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Plastics — Determination of dynamic mechanical properties —

Part 7: Torsional vibration — Non-resonance method

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 7: Vibration en torsion — Méthode hors résonance

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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 documents 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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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.

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

This second edition cancels and replaces the first edition (ISO 6721-7:1996), which has been technically revised. It also incorporates the Amendment ISO 6721-7:1996/Amd.1:2007. The main changes compared to the previous edition are as follows:

— the document has been revised editorially;

— normative references have been changed to undated.

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 7:
Torsional vibration — Non-resonance method

1 Scope

This document describes a torsional, non-resonance method for determining the components of the shear complex modulus G^* of solid polymers in the form of bars or rods at frequencies typically in the range 0,001 Hz to 100 Hz. Higher-frequency measurements can be made, but significant errors in the dynamic properties measured are likely to result (see 10.2.1 and 10.2.2). The method is suitable for measuring dynamic storage moduli ranging from about 10 MPa, which is typical of values obtained for stiff rubbers, to values of about 10 GPa which are representative of fibre-reinforced plastics. Although materials with moduli less than 10 MPa can be studied, more accurate measurements of their dynamic properties can be made using simple shear (see ISO 6721-6) or torsional deformations of thin layers between parallel plates.

This method is particularly suited to the measurement of loss factors greater than 0,02 and may therefore be conveniently used to study the variation of dynamic properties with temperature and frequency through most of the glass-rubber relaxation region (see ISO 6721-1). The availability of data determined over wide ranges of both frequency and temperature enable master plots to be derived, using frequency-temperature shift procedures, which display dynamic properties over an extended frequency range at different temperatures.

NOTE Although loss factors below 0,1 can be more accurately determined using the torsion pendulum (see ISO 6721-2), the method described in this document enables a much wider and continuous frequency range to be covered.

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 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 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 http://www.electropedia.org/

4 Principle

The specimen is subjected to a sinusoidal torque or angular displacement at a frequency significantly below the fundamental torsion resonance frequency (see 10.2.1). The amplitudes of the torque and displacement cycles applied to the specimen and the phase angle between these cycles are measured.

The storage and loss components of the shear complex modulus and the loss factor are calculated using formulae given in Clause 10.

5 Test device

5.1 Loading assembly

5.1.1 General

The requirements on the apparatus are that it shall permit measurements of the amplitudes of, and phase angle between, the torque and angular displacement cycles for a specimen subjected to a sinusoidal torque or displacement. Various designs of apparatus are possible, as illustrated schematically in Figures 1 a) and 1 b). In Figure 1 a), a sinusoidal angular displacement is generated by the drive unit D and applied to one end of the specimen S through the moving clamp C_1. The amplitude and frequency of the angular displacement are variable and monitored by the rotary displacement transducer R. The specimen is held at the opposite end by a fixed clamp C_2 and thus undergoes sinusoidal torsional deformations. The sinusoidal torque applied in deforming the specimen is monitored by a torque transducer T connected to C_2. The members between the clamp C_1 and D and between C_2 and T should be much stiffer than the specimen and should have a low thermal conductance if the specimen is to be enclosed in a temperature-controlled cabinet. Where tests are carried out at elevated temperatures, a facility shall be included in the loading assembly to avoid buckling of the specimen resulting from thermal expansion.

While each member of the loading assembly may have a much higher stiffness than the specimen, the presence of clamped or bolted connections can significantly increase the apparatus compliance. It may then be necessary to apply a compliance correction as described in 10.2.3.

Various other loading assemblies may be employed as alternatives to that detailed above. For example, the torque on the specimen may be calculated from the current supplied to the drive unit, thus eliminating the need for a separate torque transducer. With this method [Figure 1 b)], it should be recognized that part of the torque generated by the drive current is used to accelerate the drive shaft and also to deform any drive shaft suspension (Su) in parallel with the specimen. That part of the generated torque used to deform the specimen shall be determined with the aid of a separate calibration with the specimen absent. Alternatively, the suspension member may be replaced by an air bearing, thereby making the torsional rigidity of the suspension zero.

5.1.2 Clamps

The clamps shall be capable of gripping the test specimen with a force which is sufficient to prevent the specimen from slipping during the torsional deformation and maintaining the force at low temperatures.

The separation between the two clamps should preferably be variable so that specimens of different length can be accommodated and length corrections may be determined (see 10.2.4). A facility to permit small variations in the clamp separation would also allow for thermal expansion of the specimens and is necessary to avoid errors in the apparent moduli due to buckling of the specimens at high temperatures.

Any misalignment of the clamps with respect to the force transducer will produce a lateral component of the torque applied to the transducer during loading of the specimen. The alignment of the loading assembly and test specimen shall be such that any lateral component recorded by the transducer is less than 1 % of the applied torque.

5.1.3 Transducers

The term transducer in this document refers to any device capable of measuring the applied torque or displacement, or the ratio of these quantities, as a function of time. The calibrations of the transducers shall be traceable to national standards for the measurement of torque and length. The calibrations shall be accurate to ±2 % of the minimum torque and displacement cycle amplitudes applied to the specimen for the purpose of determining dynamic properties.

5.2 Electronic data-processing equipment

Data-processing equipment shall be capable of recording the torque and displacement cycle amplitudes to an accuracy of ±1 % the phase angle between the torque and displacement cycles to an accuracy of ±0,1° and the frequency to an accuracy of ±10 %.

5.3 Temperature measurement and control

According to ISO 6721-1.

5.4 Devices for measuring test specimen dimensions

According to ISO 6721-1.

6 Test specimens

6.1 General

According to ISO 6721-1.

6.2 Shape and dimensions

Test specimens in the form of rectangular Bars or cylindrical rods are recommended. The width and thickness of the bars and the diameter of the rods shall not vary along the specimen length by more than 2 % of a mean value.

Dimensions of the specimens are not critical, although length corrections for clamping effects can be minimized by increasing the length of the specimen and, for rectangular specimens, these corrections become negligible for certain values of b/h (see 10.1 and 10.2.4). For test conditions under which the storage moduli are high (≥1 GPa), sufficiently long, thin specimens shall be employed so that angular displacements are generated that may be measured with high accuracy. Alternatively, when the storage moduli are low (<100 MPa), relatively short, thick specimens may be required to achieve sufficient accuracy in the measurement of torque. A variation in dynamic properties may be observed between specimens of different thickness prepared by injection moulding owing to slight differences which may be present in the structure of the polymer in each specimen.

6.3 Preparation

According to ISO 6721-1.

7 Number of specimens

According to ISO 6721-1.

8 Conditioning

According to ISO 6721-1.

9 Procedure

9.1 Test atmosphere

According to ISO 6721-1.

9.2 Measuring the cross-section of the specimen

According to ISO 6721-1.

9.3 Clamping the specimen

Mount the specimen between the clamps using a clamping force that is sufficient to prevent slip under all test conditions. If measurements are observed to depend upon clamp pressure, then a constant pressure should preferably be used for all measurements, especially when applying a length correction (see 10.2.4).

If measurements are observed to depend upon clamp pressure, then the clamped area of the specimen is probably too small. A larger clamp face or a wider specimen should eliminate this problem.

9.4 Varying the temperature

According to ISO 6721-1.

9.5 Performing the test

A dynamic torque shall be applied by the drive motor which yields torque and displacement amplitudes for the specimen that can be measured to the accuracy specified in 5.1.3.

If the maximum shear strain within the specimen exceeds the limit for linear behaviour, then the derived dynamic properties will depend on the magnitude of the applied displacement. The limiting strain varies with the composition of the polymer and the temperature and is typically in the region of 0,2 % for glassy plastics. The dynamic strain range for linear behaviour can be explored by varying the dynamic displacement amplitude at a constant frequency and recording any change in dynamic stiffness with strain amplitude. A low frequency should be used for this purpose to minimize any temperature increase caused by mechanical loss. However, it should be noted that, because of the non-uniform strain in the specimen in this test, the onset of non-linear behaviour will be less apparent than in tests where the strain distribution is uniform. If non-linear behaviour is detected in the strain range of interest, the dynamic strain limit should be recorded in the test report.

The amplitudes of, the phase difference between and the frequency of the torque and displacement signals and the temperature of the test shall be recorded. Where measurements are to be made over ranges of frequency and temperature, it is recommended that the lowest temperature be selected first and measurements be made with increasing frequency, keeping the temperature constant. The frequency range is then repeated at the next higher temperature (see ISO 6721-1).

For test conditions under which the polymer exhibits medium or high loss (for example in the glass-rubber transition region), the energy dissipated by the polymer may raise its temperature sufficiently to give a significant change in dynamic properties. Any temperature rise will increase rapidly with increasing strain amplitude and frequency. If the data processing electronics is capable of analysing the transducer outputs within the first few cycles, then the influence of any temperature rise will be minimized. Subsequent measurements will then change with time as the specimen temperature continues to rise, and such observations will indicate the need to exercise some caution in the presentation and interpretation of results.

10 Expression of results

10.1 Symbols

L_a	length of specimen between the two clamps, in metres
l	length correction term for clamping, in metres
b	width of rectangular specimen, in metres
h	thickness of rectangular specimen, in metres
r	radius of cylindrical specimen, in metres
f	measurement frequency, in hertz
θ_A	measured amplitude of the dynamic angular displacement, in radians
T_A	measured amplitude of the dynamic torque applied to the specimen, in newton metres
δ_{Ga}, δ_G	measured phase difference and corrected phase difference, respectively, between the torque and angular-displacement cycles, in degrees
Γ_a, Γ	measured absolute value and corrected absolute value, respectively, of the torsional complex stiffness of the specimen, in newton metres per radian ($N \cdot m \cdot rad^{-1}$)
G'_a, G'	apparent shear storage modulus and corrected shear storage modulus, respectively, in pascals
G''	shear loss modulus, in pascals
$\tan \delta_{Ga}$, $\tan \delta_G$	apparent shear loss factor and corrected shear loss factor, respectively
κ	shape factor giving the ratio of torsional complex stiffness to shear complex modulus per unit length of specimen, in metres to the power four per radian ($m^4 \cdot rad^{-1}$)
I_p	polar second moment of area of the cross-section per unit specimen length, in metres to the power four (m^4)
Γ_T	torsional stiffness of the torque transducer, in newton metres per radian ($N \cdot m \cdot rad^{-1}$)
I_T	moment of inertia of that part of the loading assembly between the torque transducer and the test specimen, in kilogram square metres ($kg \cdot m^2$)
Γ_∞	measured torsional stiffness, in newton metres per radian ($N \cdot m \cdot rad^{-1}$), of a steel test specimen whose crosssectional dimensions and length are such that it is at least 100 times stiffer than the stiffest polymer specimen to be tested

NOTE The magnitude of Γ_∞ will give an estimate of the torsional stiffness of the loading assembly, which is equivalent to a spring connected in series with the specimen and will enable a correction for apparatus compliance to be deduced (see 10.2.3).

10.2 Calculation of the shear storage modulus G'

10.2.1 General

An approximate value for the storage modulus G'_a is determined from Formula (1).

$$G'_a = \frac{T_A}{\theta_A} \times \frac{L_a}{\kappa} \cos\delta_{Ga} = \Gamma_a \times \frac{L_a}{\kappa} \cos\delta_{Ga} \tag{1}$$

where the shape constant κ is calculated from the following formulae:

Rectangular bar

$$\kappa = \frac{bh^3}{3}\left(1 - 0{,}63\frac{h}{b}\right) \qquad \text{for } 0 < \frac{h}{b} < 0{,}6 \tag{2}$$

$$\kappa = \frac{bh^3}{3} \times \frac{0{,}843}{\left(1 + h^2/b^2\right)} \qquad \text{for } 0{,}6 \le \frac{h}{b} \le 1 \tag{3}$$

Cylindrical rod

$$\kappa = \frac{\pi r^4}{2} \tag{4}$$

10.2.2 Avoidance of specimen resonance

Formula (1) becomes invalid as the drive frequency approaches the fundamental torsional resonance frequency f_s of the specimen given approximately by

$$f_s = \frac{1}{2L_a}\left[\frac{\kappa G'_a}{\rho I_p}\right]^{1/2} \tag{5}$$

where ρ is the polymer density in kilograms per cubic metre and I_p is given by the following formulae:

Rectangular bar

$$I_p = \frac{bh}{12}\left(b^2 + h^2\right) \tag{6}$$

Cylindrical rod

$$I_p = \frac{\pi r^4}{2} \tag{7}$$

Errors in the use of Formula (1) become significant at applied frequencies such that

$$f \ge 0{,}08 f_s \tag{8}$$

Calculations of dynamic properties shall therefore be confined to frequencies below that given by the equality in Formula (8).

10.2.3 Correction for transducer resonance

At sufficiently high frequencies, the applied deformation will excite the torque transducer into resonance. The resonance frequency f_T is given by Formula (9).

$$f_T = \frac{1}{2\pi}\left[\frac{\Gamma_T}{I_T}\right]^{1/2} \tag{9}$$

The transducer output will have a significant error for all applied frequencies such that

$$f > 0{,}1 f_T \tag{10}$$

The resonance frequency f_T of the torque transducer and supported inertia member can be determined directly by recording the natural frequency of the transducer output after applying a torque impulse to the attached clamp without specimen.

The torsional stiffness of the specimen corrected for transducer resonance is given to a good approximation by Formula (11).

$$\Gamma = \Gamma_a\left[1 - \frac{4\pi I_T f^2}{\Gamma_T}\right] = \Gamma_a\left[1 - \frac{f^2}{f_T^2}\right] \tag{11}$$

It is recommended that Formulae (9) and (10) be used to select a torque transducer whose resonance frequency is above the frequency range for which a correction to the torque measurement is necessary.

10.2.4 Correction for apparatus compliance

If Γ_a exceeds 0,02 Γ_∞ the torsional compliance of the test assembly is not negligible and the measured angular displacement differs significantly from that of the specimen. The following correction shall then be applied:

$$\Gamma \cos\delta_G = \frac{\Gamma_a\left(\cos\delta_{Ga} - \Gamma_a / \Gamma_\infty\right)}{1 - 2\left(\Gamma_a / \Gamma_\infty\right)\cos\delta_{Ga}} \tag{12}$$

where δ_G is given by Formula (14). The value of $\Gamma \cos \delta_G$ obtained from Formula (12) shall be used in place of $\Gamma_a \cos \delta_{Ga}$ in Formula (1) to give a more accurate estimate for G'_a.

NOTE The compliance correction is not necessary if the displacement transducer is located so as to measure the relative angular displacement of the two clamps.

10.2.5 Application of a length correction

Using the measured clamp separation L_a for the specimen length in Formula (1) takes no account of some deformation of the specimen within the clamps or, in the case of rectangular bars, of restraints provided by the clamps on the out-of-plane warping of specimen cross-sections. These two effects may be allowed for by applying a small correction to L_a such that the effective length is $L_a + l$. Assuming that l is independent of L_a, Formula (1) yields

$$G' = \Gamma \times \frac{(L_a + l)}{\kappa}\cos\delta_G = G'_a \times \frac{(L_a + l)}{L_a} \tag{13}$$

Here G'_a is the apparent storage modulus corrected for apparatus compliance if necessary. A value for l may be determined from measurements of G'_a for a series of clamp separations L_a. From Formula (13), a plot of L_a/G'_a against L_a enables l to be determined from the intercept at $L_a/G'_a = 0$ and G' from the gradient.

NOTE The value of l will vary with the cross-sectional shape and dimensions of the specimen and with temperature if this causes significant changes in dynamic modulus. For rectangular specimens, the value of l can by either positive or negative depending on whether the effect of deformation within the clamps or of warping restraint is dominant. At a certain b/h ratio, which depends on the clamp design and other instrumental factors, these effects cancel to give a zero length correction.

10.3 Calculation of the shear loss factor tan δ_G

An approximate value for the shear loss factor is given by tan δ_{Ga}. If Γ_a exceeds 0,02 Γ_∞ the compliance of the loading assembly will influence the accuracy of the phase angle measurement. The loss factor shall then be obtained using Formula (14).

$$\tan\delta_G = \frac{\tan\delta_{Ga}}{1-\left[\left(\Gamma_a/\Gamma_\infty\right)\cos\delta_{Ga}\right]} \tag{14}$$

NOTE If the origin of the source of compliance in the loading assembly arises through clamped or bolted connections, there can be a contribution from friction to the measured phase angle δ_{Ga}. The magnitude of the resulting error increases with the ratio Γ_a/Γ_∞. This source of error can be avoided by locating the displacement transducer so that the relative displacement of the upper and lower clamps is measured.

10.4 Calculation of the shear loss modulus

The loss modulus G'' shall be calculated from Formula (15).

$$G'' = G'\tan\delta_G \tag{15}$$

10.5 Presentation of data as a function of temperature

According to ISO 6721-1.

11 Precision

The precision of this test method is not known because interlaboratory data are not available.

12 Test report

The test report shall include the information given in the test report of ISO 6721-1 plus the following:

a) reference to this document, i.e. ISO 6721-7;

b) the maximum dynamic strain amplitude, given approximately by $\theta_A h/L_a$ for rectangular bar specimens and by $\theta_A r/L_a$ for cylindrical rods.

