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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

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Contents

Page

Foreword

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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).

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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, 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 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 $\tan\delta$. 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

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

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

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

3.3
complex shear viscosity
η^*
ratio of dynamic stress, given by $\sigma(t) = \sigma_0 \exp(i\omega t)$, and dynamic rate of strain where the shear strain $\gamma(t)$ is given by $\gamma(t) = \gamma_0 \exp\{i(\omega t - \delta)\}$, 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
dynamic shear viscosity
η'
real part of the complex shear viscosity

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

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). 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) 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) or cone-and-plate (see 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.

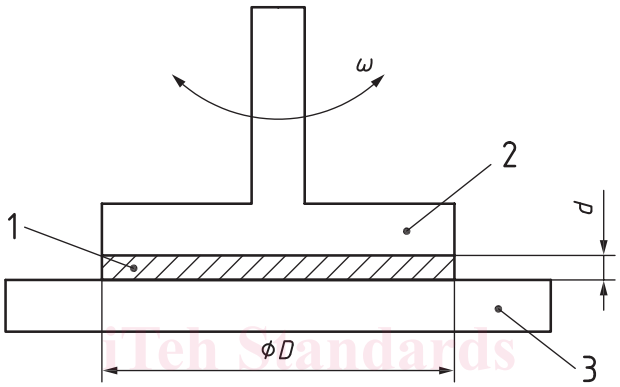
The torque required to overcome the viscoelastic resistance of the specimen shall be determined, for example, by connecting a torque-measuring device to one of the plates or one of cone and plate.

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.

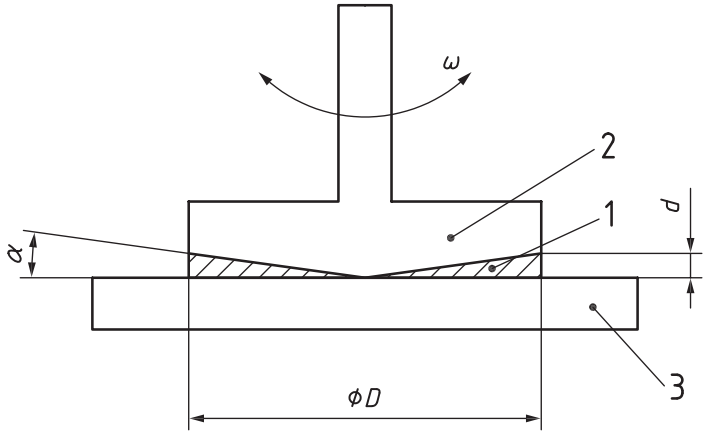
The apparatus shall be capable of measuring the angular frequency to within ±2 % of the absolute value.



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.

The truncation gap shall not exceed 0,05 mm.

The independence of the shear rate on the radius is a fundamental advantage of the cone/plate geometry. To achieve this, the gap shall be set to a height at which the virtual cone tip touches the plate for the given cone angle.

This creates some limitations for the application of the cone/plate geometry related to:

— particle sizes: The truncation gap should be not less than 10 times the particle size;

— thermally induced volume changes (shrinkage): If the virtual cone tip does not touch the plate anymore, deviations from the independence of the shear rate on the radius will occur.

If the deviation of the uniform shear rate caused by such limitations is not acceptable, the plate/plate geometry can be used.

5.5 Calibration

The rheometer and test geometries shall be calibrated periodically by measuring the torque, angular-displacement, angular-frequency and temperature response of the machine and the relevant dimensions of the geometries, or checked by using reference liquids of known viscosity or complex viscosity, in accordance with the instrument manufacturer's instructions. It is preferable that the viscosities of the reference liquids used for checking the calibration span the range in viscosity values of the specimens that are to be measured.

It is preferable that calibration be carried out at the test temperature.

NOTE Guidance on verification of the performance of the instrument is given in Annex B.

6 Sampling

The sampling procedure, including any special methods of specimen preparation and introduction into the rheometer, shall be as specified in the relevant materials standard or as otherwise agreed.

As the test specimens are typically small, being of the order of 3 g to 5 g, it is essential that they are representative of the material being sampled.

If samples or specimens are hygroscopic or contain volatile ingredients, then they shall be stored to prevent or minimize any changes in viscosity. Drying of samples may be required prior to preparing test specimens.

The test specimens shall be in the form of a disc when produced by injection or compression moulding or by cutting from sheet. Alternatively, they may be formed by placing pellets or liquid or molten polymer between the plates or cone and plate. The specimen may be introduced in the molten state only if it is not sensitive to oxidation or loss of volatile matter.

The specimen shall not contain any visible impurities or air bubbles. The specimen shall not show any obvious discolouration prior to or after testing.

7 Procedure

7.1 Test temperature

Generally, because of the temperature dependence of viscosity, measurements for comparison purposes shall be carried out at the same temperature. Details shall be as specified in the relevant materials standard or as otherwise agreed.

7.2 Zeroing the gap

Allow the apparatus to come to thermal equilibrium at the desired test temperature. The suggested equilibrium time is 15 min to 30 min. Bring the plates or cone and plate into contact with each other. Set the gap indicator to zero.

7.3 Introducing the test specimen

The specimen shall be loaded into the instrument in either the solid or the molten state as specified in Clause 6. It shall completely fill the gap between the two plates or cone and plate. Any excess material round the edges of the plates or cone and plate shall be removed before testing is started. The specimen may need to be slightly squeezed after trimming to promote good contact, but precautions shall then be taken to ensure that the specimen does not extend beyond the edges of the plates and that the specimen edge is only slightly convex.

The specimen and the measurement geometry assembly shall then be allowed to reach thermal equilibrium at the test temperature. This period of time is referred to as the preheat time. For any particular instrument, measurement geometry assembly with specimen, polymer type, sample thickness, loading procedure, and test temperature, the preheat time shall be determined by repeating the measurement but using a preheat time that is 10 % greater (see note). If there is no change in the measured values of the complex shear modulus G^*, shear storage modulus G', and shear loss modulus G'', then the preheat time is sufficient for thermal equilibrium to have been established.

NOTE This check can be incorporated into the time-sweep test for thermal stability of the sample (see 7.6).

When the instrument and specimen have reached the test temperature, measure the specimen thickness, d, which is equivalent to the measurement gap for parallel plates. For the cone-and-plate geometry, the specimen thickness is determined by the truncation gap, d_t, and the cone angle, α, (see 5.4). These values of the specimen thickness shall be used in all calculations.