ISO/FDIS 6721-10:2025(E)

ISO/TC 61/SC 5/WG 8

Secretariat: DIN

Date: 2025-01-22

Plastics — Determination of dynamic mechanical properties —

Part 10:
Complex shear viscosity using a parallel-plate and a cone-and-plate oscillatory rheometer

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 10: Viscosité complexe en cisaillement à l'aide d'un rhéomètre à oscillations à plateaux parallèles ou à géométrie cône/plan

FDIS stage

Contents

Foreword v
1 Scope 1
2 Normative references 1
3 Terms and definitions 1
4 Principle 2
5 Apparatus 2
6 Sampling 6
7 Procedure 7
8 Expression of results 10
9 Precision 13
10 Test report 14
Annex A (informative) Uncertainty limits 16
Annex B (informative) Verification of rheometer performance 19
Bibliography 24

Foreword v
1 Scope 1
2 Normative references 1
3 Terms and definitions 1
4 Principle 2

5 Apparatus 2
5.1 Measurement apparatus 2
5.2 Temperature-controlled enclosure 4
5.3 Temperature measurement and control 4
5.4 Measurement geometry 4
5.4.1 Parallel plates geometry 4
5.4.2 Cone and plate geometry 5
5.5 Calibration 5
6 Sampling 5
7 Procedure 6
7.1 Test temperature 6
7.2 Zeroing the gap 6
7.3 Introducing the test specimen 6
7.4 Conditioning the test specimen 6
7.5 Test mode (controlled stress or controlled strain) 6
7.5.1 Controlled strain mode 6
7.5.2 Controlled stress mode 7
7.6 Determination of thermal stability of sample material 7
7.7 Determination of region of linear-viscoelastic behaviour 7

7.7.1 In the controlled-strain mode 7
7.7.2 In the controlled-stress mode 7
7.7.3 Confirmation of linear-viscoelastic behaviour 8
7.8 Frequency sweep 8
7.9 Temperature sweep 8
7.10 Air entrapment 8
8 Expression of results 8
8.1 Symbols used 8
8.2 Calculation of complex shear modulus and complex shear viscosity 9
9 Precision 10
10 Test report 12
Annex A (informative) Uncertainty limits 13
Annex B (informative) Verification of rheometer performance 16
B.1 General 16
B.2 Verification of the gap separation of the rheometer plates or cone and plate 16
B.3 Verification of the strain amplitude 17
B.4 Verification of the frequency response 17
B.5 Verification of angular velocity 17
B.6 Verification of torque measurement 18
B.6.1 Verification of torque measurement using mechanical means 18
B.6.2 Verification of torque measurement using reference fluids 18
B.7 Verification of temperature measurement 18
B.8 Verification of the phase shift 19
B.9 Verification of the rheometer using reference fluids 19
B.9.1 General 19
B.9.2 Newtonian fluids 19
B.9.3 Non-Newtonian fluids 20
Bibliography 21

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at ~~www.iso.org/patents.~~www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see ~~www.iso.org/iso/foreword.html~~www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This fourth edition cancels and replaces the third edition (ISO 6721-~~-~~10:2015), which has been technically revised.

The main changes are as follows:

— ~~—~~ rheometer geometry has been described in detail for both parallel-plate and cone-and-plate geometry;

— ~~— in 7.5,~~in 7.5, controlled stress mode and controlled strain mode have been defined in separate subclauses.

A list of all parts in the ISO 6721 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Plastics — Determination of dynamic mechanical properties —

Part 10:
Complex shear viscosity using a parallel-plate and a cone-and-plate oscillatory rheometer

1 Scope

This document specifies the general principles of a method for determining the dynamic rheological properties of polymer melts at angular frequencies typically in the range of 0,01 rad·s^{-1} to 100 rad·s^{-1} by means of an oscillatory rheometer with a parallel-plate or a cone-and-plate geometry. Angular frequencies outside this range can also be used.

The method is applicable ~~to determine~~for determining values of the following dynamic rheological properties: complex shear viscosity η*, dynamic shear viscosity η', the out-of-phase component of the complex shear viscosity η”, complex shear modulus G*, shear loss modulus G”, shear storage modulus G', phase angle δ, and loss factor tanδ. It is suitable for measuring complex shear viscosity values typically up to ~10 MPa·s.

NOTE The shear loss modulus G´´ is sometimes also called viscous shear modulus and the shear storage modulus G´ is sometimes also called elastic shear modulus.

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.

ISO 472, *Plastics — Vocabulary*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 6721-1, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6721-1, ISO 5725-1, ISO 472, and the following apply.

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

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

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

3.1 ~~**3.1**~~
controlled-strain mode
testing by applying a sinusoidal angular displacement of constant amplitude

3.2 ~~3.2~~
controlled-stress mode
testing by applying a sinusoidal torque of constant amplitude

3.3 ~~3.3~~
complex shear viscosity
η^*
ratio of dynamic stress, given by $\sigma(t) = \sigma_0 \exp(\mathrm{i}\omega t)$, and dynamic rate of strain where the shear strain ~~$\gamma(t)$~~ $\gamma(t)$ is given by $\gamma(t) = \gamma_0 \exp\{\mathrm{i}(\omega t - \delta)\}$, of a viscoelastic material that is subjected to a sinusoidal vibration, where σ_0 and γ_0 are the amplitudes of the stress and strain cycles, ω is the angular frequency, δ is the phase angle between the stress and strain, and t is time

Note 1 to entry: It is expressed in pascal seconds.

3.4 ~~3.4~~
dynamic shear viscosity
η'
real part of the complex shear viscosity

Note 1 to entry: It is expressed in pascal seconds.

3.5 ~~3.5~~
out-of-phase component of the complex shear viscosity
η''
imaginary part of the complex shear viscosity

Note 1 to entry: It is expressed in pascal seconds.

4 Principle

The specimen is held between two concentric, circular parallel plates or cone-and-plate (see ~~Figure 1 and 2).~~ Figure 1 and 2). The thickness of the specimen is small compared with the diameter of the plates.

One of the plates or the cone is subjected to either a sinusoidal torque or a sinusoidal angular displacement of constant angular frequency. These are referred to as “controlled-stress” or “controlled-strain” test modes, respectively. When using the controlled-stress mode, the resultant displacement and the phase shift between the torque and displacement are measured. When using the controlled-strain mode, the resultant torque and the phase shift between the displacement and torque are measured.

The complex shear modulus G^*, shear storage modulus G', shear loss modulus G'', phase angle δ, and corresponding shear viscosity terms (see ~~Clause 3)~~ Clause 3) are determined from the measured torque and displacement and the specimen dimensions. In deriving these values, it is assumed that the specimen exhibits a linear-viscoelastic response.

The mode of oscillation used is designated as oscillatory mode I (see ISO 6721-1).

5 Apparatus

5.1 Measurement apparatus

The measurement apparatus shall consist of two concentric, rigid, circular parallel plates (see ~~Figure 1)~~ Figure 1) or cone-and-plate (see ~~Figure 2)~~ Figure 2) between which the specimen is placed. One of these plates or one side of cone-and-plate shall be made to oscillate at a constant angular frequency while the other remains at rest.

The range of complex shear viscosity values that can be measured is dependent on the specimen dimensions determined by the diameter of the geometry used and also the specification of the measuring instrument. For a specimen of given dimensions, the upper limit of the range is limited by the machine's torque capacity, angular-displacement resolution, motor inertia, and compliance. However, corrections can be made for compliance effects.

The requirements on the apparatus are that it shall permit measurement of the amplitudes of the torque and the angular displacement and the phase difference between them for a specimen subjected to either a sinusoidal torque or a sinusoidal displacement of constant angular frequency.

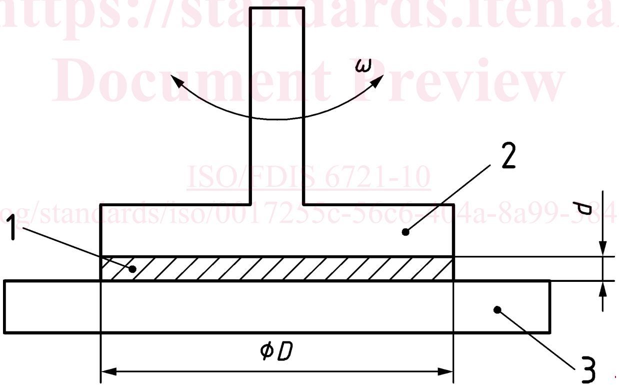
The torque required to overcome the viscoelastic resistance of the specimen shall be determined, for example, by connecting a torque-measuring device to one of the plates or one of cone and plate.

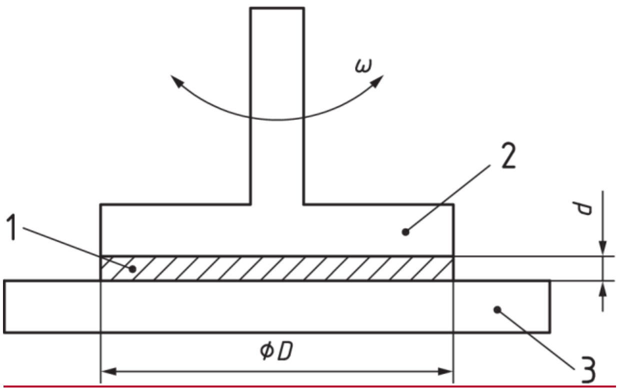
An angular-displacement measuring device shall be fitted on the moving plate or moving side of the cone-and-plate, thus permitting determination of its angular displacement and angular frequency

The apparatus shall be capable of measuring the torque to within ±2 % of the minimum torque amplitude used to determine the dynamic properties.

The apparatus shall be capable of measuring the angular displacement to within $\pm 20 \times 10^{-6}$ rad.

The apparatus shall be capable of measuring the angular frequency to within ±2 % of the absolute value.





Key

1 test specimen

2 moving plate

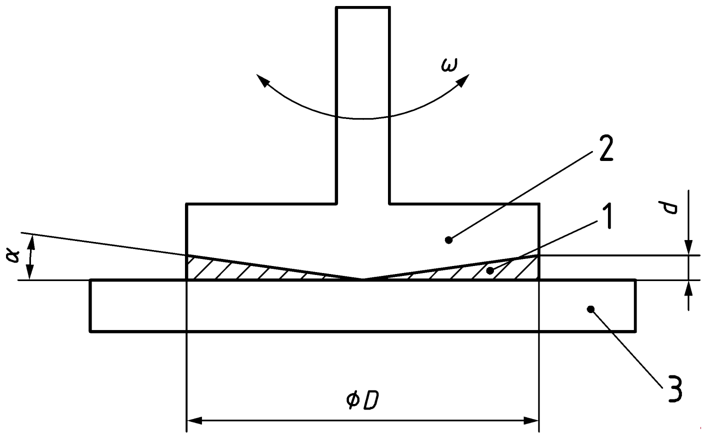
3 fixed plate

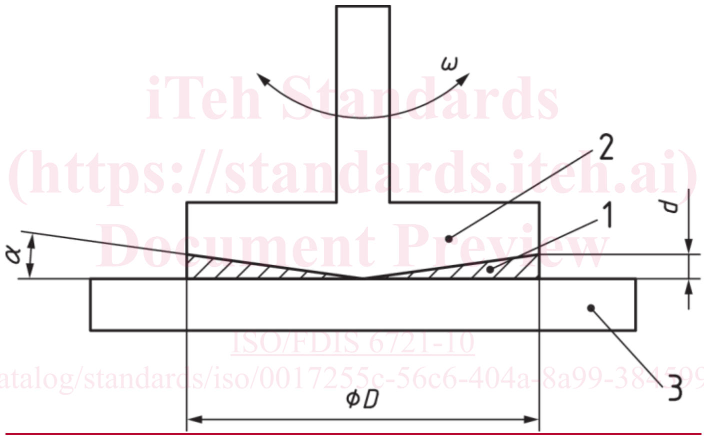
ω angular frequency (rad/sec)

d specimen thickness (mm)

D diameter of plate (mm)

Figure 1 — Parallel-plate rheometer geometry





Key

1 test specimen

2 moving cone

3 fixed plate

α cone angle (°)

ω angular frequency (rad/s)

d specimen thickness (mm)

D diameter of plate (mm)

Figure 2 — Cone-and plate rheometer geometry

5.2 Temperature-controlled enclosure

Heating may be provided by the use of forced convection, radio-frequency heating, or other suitable means.

An enclosure surrounding the measurement geometry assembly can be used to provide specific test environments. For example, samples which are sensitive to oxidation shall be measured in an inert atmosphere.

Check that the enclosure is not in contact with the measurement geometry assembly.

5.3 Temperature measurement and control

The test temperature shall preferably be measured using a device that is either in contact with or embedded in the fixed cone or plate.

The test temperature shall be accurate to within ±0,5 °C of the set temperature for set temperatures up to 200 °C, within ±1,0 °C for temperatures in the range 200 °C to 300 °C, and within ±1,5 °C for temperatures above 300 °C.

The temperature-measuring device shall have a resolution of 0,1 °C or better and shall be calibrated using a device accurate to within ±0,1 °C.

5.4 Measurement geometry

5.4.1 Parallel plates geometry

The measurement geometry assembly comprises two concentric, circular parallel plates with the specimen held between them. The plates shall have a surface finish corresponding to a maximum roughness of S = 0,25 µm and shall have no visible imperfections.

The results may be dependent on the type of material that is used to form the surfaces of the plates. This can be identified by testing using plates with different surface materials. Different surface materials shall be considered when sample slippage on the plates is suspected.

The plate diameter, D, is typically in the range of 20 mm to 50 mm. It shall be measured to within ±0,01 mm.

The specimen thickness, d, is defined by the measurement gap for plates and shall be determined to within ±0,01 mm. It is recommended that the specimen thickness lies in the range of 0,5 mm to 3 mm and that the ratio of the plate diameter to the specimen thickness lie in the range of 10 to 50 in order to minimize errors in the determination of properties. For low-viscosity polymeric liquids, it may be necessary to employ dimensions outside these recommended ranges. The total variation in the measurement gap for plates due to non-parallelism of the plates shall be less than ±0,01 mm. Variation in the measurement gap for plates during testing shall be less than ±0,01 mm.

The plates shall be sufficiently flat to enable the requirement on the total variation in the measurement gap for plates due to non-parallelism of the plates be less than ±0,01 mm.

5.4.2 Cone and plate geometry

The angle between the cone and plate shall be less than 5°. The specimen assembly comprises concentric, circular cone and plate with the specimen held between them. The surface finish of cone and plate shall be in accordance with that of parallel plates.

The results may be dependent on the type of material that is used to form the surfaces of the cone and plate. This can be identified by testing using plates with different surface materials. Different surface materials shall be considered when sample slippage on the cone or plates is suspected.

The diameter of cone and plate, and the specimen thickness at peripheral of cone and plate are in accordance with parallel plates. The total variation in the cone and plate around peripheral due to non-concentricity of the cone and plate shall be less than ±0,01 mm. Variation in the cone and plate around peripheral during testing shall be less than ±0,01 mm.