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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

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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.

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 on 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 the following URL: 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 6721-12:2009), which has been technically revised.

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

— NOTE 1and NOTE 2 in Clause 1 have been removed;

— the normative references have been updated;

— the description for alternate loading assemblies has been added and the accuracy specification for apparatus has been updated;

— the preferred test specimen sizes have been removed and the requirement for the ratio of thickness/width of the specimen has been added;

— the document has been revised editorially.

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 12:
Compressive vibration — Non-resonance method

1 Scope

This document 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 applicable for measuring dynamic storage moduli of semi-rigid polymers in the range 1 MPa to 1 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).

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:

— IEC Electropedia: available at https://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

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

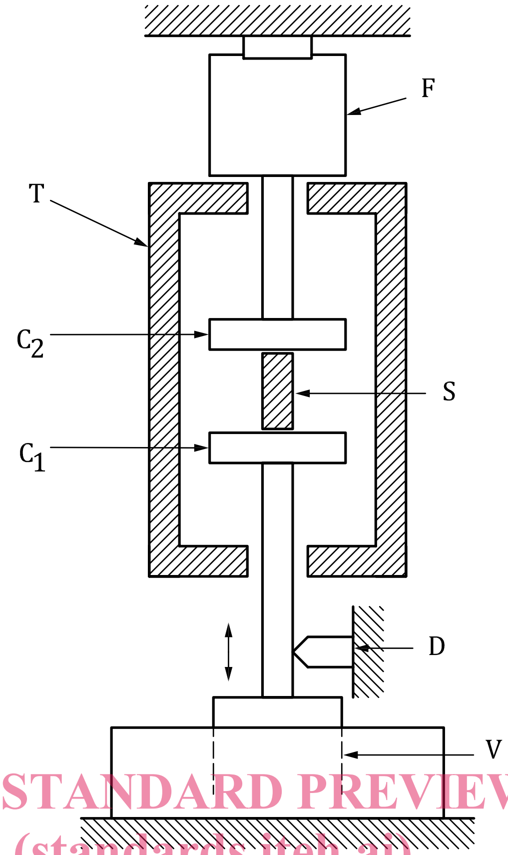
5.1.1 General

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; an example of 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.

Various other loading assemblies may be employed as alternatives to that detailed above. For example, the force on the specimens may be calculated from the current supplied to the vibrator.



Key

F	force transducer	S	test specimen
T	temperature-controlled enclosure	D	displacement transducer
C_1, C_2	compression plates	V	vibrator

NOTE The arrangement of vibrator V and transducers for displacement D and force F can also be reversed. The horizontal configuration of apparatus is also available.

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

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 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.

NOTE If the transducer is part of the particular instrument and cannot be removed, the calibration is involved with that of the electronics (see 5.2).

5.2 Electronic data-processing equipment

Data-processing equipment shall be capable of recording the force and displacement cycle amplitudes to an accuracy of ±0,5 %, the phase angle between the force and displacement cycles to an accuracy of ±0,05° and the frequency to an accuracy of ±1 %.

5.3 Temperature measurement and control

According to ISO 6721-1.

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

According to ISO 6721-1.

The devices used for measuring the dimensions of the test specimen shall be capable of determining these quantities to ±0,5 % or ±0,05 mm.

6 Test specimens

6.1 Shape and dimensions

The test specimen shall be in the shape of a right prism or cylinder. The cross-sectional area shall be selected so that the apparatus range is able to generate compressive displacements and to detect forces with adequate accuracy.

The shape factor of the dimensions of the specimen should be considered. The ratio of length/principal width (or diameter) of the test specimen shall be in the range of 1 to 2. Take care to consider buckling or barrelling of the test specimen during preloading compression for test specimens of which the ratio of length/width (or diameter) is larger (see 9.4). In order to prevent buckling or barrelling, the ratio shall preferably be 1 and not exceed 2.

NOTE The size and the ratio of length/width (or diameter) of the test specimen can influence on the test results.

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 of the test specimen 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

According to ISO 6721-1.

8 Conditioning

According to ISO 6721-1.

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 ±0,5 % or ±0,05 mm, whichever is smaller.

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. The static compressive force applied to the specimen shall be sufficient to obtain a correct value of the modulus.

NOTE 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 artefact caused by a take-up of slack and alignment of the specimen.

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 changing frequency, keeping the temperature constant. The frequency range is then repeated at the next higher temperature (see ISO 6721-1).

10 Expression of results

10.1 Calculation of the compressive storage modulus E'

The compressive storage modulus E' is determined from the Formula (1).

$$E' = \frac{\Delta F_A}{s_A} \times \frac{L}{A} \cos\ \delta \tag{1}$$

where

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

A is the cross-sectional area of the test specimen, in square metres;

f is the measurement frequency, in hertz;

s_A is the measured amplitude of the dynamic displacement, in metres;

ΔF_A is the measured amplitude of the dynamic force, in newtons;

δ is the measured phase difference between the force and displacement cycles, in radians;

E' is the compressive storage modulus in pascals.

NOTE 1 Formula (1) becomes invalid as the drive frequency approaches the fundamental resonance frequency of the specimen. Also, at sufficiently high frequencies, the applied deformation excites 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. 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.

10.2 Loss factor tan δ

A value for the loss factor tan δ 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.

10.3 Compressive loss modulus E''

The loss modulus E'' is given by Formula (2).

$$E'' = E' \tan \delta \tag{2}$$

where

E' is the compressive storage modulus, in pascals;

E'' is the compressive loss modulus, in pascals;

$\tan \delta$ is the compressive loss factor.

10.4 Presentation of data as a function of temperature

According to ISO 6721-1.

