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Plastics — Determination of dynamic mechanical properties —

Part 4:
Tensile vibration — Non-resonance method

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 4: Vibration en traction — Méthode hors résonance

Reference number
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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 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 6721-4 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

This second edition cancels and replaces the first edition (ISO 6721-4:1994), of which it constitutes a minor revision. The main change is the updating of the normative references.

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*

Plastics — Determination of dynamic mechanical properties —

Part 4:
Tensile vibration — Non-resonance method

1 Scope

This part of ISO 6721 describes a forced, non-resonance method for determining the components of the tensile complex modulus E^* 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 0,01 GPa to 5 GPa. Although materials with moduli outside this range may be studied, alternative modes of deformation should yield higher accuracy [i.e. a shear mode for $E' <$ 0,01 GPa (see ISO 6721-6) and a flexural mode for $E' >$ 5 GPa (see ISO 6721-3 or ISO 6721-5)].

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 6721-1:2001, 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 referenced documents are indispensable for the application 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 6721-1:2001, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles*

ISO 6721-3, *Plastics — Determination of dynamic mechanical properties — Part 3: Flexural vibration — Resonance-curve method*

ISO 6721-5, *Plastics — Determination of dynamic mechanical properties — Part 5: Flexural vibration — Non-resonance method*

ISO 6721-6, *Plastics — Determination of dynamic mechanical properties — Part 6: Shear vibration — Non-resonance method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6721-1:2001, Clause 3, apply.

4 Principle

The specimen is subjected to a sinusoidal tensile force or deformation at a frequency significantly below the fundamental resonance frequency for the clamped/free longitudinal mode (see 10.2.2). The amplitudes of the force and displacement cycles applied to the specimen and the phase angle between these cycles are measured. The storage and loss factor are calculated using equations 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 the phase angle between, the force and displacement cycles for a specimen subjected to a sinusoidal tensile force or deformation. Various designs of apparatus are possible: a suitable version is shown schematically in Figure 1. A sinusoidal force is generated by the vibrator V and applied to one end of the specimen S by means of the clamp C_1. The amplitude and frequency of the vibrator table displacement are variable and monitored by the transducer D. The member between V and C_1 shall be much stiffer than the specimen and shall have a low thermal conductance if the specimen is to be enclosed in a temperature-controlled cabinet.

NOTE Whilst each member of the load 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 10.2.4.

At the other end of the specimen, a second clamp C_2 is connected to a force transducer F which is supported by a rigid frame. The member between C_2 and F shall also have sufficient stiffness and low thermal conductance.

5.1.2 Clamps

The clamps shall be capable of gripping the test specimen with sufficient force to prevent the specimen from slipping during the tensile deformation and maintaining the force at low temperatures. Any misalignment of the clamps with respect to the force transducer will produce a lateral component of the force 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 tensile force. A clamp design with self-aligning faces is recommended since this will maintain alignment of the specimen axis with the axis of the load assembly independently of specimen thickness.

The derivation of a length correction (see 10.2.5) requires measurements of specimen stiffness for different values of the specimen length as defined by the clamp separation. These may be made on a single specimen if one of the clamps has a hole in the centre of its base through which the specimen may pass as the clamp separation is reduced.

5.1.3 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 calibrations of the transducers shall be traceable to national standards for the measurement of force and length. The calibrations shall be accurate to ± 2 % of the minimum force and displacement cycle amplitudes applied to the specimen for the purpose of determining dynamic properties.

5.2 Electronic data-processing equipment

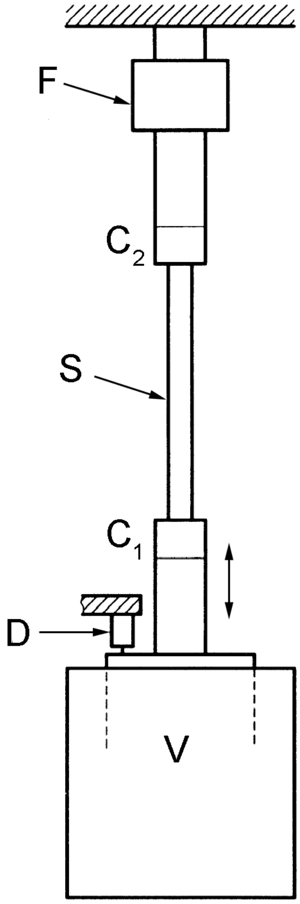
Data-processing equipment shall be capable of recording the force and displacement cycle amplitudes to an accuracy of ± 1 %, the phase angle between the force and displacement cycles to an accuracy of ± 0,1° and the frequency to an accuracy of ± 10 %.

5.3 Temperature measurement and control

See ISO 6721-1:2001, Subclauses 5.3 and 5.5.

5.4 Devices for measuring test specimen dimensions

See ISO 6721-1:2001, Subclause 5.6.



Key

F	force transducer
C_1, C_2	clamps
S	test specimen
D	displacement transducer
V	vibrator

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

6 Test specimens

6.1 General

See ISO 6721-1:2001, Clause 6.

6.2 Shape and dimensions

Test specimens of rectangular cross-section are recommended to facilitate load introduction. The width and thickness shall not vary along the specimen length by more than 3 % of the mean value. Where high accuracy in results is required, a specimen length is recommended which will permit a clamp separation of about 100 mm or more in order to achieve adequate accuracy in the determination of the dynamic tensile strain. It is also recommended that the length of the specimen between the clamps be greater than six times the specimen width in order to make the constraint by the clamps to free lateral contraction of the specimen negligible.

Cross-sectional dimensions are not critical. For test conditions under which the polymer exhibits glassy behaviour, the cross-sectional area shall be selected sufficiently small so that the vibrator is able to generate tensile displacements that may be measured with adequate accuracy. Alternatively, when the polymer exhibits rubbery behaviour, a larger cross-sectional area may be necessary to achieve sufficient accuracy in the measurement of force.

NOTE 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.

6.3 Preparation

See ISO 6721-1:2001, Subclause 6.3.

7 Number of specimens

See ISO 6721-1:2001, Clause 7.

8 Conditioning

See ISO 6721-1:2001, Clause 8.

9 Procedure

9.1 Test atmosphere

See ISO 6721-1:2001, Subclause 9.1.

9.2 Measurement of specimen cross-section

See ISO 6721-1:2001, Subclause 9.2.

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 10.2.5).

NOTE 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

See ISO 6721-1:2001, Subclause 9.4.

9.5 Performing the test

A static tensile force shall be applied to the specimen that is sufficient to prevent buckling under the decreasing part of the superimposed dynamic load. A dynamic force shall then be applied which yields force and displacement signal amplitudes which can be measured by the transducers to the accuracy specified in 5.1.3.

NOTE If the tensile 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.

The amplitudes of, the phase difference between and the frequency of the force 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 6721-1:2001, 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 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 the specimen between clamps, in metres
l	length correction term, in metres
b	specimen width, in metres
d	specimen thickness, in metres
f	measurement frequency, in hertz
s_A	measured amplitude of the dynamic displacement, in metres
ΔF_A	measured amplitude of the dynamic force, in newtons
δ_{Ea}, δ_E	measured phase difference and corrected phase difference, respectively, between the force and displacement cycles, in degrees
k_a, k	measured magnitude and corrected magnitude, respectively, of the complex stiffness of the specimen, in newtons per metre
E'_a, E'	apparent tensile storage modulus and corrected tensile storage modulus, respectively, in pascals
E''	tensile loss modulus, in pascals
$\tan\delta_{Ea}$, $\tan\delta_E$	apparent tensile loss factor and corrected tensile loss factor, respectively
k_F	stiffness of the force transducer, in newtons per metre

m_F	mass of that part of the loading assembly between the force transducer and the test specimen, in kilograms
k_∞	measured stiffness, in newtons per metre, of a steel test specimen whose cross-sectional dimensions are the maximum that the clamps can accommodate (see Note). This specimen shall be at least 100 times stiffer than the stiffest polymer specimen to be tested

NOTE 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 specimen, and will enable a correction for apparatus compliance to be deduced (see 10.2.4).

10.2 Calculation of the tensile storage modulus E'

10.2.1 General

An approximate value for the tensile storage modulus E'_a is determined from the equation

$$E'_a = \frac{\Delta F_A}{s_A} \times \frac{L_a}{bd} \cos \delta_{Ea} = \frac{k_a L_a}{bd} \cos \delta_{Ea} \tag{1}$$

10.2.2 Avoidance of specimen resonance

Equation (1) becomes invalid as the drive frequency approaches the fundamental longitudinal resonance frequency f_s of the specimen, given approximately by

$$f_s = \frac{1}{2L_a} \left(\frac{E'_a}{\rho} \right)^{1/2} \tag{2}$$

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

$$f \geqslant \frac{0{,}02}{L_a} \left(\frac{E'_a}{\rho} \right)^{1/2} \tag{3}$$

Calculations of dynamic properties shall therefore be confined to frequencies below that given by the equality in Equation (3).

10.2.3 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

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

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

$$f > 0{,}1\, f_F \tag{5}$$

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 specimen.

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

$$k = k_\mathrm{a}\left(1 - \frac{4\pi^2 m_\mathrm{F} f^2}{k_\mathrm{F}}\right) = k_\mathrm{a}\left(1 - \frac{f^2}{f_\mathrm{F}^2}\right) \tag{6}$$

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.

10.2.4 Correction for apparatus compliance

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

$$k \cos\delta_\mathrm{E} = \frac{k_\mathrm{a}\left(\cos\delta_\mathrm{Ea} - k_\mathrm{a}/k_\infty\right)}{1 - 2\left(k_\mathrm{a}/k_\infty\right)\cos\delta_\mathrm{Ea}} \tag{7}$$

where δ_E is given by Equation (10). The value of $k\cos\delta_\mathrm{E}$ obtained from Equation (7) shall be used in place of $k_\mathrm{a}\cos\delta_\mathrm{Ea}$ in Equation (1) to give a more accurate estimate for E'_a.

NOTE The compliance correction is unnecessary if the displacement transducer is located so as to measure the change in clamp separation or if extensometers are attached to the specimen.

10.2.5 Application of a length correction

Using the measured clamp separation L_a for the specimen length in Equation (1) takes no account of some distortion of the specimen within and around the clamp. Applying a small correction to L_a such that the effective length is $L_\mathrm{a} + l$ and assuming l is independent of L_a yields from Equation (1)

$$E' = \frac{k\left(L_\mathrm{a} + l\right)}{bd}\cos\delta_\mathrm{Ea} \tag{8}$$

where a correction for apparatus compliance has been applied where necessary using Equation (7). A value for the length correction l may be determined from measurements of specimen stiffness k for a series of different clamp separations L_a. From Equation (8), a plot of $1/(k\cos\delta_\mathrm{Ea})$ against L_a enables l to be determined from the intercept at $1/(k\cos\delta_\mathrm{Ea}) = 0$ and E' from the gradient.

NOTE 1 The value for l will vary with the cross-sectional dimensions of the specimen and with temperature if this causes significant changes in dynamic modulus.

NOTE 2 The derivation of a length correction is unnecessary if the dynamic strain is measured using extensometers attached to the specimen.

10.3 Calculation of the tensile loss factor tanδ_E

An approximate value for the tensile loss factor is tanδ_Ea.

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

$$\tan\delta_\mathrm{E} = \frac{\tan\delta_\mathrm{Ea}}{1 - \left(\dfrac{k_\mathrm{a}}{k_\infty \cos\delta_\mathrm{Ea}}\right)} \tag{9}$$

NOTE 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_Ea. 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 change in the clamp separation is measured or by attaching extensometers to the specimen.

10.4 Calculation of the tensile loss modulus

The loss modulus E'' shall be calculated from

$$E'' = E' \tan \delta_{\mathrm{E}} \tag{10}$$

10.5 Varying the temperature

See ISO 6721-1:2001, 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 include the following information:

a) a reference to this part of ISO 6721;

b) to m) see ISO 6721-1:2001, Clause 12;

n) the dynamic strain amplitude given approximately by $s_{\mathrm{A}}/L_{\mathrm{a}}$.

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