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Plastics — Determination of dynamic mechanical properties —

Part 6:
Shear vibration — Non-resonance method

Plastiques — Détermination des propriétés mécaniques dynamiques —
Partie 6: Vibration en cisaillement — Méthode hors résonance

Reference number
ISO 6721-6:1996(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 6721-6 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *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: Dynamic shear viscosity using a parallel-plate oscillatory rheometer*

International Organization for Standardization
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Plastics — Determination of dynamic mechanical properties —

Part 6:
Shear vibration — Non-resonance method

1 Scope

This part of ISO 6721 describes a forced, non-resonance method for determining the components of the shear complex modulus G^* of polymers at frequencies typically in the range 0,01 Hz to 100 Hz. The method is suitable for measuring dynamic storage moduli in the range 0,1 MPa to 50 MPa. Although materials with moduli greater than 50 MPa may be studied, more accurate measurements of their dynamic shear properties can be made using a torsional mode of deformation (see parts 2 and 7 of ISO 6721).

This method is particularly suited to the measurement of loss factors greater than 0,1 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 6721-1:1994, subclause 9.4). The availability of data determined over wide ranges of both frequency and temperature enables master plots to be derived, using frequency/temperature shift procedures, which display dynamic properties over an extended frequency range at different temperatures.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 6721. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 6721 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 6721-2:1994, *Plastics — Determination of dynamic mechanical properties — Part 2: Torsion-pendulum method.*

ISO 6721-7:1996, *Plastics — Determination of dynamic mechanical properties — Part 7: Torsional vibration — Non-resonance method.*

3 Definitions

See ISO 6721-1:1994, clause 3.

4 Principle

A test-specimen assembly is subjected to a sinusoidal shear force or deformation at a frequency significantly below the fundamental shear resonance frequency (see 10.2.1). The amplitudes of the force and displacement cycles applied to the test-specimen assembly 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 equations given in clause 10 of this part of ISO 6721.

5 Apparatus

5.1 Loading assembly

The requirements for the loading assembly are that it shall permit measurements of the amplitudes of, and phase angle between, the force and displacement cycles for a test-specimen assembly subjected to a sinusoidal shear force or deformation. Various designs of apparatus are possible: a suitable version is shown schematically in figure 1. The shear test-specimen assembly consists of two identical specimens S of the polymer bonded to metal end-pieces P_1 and P_2. A sinusoidal force is generated by the vibrator V and applied to the two outer end-pieces P_1 of the test-specimen assembly through the clamping device C_1 of the shear load stage. The amplitude and frequency

of the vibrator table displacement are variable and monitored by the transducer D. The test-specimen assembly is held at its centre P_2 by a fixed clamp C_2, and thus each specimen S of the polymer is subjected to simple shear deformations of equal magnitude. The sinusoidal force applied in deforming the test-specimen assembly is monitored by a force transducer F connected to C_2. The members between the clamps C_1 and V, and between C_2 and F, shall be much stiffer than the test-specimen assembly and shall have a low thermal conductance if the test-specimen assembly is to be enclosed in a temperature-controlled cabinet.

NOTE 1 While each member of the loading assembly may have a much higher stiffness than the test-specimen assembly, 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 section 10.2.3.

A clamping arrangement may be used in which a single specimen of the polymer is subjected to a simple shear deformation, but precautions shall then be taken to ensure that any torque in the loading assembly resulting from the application of load to the specimen does not influence the measurements of the dynamic shear force and displacement. Measurements of the deformation of the specimen may also be made by locating the displacement transducer so as to measure the relative displacement of the two parts C_1 and C_2 of the load stage. The magnitude of the correction for the compliance of the loading assembly will then become small or negligible (see 10.2.3).

5.1.1 Load stage

The shear load stage shall be capable of gripping the test-specimen assembly with sufficient force to prevent any relative movement between the metal blocks P of the test-specimen assembly and the load stage clamps, and to maintain the force at low temperatures. Any misalignment of the load stage with respect to the force transducer will produce a lateral component of the force applied to the transducer during loading of the test-specimen assembly. The alignment of the loading assembly and test-specimen assembly shall be such that any lateral component recorded by the transducer is less than 1 % of the applied longitudinal force.

5.1.2 Transducers

The term transducer in this part of ISO 6721 refers to any device capable of measuring the applied force or displacement, or the ratio of these quantities, as a function of time. The calibration of the transducers shall be traceable to national standards for the measurement of force and length. The calibration shall be accurate to ± 2 % of the minimum force and displacement cycle amplitudes applied to the test-specimen assembly for the purpose of determining dynamic properties.

5.2 Electronic data-processing equipment

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

5.3 Temperature measurement and control

See ISO 6721-1:1994, subclauses 5.3 and 5.5.

5.4 Devices for measuring test specimen dimensions

See ISO 6721-1:1994, subclause 5.6.

6 Test assembly

See ISO 6721-1:1994, clause 6.

6.1 Shape and dimensions

A suitable design for the shear test-specimen assembly is shown in figure 2. Here the metal end-pieces P are cylindrical, but any cross-sectional shape is suitable as long as the end-pieces can be clamped rigidly in the shear load stage. The dimensions of the end-pieces and the polymer specimens S shall be chosen such that the deformation of the end-pieces under an applied load is negligible in comparison with that of the specimens. For a polymer whose shear modulus is less than 100 MPa, this will mean that the thickness of the end-pieces may be comparable with the thickness L of the specimens.

The cross-sectional shape of the polymer specimens in the plane of their bonded faces is not critical, although a rectangular section is recommended in order to simplify the application of a term representing the contribution to the specimen deformation from bending — see equation (1) in 10.2. The specimens may then be conveniently cut from a sheet of the polymer and bonded to the end-pieces to construct the shear test-specimen assembly. The dimensions of each polymer specimen shall not vary by more than 3 % of the mean value. This dimension shall be sufficiently large to allow adequate accuracy to be achieved in the determination of dynamic strain and hence dynamic moduli — see equation (1) in 10.2. In addition, it is recommended that the dimension h of the polymer in the direction of the applied load should be greater than $4L$ in order to make the correction for bending negligible.

NOTE 2 A variation in dynamic properties may be observed between specimens of different thickness prepared by injection moulding owing to differences which may be present in the structure of the polymer in each specimen.

6.2 Preparation of polymer specimens

See ISO 6721-1:1994, subclause 6.2.

7 Number of test assemblies

See ISO 6721-1:1994, clause 7, reading "test-specimen assemblies" for "test specimens".

8 Conditioning

See ISO 6721-1:1994, clause 8.

9 Procedure

9.1 Test atmosphere

See ISO 6721-1:1994, subclause 9.1.

9.2 Measuring the cross-section of the polymer specimen

See ISO 6721-1:1994, subclause 9.2.

9.3 Clamping the test assembly

Mount the test-specimen assembly in the load stage using a clamping force that is sufficient to prevent relative movement between each clamp and the associated end-piece under all test conditions.

9.4 Varying the temperature

See ISO 6721-1:1994, subclause 9.4.

9.5 Performing the test

Apply to the shear test-specimen assembly a dynamic force which yields force and displacement signal amplitudes which can be measured by the transducers to the accuracy specified in 5.1.2.

NOTE 3 If the shear strain exceeds the limit for linear behaviour, then the derived dynamic properties will depend on the magnitude of the applied strain. This limit varies with the composition of the polymer and the temperature, and is typically in the region of 0,2 % for glassy plastics.

Record the amplitudes of, the phase difference between and the frequencies of the force and displacement signals, as well as the temperature of the test. Where measurements are to be made over ranges of frequency and temperature, it is recommended that the lowest temperature be selected first and measurements made with increasing frequency, keeping the temperature constant. The frequency range is then repeated at the next higher temperature (see ISO 6721-1:1994, subclause 9.4).

For those 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

A	bonded area, in square metres, of the specimens
f	measurement frequency, in hertz
f_F	resonance frequency, in hertz, of the force transducer
f_S	resonance frequency, in hertz, of the test-specimen assembly
G'_a, G'	apparent and corrected shear storage modulus, in pascals
G''	shear loss modulus, in pascals
h	mean of the specimen heights, in metres, in the direction of the applied load
k_a, k	measured and corrected magnitude, in newtons per metre, of the complex stiffness of the test-specimen assembly
k_F	stiffness of the force transducer, in newtons per metre
k_∞	measured stiffness, in newtons per metre, of a metal bar whose cross-sectional dimensions are the same as those of the end-pieces of the shear test-specimen assembly (see note 4). This bar shall be at least 100 times stiffer than the stiffest polymer specimen to be tested.
L	mean of dimension, in metres, of each polymer specimen between bonded faces

m_F mass, in kilograms, of that part of the loading assembly between the force transducer and the test-specimen assembly

s_A measured amplitude, in metres, of the dynamic displacement

$\tan \delta_{Ga}$, $\tan \delta_G$ apparent and corrected shear loss factor

δ_{Ga}, δ_G measured and corrected phase difference, in degrees, between the force and displacement cycles

ΔF_A measured amplitude, in newtons, of the dynamic force

NOTE 4 The magnitude of k_∞ will give an estimate of the stiffness of the loading assembly which is equivalent to a spring connected in series with the shear test-specimen assembly and will enable a correction for apparatus compliance to be deduced (see 10.2.3).

10.2 Calculation of the shear storage modulus G'

An approximate value for the shear storage modulus G'_a, is determined from the equation:

$$G'_a = \frac{\Delta F_A}{s_A} \times \frac{L}{A} \times \left[1 + \frac{L^2}{h^2} \times \frac{G'}{E'}\right] \cos \delta_{Ga}$$

$$= \frac{k_a L}{A} \left[1 + \frac{L^2}{h^2} \times \frac{G'}{E'}\right] \cos \delta_{Ga} \quad \ldots (1)$$

The term in square brackets accounts for a contribution from bending to the deformation of the specimen. Values for G'/E' typically range from 0,37 for isotropic glassy or semicrystalline polymers to 0,33 for rubbers.

10.2.1 Avoidance of test assembly resonance

Equation (1) becomes invalid as the drive frequency approaches the fundamental shear resonance frequency f_S of the test-specimen assembly given approximately by

$$f_s = \frac{1}{2L}\left(\frac{G'_a}{\rho}\right)^{1/2} \quad \ldots (2)$$

where ρ is the polymer density in kilograms per cubic metre.

An error in the use of equation (1) becomes significant at applied frequencies such that

$$f \geqslant \frac{0{,}04}{L}\left(\frac{G'_a}{\rho}\right)^{1/2} = 0{,}08 f_s \quad \ldots (3)$$

Calculations of dynamic properties shall therefore be confined to frequencies below $0{,}08 f_S$.

10.2.2 Correction for transducer resonance

At sufficiently high frequencies, the applied deformation will excite the force transducer into resonance. The resonance frequency f_F is given by

$$f_F = \frac{1}{2\pi}\left(\frac{k_F}{m_F}\right)^{1/2} \quad \ldots (4)$$

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

$$f > 0{,}1 f_F \quad \ldots (5)$$

The resonance frequency f_F of the force transducer and supported mass m_F can be determined directly by recording the natural frequency of the transducer output after striking the attached clamp without the test-specimen assembly.

The test-specimen assembly stiffness corrected for transducer resonance is given to a good approximation by the equation

$$k = k_a\left(1 - \frac{4\pi^2 m_F f^2}{k_F}\right) = k_a\left(1 - \frac{f^2}{f_F^2}\right) \quad \ldots (6)$$

It is recommended that equations (4) and (5) be used to select a force transducer whose resonance frequency is above the frequency range for which a correction to the force measurement is necessary.

10.2.3 Correction for apparatus compliance

If k_a is greater than $0{,}02 k_\infty$, then the compliance of the test-specimen assembly is not negligible and the measured displacement differs significantly from that of the assembly. The following correction shall then be applied:

$$k \cos \delta_G = \frac{k_a(\cos \delta_{Ga} - k_a/k_\infty)}{1 - 2(k_a/k_\infty)\cos \delta_{Ga}} \quad \ldots (7)$$

where δ_G is given by equation (8).

The value of $k \cos \delta_G$ obtained from equation (7) shall be used in place of $k_a \cos \delta_{Ga}$ in equation (1) to give a more accurate estimate for G'_a.

NOTE 5 The compliance correction is unnecessary if the displacement transducer is located so as to measure the relative displacement of the two parts of the shear stage.

10.3 Calculation of the shear loss factor tan δ_G

An approximate value for the shear loss factor is tan δ_{Ga}.

If k_a is greater than $0{,}02k_\infty$ then the compliance of the loading assembly will influence the accuracy of the phase angle measurement. The loss factor shall then be obtained using the equation

$$\tan \delta_G = \frac{\tan \delta_{Ga}}{1-(k_a/k_\infty \cos \delta_{Ga})} \quad \ldots (8)$$

NOTE 6 If the origin of the source of compliance in the loading assembly arises through clamped or bolted connections, there may be a contribution from friction to the measured phase angle δ_{Ga}. The magnitude of the resulting error increases with the ratio k_a/k_∞. This source of error can be avoided by locating the displacement transducer so that the relative displacement of the two parts of the load stage is measured.

10.4 Calculation of the shear loss modulus

Calculate the loss modulus G'' from the equation

$$G'' = G' \tan \delta_G \quad \ldots (9)$$

10.5 Presentation of data as a function of temperature

See ISO 6721-1:1994, subclause 9.4.

11 Precision

The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added at the following revision.

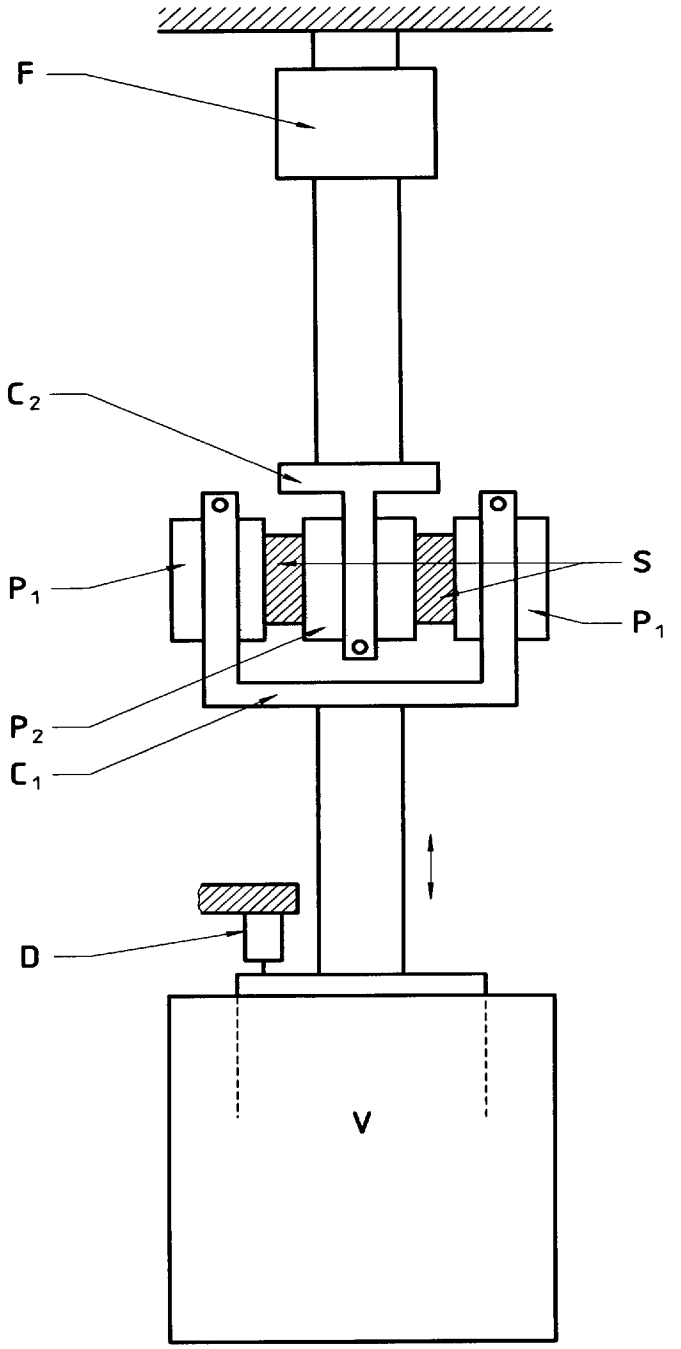
12 Test report

The test report shall contain the following information:

a) a reference to this part of ISO 6721;

b) to m) see ISO 6721-1:1994, clause 12;

n) the dynamic shear strain amplitude, given approximately by s_A/L.



Key:

C_1, C_2	Clamps	P_1, P_2	Metal end-pieces
D	Displacement transducer	S	Polymer specimens
F	Force transducer	V	Vibrator

Figure 1 — Schematic diagram of a suitable loading assembly for determining dynamic moduli by a shear-forced non-resonance method

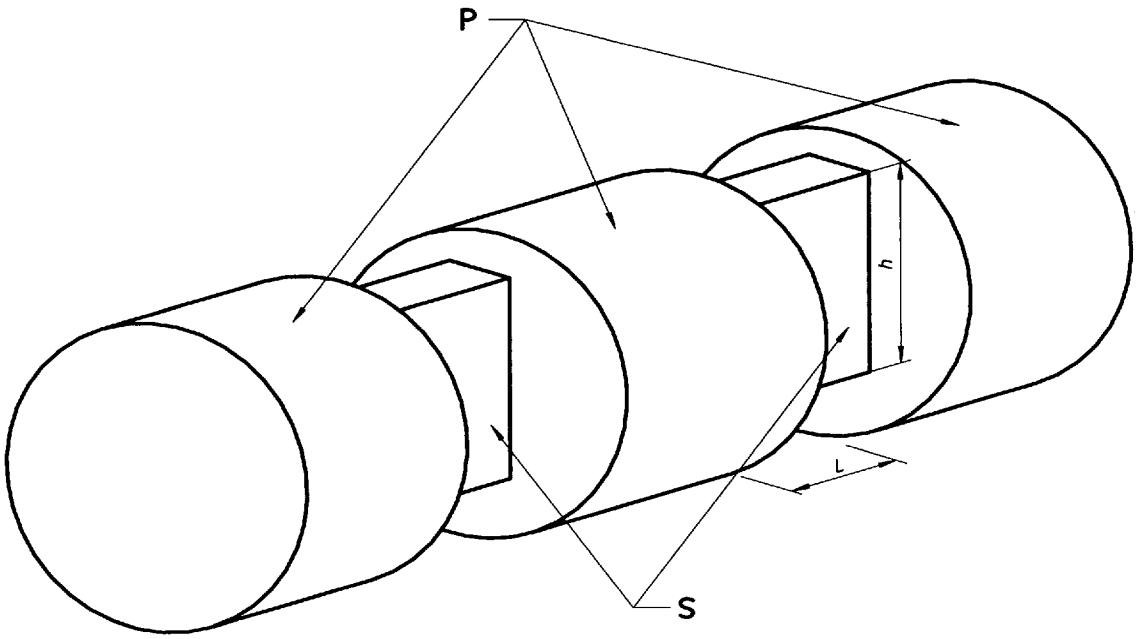


Figure 2 — Shear test-specimen assembly suitable for use with the apparatus shown in figure 1
(Specimens of the polymer S are bonded to metal end-pieces P which are clamped in the shear load stage. L and h are the width and height, respectively, of each polymer specimen.)

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