 Precision

The precision of this test method cannot be determined universally, since the method covers a wide variety of bituminous binders and test conditions such as temperature, frequency or plate geometry. Based on repeatability and reproducibility data of recent large scale round robin tests (as given in the Table 2 and Table 3 below), an estimate of the precision for the complex shear modulus $|G^*|$ and phase angle δ can be derived as given in Table 1. Precision values are given as repeatability r (= 2,77 · s_r), and reproducibility R (= 2,77 · s_R) where s_r is the standard deviation under repeatability conditions and s_R is the standard deviation under reproducibility conditions.

Table 1 — Estimated precision for complex shear modulus $|G^*|$ and phase angle δ

	Repeatability r = 2,77 · s_r	Reproducibility R = 2,77 · s_R
$|G^*|$ (precision expressed in % of the mean value)	15 %	30 %
δ (precision expressed in absolute values)	2°	4°

The values given in Table 1 are independent of the type of binder (paving grade or polymer modified), state of binder (fresh, aged RTFOT [EN 12607-1] or RTFOT + PAV [EN 12607-1 + EN 14769]) as well as specimen geometry (25 mm or 8 mm diameter), as well as test temperatures and frequencies.

If ageing conditioning or recovery procedures are carried out before DSR testing, these procedures will have an effect on the final test result, and therefore also on the precision of the results.

NOTE 1   Round robin results with estimates (typical ranges covering different test conditions) for repeatability r (= 2,77 · s_r), and reproducibility R (= 2,77 · s_R) from BNPétrole, France are summarized in Table 2 with reference to type and grade of the bituminous binder and year of round robin. Precision for $|G^*|$ is expressed in % from the mean value and precision for δ is expressed in absolute values. Measurements include temperatures from 10 °C to 65 °C at frequency 1,59 Hz, with 25 mm and 8 mm plate geometry. Note that the RTFOT procedure was individually carried out by each participating laboratory, which is why the results are influenced by ageing conditions in addition to DSR testing.

Table 2 — Round robin results from BNPétrole, France

Bitumen	Year	$|G^*|$		δ	
		r	R	r	R
50/70 (after RTFOT)	2017	15 % to 22 %	32 % to 44 %	1° to 2°	2° to 5°
20/30 (after RTFOT)	2018	13 % to 21 %	30 % to 44 %	1° to 2°	3° to 4°
PMB 45/80–55 (after RTFOT)	2019	13 % to 21 %	25 % to 50 %	1° to 2°	1° to 3°
PMB 45/80–55 (fresh binder)	2020	6 % to 12 %	20 % to 43 %	0,5° to 1°	1° to 3°

NOTE 2   Round robin results with estimates for repeatability r (= 2,77 · s_r) and reproducibility R (= 2,77 · s_R) from Eurobitume Deutschland are summarized in Table 3 with reference to bituminous binder and year of round robin.

Precision for $|G*|$ is expressed in % from the mean value and precision for δ is expressed in absolute values. Measurements at 60 °C, frequency 1,59 Hz applying a 25 mm plate geometry. Note that the RTFOT procedure was individually carried out by each participating laboratory, which is why the results are influenced by ageing conditions in addition to DSR testing.

Table 3 — Round robin results from Eurobitume Deutschland

Bitumen	Year	$|G*|$		δ	
		r	*R*	*r*	*R*
PMB 25/55–55	2018	5 %	30 %	0,2°	1,2°
	2019	6 %	21 %	0,2°	3,7°
PMB 25/55–55 (after RTFOT)	2018	5 %	37 %	0,4°	1,5°
	2019	6 %	27 %	0,2°	1,7°
PMB 40/100–65	2018	8 %	37 %	0,7°	3,2°
	2019	6 %	18 %	0,3°	1,1°
PMB 40/100–65 (after RTFOT)	2018	5 %	30 %	0,3°	1,4°
	2019	8 %	26 %	0,5°	1,7°
70/100	2018	5 %	30 %	0,3°	4,2°
	2019	5 %	31 %	0,3°	0,9°
70/100 (after RTFOT)	2018	5 %	24 %	0,3°	1,0°
	2019	7 %	36 %	0,4°	1,2°

11 Test report

The test report shall contain at least the following information:

a) reference to this document including its year of publication;

b) type and complete identification of the sample under test;

c) storage time of the specimen;

d) diameter of upper and lower rheometer plate and testing gap;

e) testing conditions followed, including equilibration time;

f) result of the test (see Clause 9);

g) any deviation, by agreement or otherwise, from the procedure specified, and unusual features observed;

h) date of the test.

Annex A
(informative)

Temperature verification procedure

Thermal gradients within the rheometer and the difficulty of calibrating the DSR temperature instrument while it is mounted in the rheometer mean that a temperature verification of the DSR temperature transducer is required. To this end, temperature measurements obtained from a dummy specimen into which a calibrated thermal sensor, measuring to an accuracy of ± 0,02 °C, has been inserted, and the DSR temperature transducer can be compared. A dummy specimen of bituminous binder or silicon wafer may be used.

Alternatively, specially devised thermometers may be used that can be inserted between the plates to verify the temperature.

Prepare the dummy specimen or use the silicon wafer following standard procedures. Use the dummy specimen only for temperature verification measurements. (DSR measurements are not valid if a temperature detector is inserted into the asphalt binder.) Adjust the temperature in the chamber to the minimum temperature that will be used for testing and allow the chamber to come to thermal equilibrium. Read the DSR temperature and the temperature of the dummy specimen. Increase the temperature in increments of not more than 6 °C and repeat the measurements to cover the range of test temperatures. The difference between the temperature probe and the temperature indicated by the DSR transducer varies with temperature and depends on testing geometry.

Apply an appropriate temperature correction to the temperature measurement indicated by the DSR transducer if both readings do not agree within ± 0,1 °C.

Annex B
(informative)

Determining equilibration time

It takes some time for the temperature of the bituminous binder specimen to reach thermal equilibrium. This equilibrium time varies for different rheometers, depending upon the design of the rheometer and the type of heating-cooling unit and medium.

The equilibrium time can be determined by monitoring $|G^*|$ with time. The phase angle is not very sensitive to changes in temperature and should not be used for this purpose. To determine the equilibrium time, mount a binder specimen in the DSR and run a test at a given frequency according to the described procedure. Record the time required to bring $|G^*|$ to a constant value and add between 2 min to 5 min. The sum is the equilibration time.

NOTE   Prolonged measurements can be influenced by internal heat in the specimen generated by the vibration.

Annex C
(normative)

Determination of the linear viscoelastic (LVE) range

The linear viscoelastic range should be determined for each selected geometry.

It is necessary to use a specific test specimen for this determination.

For a given geometry, determination of the linear viscoelastic range on all the selected temperature ranges shall consist, at least, of carrying out:

— shear strain (or shear stress) sweep at the lowest temperature and at the highest frequency;

— shear strain (or shear stress) sweep at the highest temperature and at the lowest frequency;

in order to select the adequate shear strain (or shear stress) level.

NOTE   For 25 mm parallel-plates the shear strain sweep can be performed in the approximate range from 0,01 % to 100 % and in the approximate range from 0,001 % to 10 % for the 8 mm parallel-plates with logarithmic distribution of data points.

To remain in the linear viscoelastic range, the values of G' and G'' shall not differ by more than 5 % of the linear regression line fitted to the first stable values over the shear stress or shear strain range chosen. Values of G' and G'' at very low shear strain (or shear stress) levels can be affected by the limited angular resolution of the instrument.

For graphical display of G' and G'', a logarithmic scale is recommended.

If the binder is similar to one of the binders already tested on the same apparatus, it is possible to carry out a simplified procedure for linear viscoelastic range determination, for example by selecting only one temperature for each geometry.

Annex D
(normative)

Determining rheological parameters TX and δ_{TX}

Rheological measurements often result in a large number of data. In order to condense the information to a simplified form, some calculations may be performed on the measurements to provide values of temperature and the corresponding phase angle for a given shear modulus $|G^*| = Y$ at certain measuring conditions. The temperature $T(|G^*| = Y)$ shall be determined from two individual data points by logarithmic interpolation according to Formula (D.1). One data point shall be above and one below the key value Y with both data points not more than 10 °C apart from each other. The corresponding phase angle $\delta_{T(|G^*| = Y)}$ is determined from the same data points by linear interpolation according to Formula (D.2).

$$T\left(\left|G*\right| = Y\right) = \frac{T\left(\left|G*\right| > Y\right) - T\left(\left|G*\right| < Y\right)}{\lg\left(\left|G*\right| > Y\right) - \lg\left(\left|G*\right| < Y\right)}\left(\lg\left(Y\right) - \lg\left(\left|G*\right| < Y\right)\right) + T\left(\left|G*\right| < Y\right) \tag{D.1}$$

$$\delta_{T\left(\left|G*\right| = Y\right)} = \frac{\delta_{T\left(\left|G*\right| > Y\right)} - \delta_{T\left(\left|G*\right| < Y\right)}}{T\left(\left|G*\right| > Y\right) - T\left(\left|G*\right| < Y\right)}\left(T\left(\left|G*\right| = Y\right) - T\left(\left|G*\right| > Y\right)\right) + \delta_{T\left(\left|G*\right| > Y\right)} \tag{D.2}$$

Two explicit values for Y are chosen for calculation of $T(|G^*| = Y)$ and $\delta_{T(|G^*| = Y)}$ to provide information about the viscoelastic behaviour:

— at intermediate service temperature: $|G^*| = 5$ MPa at frequency = 1,59 Hz;

— at high service temperature: $|G^*| = 15$ kPa at frequency = 1,59 Hz.

For the purpose of use in product standards such as EN 14023, the temperatures TX, where X is 0, 1, 2, 3 or 4 and corresponding phase angles δ_{TX} specified in Table D.1 below may be calculated using Formula (D.1) and Formula (D.2). Different rheometer plates and aging conditions are used to evaluate the viscoelastic behaviour, the durability of viscoelastic behaviour and the temperature sensitivity based on measurements with a frequency of 1,59 Hz (10 rad · s^{-1}).

Table D.1 — Definitions of temperatures $T0$, $T1$, $T2$, $T3$ and $T4$ with respect to measuring geometry, $|G^*|$ value at 1,59 Hz (10 rad s^{-1}) and stage of binder

Temperature	Measuring geometry (parallel plate)	$|G^*|$ value at 1,59 Hz (10 rad · s^{-1})	Stage of binder (conditioning)
$T0$	25 mm plate	$|G^*| = 15$ kPa	Original
$T1$	8 mm plate	$|G^*| = 5$ MPa	EN 12607-1
$T2$	25 mm plate	$|G^*| = 15$ kPa	EN 12607-1
$T3$	8 mm plate	$|G^*| = 5$ MPa	EN 12607-1 + EN 14769
$T4$	25 mm plate	$|G^*| = 15$ kPa	EN 12607-1 + EN 14769

Report the value of TX in °C, to the nearest 0,1 °C, and the value of δ_{TX} in degrees (°), to the nearest 0,1 °.

NOTE   Round robins organized by BNPétrole have provided the following precision estimates on $T1$ and $T2$ that are given in Table D.2 and Table D.3.

Table D.2 (informative) — Round robin results with estimates for repeatability, r, and reproducibility, R, for $T(|G^*| = 5\ MPa)$ and $\delta_{T(|G^*| = 5\ MPa)}$ measured with 8 mm parallel plate from BNPétrole, France with reference to bituminous binder and year of round robin; for the years 2017, 2018 and 2019 the values correspond to $T1$ and δ_{T1}

| Bitumen	Year	$T(|G^*| = 5\ MPa)$		$\delta_{T(|G^*| = 5\ MPa)}$	
		Repeatability r	Reproducibility R	Repeatability r	Reproducibility R
50/70 (after RTFOT)	2017	1,1 °C	2,6 °C	1,8°	5,8°
20/30 (after RTFOT)	2018	1,5 °C	3,0 °C	1,6°	3,3°
PMB 45/80–55 (after RTFOT)	2019	0,9 °C	2,7 °C	1,3°	3,1°
PMB 45/80–55 (fresh binder)	2020	0,5 °C	1,8 °C	1,0°	2,3°

Table D.3 (informative) — Round robin results with estimates for repeatability, r, and reproducibility, R, for $T(|G^*| = 15\ kPa)$ and $\delta_{T(|G^*| = 15\ kPa)}$ measured with 25 mm parallel plate from BNPétrole, France with reference to bituminous binder and year of round robin; for the years 2017, 2018 and 2019 the values correspond to $T2$ and δ_{T2}, for the year 2020 the values correspond to $T0$ and δ_{T0}

Bitumen	Year	$T(|G^*| = 15\ kPa)$		$\delta_{T(|G^*| = 15\ kPa)}$	
		Repeatability r	Reproducibility R	Repeatability r	Reproducibility R
50/70 (after RTFOT)	2017	1,1 °C[a]	2,2 °C[a]	0,8°[a]	1,3°[a]
20/30 (after RTFOT)	2018	1,5 °C[a]	2,8 °C[a]	1,3°[a]	2,2°[a]
PMB 45/80–55 (after RTFOT)	2019	1,0 °C	1,8 °C	0,5°	1,6°
PMB 45/80–55 (fresh binder)	2020	0,7 °C	2,4 °C	0,5°	1,1°

[a]   Definition of $T2$ was in 2017 and 2018 with $|G^*| = 50$ kPa, 25 mm and is therefore the reference to the stated values of r and R. In 2019 the definition of $T2$ was changed to $|G^*| = 15$ kPa, 25 mm, which will be the future reference.

Annex E
(informative)

Flow chart
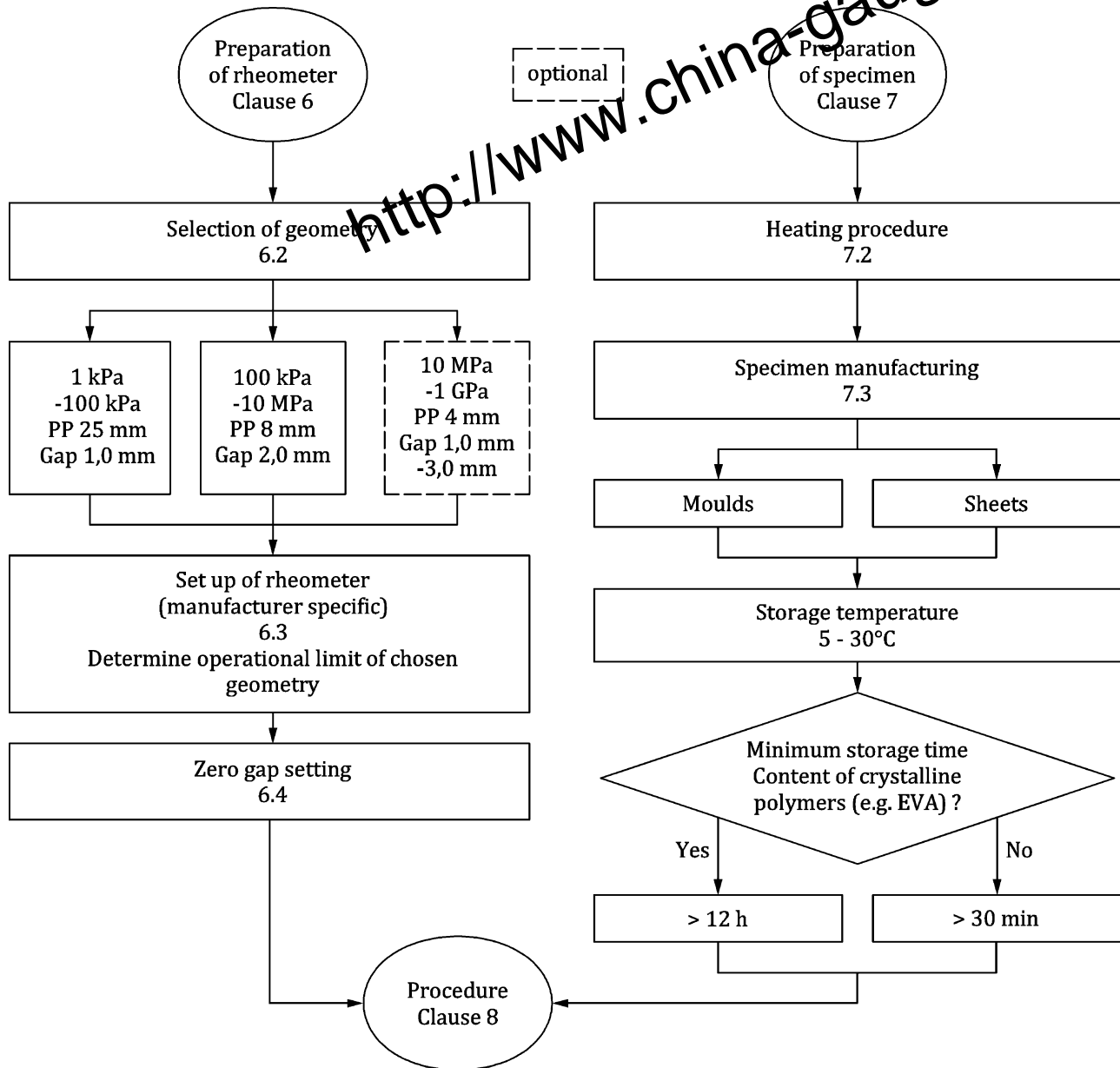
Figure E.1 — Flow chart for preparation of rheometer and preparation of specimen

Procedure
Clause 8

optional

Preparation of rheometer plates
8.2

Specimen conditioning at 5 °C

Select installation temperature

Maintain selected T ± 1 °C for > 5 min

Specimen placing onto rheometer

Selected gap setting + 5 %
8.3

Normal force ≤ 1 N
during gap setting

Normal force
not monitored:
minimum waiting
time 5 min

Trimming of excess material

Setting of the testing gap

Temperature and frequency selection
8.4

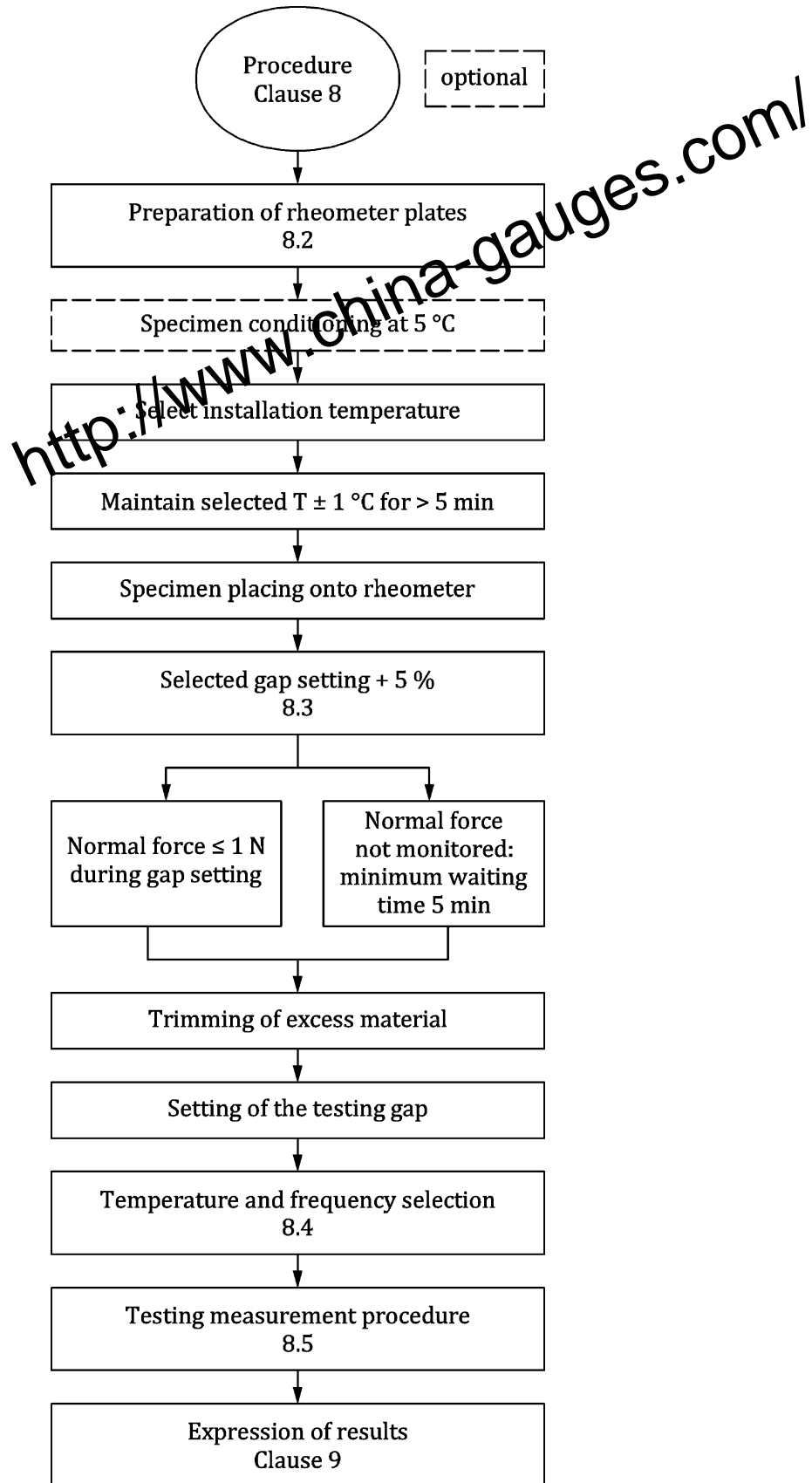Testing measurement procedure
8.5

Expression of results
Clause 9

Figure E.2 — Flow chart for test procedure

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[3] EN 14023, *Bitumen and bituminous binders - Specification framework for polymer modified bitumens*

[4] EN 14769, *Bitumen and bituminous binders - Accelerated long-term ageing conditioning by a Pressure Ageing Vessel (PAV)*

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