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Plastics — Determination of dynamic mechanical properties —

Part 12:
Compressive vibration — Non-resonance method

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 12: Vibration en compression — 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 6721-12 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

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*

Plastics — Determination of dynamic mechanical properties —

Part 12:
Compressive vibration — Non-resonance method

1 Scope

This part of ISO 6721 describes a compressive vibration, non-resonance method for determining the components of the compressive 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 of semi-rigid polymers in the range 1 MPa to 1 GPa (see Notes 1 and 2).

NOTE 1 The method is applicable to the following semi-rigid polymers:

— low-density polyethylene, ultra-high-molecular-weight polyethylene, polybutylene, and ethylene/propylene/diene copolymer;

— polytetrafluoroethylene, plasticized poly(vinyl chloride), and ethylene/vinyl acetate copolymer;

— thermoplastic elastomers and polyurethane.

NOTE 2 The measurement of dynamic properties of materials with different ranges of moduli can be made using alternative modes of deformation (i.e. a shear mode for 0,1 MPa $< E' <$ 50 MPa, a tensile mode for 0,01 GPa $< E' <$ 5 GPa, a torsional mode for 10 MPa $< E' <$ 10 GPa, and flexural modes for $E' >$ 5 GPa.

This method is particularly suited to the measurements of dynamic moduli and loss factors of semi-rigid plastics in the shape of a right-angled prism, cylinder or tube and can 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 present 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 291, *Plastics — Standard atmospheres for conditioning and testing*

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

ISO 294-1, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles, and moulding of multipurpose and bar test specimens*

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

ISO 472, *Plastics — Vocabulary*

ISO 6721-1:2001, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles*

ISO 10724-1, *Plastics — Injection moulding of test specimens of thermosetting powder moulding compounds (PMCs) — Part 1: General principles and moulding of multipurpose test specimens*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6721-1 and ISO 472 apply.

4 Principle

The specimen is subjected to a sinusoidal compressive force or deformation at a frequency significantly below the fundamental resonance frequency for the clamped/free longitudinal mode. 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 compressive complex modulus and the loss factor are calculated.

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 compressive 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 compression plate 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 chamber.

NOTE Whilst each member of the loading assembly might have a much higher stiffness than the specimen, the presence of bolted connections can significantly increase the apparatus compliance.

At the other end of the specimen, a second compression plate 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 Compression plates

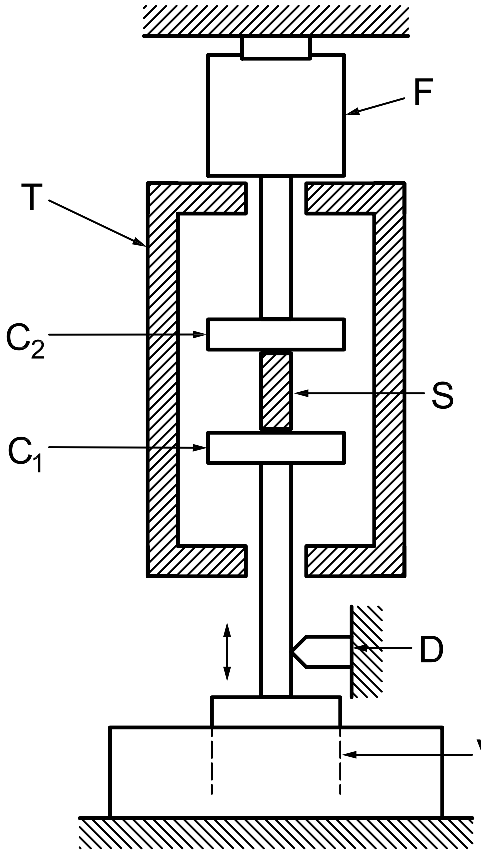
Compression plates, for supporting the test specimen and applying the compressive vibration force to the test specimen, shall have a surface finish corresponding to an average surface roughness of $Ra = 0{,}25$ µm and shall have no visible imperfections. The surfaces shall be parallel to each other in a plane normal to the loading axis.

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 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 %.



Key

F force transducer

T temperature-controlled enclosure

C_1, C_2 compression plates

S test specimen

D displacement transducer

V vibrator

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

5.3 Temperature measurement and control

The test specimen and the compression plates shall be enclosed in a temperature-controlled enclosure containing air or a suitable inert gas for purging purposes.

The enclosure shall be designed so that its temperature can be varied over the range sufficient for the materials under test (e.g. −100 °C to +250 °C). It is recommended that the chamber be equipped with temperature-programming facilities.

The devices used for measuring the temperature of the air or the inert gas surrounding the specimen shall be capable of determining the temperature to ±0,5 °C.

5.4 Devices for measuring test specimen dimensions

See ISO 6721-1:2001, Subclause 5.6.

The devices used for measuring the dimensions of the test specimen shall be capable of determining these quantities to ±0,5 %.

6 Test specimens

6.1 Shape and dimensions

The test specimen shall be in the shape of a right prism or cylinder whose length is twice its principal width or diameter. The preferred specimen sizes are (4 ± 0,2) mm × (4 ± 0,2) mm × (8 ± 2) mm (prism) or (5 ± 0,2) mm in diameter and (10 ± 2) mm in length (cylinder).

The relationship between the dimensions of the test specimen and the size of the product will determine the possibility of using preferred test specimens. If the use of the preferred test specimen is impossible, it should be noted that the ratio of length/width (or diameter) of the test specimen has very significant influence on the test results.

When the ratio of length/width (or diameter) of the test specimen is smaller, barrelling of the specimen under compression and the shape factor of the dimensions of the specimen should be considered. On the other hand, take care to consider buckling of the test specimen during compression for test specimens of which the ratio of length/width (or diameter) is larger.

6.2 Preparation

Test specimens shall be prepared in accordance with the relevant material specification. When none exists, or if otherwise agreed by the interested parties, test specimens can be machined from specimens that are compression moulded or injection moulded from materials in accordance with ISO 293, ISO 294-1, ISO 295 or ISO 10724-1, as appropriate. Great care shall be taken in machining the ends so that smooth, flat, parallel surfaces, perpendicular to the longest axis of the specimen, result.

It is recommended to machine the end surfaces of the test specimen within the parallelism of 0,025 mm/100 mm using a lathe or a milling machine.

When the material shows a significant difference in compressive properties in two principal directions, it shall be tested in these two directions.

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 wide range for quality control purposes, one test specimen is sufficient. In all other cases, at least two test specimens shall be tested.

8 Conditioning

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

9 Procedure

9.1 Test atmosphere

The test temperature and the gas supply (air or inert gas) shall be chosen according to the specific type of test and the purpose of the test.

9.2 Measuring the dimensions of and mounting the test specimen

Before the test, measure the width and thickness, or diameter, of each test specimen at three points along its length, and calculate the mean value of the cross-sectional area.

Measure the length of each test specimen to 1 % accuracy.

Mount the test specimen between the compression plates, taking care to align the centre-line of its long axis with the centre-line of the loading assembly and to ensure that the ends of the test specimen are parallel with the surfaces of the compression plates.

9.3 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.

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.

9.4 Performing the test

A static compressive force shall be applied to the test specimen that is sufficient to maintain the load under the decreasing part of the superimposed dynamic load. Also, the static compressive force shall be sufficient to prevent a toe effect. A further check may be carried out to confirm that measurements have been made within the linear-viscoelastic region.

In some cases of compression tests for plastics, a toe region is observed in a typical relationship between the compressive stress and the strain. This does not represent a property of the material and is an artifact caused by a take-up of slack and alignment of the specimen. The static compressive force applied to the specimen shall be sufficient to obtain a correct value of the modulus.

Take care to ensure that the static compressive force preloaded on to the specimen does not lead to buckling or barrelling of the specimen. If the compressive strain exceeds the limit for linear behaviour, then the derived dynamic properties will depend on the magnitude of the applied strain. Assuming linear viscoelastic behaviour, then, for an applied sinusoidal displacement or compressive stress, the resultant output of stress or displacement, respectively, will also be sinusoidal.

Record the amplitudes of the force and displacement signals, the phase difference between them and their frequency, 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 that 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).

10 Expression of results

10.1 Symbols

L length of the test specimen between compressive plates, in metres

A cross-sectional area of the test specimen, in square 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_a, δ measured phase difference and corrected phase difference, respectively, between the force and displacement cycles, in degrees

E'_a, E' apparent compressive storage modulus and corrected compressive storage modulus, respectively, in pascals

E'' compressive loss modulus, in pascals

$\tan\delta$ compressive loss factor

10.2 Calculation of the compressive storage modulus E'

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

$$E'_a = \frac{\Delta F_A}{s_A} \times \frac{L}{A} \cos\delta_a \quad (1)$$

NOTE 1 Equation (1) becomes invalid as the drive frequency approaches the fundamental resonance frequency of the specimen. Also, at sufficiently high frequencies, the applied deformation will excite the transducer into resonance.

NOTE 2 If the length of the specimen between the compressive plates cannot be measured directly, a correction for the compliance of the test assembly is necessary to obtain the corrected dynamic storage modulus E'. Also, if the source of compliance arises in the loading assembly, there might be an influence on the measured phase angle δ_a. However, the correction for apparatus compliance can be negligible for the measurement of dynamic properties of semi-rigid plastics when each member of the loading assembly has a much higher stiffness than the specimen.

10.3 Calculation of the loss factor $\tan\delta$

A value for the loss factor $\tan\delta$ is calculated using the phase angle δ, the phase difference between the dynamic stress and the dynamic strain in a viscoelastic material subjected to a sinusoidal oscillation. The phase angle is expressed in radians.

10.4 Calculation of the compressive loss modulus E''

The loss modulus E'' shall be calculated from

$$E'' = E' \tan\delta \quad (2)$$

10.5 Presentation of data as a function of temperature

See ISO 6721-1:2001, Clause 10.

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.

11.1 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_A/L;

o) any test conditions agreed upon that deviate from this part of ISO 6721.

Bibliography

[1] ISO 604, *Plastics — Determination of compressive properties*

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