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ISO 6721-10

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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

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Contents

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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*.

This second edition cancels and replaces the first edition (ISO 6721-10:1997), which has 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*
- *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.

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Plastics — Determination of dynamic mechanical properties —

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

1 Scope

This part of 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 rad·s^{-1} to 100 rad·s^{-1} by means of an oscillatory rheometer with a parallel-plate geometry. Angular frequencies outside this range can also be used (see note 1). The method is used to determine 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'' 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 measurement range is limited by the specification of the measuring instrument and also by the response of the specimen. When testing using angular frequencies lower than 0,1 rad·s^{-1} the test time can increase significantly as the time taken to obtain a single measurement is proportional to the reciprocal of the angular frequency. Consequently, when testing at low angular frequencies degradation or polymerization 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 normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 6721. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 6721 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 472:1999, *Plastics — Vocabulary.*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

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

3 Terms and definitions

For the purposes of this part of ISO 6721, the terms and definitions given in ISO 6721-1:1994, ISO 5725-1:1994 and ISO 472:1999 apply, plus the following:

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 $\sigma(t) = \sigma_0 \exp \mathrm{i}\omega t$, and dynamic rate of strain $\dot{\gamma}(t)$, where the shear strain $\gamma(t)$ is given by $\gamma(t) = \gamma_0 \exp \mathrm{i}(\omega t - \delta)$, 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

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.

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 Principle

The specimen is held between two concentric, circular parallel plates (see Figure 1). The thickness of the specimen is small compared with the diameter of the plates.

The specimen 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 6721-1:1994, clause 4).

5 Apparatus

5.1 Measurement apparatus

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

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.

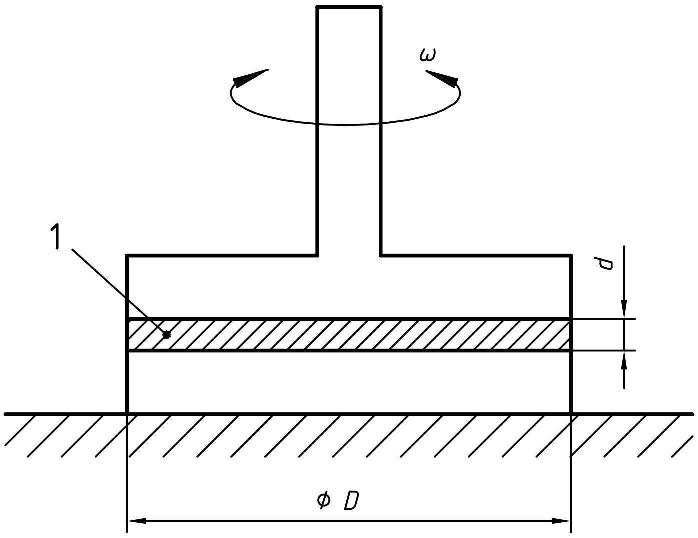
A torque-measuring device shall be connected to one of the plates, thus permitting measurement of the torque required to overcome the viscoelastic resistance of the specimen.

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 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

ω = angular frequency
d = specimen thickness
D = diameter of plate

Figure 1 — Parallel-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 environmental chamber surrounding the plate/specimen assembly can be used to provide specific test environments, for example a nitrogen atmosphere.

Check that the chamber is not in contact with the plate/specimen assembly.

5.3 Temperature measurement and control

The test temperature should 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, 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 and shall be calibrated using a device accurate to within ± 0,1 °C.

5.4 Plate/specimen assembly

The plate/specimen 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 Ra = 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.

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

The specimen thickness d is defined by the plate separation and shall be determined to within ± 0,01 mm. It is recommended that the specimen thickness lie in the range 0,5 mm to 3 mm and that the ratio of the plate diameter to the specimen thickness lie in the range 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 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 instrument manufacturer's instructions. It is preferable that the complex viscosities of reference liquids used for calibration lie in the same range as those of the specimens to be measured.

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

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. 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 must 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 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. Any excess material round the edges of the plates 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.

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, plate/specimen assembly 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). 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 plate separation (see 5.4). This value of the specimen thickness shall be used in all calculations.

7.4 Conditioning the test specimen

The test specimen may be conditioned before testing by holding it at zero shear at the test temperature for a specified period of time and/or by pre-shearing.

7.5 Test mode (controlled stress or controlled strain)

Measurements are made using instruments either in a controlled-strain mode or in a controlled-stress mode.

In the controlled-strain mode, a sinusoidal displacement is produced at constant angular frequency, and the resultant sinusoidal torque and the phase shift between the torque and displacement are measured.

In the controlled-stress mode, a sinusoidal torque is applied at constant angular frequency, and the resultant sinusoidal displacement and the phase shift between the torque and displacement are measured.

Measurement of the dynamic rheological properties of specimens in accordance with this part of ISO 6721 is restricted to the linear-viscoelastic region of behaviour. Linear-viscoelastic behaviour is defined, for the purposes of this part of ISO 6721, as behaviour in which the viscosity or modulus is independent of the applied stress or strain. This assumption is necessary for the analysis of the test data. It is therefore necessary for the amplitude of oscillation in the controlled-stress or controlled-strain modes to be set such that the deformation of the specimen occurs within the linear-viscoelastic region.

For methods of determining the limits of the linear-viscoelastic behaviour region, see 7.7.

7.6 Determination of thermal stability of sample material

Before testing a particular material, carry out a timed run at the test temperature to determine the thermal stability of the material. The run shall be made using the same plate/specimen assembly geometry, and angular frequencies and torque or angular displacement similar to those to be used in subsequent testing. It may be necessary to carry out runs at more than one frequency of oscillation (see note 1). The thermal-stability time is defined as the time taken from the start of the run to the point in time at which any of the measured values of G^*, G' and G'' have changed by 5 % from their initial value (see note 2). It shall be expressed as a time at a given temperature and angular frequency, for example 500 s at 250 °C and 1 rad/s. Subsequent measurements on new specimens from the same sample at that temperature shall be completed in a time shorter than the thermal-stability time.

NOTE 1 Specimen-degradation effects on rheological properties are normally most easily identifiable when testing at low frequencies of oscillation.

NOTE 2 It may be necessary to discard initial spurious results when determining the initial modulus values.

For some materials, it may not be possible to obtain the desired results within the thermal-stability time due to rapid degradation or crosslinking of the material. In such cases, the test report shall state the percentage change in modulus occurring over the duration of the test, this value having been determined from timed runs.

7.7 Determination of region of linear-viscoelastic behaviour

7.7.1 In the controlled-strain mode

When working in the controlled-strain mode, determine the maximum permissible amplitude of oscillation by performing a strain run. The strain run shall be made using the same plate/specimen assembly geometry, and angular frequency and temperature similar to those to be used in subsequent testing. It may be necessary to carry out strain measurements at more than one oscillation frequency to check for any dependence of the limit of linear-viscoelastic behaviour on the angular frequency. Test the specimen by increasing the amplitude of oscillation over a range of values, preferably commencing with a strain, measured at the edge of the plate, of not more than 1 %.

Measure the complex shear modulus G^*, shear storage modulus G' and shear loss modulus G'' as functions of the amplitude of oscillation to determine the maximum permissible amplitude of oscillation for measurements within the linear-viscoelastic region.

The maximum value of the strain to be used in actual testing shall be less than the lowest value of the strain at which a difference of 5 % occurred in the values of any of the parameters G^*, G' or G'' compared with their values in the linear-viscoelastic region. If it is not possible to determine properties within the linear-viscoelastic region, this shall be stated in the test report.

NOTE For some materials, the linear-viscoelastic region is confined to very small strains. The associated measurement errors prevent properties being determined reliably in this region.

7.7.2 In the controlled-stress mode

When working in the controlled-stress mode, determine the range of linear-viscoelastic behaviour by performing a stress run. The stress run shall be made using the same plate/specimen assembly geometry, and angular frequency and temperature similar those to be used in subsequent testing. It may be necessary to carry out measurements at more than one frequency to check for any dependence of the limit of linear-viscoelastic behaviour on the angular frequency. Test the specimen by increasing the torque over a range of values, preferably commencing with a torque, measured at the edge of the plate, of not more than 1 %.

Measure the complex shear modulus G^*, shear storage modulus G' and shear loss modulus G'' as functions of the torque to determine the maximum permissible torque for measurements within the linear-viscoelastic region.

The maximum value of the applied torque to be used in actual testing shall be less than the lowest value of the torque at which a deviation of 5 % occurred in the values of any of the parameters G^*, G' or G'' compared with their values in the linear-viscoelastic region. If it is not possible to determine properties within the linear-viscoelastic region, this shall be stated in the test report (see note to 7.7.1).

7.7.3 Confirmation of linear-viscoelastic behaviour

A further check may be carried out to confirm that measurements have been made within the linear-viscoelastic region. Assuming such behaviour, then for an applied sinusoidal displacement or torque the resultant output of torque or displacement, respectively, will also be sinusoidal. A non-sinusoidal output indicates that the behaviour is non-linear. In such cases, the assumptions made in the analysis of the experimental data are not valid and consequently the modulus and viscosity values determined are incorrect. If such checks have been made, this shall be stated in the test report.

7.8 Frequency run

When carrying out a frequency run on a specimen, it is necessary to check for degradation, particularly when testing at low frequencies, and for distortion or fracture of the specimen at high frequencies.

Changes in the specimen due, for example, to degradation, crosslinking or melt fracture can affect the test results. The effect of such changes can be identified by carrying out at the end of a test a repeat measurement on the same specimen at the conditions used at the start of the test, and then comparing the results.

If distortion of the specimen at the edges of the plates, commonly known as melt fracture, occurs, then those measurements shall be invalid. However, results obtained prior to distortion of the specimen remain valid.

7.9 Temperature run

When carrying out a temperature run from a low to a high temperature, it is necessary to ensure good physical contact between the specimen and the plates, preferably by melting the specimen between the plates prior to testing, and cooling to the starting temperature as necessary.

When performing temperature runs, consideration shall be given to applying a correction for the change in dimensions of the assembly due to thermal expansion. Some systems have software which makes this correction automatically using the coefficient of linear expansion of the fixture.

See also 7.8 concerning degradation and distortion of the specimen.

8 Expression of results

8.1 Symbols used

D	plate diameter, expressed in metres;
d	plate separation, expressed in metres;
T	torque, expressed in newton metres;
θ	angular displacement, expressed in radians;
ω	angular frequency (of oscillation), expressed in radians per second ($\omega = 2\,\pi f$ where f is the frequency in Herz);
σ	shear stress, expressed in pascals;
γ	shear strain, expressed as a dimensionless number;
G'	shear storage modulus, expressed in pascals;
G''	shear loss modulus, expressed in pascals;
G^*	complex shear modulus, expressed in pascals;
η'	dynamic shear viscosity, expressed in pascal seconds;
η''	out-of-phase component of the complex shear viscosity, expressed in pascal seconds;
η^*	complex shear viscosity, expressed in pascal seconds;
δ	phase shift or loss angle, expressed in radians;
t	time, expressed in seconds.

8.2 Calculation of complex shear modulus and complex shear viscosity

For a linear-viscoelastic fluid subjected to a harmonic stress $\sigma(t)$,

$$\sigma(t) = \sigma_0 \exp \mathrm{i}\omega t$$

resulting in a harmonic strain $\gamma(t)$ given by

$$\gamma(t) = \gamma_0 \exp \mathrm{i}(\omega t - \delta)$$

where

σ_0 and γ_0 are the amplitudes of the stress and strain, respectively;

ω is the angular frequency;

δ is the phase shift or loss angle between the stress and strain,

The following quantities can be defined:

— shear storage modulus	$G' = \sigma_0 \cos(\delta)/\gamma_0$
— shear loss modulus	$G'' = \sigma_0 \sin(\delta)/\gamma_0$
— complex shear modulus	$G^* = G' + \mathrm{i}G''$
— dynamic shear viscosity	$\eta' = G''/\omega$
— out-of-phase component of the complex shear viscosity	$\eta'' = G'/\omega$
— complex shear viscosity	$\eta^* = \eta' - \mathrm{i}\eta''$

where i is defined by $\mathrm{i} = \sqrt{-1}$.

Furthermore, it can be shown that

$$G^* = \mathrm{i}\omega\eta^*$$

For forced oscillation of a parallel-plate assembly and assuming a linear-viscoelastic specimen response, the equation of motion governing the flow is given by

$$T_0 = (\pi D^4/32d)\,[G'(\omega) + \mathrm{i}G''(\omega)]\,\theta_0 \exp(-\mathrm{i}\delta)$$

where

T_0 is the amplitude of the torque;

θ_0 is the amplitude of the angular displacement.

This equation assumes that the inertia and compliance terms are small. The errors associated with these assumptions are generally small when testing polymer melts, particularly when using low angular frequencies and when the stiffness of the specimen is low compared with that of the machine (see note).

NOTE Corrections for the inertia and compliance terms are proposed by various authors, for example Marin[1], Walters[2] and Whorlow[3].

Thus, for a given angular frequency, the shear storage modulus G' and shear loss modulus G'' are given by

$$G' = 32dT_0 \cos(\delta)/\theta_0\pi D^4$$

and

$$G'' = 32dT_0 \sin(\delta)/\theta_0\pi D^4$$

The phase shift δ is determined experimentally.

From the relationships between the shear moduli and the viscosities presented above, the following equations can be derived:

$$\eta' = 32 d T_0 \sin(\delta)/\omega\theta_0\pi D^4$$

and

$$\eta'' = 32 d T_0 \cos(\delta)/\omega\theta_0\pi D^4$$

For an angular displacement θ, the strain γ in the specimen at the edge of the plate is given by

$$\gamma = \theta D/2d$$

Generally, commercial instruments use software to determine the values of the complex shear modulus and its real and imaginary components from the raw data. The method by which they obtain and analyse the raw data of torque and amplitude and the phase shift between the sinusoidal torque and amplitude functions may vary. Also, the corrections for inertia and compliance effects, if applied, may vary. Specification of these procedures is currently outside the scope of this part of ISO 6721. Where procedures differ fundamentally from that presented here, the basis on which the instrument determines modulus and viscosity values shall be stated in the test report.

9 Precision

The following precision data were determined during a test programme carried out in 1995[4] comprising ten laboratories in total making measurements at three angular frequencies. s_r and s_R are the repeatability and reproducibility standard deviations, respectively. r and R are the repeatability and reproducibility limits, respectively (see ISO 5725-1). m is the mean modulus value at the given angular frequency.

Two materials were used for testing in the round robin: an unfilled high-density polyethylene (PE-HD) and an unfilled polypropylene (PP). PE-HD specimens were produced by compression moulding, and PP specimens by injection moulding. All samples were supplied to the participants by the organizer of the round robin. The instruments used included both controlled-strain and controlled-stress type rheometers.

Polyethylene at 190 °C, G'					
Angular frequency	**Mean, m**	**Repeatability**		**Reproducibility**	
rad·s^{-1}	Pa	s_r (%)	r (%)	s_R (%)	R (%)
1	6 130	3,0	8,4	9,4	26,4
10	25 500	2,0	5,5	8,9	24,8
100	91 300	1,6	4,4	7,9	22,2

Polyethylene at 190 °C, G''					
Angular frequency	**Mean, m**	**Repeatability**		**Reproducibility**	
rad·s^{-1}	Pa	s_r (%)	r (%)	s_R (%)	R (%)
1	8 320	1,6	4,5	8,5	23,9
10	28 900	1,3	3,7	8,4	23,4
100	79 600	1,2	4,3	7,5	21,0

Polypropylene at 210 °C, G'					
Angular frequency	**Mean, m**	**Repeatability**		**Reproducibility**	
rad·s^{-1}	Pa	s_r (%)	r (%)	s_R (%)	R (%)
1	1 590	3,4	9,4	11,3	31,7
10	12 000	2,5	6,9	10,5	29,3
100	47 800	2,5	7,1	9,3	26,1

Polypropylene at 210 °C, G''					
Angular frequency	**Mean, m**	**Repeatability**		**Reproducibility**	
rad·s^{-1}	Pa	s_r (%)	r (%)	s_R (%)	R (%)
1	3 880	2,4	6,6	10,3	28,8
10	15 400	2,4	6,8	9,5	26,7
100	36 000	2,7	7,7	9,1	25,6

The uncertainties of measurements carried out in accordance with this part of ISO 6721 are detailed in annex A.

10 Test report

The test report shall include the following information:

a) a reference to this part of ISO 6721;

b) a test reference number;

c) the date of the test;

d) all details necessary to identify the operator;

e) all details necessary for identification of the material tested;

f) details of preparation and loading of the specimen between the plates;

g) a description of the rheometer used, including the plate diameter, plate separation and the material forming the plate surface;

h) the test temperature, in degrees Celsius;

i) the preheating time, in seconds;

j) details of the equilibrating time and any pre-shear conditioning;

k) the thermal-stability time, in seconds;

l) the test duration, in seconds;

m) for testing using the controlled-stress mode, the value of the stress at the edge of the plate, expressed in pascals;

   or

   for testing using the controlled-strain mode, the value of the strain at the edge of the plate, expressed as a dimensionless number;

n) any of the following, presented as functions of strain amplitude, angular frequency, temperature or time, as necessary:

   - the shear storage modulus G', in pascals,
   - the shear loss modulus G'', in pascals,
   - the complex shear modulus G^*, in pascals,
   - the dynamic shear viscosity η', in pascal seconds,
   - the out-of-phase component of the complex viscosity η'', in pascal seconds,
   - the complex viscosity η^*, in pascal seconds,
   - the phase shift or loss angle δ, in radians,
   - tan δ;

o) details of any corrections applied to the data;

p) any visual observations of melt fracture or degradation of the specimen;

q) any test conditions agreed on that deviate from this part of ISO 6721.

Annex A
(informative)

Uncertainty limits

Given that the storage and loss moduli are given by the expressions

$$G' = \frac{32 d T_0 \cos\delta}{\theta_0 \, \pi D^4} \tag{A.1}$$

and

$$G'' = \frac{32 d T_0 \sin\delta}{\theta_0 \, \pi D^4} \tag{A.2}$$

respectively, then the corresponding fractional uncertainties are given by the expressions

$$\underline{G'} = \left[\underline{d}^2 + \underline{T_0}^2 + (\underline{\cos\delta})^2 + \underline{\theta_0}^2 + 4\underline{D}^2 \right]^{0,5} \tag{A.3}$$

and

$$\underline{G''} = \left[\underline{d}^2 + \underline{T_0}^2 + (\underline{\sin\delta})^2 + \underline{\theta_0}^2 + 4\underline{D}^2 \right]^{0,5} \tag{A.4}$$

where the underlining indicates the fractional uncertainty in each of the terms. The fractional uncertainty is determined as the ratio of the uncertainty in the value of the term, due for example to its calibration and/or resolution, to the value of the term. For example, for the measurement of an angular displacement of 20×10^{-4} rad at, say, an accuracy of $\pm 20 \times 10^{-6}$ rad, the fractional uncertainty of the measurement is 0,01.

To include contributions due to the uncertainty in the measurement of angular frequency, temperature dependence and the effects of degradation and non-linear viscoelasticity, then the above expressions for the uncertainties in the measurement of G' and G'' can be rewritten as follows:

$$\underline{G'} = \left[\underline{d}^2 + \underline{T_0}^2 + (\underline{\cos\delta})^2 + \underline{\theta_0}^2 + 4\underline{D}^2 + \underline{F}^2 + \underline{\phi}^2 + \underline{\mathrm{DE}}^2 + \underline{\mathrm{SL}}^2 \right]^{0,5} \tag{A.5}$$

and

$$G'' = \left[\underline{d}^2 + \underline{T_0}^2 + (\underline{\sin\delta})^2 + \underline{\theta_0}^2 + 4\underline{D}^2 + \underline{F}^2 + \underline{\phi}^2 + \underline{\mathrm{DE}}^2 + \underline{\mathrm{SL}}^2 \right]^{0,5} \tag{A.6}$$

where

$\underline{F}$ is the fractional uncertainty due to the error in the angular frequency;

$\underline{\phi}$ is the fractional uncertainty due to the effect of temperature on the viscosity;

$\underline{\mathrm{DE}}$ is the fractional uncertainty due to degradation effects;

$\underline{\mathrm{SL}}$ is the fractional uncertainty due to the effect of non-linear-viscoelastic behaviour.

The fractional uncertainties in each of these parameters were determined using the values for each of the parameters and the tolerances specified below:

Plate diameter, D	25 mm
Specimen thickness, d	1,5 mm
Strain at edge of plate	5 %

Torque	± 2 % absolute
Angular displacement	$\pm 20 \times 10^{-6}$ rad
Plate diameter	$\pm 0{,}01$ mm
Plate gap	$\pm 0{,}01$ mm
Non-alignment of plates	$\pm 0{,}01$ mm
Angular frequency	± 2 % absolute
Temperature, θ	$\pm 0{,}5$ °C for $\theta \leqslant 200$ °C
	$\pm 1{,}0$ °C for 200 °C $< \theta \leqslant 300$ °C
	$\pm 1{,}5$ °C for $\theta > 300$ °C

In determining the uncertainty in measurements of G' and G'', it was also assumed that

- the terms $\cos\delta$ and $\sin\delta$ had a maximum uncertainty of 3 % for loss angles δ over the range 17° to 73° [4];
- the effect of a 2 % error in the determination of the angular frequency had a 2 % effect on the measured value of G' and G'', i.e. that $\log(G')$ and $\log(G'')$ were linear functions of log(angular frequency) with a slope of 1;
- degradation of the sample material and the error due to non-linear-viscoelastic behaviour (related to determining the limiting strain for linear-viscoelastic measurements) had either a 0 % or a 5 % effect on measured G' and G'' values representing the two extreme cases.

Given these assumptions, then the uncertainties in the measurement of G' and G'' for various values of the temperature dependence of the viscosity and the error in the temperature are given in Table A.1. For the PE-HD and PP samples used in the round robin [4], it was estimated, from temperature-run data, that the temperature dependence of the moduli was approximately 1 %/°C.

Table A.1 — Uncertainties in the measurement of the storage modulus G' and loss modulus G''

Temperature dependence of viscosity %/°C	Error in temperature ±°C	Uncertainty in measurement of G' and G'' %	
		Assuming 0 % effect for both degradation and non-linear-viscoelastic behaviour	**Assuming 5 % effect for both degradation and non-linear-viscoelastic behaviour**
0	—	4,2	8,2
1	0,5	4,2	8,2
1	1,0	4,3	8,3
3	1,0	5,2	8,8

These uncertainties do not take into account errors due to specimen inertia, and the instrument-related factors of non-parallelism and non-axial alignment of the plates, edge effects, machine inertia and machine compliance. These factors will all increase the level of uncertainty in the measurements.

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