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Plastics — Determination of dynamic mechanical properties —

Part 5: Flexural vibration — Non-resonance method

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 5: Vibration en flexion — 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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 6721-5:1996), which has been technically revised. It also incorporates the Amendment ISO 6721-5:1996/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 5:
Flexural vibration — Non-resonance method

1 Scope

This document describes a flexural, non-resonance method for determining the components of the flexural complex modulus $E_f{}^*$ of polymers at frequencies typically in the range 0,01 Hz to 100 Hz. Higher-frequency measurements can be made, but significant errors in the dynamic properties measured are likely to result (see 10.2.2 and 10.2.3). The method is suitable for measuring dynamic storage moduli in the range 10 MPa to 200 GPa.

NOTE Although materials with moduli less than 10 MPa can be studied, more accurate measurements of their dynamic-mechanical properties can be made using shear modes of deformation (see ISO 6721-6).

This method is particularly suited to the measurement of loss factors greater than 0,02 and can 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 enables master plots to be derived, using frequency/temperature shift procedures, which present dynamic properties over an extended frequency range at different temperatures.

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

A test specimen is subjected to a sinusoidal transverse force or displacement at a frequency significantly below the fundamental flexural resonance frequency (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 components of the flexural complex modulus and the loss factor are calculated using formulae given in Clause 10.

5 Apparatus

5.1 Loading assembly

5.1.1 General

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 specimen subjected to a transverse sinusoidal force or displacement. Various designs of apparatus are possible, two of them are illustrated schematically in Figures 1 and 2. In Figure 1 a), a sinusoidal displacement is generated by the vibrator V and applied to the specimen S through moving clamps C_1 located close to the opposite ends of the specimen. The amplitude and frequency of the vibrator table displacement are variable and monitored by the transducer D. The specimen is held at its centre by a fixed clamp C_2 and thus undergoes sinusoidal flexural deformations. The sinusoidal force applied in deforming the specimen 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 specimen and shall have a low thermal conductance if the specimen is to be enclosed in a temperature-controlled cabinet.

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 10.2.4.

Various other loading assemblies may be employed as alternatives to that detailed above. For example, the specimen may be simply supported and deformed in three-point flexure, as illustrated in Figure 1 b). Furthermore, the force on the specimen may be calculated from the current supplied to the vibrator, thus eliminating the need for a separate force transducer. With this method (see Figure 2), that part of the force generated by the vibrator current is used to accelerate the drive shaft and to deform the drive-shaft suspension Su in parallel with the specimen. That part of the generated force used to deform the specimen shall be determined with the aid of a separate calibration with the specimen absent.

5.1.2 Load stage

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

With the simply supported specimen [Figure 1 b)], the supports (rollers or fixed round supports) shall contact the specimen along parallel lines and have radii sufficiently large to avoid significant indentation of the specimen and thereby minimize consequent errors in the measured moduli and loss factors.

The separation between the two outer clamps and between the outer supports shall be variable so that specimens of different length can be accommodated and length corrections may be determined for the clamped specimens (see 10.2.5). A facility to permit small variations in the clamp separation [Figure 1 a)] 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 load stage 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 longitudinal force.

5.1.3 Transducers

The term transducer in this document 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 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

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 of rectangular cross-section are recommended to facilitate load introduction. The width and thickness shall not vary along the specimen length by more than 2 % of the mean value.

The dimensions of the specimens are not critical although, for isotropic materials, values of $L_a/h > 16$ for clamped specimens and $L_a/h > 8$ for simply supported specimens would make corrections for shear deformation negligible (see 10.1 and 10.2). Also, values of $L_a/b > 6$ for clamped specimens and $L_a/b > 3$ for simply supported specimens are recommended to avoid significant errors associated with constraints to deformations along the width direction (anticlastic curvature) near to the clamps or central support (see 10.1).

For test conditions under which the storage moduli are high (≥50 GPa), sufficiently long, thin specimens shall be employed so that 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 force.

NOTE A variation in dynamic properties can be observed between specimens of different thickness prepared by injection moulding owing to differences which can 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 shall be used for all measurements, especially when applying a length correction (see 10.2.5 and Note).

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 can eliminate this problem. For measurements with a sub-ambient starting temperature, it can help to fix the sample at room temperature just loosely and tighten it in the cold.

9.4 Varying the temperature

According to ISO 6721-1.

9.5 Performing the test

Apply, by means of the vibrator, a dynamic force which yields force and displacement signal amplitudes for the specimen that can be measured to the accuracy specified in 5.1.3. For simply supported specimens, also apply a static force that is sufficient to maintain the load under the decreasing part of the superimposed dynamic load.

If the maximum tensile 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.

Record the amplitudes of, the phase difference between and the frequency 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).

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 equipement 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

b	specimen width, in metres
h	specimen thickness, in metres
$E_f{}'_a$, $E_f{}'$	apparent and corrected flexural storage modulus, in pascals
E''_f	flexural loss modulus, in pascals
f	measurement frequency, in hertz
f_F	resonance frequency of the force transducer, in hertz
f_s	resonance frequency of the specimen, in hertz
G'	shear storage modulus, in pascals
k_a, k	measured and corrected magnitude of the complex stiffness of the specimen, in newtons per metre
k_F	stiffness of the force transducer, in newtons per metre
k_∞	measured stiffness of a steel test specimen whose cross-sectional dimensions and length are such that it is at least 100 times stiffer than the stiffest polymer specimen to be tested (see Note), in newtons per metre
l	length correction term, in metres, to allow for clamping
L_a	(for a clamped specimen) length of specimen between the central clamp and each outer clamp, in metres
	(for a simply supported specimen) length of specimen between the central loading line and each outer clamp support, in metres
m_F	mass of that part of the loading assembly between the force transducer and the test specimen, in kilograms
s_A	measured amplitude of the dynamic displacement, in metres
$\tan\delta_{E_{fa}}$, $\tan\delta_{E_f}$	apparent and corrected flexural loss factor
$\delta_{E_{fa}}$, δ_{E_f}	measured and corrected phase difference between the force and displacement cycles, in degrees
ΔF_A	measured amplitude of the dynamic force applied to the specimen, in newtons

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 flexural storage modulus $E_f{}'$

10.2.1 General

An approximate value $E_f{}'_a$ for the storage modulus is determined from Formulae (1) and (2):

Clamped specimen

$$E_{f}{'}_{\mathrm{a}} = \frac{\Delta F_{\mathrm{A}}}{s_{\mathrm{A}}} \times \frac{L_{\mathrm{a}}^{3}}{2bh^{3}} \times \left[1 + \frac{h^{2}}{L_{\mathrm{a}}^{2}} \times \frac{E_{f}{'}}{G'}\right] \cos\delta_{E_{f\mathrm{a}}} = k_{\mathrm{a}} \times \frac{L_{\mathrm{a}}^{3}}{2bh^{3}} \times \left[1 + \frac{h^{2}}{L_{\mathrm{a}}^{2}} \times \frac{E_{f}{'}}{G'}\right] \cos\delta_{E_{f\mathrm{a}}} \quad (1)$$

Simply supported specimen

$$E_{f}{'}_{\mathrm{a}} = \frac{\Delta F_{\mathrm{A}}}{s_{\mathrm{A}}} \times \frac{2L_{\mathrm{a}}^{3}}{bh^{3}} \times \left[1 + \frac{h^{2}}{4L_{\mathrm{a}}^{2}} \times \frac{E_{f}{'}}{G'}\right] \cos\delta_{E_{f\mathrm{a}}} = k_{\mathrm{a}} \times \frac{2L_{\mathrm{a}}^{3}}{bh^{3}} \times \left[1 + \frac{h^{2}}{4L_{\mathrm{a}}^{2}} \times \frac{E_{f}{'}}{G'}\right] \cos\delta_{E_{f\mathrm{a}}} \quad (2)$$

In these formulae, the terms in square brackets account approximately for the effects of shear deformation during the flexure. Values for E_f'/G' typically range from 2,7 for isotropic glassy or semi-crystalline polymers to 3,0 for rubbers. Higher values of E_f'/G' may be required for anisotropic materials and must be estimated from dynamic flexural and shear modulus data. It is recommended that the L_{a}/h ratios are chosen such that the magnitudes of the correction terms for shear deformation do not exceed 0,1.

NOTE The shear correction terms (h^2/L^2_{a}) (E_f'/G') and $(h^2/4L^2_{\mathrm{a}})$ (E_f'/G') in Formulae (1) and (2), respectively, are approximate since they omit a factor (the shear deflection coefficient) that accounts for the distribution of shear stress across the specimen thickness.

10.2.2 Avoidance of specimen resonance

Formulae (1) and (2) become invalid as the drive frequency approaches the fundamental flexural resonance frequency f_{s} of the specimen given approximately by the following formulae:

Clamped specimen

$$f_{\mathrm{s}} = 1{,}03 \times \frac{h}{L_{\mathrm{a}}^{2}} \times \left(\frac{E_{f}{'}_{\mathrm{a}}}{\rho}\right)^{1/2} \quad (3)$$

Simply supported specimen

$$f_{\mathrm{s}} = 0{,}71 \times \frac{h}{L_{\mathrm{a}}^{2}} \times \left(\frac{E_{f}{'}_{\mathrm{a}}}{\rho}\right)^{1/2} \quad (4)$$

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

Errors in the use of Formulae (1) and (2) become significant at applied frequencies such that

$$f \geq 0{,}08 f_{\mathrm{s}} \quad (5)$$

Calculations of dynamic properties shall therefore be confined to frequencies below $0{,}08f_{\mathrm{s}}$.

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

$$f_F = \frac{1}{2\pi} \times \left(\frac{k_F}{m_F} \right)^{1/2} \quad (6)$$

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

$$f > 0{,}1 f_F \quad (7)$$

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 Formula (8).

$$k = k_a \left(1 - \frac{4\pi^2 m_F f^2}{k_F} \right) = k_a \left(1 - \frac{f^2}{f_F^2} \right) \quad (8)$$

It is recommended that Formulae (6) and (7) 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 $0{,}02 k_\infty$, 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:

$$k \cos \delta_{E_f} = \frac{k_a \left(\cos \delta_{E_{fa}} - k_a / k_\infty \right)}{1 - 2 \left(k_a / k_\infty \right) \cos \delta_{E_{fa}}} \quad (9)$$

where δ_{E_f} is given by Formula (11).

The value of $k \cos \delta_{E_f}$ obtained from Formula (9) shall be used in place of $k_a \cos \delta_{E_{fa}}$ in Formula (1) or Formula (2) to give a more accurate estimate for $E_f{}'_a$.

NOTE The compliance correction is not necessary if the displacement transducer is located so as to measure the relative displacement of central and outer clamps or supports.

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 distortion of the specimen within and around the clamps. Applying a small correction to L_a such that the effective length is $L_a + l$, and assuming l is independent of L_a, yields from Formula (1).

$$E_f{}' = \frac{k (L_a + l)^3}{2bh^3} \times \left(1 + \frac{h^2}{L_a^2} \times \frac{E_f{}'}{G'} \right) \cos \delta_{E_{fa}} = E_f{}'_a \times \frac{(L_a + l)^3}{L_a^3} \quad (10)$$

where E_a is the apparent storage modulus corrected for apparatus compliance, if necessary, and the length correction has been ignored in the small shear-correction term.

A value for l may be determined from measurements of $E_f{'}_a$ for a series of clamp separations L_a. From Formula (10), a plot of $L_a/E_f{'}_a{}^{1/3}$ against L_a enables l to be determined from the intercept at $L_a/E_f{'}_a{}^{1/3} = 0$ and $E_f{'}$ to be determined from the gradient.

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

10.3 Calculation of the flexural loss factor $\tan\delta_{E_f}$

An approximate value for the flexural loss factor is $\tan\delta_{E_{fa}}$.

If k_a is greater than 0,02 k_∞ then the compliance of the loading assembly will influence the accuracy of the phase angle measurement. The loss factor shall then be obtained using Formula (11).

$$\tan\delta_{E_f} = \frac{\tan\delta_{E_{fa}}}{1-\left(k_a / k_\infty \cos\delta_{E_{fa}}\right)} \tag{11}$$

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 $\delta_{E_{fa}}$. 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 central and outer clamps or supports is measured.

10.4 Calculation of the flexural loss modulus $E_f{''}$

Calculate the loss modulus $E_f{''}$ from Formula (12).

$$E_f'' = E_f' \cdot \tan\delta_{E_f} \tag{12}$$

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 contain the information given in the test report of ISO 6721-1 plus the following:

a) reference to this document (i.e. ISO 6721-5);

b) the maximum dynamic strain amplitude, given approximately by $3hs_A/L_a^2$ for clamped specimens and by $3hs_A/2L_a^2$, for simply supported specimens.

