INTERNATIONAL STANDARD

ISO 6721-1

Third edition
2011-05-15

Plastics — Determination of dynamic mechanical properties —

Part 1: General principles

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 1: Principes généraux

Reference number
ISO 6721-1:2011(E)

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Published in Switzerland

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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.

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 part of ISO 6721 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6721-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

This third edition cancels and replaces the second edition (ISO 6721-1:2001), of which it constitutes a minor revision involving the following changes:

— a new subclause (9.6), covering the case when the dynamic-strain amplitude is varied, has been added to the procedure clause;

— the expression of results clause (Clause 10) and the test report clause (Clause 12) have been modified accordingly [Clause 10 by the addition of a new paragraph (the third) and Clause 12 by the addition of a new item, item n)].

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*

— *Part 11: Glass transition temperature*

— *Part 12: Compressive vibration — Non-resonance method*

Introduction

The methods specified in the first nine parts of ISO 6721 can be used for determining storage and loss moduli of plastics over a range of temperatures or frequencies by varying the temperature of the specimen or the frequency of oscillation. Plots of the storage or loss moduli, or both, are indicative of viscoelastic characteristics of the specimen. Regions of rapid changes in viscoelastic properties at particular temperatures or frequencies are normally referred to as transition regions. Furthermore, from the temperature and frequency dependencies of the loss moduli, the damping of sound and vibration of polymer or metal-polymer systems can be estimated.

Apparent discrepancies may arise in results obtained under different experimental conditions. Without changing the observed data, reporting in full (as described in the various parts of ISO 6721) the conditions under which the data were obtained will enable apparent differences observed in different studies to be reconciled.

The definitions of complex moduli apply exactly only to sinusoidal oscillations with constant amplitude and constant frequency during each measurement. On the other hand, measurements of small phase angles between stress and strain involve some difficulties under these conditions. Because these difficulties are not involved in some methods based on freely decaying vibrations and/or varying frequency near resonance, these methods are used frequently (see ISO 6721-2 and ISO 6721-3). In these cases, some of the equations that define the viscoelastic properties are only approximately valid.

Plastics — Determination of dynamic mechanical properties —

Part 1: General principles

1 Scope

The various parts of ISO 6721 specify methods for the determination of the dynamic mechanical properties of rigid plastics within the region of linear viscoelastic behaviour. This part of ISO 6721 is an introductory section which includes the definitions and all aspects that are common to the individual test methods described in the subsequent parts.

Different deformation modes may produce results that are not directly comparable. For example, tensile vibration results in a stress which is uniform across the whole thickness of the specimen, whereas flexural measurements are influenced preferentially by the properties of the surface regions of the specimen.

Values derived from flexural-test data will be comparable to those derived from tensile-test data only at strain levels where the stress-strain relationship is linear and for specimens which have a homogeneous structure.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 293, *Plastics — Compression moulding of test specimens of thermoplastic materials*

ISO 294 (all parts), *Plastics — Injection moulding of test specimens of thermoplastic materials*

ISO 295, *Plastics — Compression moulding of test specimens of thermosetting materials*

ISO 1268 (all parts), *Fibre-reinforced plastics — Methods of producting test plates*

ISO 2818, *Plastics — Preparation of test specimens by machining*

ISO 4593, *Plastics — Film and sheeting — Determination of thickness by mechanical scanning*

ISO 6721-2:2008, *Plastics — Determination of dynamic mechanical properties — Part 2: Torsion-pendulum method*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

NOTE Some of the terms defined here are also defined in ISO 472[7]. The definitions given here are not strictly identical with, but are equivalent to, those in ISO 472.

3.1
complex modulus
M^*
ratio of dynamic stress, given by $\sigma(t) = \sigma_A \exp(i2\pi ft)$, and dynamic strain, given by $\varepsilon(t) = \varepsilon_A \exp[i(2\pi ft - \delta)]$, of a viscoelastic material that is subjected to a sinusoidal vibration, where σ_A and ε_A are the amplitudes of the stress and strain cycles, f is the frequency, δ is the phase angle between stress and strain (see 3.5 and Figure 1) and t is time

NOTE 1 It is expressed in pascals (Pa).

NOTE 2 Depending on the mode of deformation, the complex modulus might be one of several types: E^*, G^*, K^* or L^* (see Table 3).

$$M^* = M' + iM'' \qquad \text{(see 3.2 and 3.3)} \qquad (1)$$

where

$$i = (-1)^{1/2} = \sqrt{-1}$$

For the relationships between the different types of complex modulus, see Table 1.

NOTE 3 For isotropic viscoelastic materials, only two of the elastic parameters G^*, E^*, K^*, L^* and μ^* are independent (μ^* is the complex Poisson's ratio, given by $\mu^* = \mu' + i\mu''$).

NOTE 4 The most critical term containing Poisson's ratio μ is the "volume term" $1 - 2\mu$, which has values between 0 and 0,4 for μ between 0,5 and 0,3. The relationships in Table 1 containing the "volume term" $1 - 2\mu$ can only be used if this term is known with sufficient accuracy.

It can be seen from Table 1 that the "volume term" $1 - 2\mu$ can only be estimated with any confidence from a knowledge of the bulk modulus K or the uniaxial-strain modulus L and either E or G. This is because K and L measurements involve deformations when the volumetric strain component is relatively large.

NOTE 5 Up to now, no measurement of the dynamic mechanical bulk modulus K, and only a small number of results relating to relaxation experiments measuring $K(t)$, have been described in the literature.

NOTE 6 The uniaxial-strain modulus L is based upon a load with a high hydrostatic-stress component. Therefore values of L compensate for the lack of K values, and the "volume term" $1 - 2\mu$ can be estimated with sufficient accuracy based upon the modulus pairs (G, L) and (E, L). The pair (G, L) is preferred, because G is based upon loads without a hydrostatic component.

NOTE 7 The relationships given in Table 1 are valid for the complex moduli as well as their magnitudes (see 3.4).

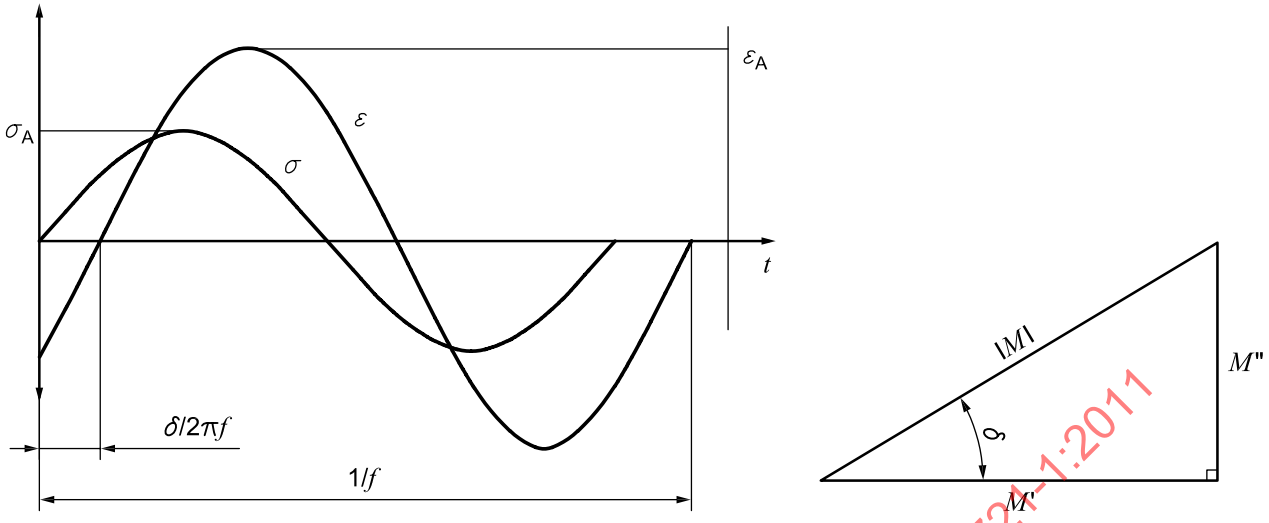
NOTE 8 Most of the relationships for calculating the moduli given in the other parts of this International Standard are, to some extent, approximate. They do not take into account e.g. "end effects" caused by clamping the specimens, and they include other simplifications. Using the relationships given in Table 1 therefore often requires additional corrections to be made. These are given in the literature (see e.g. References [1] and [2] in the Bibliography).

NOTE 9 For linear-viscoelastic behaviour, the complex compliance C^* is the reciprocal of the complex modulus M^*, i.e.

$$M^* = (C^*)^{-1} \qquad (2)$$

Thus

$$M' + iM'' = \frac{C' - iC''}{(C')^2 + (C'')^2} \qquad (3)$$



a) b)

The phase shift $\delta/2\pi f$ between the stress σ and strain ε in a viscoelastic material subjected to sinusoidal oscillation (σ_A and ε_A are the respective amplitudes, f is the frequency).

The relationship between the storage modulus M', the loss modulus M'', the phase angle δ and the magnitude $|M|$ of the complex modulus M^*.

Figure 1 — Phase angle and complex modulus

Table 1 — Relationships between moduli for uniformly isotropic materials

	G **and** μ	E **and** μ	K **and** μ	G **and** E	G **and** K	E **and** K	G **and** L[a]
Poisson's ratio, μ $1-2\mu =$[b]				$3-\frac{E}{G}$	$\frac{G/K}{1+G/3K}$	$\frac{E}{3K}$	$\frac{1}{L/G-1}$
Shear modulus, $G =$		$\frac{E}{2(1+\mu)}$	$\frac{3K(1-2\mu)}{2(1+\mu)}$			$\frac{E}{3-E/3K}$	
Tensile modulus, $E =$	$2G(1+\mu)$		$3K(1-2\mu)$		$\frac{3G}{1+G/3K}$		$\frac{3G(1-4G/3L)}{1-G/L}$
Bulk modulus, $K =$[c]	$\frac{2G(1+\mu)}{3(1-2\mu)}$	$\frac{E}{3(1-2\mu)}$		$\frac{G}{3(3G/E-1)}$			$L-\frac{4G}{3}$
Unaxial-strain or longitudinal-wave modulus, $L =$	$\frac{2G(1-\mu)}{1-2\mu}$	$\frac{E(1-\mu)}{(1+\mu)(1-2\mu)}$	$\frac{3K(1-\mu)}{1+\mu}$	$\frac{G(4G/E-1)}{3G/E-1}$	$K+\frac{4G}{3}$	$\frac{K(1+E/3K)}{1-E/9K}$	

a See Note 6 to definition 3.1.

b See Note 4 to definition 3.1.

c See Note 5 to definition 3.1.

3.2
storage modulus
M'
real part of the complex modulus M^* [see Figure 1b)]

NOTE 1 The storage modulus is expressed in pascals (Pa).

NOTE 2 It is proportional to the maximum energy stored during a loading cycle and represents the stiffness of a viscoelastic material.

NOTE 3 The different types of storage modulus, corresponding to different modes of deformation, are: E'_t tensile storage modulus, E'_f flexural storage modulus, G'_s shear storage modulus, G'_{to} torsional storage modulus, K' bulk storage modulus, L'_c uniaxial-strain storage modulus and L'_w longitudinal-wave storage modulus.

3.3
loss modulus
M''
imaginary part of the complex modulus [see Figure 1b)]

NOTE 1 The loss modulus is expressed in pascals (Pa).

NOTE 2 It is proportional to the energy dissipated (lost) during one loading cycle. As with the storage modulus (see 3.2), the mode of deformation is designated as in Table 3, e.g. E''_t is the tensile loss modulus.

3.4
magnitude $|M|$ of the complex modulus
root mean square value of the storage and the loss moduli as given by the equation

$$|M|^2 = (M')^2 + (M'')^2 = (\sigma_A/\varepsilon_A)^2 \quad (4)$$

where σ_A and ε_A are the amplitudes of the stress and the strain cycles, respectively

NOTE 1 The complex modulus is expressed in pascals (Pa).

NOTE 2 The relationship between the storage modulus M', the loss modulus M'', the phase angle δ, and the magnitude $|M|$ of the complex modulus is shown in Figure 1b). As with the storage modulus, the mode of deformation is designated as in Table 3, e.g. $|E_t|$ is the magnitude of the tensile complex modulus.

3.5
phase angle
δ
phase difference between the dynamic stress and the dynamic strain in a viscoelastic material subjected to a sinusoidal oscillation (see Figure 1)

NOTE 1 The phase angle is expressed in radians (rad).

NOTE 2 As with the storage modulus (see 3.2), the mode of deformation is designated as in Table 3, e.g. δ_t is the tensile phase angle.

3.6
loss factor
$\tan\delta$
ratio between the loss modulus and the storage modulus, given by the equation

$$\tan\delta = M''/M' \quad (5)$$

where δ is the phase angle (see 3.5) between the stress and the strain

NOTE 1 The loss factor is expressed as a dimensionless number.

NOTE 2 The loss factor $\tan\delta$ is commonly used as a measure of the damping in a viscoelastic system. As with the storage modulus (see 3.2), the mode of deformation is designated as in Table 3, e.g. $\tan\delta_t$ is the tensile loss factor.

3.7
stress-strain hysteresis loop
stress expressed as a function of the strain in a viscoelastic material subject to sinusoidal vibrations

NOTE Provided the viscoelasticity is linear in nature, this curve is an ellipse (see Figure 2).

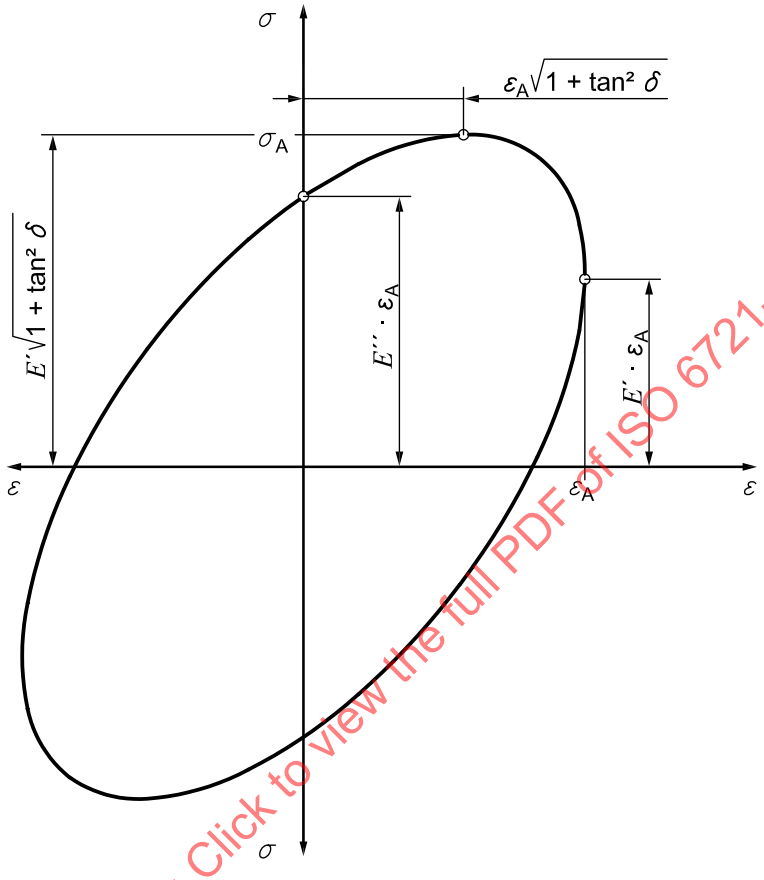


Figure 2 — Dynamic stress-strain hysteresis loop for a linear-viscoelastic material subject to sinusoidal tensile vibrations

3.8
damped vibration
time-dependent deformation or deformation rate $X(t)$ of a viscoelastic system undergoing freely decaying vibrations (see Figure 3), given by the equation

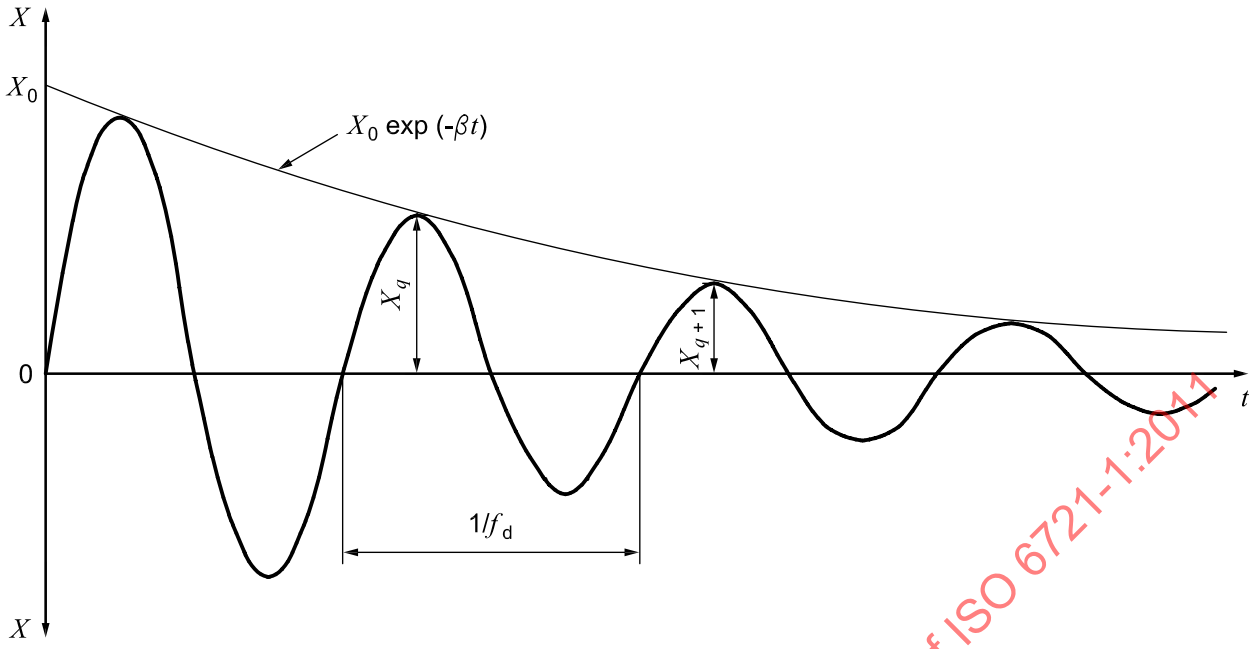
$$X(t) = X_0 \exp(-\beta t) \times \sin 2\pi f_d t \quad (6)$$

where

X_0 is the magnitude, at zero time, of the envelope of the cycle amplitudes;

f_d is the frequency of the damped system;

β is the decay constant (see 3.9)



[X is the time-dependent deformation or deformation rate, X_q is the amplitude of the qth cycle and X_0 and β define the envelope of the exponential decay of the cycle amplitudes — see Equation (6).]

Figure 3 — Damped-vibration curve for a viscoelastic system undergoing freely decaying vibrations

3.9
decay constant
β
coefficient that determines the time-dependent decay of damped free vibrations, i.e. the time dependence of the amplitude X_q of the deformation or deformation rate [see Figure 3 and Equation (6)]

NOTE The decay constant is expressed in reciprocal seconds (s^{-1}).

3.10
logarithmic decrement
Λ
natural logarithm of the ratio of two successive amplitudes, in the same direction, of damped free oscillations of a viscoelastic system (see Figure 3), given by the equation

$$\Lambda = \ln(X_q/X_{q+1}) \tag{7}$$

where X_q and X_{q+1} are two successive amplitudes of deformation or deformation rate in the same direction

NOTE 1 The logarithmic decrement is expressed as a dimensionless number.

NOTE 2 It is used as a measure of the damping in a viscoelastic system.

NOTE 3 Expressed in terms of the decay constant β and the frequency f_d, the logarithmic decrement is given by the equation

$$\Lambda = \beta/f_d \tag{8}$$

NOTE 4 The loss factor $\tan\delta$ is related to the logarithmic decrement by the approximate equation

$$\tan\delta \approx \Lambda/\pi \tag{9}$$

NOTE 5 Damped freely decaying vibrations are especially suitable for analysing the type of damping in the material under test (i.e. whether the viscoelastic behaviour is linear or non-linear) and the friction between moving and fixed components of the apparatus (see Annex B).

3.11
resonance curve
curve representing the frequency dependence of the deformation amplitude D_A or deformation-rate amplitude R_A of an inert viscoelastic system subjected to forced vibrations at constant load amplitude L_A and at frequencies close to and including resonance (see Figure 4 and Annex A)

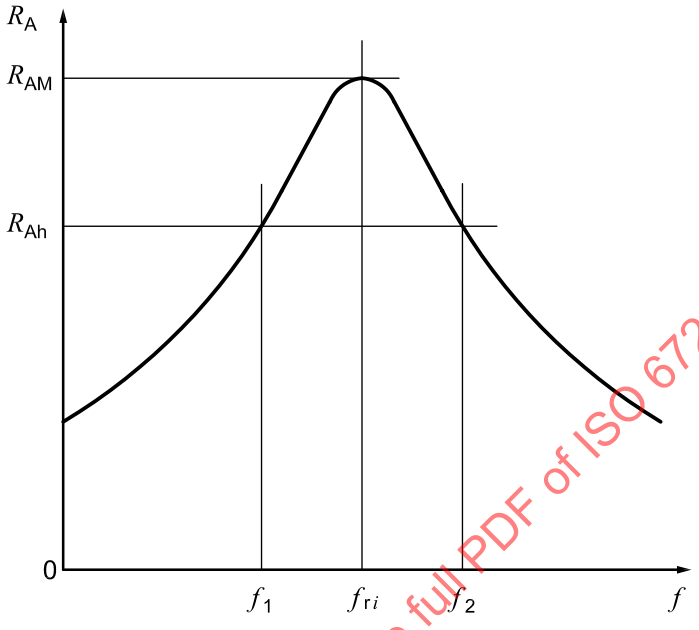


Figure 4 — Resonance curve for a viscoelastic system subjected to forced vibrations
(Deformation-rate amplitude R_A versus frequency f at constant load amplitude; logarithmic frequency scale)

3.12
resonance frequencies
f_{ri}
frequencies of the peak amplitudes in a resonance curve

NOTE 1 The subscript i refers to the order of the resonance vibration.

NOTE 2 Resonance frequencies are expressed in hertz (Hz).

NOTE 3 Resonance frequencies for viscoelastic materials derived from measurements of displacement amplitude will be slightly different from those obtained from displacement-rate measurements, the difference being larger the greater the loss in the material (see Annex A). Storage and loss moduli are accurately related by simple expressions to resonance frequencies obtained from displacement-rate curves. The use of resonance frequencies based on displacement measurements leads to a small error which is only significant when the specimen exhibits high loss. Under these conditions, resonance tests are not suitable.

3.13
width of a resonance peak
Δf_i
difference between the frequencies f_1 and f_2 of the ith-order resonance peak, where the height R_{Ah} of the resonance curve at f_1 and f_2 is related to the peak height R_{AMi} of the ith mode by

$$R_{Ah} = 2^{-1/2} R_{AM} = 0{,}707 R_{AM} \tag{10}$$

(see Figure 4)

NOTE 1 The width Δf_i is expressed in hertz (Hz).

NOTE 2 It is related to the loss factor $\tan\delta$ by the equation

$$\tan\delta = \Delta f_i / f_{ri} \qquad (11)$$

If the loss factor does not vary markedly over the frequency range defined by Δf_i, Equation (11) holds exactly when the resonance curve is based on the deformation-rate amplitude (see also Annex A).

4 Principle

A specimen of known geometry is subjected to mechanical oscillation, described by two characteristics: the mode of vibration and the mode of deformation.

Four oscillatory modes, I to IV, are possible, depending on whether the mode of vibration is non-resonant, natural (resonant) or near-resonant. These modes are described in Table 2.

The particular type of modulus depends upon the mode of deformation (see Table 3).

Table 4 indicates ways in which the various types of modulus are commonly measured. Table 5 gives a summary of the methods covered by the various parts of this International Standard.

Table 2 — Oscillatory modes

(Terms written in bold type give the designation of the mode; terms in normal type provide additional information.)

Mode of oscillation	**I**	**II**	**III**	**IV**[a]
	Forced vibration			Damped, **freely decaying amplitude**
	Constant frequency	Resonance frequency	**Resonance curve**	
Frequency	**Non-resonance**	**Resonance** (natural)	Sweep, near resonance	Approximately resonant
Load amplitude	One of the two constant, the other measured	Constant[b]	Constant	Excitation pulse
Deformation amplitude		Measured	Measured	
Inertial mass	None	Specimen and/or additional masses, depending on frequency range		

[a] The type of torsion pendulum used shall be indicated by adding the relevant letter, A or B (see ISO 6721-2:2008, Figures 1 and 2).

[b] The load must be in phase with the deformation rate.

Table 3 — Type of modulus (mode of deformation)

Designation	**Type of modulus**
E_t	Tensile
E_f	Flexural
G_s	Shear
G_{to}	Torsion
K	Bulk compression
L_c	Uniaxial compression (of thin sheets)
L_w	Longitudinal bulk wave

Table 4 — Commonly used test arrangements

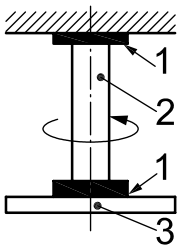
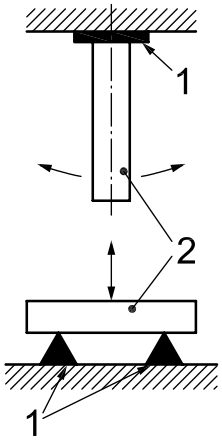
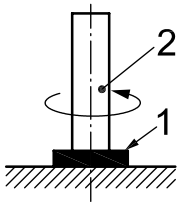
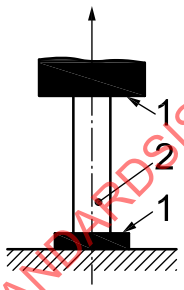
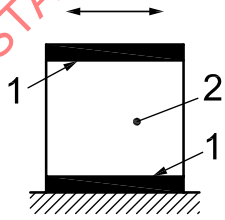
Test arrangement	Type of modulus and mode of oscillation		Relevant part of ISO 6721	Inertial mass	Typical frequency, Hz
	G_{to}	IV	Part 2	Inertial member	0,1 to 10
	E_f	III	Part 3	Specimen	10 to 1 000
	E_f	I	Part 5	None	10^{-3} to 100
	G_{to}	I	Part 7		
	E_t	I	Part 4		
	G_s	I	Part 6		
Key to figures: 1 — Clamps, pivots or supports; 2 — Specimen; 3 — Inertial member.					

Table 4 (*continued*)

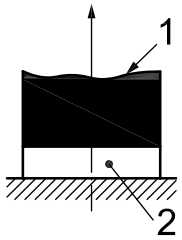
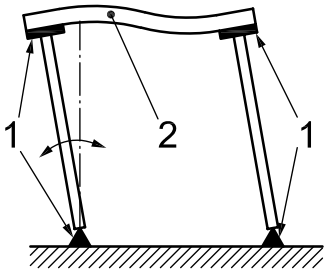
Test arrangement	Type of modulus and mode of oscillation		Relevant part of ISO 6721	Inertial mass	Typical frequency, Hz
	L_c	I	—	None	10^{-3} to 100
	E_f	I	—		10^{-3} to 10
	E_f	II	—	Specimen and arms	3 to 60
Key to figures: 1 — Clamps, pivots or supports; 2 — Specimen; 3 — Inertial member.					

Table 5 — Methods covered by the various parts of this International Standard

Mode of oscillation (see Table 2)	Type of modulus (see Table 3)						
	E_t	E_f	G_s	G_{to}	K	L_c	L_w
I	Part 4	Part 5	Part 6	Part 7			Part 8
II							
III		Part 3					
IV				Part 2			

5 Test apparatus

5.1 Type

The apparatus used is specified in detail in the relevant part of this International Standard (see the Introduction and Clause 4).

5.2 Mechanical, electronic and recording systems

See the relevant part of this International Standard.

5.3 Temperature-controlled enclosure

The test specimen and the clamps or supports shall be enclosed in a temperature-controlled enclosure containing air or a suitable inert gas.

The enclosure shall be designed so that its temperature can be varied over a range sufficient for the material under test (e.g. −100 °C to +300 °C). It is recommended that the chamber be equipped with temperature-programming facilities.

The temperature in the enclosure shall be uniform to within ±1 °C along the length of the specimen. If the constant-temperature procedure is used (see 9.5), the temperature shall be constant to within ±1 °C during the test. When a constant rate of increase (or decrease) in temperature is used (see 9.4), the rate shall not be greater than 120 °C/h and the temperature shall not vary with time by more than ±0,5 °C during a single measurement (e.g. a series of free oscillations following the starting pulse or a resonance curve).

5.4 Gas supply

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

5.5 Temperature-measurement device

The device for measuring the temperature of the air surrounding the specimen shall be capable of determining the temperature to ±0,5 °C. The use of a thermometer with a low-inertia sensor is recommended.

5.6 Devices for measuring test specimen dimensions

For the purposes of the various parts of this International Standard, the test specimen dimensions used for calculating moduli are measured at room temperature only. For the measurements of the temperature dependence of moduli, therefore, the effects of thermal expansion are not taken into account.

The devices used for measuring the length, width and thickness of the specimen (see also ISO 4593) shall be capable of determining these quantities to ±0,5 %.

6 Test specimens

6.1 General

The parameters measured by these methods are sensitive to dimensional non-uniformity of the specimen and to differences in its physical state (e.g. degree of crystallinity, orientation or internal stress). These factors should be considered when choosing the dimensions and tolerances, methods of preparation and conditioning procedures for specimens of a particular material.

The specimens (homogeneous specimens, laminated bars or strips) shall have negligible shrinkage or warpage within the temperature range of the measurements.

6.2 Shape and dimensions

See the relevant part of this International Standard.

6.3 Preparation

For the purposes of these methods, whether carried out on starting materials or finished products, the test specimens shall be prepared in accordance with the relevant material standard. They may be machined (see ISO 2818) from compression-moulded plates (see ISO 293, ISO 295 or ISO 1268) or from the finished product. Alternatively, the specimens may be injection-moulded (see ISO 294).

7 Number of test specimens

At least three test specimens shall be used for single-point measurements, i.e. measurements at a single temperature and frequency. If the temperature and/or the frequency is varied over a more-or-less wide range for quality-control purposes, one specimen is sufficient. In all other cases, at least two specimens shall be tested.

8 Conditioning

The test specimens shall be conditioned as specified in the International Standard for the material to be tested. In the absence of this information, the most appropriate conditions from ISO 291 shall be selected, unless otherwise agreed upon by the interested parties.

9 Procedure

9.1 Test atmosphere

The test temperature (or the dependence of the temperature on time), the gas supply (air or inert gas) and the relative humidity shall be chosen according to the specific type of test and the purpose of the test.

9.2 Measurement of specimen cross-section

Before the test, measure the thickness and width of each specimen to ±0,5 % at five points along its length. All specimens with visible irregularities, e.g. sink marks, or variations in thickness and/or width greater than 3 % of the average shall be rejected. With specimens of non-uniform thickness, e.g. finished parts, only the loss factor can be determined.

The procedure for measuring the dimensions of specimens of other shapes shall be agreed upon by the interested parties.

9.3 Mounting the test specimens

See the relevant part of this International Standard.

9.4 Varying the temperature

If temperature is the independent variable, the temperature of the test specimen shall be varied from the lowest to the highest temperature of interest while measuring the viscoelastic properties. The frequency of vibration may be fixed (oscillation mode I), decreased naturally with increasing temperature (oscillation modes II and IV) or swept (oscillation mode III) (see Table 2).

Tests conducted over a range of temperatures shall be performed at incremental temperature steps or at a rate of change of temperature slow enough to allow temperature equilibrium to be reached throughout the entire specimen. The time to reach equilibrium will depend on the mass of the particular specimen and the apparatus being used. Temperature rates of 1 °C/min to 2 °C/min or 2 °C to 5 °C step intervals held for 3 min to 5 min have been found suitable. For oscillation mode III, step intervals of 10 min are recommended.

The dynamic moduli of polymers are influenced in general by the state of physical ageing of the specimen at the time of measurement. The age state depends upon the thermal history of the specimen and changes with time at temperatures below the temperature θ_α corresponding to the centre of the α-relaxation region. The α-relaxation is the highest-temperature mechanical relaxation mechanism and in amorphous polymers is assigned to the glass to rubber transition. Changes in the physical age state influence the molecular mobility and hence the response of the polymer to a time-dependent load or deformation.

When measurements are made with increasing temperature, changes in age state will begin to occur within the time scale of the test as the temperature approaches θ_α. Subsequent cooling will in general establish a different state of physical ageing, and further measurements of dynamic properties will not reproduce previous values. Meaningful measurements of high accuracy will therefore require a record of the thermal history of the specimen and the heating rate if tests at elevated temperatures are carried out.

9.5 Varying the frequency

If vibrational frequency is the independent variable, the test temperature shall be fixed at the desired temperature. The vibrational frequency of the specimen shall be varied while measuring the viscoelastic properties.

9.6 Varying the dynamic-strain amplitude

If the test method allows measurements to be made over a range of strain amplitudes, it is good practice to present results for the variation of storage modulus with dynamic-strain amplitude ε_A to reveal the strain limit for linear viscoelastic behaviour. Measurements should start with the lowest strain for which accurate measurements are possible and proceed to higher strain values. These measurements should be made at a low frequency, preferably around 1 Hz.

NOTE Under dynamic loading, the temperature of viscoelastic materials can increase significantly owing to the dissipation of mechanical energy in the specimen as heat. If the temperature rise is significant, properties will change with time under load. The temperature rise increases with the loss modulus of the material, the dynamic-strain amplitude and the frequency. If the data-processing electronics is capable of analysing results within the first few cycles, 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 interpretation and presentation of the results.

10 Expression of results

Prepare a table of results, using the designation of the moduli as indicated in Table 3 for the relevant deformation mode, plus the designation of the type of oscillation as in Table 2, e.g.

E''_f (III) — Flexural loss modulus measured using resonance curves.

Average values and, if requested and possible, standard deviations for the storage modulus and the loss modulus shall be reported to two significant figures.

If the test method allows measurements to be made over a range of strain amplitude, present results of storage modulus plotted against strain amplitude.

Also prepare plots of the storage modulus and loss modulus versus temperature at different frequencies or plots of the storage modulus and loss modulus versus frequency at different temperatures, using logarithmic scales for the modulus and frequency axes.

For further details, see the relevant part of this International Standard.

11 Precision

See the relevant part of this International Standard.

12 Test report

The test report shall include the following information:

a) a reference to the relevant part of this International Standard;

b) all details necessary for complete identification of the material tested, including type, source, manufacturer's code number, form and previous history where these are known;

c) for sheets, the thickness of the sheet and, if applicable, the direction of the major axes of the specimens in relation to some feature of the sheet;

d) the date of the test;

e) the shape and dimensions of the specimens;

f) the method of preparing the specimens;

g) details of the conditioning of the specimens;

h) the number of specimens tested;

i) details of the test atmosphere if other than air;

j) a description of the apparatus used for the test;

k) the temperature programme used for the test, including the initial and final temperatures as well as the rate of linear change in temperature or the size and duration of the temperature steps;

l) the table of data prepared as specified in Clause 10;

m) the modulus versus temperature or modulus versus frequency plots prepared as specified in Clause 10;

n) where possible, a plot of storage modulus against dynamic-strain amplitude at a single frequency as specified in Clause 10.

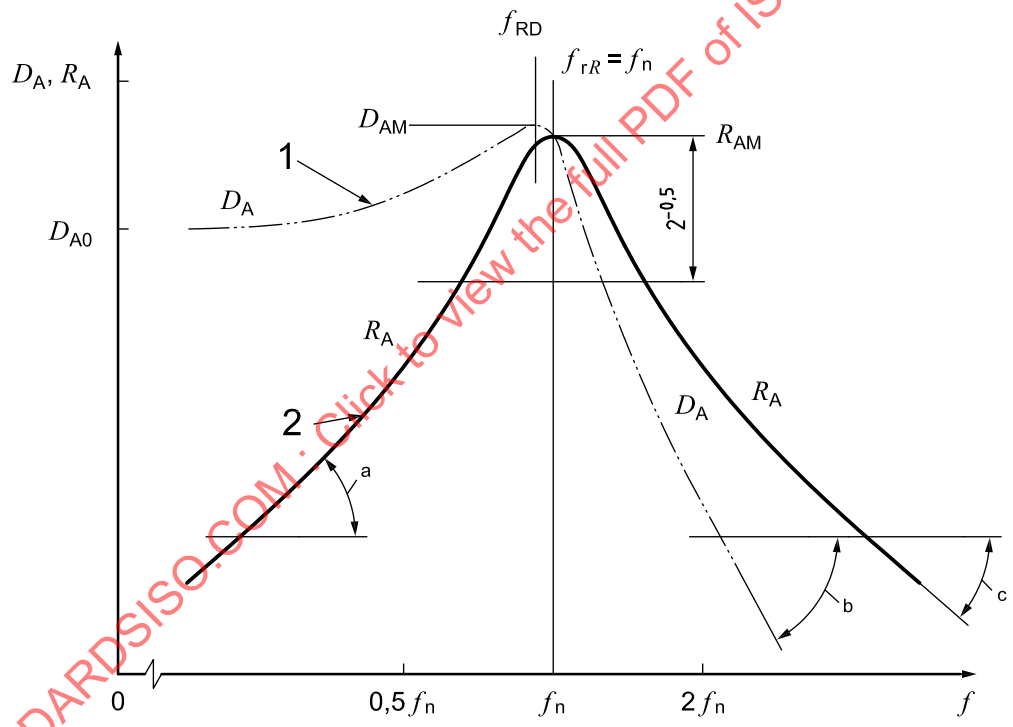
Annex A
(informative)

Resonance curves

A.1 If a mechanically inert viscoelastic system is subjected to a vibrational force with varying frequency and constant amplitude, it shows single- or multiple-resonance behaviour. This can be described in terms of the deformation amplitude D_A or deformation-rate amplitude R_A of the system.

A.2 In vibration tests, resonance behaviour is usually presented as plots of the deformation amplitude D_A of the system versus the frequency f. The characteristics of this type of resonance $D_A(f)$ are as given in A.2.1 to A.2.4.

A.2.1 For a single vibrational order at low frequencies, D_A tends to a limiting "static" amplitude D_{A0} ($f = 0$) and, at high frequencies, to a limiting slope of –2 (–40 dB/decade) when plotted using the same logarithmic scale along both axes (see Figure A.1).



The curves are plotted in terms of

1 deformation amplitude D_A versus frequency f and

2 deformation-rate amplitude R_A versus frequency

for the first vibrational order. Double-logarithmic plots have been made, assuming a loss factor $\tan\delta$ of 0,6. D_{AM} and R_{AM} are the resonance amplitudes, f_{RD} and f_{rR} are the corresponding peak frequencies and f_n is the natural frequency (peak frequency, without damping, at $\tan\delta = 0$).

[a] Slope +1.

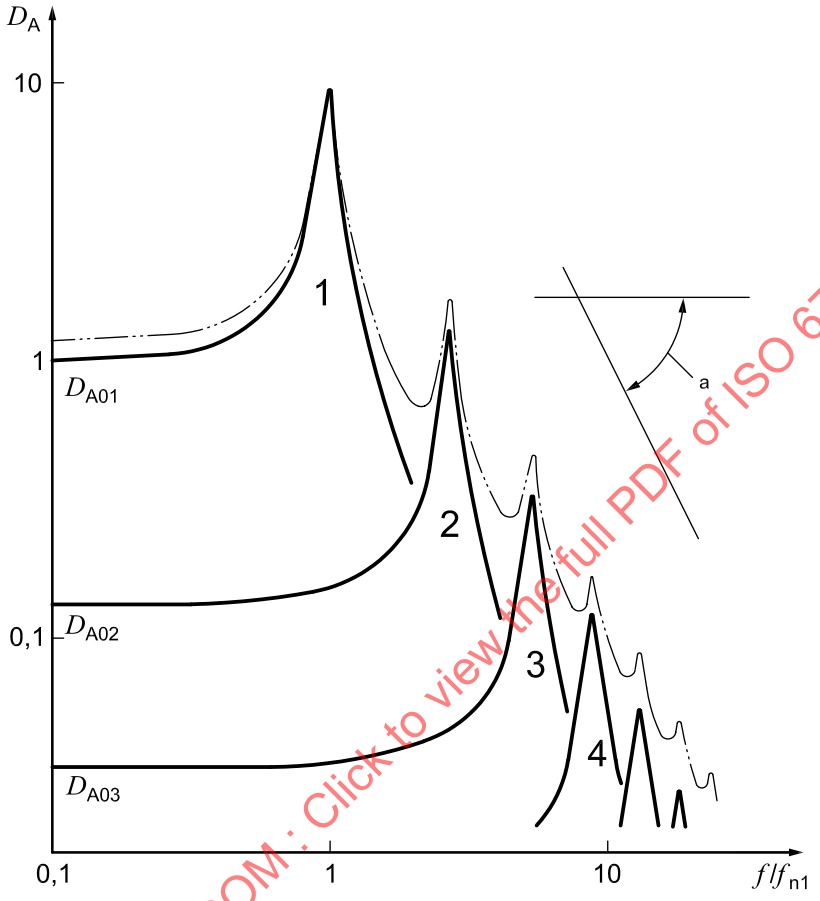
[b] Slope –2.

[c] Slope –1.

Figure A.1 — Resonance curves

A.2.2 The resonance frequency f_{RD} at the peak amplitude D_{AM} differs from the natural frequency f_n (of the same system but without damping). This last parameter, however, determines the value of the storage component M' of the complex modulus. M' can therefore only be calculated approximately from $D_A(f)$ curves.

A.2.3 Within a series of vibrational orders i, the resonance amplitudes D_{AMi} decrease markedly, approximately in proportion to $(f_{Ri})^{-2}$ (see Figure A.2).



Solid line: Single orders $i = 1, 2, 3, ...$

Dotted line: Sum of the single orders (multiple-resonance curve)

f_{n1} is the natural frequency for first-order oscillations

[a] Slope –2.

Figure A.2 — Resonance curves plotted for the deformation amplitudes D_A of oscillational order $i = 1, 2, 3$ for a flexurally vibrating specimen with both ends free and for $\tan\delta = 0,1$