



BSI Standards Publication

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

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 It supersedes BS EN 14770:2012, which is withdrawn.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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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of an identical text or by endorsement, at the latest by January 2024, and conflicting national standards shall be withdrawn at the latest by January 2024.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- b) reference to outdated standards IP PM CM-02 and XPT 66-065 removed;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 $\pm 0,1$ °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