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Plastics — Determination of dynamic mechanical properties —

Part 3: Flexural vibration — Resonance-curve method

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 3: Vibration en flexion — Méthode en résonance

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Contents

Page

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,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 6721-3:1994), which has been technically revised. It also incorporates the Technical Corrigendum ISO 6721-3:1994/Cor 1:1995.

The main changes compared to the previous edition are as follows:

— the document has been revised editorially;

— the normative references have been updated;

— the NOTE in Clause 3 has been moved to Clause 4;

— the method of specimen density measurement has been defined.

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 3: Flexural vibration — Resonance-curve method

1 Scope

This document specifies a bending-vibration method based upon resonance curves for determining the flexural complex modulus E_f^* of homogeneous plastics and the damping properties of laminated plastics intended for acoustic insulation, for example systems consisting of a metal sheet coated with a damping plastic layer, or sandwich systems consisting of two sheet-metal layers with an intermediate plastic layer. For many purposes, it is useful to determine these properties as a function of temperature and frequency.

2 Normative reference

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 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pycnometer method and titration method*

ISO 1183-2, *Plastics — Methods for determining the density of non-cellular plastics — Part 2: Density gradient column method*

ISO 1183-3, *Plastics — Methods for determining the density of non-cellular plastics — Part 3: Gas pyknometer method*

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 specimen is submitted to forced bending vibrations in the frequency range between about 10 Hz and 1 000 Hz. The resonance curve (see ISO 6721-1) is determined and, from the curve obtained, the flexural storage modulus E'_f is calculated in the range above 0,5 MPa and the loss factor given by $\tan \delta = E''_f/E'_f$ is calculated in the range between about 10^{-2} and 10^{-1} (see NOTE). The test frequency can be varied by making measurements at more than one vibrational order. The measurement range for the flexural loss modulus E''_f is determined by that of the loss factor and by the value of the storage modulus.

The mode of oscillation used is designated oscillation mode III (see ISO 6721-1) and the type of modulus measured is designated E_f.

The test is performed on rectangular bars, either mounted vertically with the upper end clamped and the other end free (method A) or suspended horizontally by fine fibres at vibrational nodes (method B) (see Figure 1). Method A is suitable for testing specimens of most types of plastic, including relatively soft materials, whereas method B is particularly suitable for testing rigid (i.e. dimensionally stable) specimens, for example sheet metal covered by a plastic layer for damping purposes.

NOTE As stated in ISO 6721-1, frequencies derived from resonance curves based on deformation-rate amplitude measurements are exactly related to dynamic properties. For the recommended range of the loss factor of this document, i.e. $\tan \delta < 0{,}1$, resonance curves based upon deformation amplitudes are also related to dynamic properties of the material.

5 Test apparatus

5.1 General

The apparatus consists of devices for clamping (method A) or suspending (method B) the specimen, electronic devices (frequency generator and recording device) for exciting the specimen to forced bending vibration, and for measuring the frequency as well as the velocity amplitude of the specimen. For excitation and detection of the vibrations two electromagnetic transducers are situated near the ends of the specimen. The specimen, the clamping or supporting device and the electromagnetic transducers are enclosed in a temperature-controlled chamber (see Figure 1).

5.2 Clamps or suspension fibres

If the specimen is clamped at one end, the clamp shall be designed to hold the upper end of the specimen securely and tightly [see Figure 1 a)]. It shall be constructed so that no additional damping of the system occurs.

There are two causes of additional damping.

— Friction between the test specimen and the clamp: This can be detected by stimulating freely decaying oscillations of the relevant vibrational order. As explained in ISO 6721-1, the type of decay is indicative of different types of deviation from linear viscoelastic behaviour.

— Vibration of the clamp: The clamp shall be rigidly mounted on a heavy mass, which acts as a counterweight to the oscillating test specimen. This requires a heavy rigid stand within the temperature-controlled chamber (see Figure 1).

If the specimen is tested in the horizontal position, it shall be supported by two fine fibres at vibrational nodes (see 9.4.2).

5.3 Exciter and detector

The frequency generator shall be capable of exciting the specimen with the aid of the electromagnetic transducer to oscillations within the frequency range of 10 Hz to 1 000 Hz with a constant force amplitude.

The detector shall be capable of measuring the deformation or deformation-rate amplitude (see NOTE in Clause 4) of the specimen and the frequency of the oscillation, thereby permitting the recording of the resonance curve.

The amplitude of the exciter and the sensitivity of the detector shall not vary with frequency by more than 0,5 % within the range of a single-resonance peak, i.e. for any 10 % variation of the frequency.

A tracking filter shall be used to minimize noise at the detector. Frequencies shall be measured with a resolution of at least 0,1 % (see 11.2).

Two small, thin steel plates shall be adhesively bonded at the ends of the specimen to permit the excitation and detection of the vibrations by means of suitable electromagnetic transducers (see 6.3).

5.4 Temperature-controlled enclosure

According to ISO 6721-1.

5.5 Gas supply

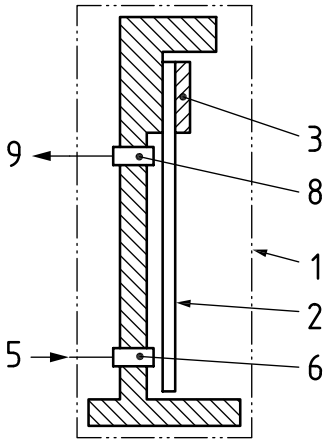
Supply of air or other suitable inert gas for purging purposes.

5.6 Temperature-measurement device

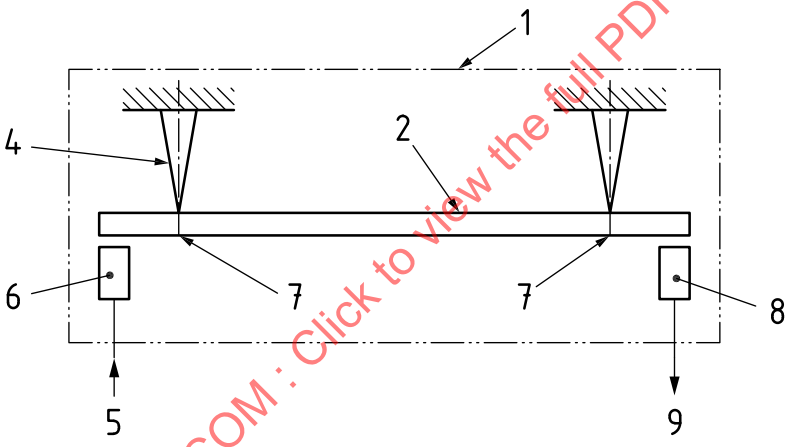
According to ISO 6721-1.

5.7 Devices for measuring test specimen dimensions

According to ISO 6721-1.



a) Specimen mounted vertically with upper end clamped (Method A)



b) Specimen suspended horizontally at vibrational nodes (Method B)

Key

1	temperature-controlled enclosure	6	exciter
2	test specimen	7	vibrational nodes
3	clamp	8	detector
4	fine fibres	9	to amplifier
5	from frequency generator		

Figure 1 — Schematic diagrams of test apparatus for methods A and B

6 Test specimens

6.1 General

According to ISO 6721-1.

6.2 Shape and dimensions

Specimens shall be rectangular bars or strips thick enough to give sufficient bending stiffness, which is critical for the resonance frequency. On the other hand, the thickness shall be sufficiently small when compared to the wavelength of the bending vibration. The specimen thickness shall also be limited to avoid effects due to shear deformation and rotatory inertia if accurate values of E' are required. Length-to-thickness ratios of less than 50 shall be avoided if values of E' are required to be accurate to within ±5 %, from measurements up to the sixth order of homogeneous, isotropic specimens.

The thickness of the layers of a multilayer system will depend on the purpose for which the system was designed. When comparing various systems by the bending-vibration test, the preferred ratio of the mass of the plastic layer to the mass of the basic sheet material is 1:5.

The width of the specimens shall be less than one-half of the wavelength used in order to avoid lateral resonance vibrations. A width of 10 mm should be suitable in most cases.

The length of the specimens depends on the desired frequency. For specimens clamped at one end, the length shall be sufficiently large to avoid the clamp influencing the vibration significantly. A free length of 180 mm should be used. If the specimen is not clamped, its length shall be 150 mm.

6.3 Preparation

According to ISO 6721-1.

Small, thin, light steel plates shall be adhesively bonded to the specimens near their ends to allow excitation and detection of the vibrations by means of electromagnetic transducers. To avoid errors in E' greater than 4 %, the ratio of the added mass to the specimen mass shall not exceed 1 %. To avoid the steel plates causing additional stiffness, they shall not extend along more than 2 % of the length of the specimen. The distance between the steel plates shall be large enough to avoid cross-talk between exciter and detector.

Multilayer specimens shall be fabricated with the thickness and by the production techniques to be used in the projected end-use. For example, for a plastic material on steel sheet, the plastic can be applied to the metal by spraying, as a mastic or as an adhesively bonded sheet.

7 Number of test 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 Measurement of specimen cross-section

According to ISO 6721-1.

9.3 Measurement of specimen density

Measure the density of the test specimen, ρ (kg m^{-3}) to an accuracy of ±0,5 % using one of the procedures described in ISO 1183-1 or ISO 1183-2 or ISO 1183-3.

9.4 Mounting the test specimens and adjustment of the transducers

9.4.1 Method A

Clamp the specimen so that the clamping force is high enough to avoid additional damping from friction between the specimen and the clamp (see 5.2). Measure the free length L of the specimen to ±0,5 %.

9.4.2 Method B

Measure the length of the specimen to ±0,2 %. Calculate the distance L_i of the first nodes from the ends of the specimen, using either Formula (1) or Formula (2):

$$L_i / l = 0{,}224 \quad \text{for } i = 1 \tag{1}$$

$$L_i / l = 0{,}660/(2i+1) \quad \text{for } i > 1 \tag{2}$$

where

l is the length of the specimen;

i is the vibrational order.

Mount the specimen by fine, preferably non-metallic, fibres at the calculated positions of the vibrational nodes.

9.4.3 Adjustment of the transducers

After clamping or supporting the specimen, adjust the detector and exciter transducers so that they are just far enough from the specimen to avoid any noticeable effect on the resonance frequency. With the usual test apparatus, the recommended distance is greater than 3 mm for i = 1. Gaps of 1 mm or less can be used with higher orders.

9.5 Varying the temperature

According to ISO 6721-1.

9.6 Varying the frequency

According to ISO 6721-1.

9.7 Recording the resonance curve

Excite the specimen using the frequency generator and determine the amplitude (or the effective value) of the deformation or deformation rate. By varying the frequency, record the resonance curve.

Measure the amplitude to ±0,5 %, the resonance frequencies to at least ±0,1 % and the width of the resonance peaks to ±1 % of the value of the peak width (see 11.2).

Usually, it is possible to measure the resonance curve in the range from the first to the sixth or seventh order of the vibration.

When a clamped specimen is used (method A), the first order is the one mostly affected by damping in the clamp, and the amplitudes of the vibrations of higher orders decrease rapidly with frequency. Therefore, intermediate orders shall be chosen for the measurements.

Ensure that the type of amplitude decay measured does not include either friction between moving and fixed parts of the apparatus or non-linear behaviour of the materials under test (see ISO 6721-1).

10 Expression of results

10.1 Symbols

E'_f flexural storage modulus, expressed in pascals

E''_f flexural loss modulus, expressed in pascals

$\tan \delta_f$ flexural loss factor (dimensionless number)

ϱ specimen density, expressed in kilograms per cubic metre

l length of the specimen (method B), expressed in metres

L free length of the specimen (method A), expressed in metres

h thickness of the specimen, expressed in metres

f frequency of the oscillation, expressed in hertz

i order of the vibration

f_{ri} resonance frequency at the order i of the vibration (see ISO 6721-1), expressed in hertz

Δf_i width of i^{th} order resonance peak (see ISO 6721-1), expressed in hertz

k_i^2 numerical factor given by one of the Formulae (3) to (5) or (6) to (8):

For method A:

$$k_1^2 = 3{,}52 \text{ for } i = 1 \tag{3}$$

$$k_2^2 = 22{,}0 \text{ for } i = 2 \tag{4}$$

$$k_i^2 = \left(i - 1/2\right)^2 \pi^2 \text{ for } i > 2 \tag{5}$$

For method B:

$$k_1^2 = 22{,}4 \text{ for } i = 1 \tag{6}$$

$$k_2^2 = 61{,}7 \text{ for } i = 2 \tag{7}$$

$$k_i^2 = (i+1/2)^2 \pi^2 \text{ for } i > 2 \tag{8}$$

10.2 Calculation of flexural storage modulus, E'_f

The flexural storage modulus E'_f is given by Formula (9).

$$E'_f = \left[4\pi(3\varrho)^{\frac{1}{2}} \, l^2/h\right]^2 \left(\frac{f_{ri}}{k_i^2}\right)^2 \tag{9}$$

10.3 Calculation of flexural loss factor, $\tan \delta_f$

The flexural loss factor $\tan \delta_f$ shall be calculated from the width Δf_i of the resonance peak and the natural frequency f_{ri} by Formula (10) (see ISO 6721-1).

$$\tan\delta_f = \Delta f_i / f_{ri} \tag{10}$$

NOTE As defined in ISO 6721-1, the width Δf_i of any peak in the resonance curve is based upon an attenuation a of the deformation-rate amplitude of $2^{1/2}$. Highly damping materials, however, often show resonance peaks smaller than this attenuation. In this case, with reduced precision, the resonance curves are analysed to calculate the loss factor using a lower attenuation a nearer to the value 1 or using a curve-fitting process, using the formulae given in ISO 6721-1.

Alternatively, the mode of oscillation may be changed from mode number III to IV (see ISO 6721-1). Switching off the exciter at the natural frequency of the relevant vibrational order, freely decaying oscillations are stimulated and may be analysed according to ISO 6721-1. This measuring principle may also be useful in the case of very weakly damping materials with extremely small resonance-peak widths (see 11.2).

10.4 Calculation of flexural loss modulus, E''_f

The flexural loss modulus E''_f can be calculated from the flexural storage modulus E'_f and the flexural loss factor ($\tan \delta_f$) using the Formula (11).

$$E''_f = E'_f \tan\delta_f \tag{11}$$

10.5 Plotting the complex modulus as a function of temperature

If the complex modulus is measured as a function of temperature, not only E'_f and E''_f, but also the frequency at which they are measured, shall be plotted as a function of temperature, because the resonance frequency of a given plastic specimen is shifted to lower values as the storage modulus decreases with increasing temperature. However, the values of the components which are temperature-dependent at constant frequency can be obtained by interpolation if curves have been plotted at several different frequencies (i.e. for several different vibrational orders).

11 Precision

11.1 Storage modulus

If all instructions regarding the determination of each individual parameter in Formula (9) have been followed (see 5.3, 9.2, 9.3, 9.4.1, 9.4.2 and 9.7), the precision of measurement of E'_f will be between ±3 % and ±5 % up to vibrational order 4. For higher-order vibrational modes, it becomes necessary to consider the effect of shear deformation. In the case of laminated systems, E'_f represents an average modulus for the system (the flexural mean).

11.2 Loss factor

The precision of measurement of the loss factor ($\tan \delta_f$) depends on its value and the frequency resolution of the measuring equipment. The relationship between the coefficient of variation V_f of the frequency measurement and the coefficient of variation V_δ of the loss factor is given by the Formula (12).

$$V_\delta = \sqrt{2}\ V_f / \tan\delta_f \tag{12}$$

Using a value of 0,1 % for V_f (see 9.7), the coefficient of variation V_δ of the loss factor is 1,4 % when $\tan \delta_f$ = 0,1 and 14 % when $\tan \delta_f$ = 0,01. For precise measurements of $\tan \delta_f$ and thus of E''_f [see Formula (11)], the frequency resolution should be higher than that necessary for the measurement of E'_f.

For small values of the loss factor, freely decaying vibrations stimulated at the resonance frequencies shall be used.

11.3 Precision of the methods

The precision of the two methods described in this document is not known because inter-laboratory data are not available. See also Annex A.

12 Test report

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

a) a reference to this document, i.e. ISO 6721-3:2021;

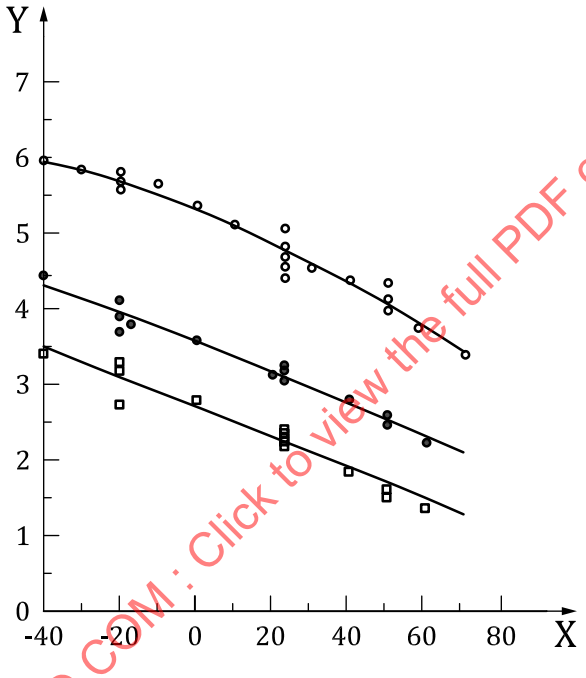
b) the method used (A o r B).

Annex A
(informative)

Interlaboratory testing

During the preparation of the original version of this document, ISO 6721:1983[6], an interlaboratory test was performed in 1976. Four countries (France, Japan, Germany and Italy) participated in this test. The thermoplastics examined were PMMA, PVC and PE-HD. The results of the interlaboratory test are shown in Figure A.1 to Figure A.3.

The reproducibility was found to be ±5 % for E'_f and ±3 × 10^{-3} for tan δ_f.



Key

X temperature θ (°C)

Y flexural storage modulus E'_f (MPa) x10^{-3}

PMMA

PVC

PE-HD

Figure A.1 — Real part E'_f of the flexural complex modulus versus temperature for poly(methyl methacrylate) (PMMA), poly(vinyl chloride) (PVC) and polyethylene (PE-HD), measured at 300 Hz