INTERNATIONAL STANDARD

ISO 6721-10

First edition
1997-08-01

Plastics — Determination of dynamic mechanical properties —

Part 10:
Complex shear viscosity using a parallel-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

Reference number
ISO 6721-10:1997(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 6721-10 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

Together with the other parts of ISO 6721, it cancels and replaces ISO 537:1989 and ISO 6721:1983, which have been technically revised.

ISO 6721 consists of the following parts, under the general title *Plastics — Determination of dynamic mechanical properties*:

— *Part 1: General principles*

— *Part 2: Torsion-pendulum method*

— *Part 3: Flexural vibration — Resonance-curve method*

— *Part 4: Tensile vibration — Non-resonance method*

— *Part 5: Flexural vibration — Non-resonance method*

— *Part 6: Shear vibration — Non-resonance method*

— *Part 7: Torsional vibration — Non-resonance method*

— *Part 8: Longitudinal and shear vibration — Wave-propagation method*

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

Printed in Switzerland

— *Part 9: Tensile vibration — Sonic-pulse propagation method*

— *Part 10: Complex shear viscosity using a parallel-plate oscillatory rheometer*

Annex A of this part of ISO 6721 is for information only.

This page intentionally left blank

Plastics — Determination of dynamic mechanical properties —

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

1 SCOPE

This part of the International Standard ISO 6721 specifies the general principles of a method for determining the dynamic rheological properties of polymer melts at angular frequencies typically in the range 0,01 - 10 Hz by means of an oscillatory rheometer with a parallel plate test geometry. Frequencies outside this range can be used (see Note 1). The method is used to determine values of the 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'' and shear storage modulus G'. It is suitable for measuring complex shear viscosity values typically up to approximately 10 MPa.s (see Note 2).

[NOTE 1: The angular frequency range of measurement is limited by the specification of the measuring instrument and also by the response of the specimen. When testing using frequencies lower than 0,01 Hz the testing time can increase significantly as the time taken to obtain a single datum is proportional to the reciprocal of the test frequency. Consequently, when testing at low frequencies degradation or polymerisation of the specimen is more likely to occur and have an effect on the results. At high angular frequencies the specimen may distort or fracture at the edge consequently invalidating the test results.]

[NOTE 2: The range of complex shear viscosity values that can be measured is dependent on the specimen dimensions 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 and compliance. However, correction can be made for compliance effects.]

2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 472: 1988, Plastics - Vocabulary.

ISO 6721-1: 1994, Plastics - Determination of dynamic mechanical properties - Part 1: General principles.

3 DEFINITIONS

For the purposes of this part of ISO 6721, the definitions given in ISO 6721-1:1994 and ISO 472: 1988 apply. In addition the following definitions apply:

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^*: The ratio of dynamic stress, given by $\sigma(t) = \sigma_0 \exp i\omega t$, and dynamic rate of strain $\dot{\gamma}(t)$, 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 the angular frequency, δ is the phase angle between the stress and strain and t is time.

It is expressed in Pascal.seconds.

3.4 dynamic shear viscosity, η': The real part of the complex shear viscosity.

The dynamic shear viscosity is expressed in Pascal.seconds.

A torque measuring device shall be connected to one of the plates thus permitting measurement of the torque required to overcome the viscoelastic resistance of the specimen.

An angular displacement measuring device shall be fitted to the moving plate thus permitting determination of its angular displacement and angular frequency.

The apparatus shall have an accuracy of torque measurement to within ± 2% of the minimum torque amplitude used to determine the dynamic properties.

The apparatus shall have an accuracy of angular displacement measurement to within $\pm 20 \times 10^{-6}$ radians.

The apparatus shall have an accuracy of angular frequency measurement to within ± 2% of the absolute value.

5.2 Temperature-controlled enclosure

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

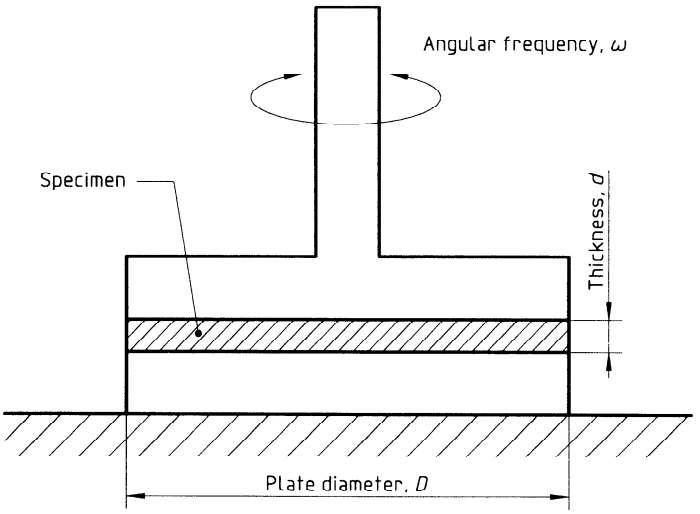

Figure 1 — Parallel-plate rheometer test geometry

3.5 **out-of-phase component of the complex shear viscosity, η":** The imaginary part of the complex shear viscosity.

The out-of-phase component of the complex shear viscosity is expressed in Pascal.seconds.

4 PRINCIPLES

The specimen shall be held between two concentric, circular, parallel plates, Figure 1. The thickness of the specimen shall be small compared with the diameter of the plates.

The specimen shall be 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' testing modes respectively. In using the controlled-stress mode the resultant displacement and the phase shift between the torque and displacement are measured. In 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", the phase angle δ and the corresponding complex shear viscosity parameters (see clause 3) are determined from the torque and displacement functions and the specimen dimensions. In deriving these terms it is assumed that the specimen exhibits a linear viscoelastic response.

The mode of oscillation used is designated as oscillatory mode I (see ISO 6721-1:1994, clause 4).

5 APPARATUS

5.1 Measuring apparatus

The measuring apparatus shall consist of two concentric, rigid, circular parallel plates between which the specimen is placed, Figure 1. One of these plates shall be made to oscillate at a constant angular frequency while the other remains at rest.

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

An environmental chamber surrounding the test geometry can be used to provide specific test environments, for example a nitrogen atmosphere.

It shall be checked that the chamber is not in contact with the specimen, plate geometries or their supports.

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 plate.

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

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

5.4 Test geometry

The test geometry is defined by two concentric, circular parallel plates between which the specimen is held. The plates shall have a surface finish corresponding to a maximum roughness of R_a = 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 of different surface materials.

The diameter of the plates D shall be determined to ± 0,01 mm and is typically in the range 20 - 50 mm.

The specimen thickness d is defined by the plate separation and shall be determined to ± 0,01 mm. It is recommended that the specimen thickness should lie in the range 0,5 - 3 mm and the ratio of plate diameter to specimen thickness should lie in the range 10 to 50 in order to minimize errors in the determination of properties. For low viscosity

polymer liquids it may be necessary to employ dimensions outside these recommended ranges. The total variation in the plate separation due to non-parallelism of the plates shall be less than ± 0,01 mm. Variation in the plate separation during testing shall be less than ± 0,01 mm.

5.5 Calibration

The rheometer shall be calibrated periodically by measuring the torque, angular displacement and angular frequency response of the machine or by using reference liquids of known complex viscosity, in accordance with the instructions of the instrument manufacturer. It is preferable that the complex viscosity of reference liquids used for calibration shall have values in the same range as that of the specimens to be measured.

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

6 SAMPLING

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

As the test specimens are typically small, being of the order of 3 - 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 minimise any effects on 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 into the test geometry. The sample may be loaded in the molten state only if it is not sensitive to oxidation or loss of volatiles.

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 otherwise by agreement.

7.2 Loading the test specimen

The specimen shall be loaded into the instrument in either its solid or molten state in accordance with clause 6. The specimen shall completely fill the gap between the two plates. Any excess material around the edges of the plates shall be removed before testing is commenced. Specimens may need to be slightly squeezed after trimming to promote good contact but precautions should then be taken to ensure that the specimen does not extend beyond the edges of the plate.

The specimen and plates 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, test geometry, 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 3). If there is no further 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 occurred.

[NOTE 3: This test can be incorporated into the time sweep test for thermal stability of the sample (see clause 7.5)]

When the instrument and specimen have reached the test temperature the specimen thickness d, which is equivalent to the plate separation, shall be determined (see subclause 5.4). This value for the specimen thickness shall be used for calculations.