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Plastics — Determination of dynamic mechanical properties —

Part 9: Tensile vibration — Sonic-pulse propagation method

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 9: Vibration en traction — Méthode de propagation de signaux acoustiques

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Contents

Page

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 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 6721-9:1997), which has been technically revised. It also incorporates the Amendment ISO 6721-9:1997/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 9:
Tensile vibration — Sonic-pulse propagation method

1 Scope

This document describes a pulse propagation method for determining the storage component of the complex tensile modulus *E*' of polymers at discrete frequencies typically in the range 3 kHz to 10 kHz. The method is suitable for measuring materials with storage moduli in the range 0,01 GPa to 200 GPa and with loss factors below 0,1 at around 10 kHz. With materials having a higher loss, significant errors in velocity measurement are introduced through decay of amplitude.

The method allows measurements to be made on thin films or fine fibres and long specimens, typically tapes of 300 mm × 5 mm × 0,1 mm or fibres of 300 mm × 0,1 mm (diameter).

This method may not be suitable for cellular plastics, composite plastics and multilayer products.

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 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 and the following 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/

3.1
longitudinal sonic pulse
single sonic pulse where the deformations coincide with the direction of propagation of the pulse

4 Principle

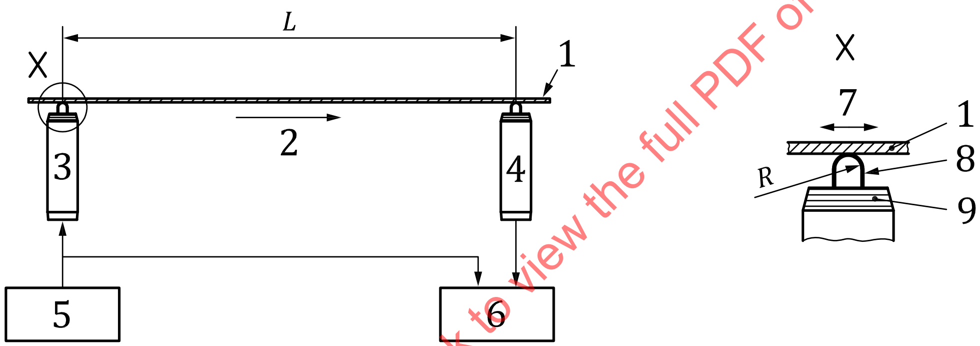
Measurements are made of the velocity of a single longitudinal sonic wave in a longitudinal thin specimen. The velocity of the longitudinal sonic wave is determined by measuring the transit time of a sonic pulse between two transducers attached to the specimen over a frequency range from 3 kHz to 10 kHz. A longitudinal sonic pulse is transmitted along the length of the specimen. The velocity is independent on the specimen geometry, if the sonic velocity is measured in a long, thin specimen. The tensile storage modulus is calculated from the product of specimen density and the square of sonic velocity.

5 Test device

5.1 Apparatus

The requirements for the apparatus shall enable measurement of the velocity of a longitudinal sonic pulse in a specimen.

Figure 1 shows schematically an example for measuring pulse velocity in a test specimen between the transmitting and receiving transducers.



Key

1	test specimen	7	direction of vibration
2	sonic pulse	8	transducer edge
3	transmitting transducer	9	transducer
4	receiving transducer	*R*	radius of transducer edge
5	transducer drive unit	*L*	transducer separation distance (m)
6	arrival time measuring equipment		

Figure 1 — Schematic diagram of suitable apparatus for measuring sonic pulse velocity between a transmitting and a receiving transducer

5.2 Transducers

Two piezoelectric transducers having a resonant frequency in the range from about 3 kHz to 10 kHz shall be mounted on the frame so that the direction of the vibration of each transducer accurately coincides with the direction to the position of the other transducer. A mechanical pulse having longitudinal displacement in the test specimen is generated by the transmitting transducer. A pulse propagated through a test specimen is detected by the receiving transducer. One transducer shall be movable so that the distance between them can be varied from about 50 mm to 500 mm, and accurately measured to ± 0,5 % of the distance between the transducers.

5.3 Transducer drive unit

This unit shall provide a suitable pulse voltage for the transmitting transducer to produce a sonic pulse.

5.4 Pulse arrival time measuring equipment

This instrument shall be capable of measuring the time interval between two pulses, one from transducer drive unit and the other from receiving transducer to an accuracy of ± 0,5 µs.

NOTE The time interval will depend upon the distance between the transmitting and receiving transducers and the sonic wave velocity in the material.

5.5 Temperature measurement and control equipment

According to ISO 6721-1.

6 Test specimens

6.1 General

The test specimens shall be in accordance with ISO 6721-1.

6.2 Shape and dimensions

Test specimens in the form of a thin film or fibre are suitable. The dimensions are not critical, however, specimens 200 mm to 500 mm in length, 1 mm to 10 mm in width, and 0,1 mm to 1 mm in thickness or diameter are suitable.

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 Mounting the specimen

Place a test specimen in the apparatus so that the received pulse has a suitable amplitude to determine its arrival time. Poor contact between the test specimen and the transducers makes the pulse energy inadequate for determining the sonic pulse propagation time.

For not rigid specimens, apply a small tension to the specimen to make it tight, but not stretched.

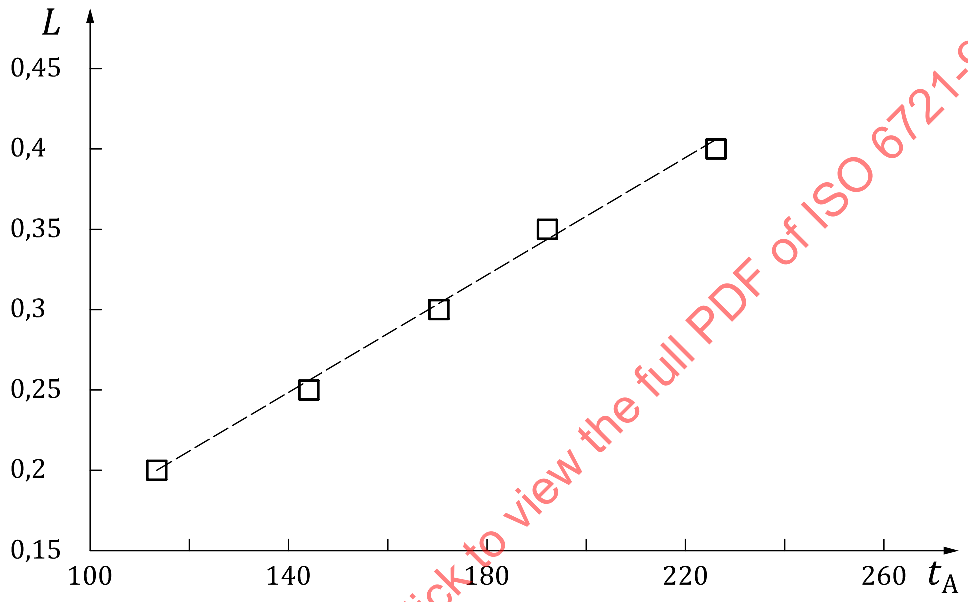
9.3 Performing the test

9.3.1 Position the transducers so that the longitudinal sonic pulse will be transmitted through the test specimen. The separation distance between them is determined so that the sonic pulse arrival time shall be at least 100 µs for good accuracy (See Note). Measure the separation distance of the transducers, L (m) to an accuracy of ± 0,5 %.

NOTE Since most plastic materials will have a pulse velocity between 1 and 2,5 × 10^3 m·s^{-1}, the minimum separation distance will generally be 0,1 m to 0,25 m.

9.3.2 Apply a sonic pulse to the specimen, and measure the arrival time t_A (s) to an accuracy of ± 0,5 µs.

9.3.3 Repeat the measurements described in 9.3.1 and 9.3.2 for the same specimen at least three different separation distances between the transducers. In order to get good accuracy, the values of the distance should be selected so as to be distributed uniformly within the whole range of the distance suitable for determining the arrival time (See Figure 2).



Key

t_A observed arrival time (µs)

L transducer separation distance (m)

□ observed values

– – – – linear regression

Figure 2 — Example of linear regression of distance and arrival time

9.3.4 Measure the density of the test specimen, ρ (kg·m^{-3}) at the same temperature as that the pulse arrival time is measured 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 Varying the temperature

According to ISO 6721-1.

10 Calculation and expression of results

10.1 Symbols

E' (Pa)	tensile storage modulus
L (m)	transducer separation distance
L_i (m)	transducer separation distance for ith measurement
t_A (s)	observed pulse arrival time
t_{Ai} (s)	observed pulse arrival time corresponding to L_i
v_L (m·s^{-1})	longitudinal wave velocity in the test specimen
ρ (kg·m^{-3})	density of the test specimen

10.2 Calculation of the longitudinal wave velocity

The wave velocity, v_L is given by the slope, $\Delta L/\Delta t_A$ of the plots of L_i vs. t_{Ai}. The value of the slope shall be obtained from at least three measurements at different distance of the transducer separation, analysing the data with linear regression (See Figure 2).

$$v_L = \Delta L / \Delta t_A \quad (1)$$

10.3 Calculation of the tensile storage modulus, *E*'

The tensile storage modulus, E' is calculated using Formula (2).

$$E' = \rho v_L^2 \quad (2)$$

10.4 Significant figures

Calculate the wave velocity, v_L and the tensile storage modulus, E' to three significant figures.

11 Precision

The precision of this test method is not known because interlaboratory data are not available due to the difficulty in finding laboratories with test equipment capable of operating at the same frequency. It is recognized that the properties of thermoplastics are time-dependent, and the pulse propagation time and hence the dynamic modulus depend closely on the frequency of the sonic pulse used. For information purposes, however, the within-laboratory standard deviation has been determined using data from one laboratory which tested four different materials (see Annex A).

12 Test report

The test report shall contain the following information:

a) a reference to this document, i.e. ISO 6721-9;

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 films or sheets, their thickness and, if applicable, the direction of the major axes of the test specimens in relation to some feature of the films or sheets;

d) the shape and dimensions of the test specimens;

e) the method of preparing the test specimens;

f) details of the conditioning of the test specimens;

g) the number of test specimens tested;

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

i) sonic wave frequency used;

j) the method of density measurement of test specimens;

k) the individual test results, if required;

l) the mean values of the test results;

m) the date of the test.

Annex A
(informative)

Precision

A.1 For the reasons outlined in Clause 11, the precision of this method is not known. However, Table A.1 gives repeatability data based on testing performed on identical test pieces in the same laboratory under the same conditions by the same operator using the same equipment within short intervals of time.

The densities of specimens were measured by use of a density gradient column in accordance with ISO 1183-2. The v_L and E' values reported are average values obtained from 10 measurements. It can be seen that a coefficient of variation of up to about 3 % can be expected within a particular laboratory.

Table A.1 — Repeatability data for tensile storage modulus E' measured at 10 kHz

Material	**Wave velocity, v_L** m·s^{-1}	**Specimen density, ρ** kg·m^{-3}	**Average value of E'** GPa	**Standard deviation**	**Coefficient of variation** %
PE-UHMW	1,366 × 10^3	944,8	1,762	0,009 3	0,53
PP	1,768 × 10^3	895,4	2,811	0,054 2	1,93
Uniaxially stretched PP	3,505 × 10^3	898,7	11,10	0,280	2,50
PEEK	1,803 × 10^3	1 302	4,232	0,032 8	0,77

A.2 When this method is used with certain materials, consideration should be given to various factors that can lead to a decrease in the repeatability of the measured values. Such factors include the following:

a) poor contact between the specimen and the transducers, which may give rise to a low amplitude of the sonic pulse and associated errors in determining the pulse propagation time;

b) anisotropy in the properties of the material, caused by molecular orientation, which will give rise to a dependence of the pulse propagation time on direction in the specimen;

c) the presence of filler or reinforcement in the material such that the distribution or orientation of the filler or reinforcement affects the pulse propagation time.

Since the properties of thermoplastics are time-dependent[1], the pulse propagation time and hence the dynamic modulus depend closely on the frequency of the sonic pulse used[2]. It is therefore not possible to make accurate comparisons of results obtained using different frequencies.