INTERNATIONAL STANDARD

ISO 6721-8

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Plastics — Determination of dynamic mechanical properties —

Part 8:

Longitudinal and shear vibration — Wave-propagation method

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 8: Vibrations longitudinale et en cisaillement — Méthode de propagation des ondes

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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.

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.

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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*

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Plastics — Determination of dynamic mechanical properties —

Part 8:
Longitudinal and shear vibration — Wave-propagation method

1 SCOPE

This part of the International Standard ISO 6721 describes an ultrasonic wave propagation method for determining the storage components of the longitudinal complex modulus L* and the shear complex modulus G* of polymers at discrete frequencies typically in the range 0.5 MHz to 5 MHz. 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 1 MHz. With materials that have a higher loss, significant errors in velocity measurement are introduced through waveform distortion and can only be reduced using procedures that are outside the scope of this standard.

The method allows measurements to be made on small specimens, typically 50 mm x 20 mm x 5 mm, or small regions of larger specimens or sheets. It is therefore possible to obtain information on the homogeneity or anisotropy (see clause 10.5) of modulus in a specimen.

2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to use the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1183:1987, Plastics - Methods for determining the density and relative density of non-cellular plastics.

ISO 6721-1:1994, Plastics - Determination of dynamic mechanical properties - Part 1: General principles.

3 DEFINITIONS

See ISO 6721-1, 4.

3.1 LONGITUDINAL MODULUS

The ratio of a uniaxial tensile or compressive stress applied to a specimen to the resulting uniaxial strain when the strain in a plane transverse to the axis of applied stress is zero. See table 5 in ISO 6721-1 for relationships between this and other moduli.

3.2 LONGITUDINAL ACOUSTIC WAVE

A sound wave in which the particle displacement is in the direction of wave propagation.

3.3 TRANSVERSE ACOUSTIC WAVE

A sound wave in which the particle displacement is perpendicular to the direction of wave propagation.

3.4 BULK WAVE

The mode of propagation of an acoustic wave in a material whose boundaries normal to the direction of propagation are infinitely remote. This mode is realised in practice for waves whose wavelength is much less than the dimensions of the specimen transverse to the direction of propagation. In practice, the acoustic wave frequency is then ultrasonic.

4 PRINCIPLE

Measurements are made of the velocity of longitudinal and transverse acoustic waves in a specimen and the specimen density. The frequency of the wave is chosen so that its wavelength in the specimen is significantly less than the specimen dimensions in a plane transverse to the direction of wave propagation. The wave then propagates as a bulk wave. The longitudinal and shear storage moduli are given by the product of the material density and the square of the longitudinal and the shear wave velocities respectively.

Two methods are described in this international standard for measuring wave velocities. In the immersion method, the specimen intercepts a beam of longitudinal acoustic wave pulses passing between a transmitting and receiving transducer in a bath of a suitable liquid. At normal incidence, longitudinal wave pulses are excited in the specimen. As the angle of incidence is increased, the amplitude of the longitudinal refracted wave decreases and a refracted transverse (shear) wave is generated. Longitudinal and transverse wave velocities are deduced from measurements of differences in pulse transit times with and without the specimen in the beam and a knowledge of the velocity of sound in the liquid.

In the transducer contact method, the specimen is sandwiched between two transducers, one launching and the other receiving acoustic wave pulses. For the determinaion of longitudinal and transverse wave velocities, transducer pairs having longitudinal and transverse polarisations, respectively, are used. Wave velocities are again obtained from measurements of differences in pulse transit times with and without the specimen in the beam.

5 TESTING DEVICE

5.1 APPARATUS

The requirements of the apparatus are that it shall enable measurement of the velocities of longitudinal and transverse ultrasonic waves in a specimen. Two methods are described in this International Standard.

5.1.1 Method A: Immersion method

Figure 1a shows, schematically, suitable apparatus for measuring velocity by an immersion method. Two ultrasonic transducers are mounted coaxially in a bath containing a liquid, one acts as a transmitter T of longitudinal ultrasonic wave pulses and the other as a receiver R. The transmitter is driven by a series of high-voltage, short-duration electrical pulses from the transducer drive unit. A pulse repetition interval of about 1 ms is satisfactory. Acoustic pulses launched by the transmitter travel through the liquid and the specimen and are detected by the receiving transducer. The specimen is mounted on a turntable, located between the transducers T and R, such that the angle of incidence of the acoustic beam can be varied and measured to ± 0.5°. The specimen can be removed from the beam. The receiving transducer is connected to electronic equipment that will enable measurement of the difference in the arrival times of pulses received with and without the specimen in the beam. An oscilloscope, whose timebase is accurately calibrated and triggered by the transducer drive unit, is suitable for this purpose.

The receiving transducer may be replaced by a reflecting surface, such as a metal block, positioned normal to the axis of the transmitter as shown in figure 1b. The transmitting transducer is now used to detect the beam of pulses reflected back through the liquid and the specimen and is connected to the transit-time measuring equipment (see note 1).

(Note 1. This test arrangement may be more appropriate if the specimen is only available as a thin sheet since the transit time in the specimen is twice that obtained using the transmitter and receiver arrangement.)

5.1.2 Method B. Transducer contact method

Figure 2a shows a method for measuring wave velocity by direct contact between the transmitting and receiving transducers and the surfaces of the specimen. For the determination of the longitudinal wave velocity, transducer pairs that launch and receive longitudinal acoustic waves are used whilst, for the determination of the transverse wave velocity, shear (transverse) wave transducers are employed. The transducer separation can be varied to accommodate specimens of different thickness including direct contact between the two transducers. A coupling fluid is necessary to maximise the pulse amplitude transmitted to the specimen and to the receiver.

The receiver may be replaced by a reflecting surface in contact with the specimen as shown in figure 2b. The transmitting transducer is now used to detect the beam of pulses reflected back through the specimen and is connected to the transit time measuring equipment (see note 1).

5.2 TRANSDUCERS

When driven by the transducer drive unit, the transmitter should produce a short pulse at its natural frequency that has a duration of around three or four cycles. A suitable waveform is shown in figure 3. Pulses of longer duration are satisfactory but may not allow measurement of wave velocities by timing the interval between pulses that have been internally reflected by the specimen surfaces owing to an overlap of those pulses.

In either of the test arrangements shown in figure 2, the transmitter should possess a suitable buffer material located between the acoustic resonating device and the surface of the transmitter in order to prevent the contact with the specimen, the receiver or the reflector from influencing the acoustic performance of the transmitter and hence the shape of the pulses generated.

5.3 TRANSIT-TIME MEASURING EQUIPMENT

Data processing equipment shall be capable of measuring the time interval between two received pulses to an accuracy of ± 0.5% of the time interval (see note 2).

(Note 2. The time interval between received pulses will depend upon the thickness of the specimen and the wave velocity in the material. For attenuating materials, such as most polymers, where specimens of only a few millimetres in thickness can be used, time intervals will be in the region of one microsecond.)

The use of a digital storage oscilloscope having a high sampling rate or an oscilloscope whose time base is triggered by the transducer drive unit through an accurate digital delay circuit are suitable for this purpose.

5.4 TEMPERATURE MEASUREMENT AND CONTROL

See ISO 6721-1, sub clause 5.5 and note 3.

(Note 3. The determination of wave velocity using the methods described in this standard involve measuring the time interval between two received pulses. When these pulses are obtained with and without the specimen in the acoustic beam, it is important that the temperature of the apparatus has not changed significantly between the two measurements. As general guidance, any temperature change should be less than 0.5 °K using the transducer contact method and less than 0.2 °K using the immersion method.)

6 TEST SPECIMENS

See ISO 6721-1, clause 6.

6.1 SHAPE AND DIMENSIONS

Test specimens in the shape of a bar or plate are suitable. The surfaces normal to the wave direction must be smooth, plane and parallel over an area comparable with the area of the faces of the transmitter and receiver. The dimension d of the specimen in the wave direction shall not vary by more than ± 0.2% over this area.

In order to ensure that it is the bulk wave velocity that is measured (see clause 4.4), the dimensions transverse to the wave direction shall be greater than 3 x the longitudinal pulse wavelength in the specimen. The wavelength λ(m) can be calculated from a knowledge of the pulse frequency f(Hz) and the longitudinal wave velocity in the specimen v_L(ms^{-1}) using the equation

$$\lambda = \frac{v_L}{f} \qquad (1)$$

6.2 PREPARATION

See ISO 6721-1, sub clause 6.2.

7 NUMBER OF SPECIMENS

See ISO 6721-1, clause 7.

8 CONDITIONING

See ISO 6721-1, clause 8.

9 PROCEDURE

9.1 TEST ATMOSPHERE

See ISO 6721-1, sub clause 9.1.

9.2 MEASURING THE SPECIMEN DIMENSION

Measure the dimension of the specimen in the direction of wave propagation at 3 points within the area through which the ultrasonic beam will travel. If these measurements vary by more than ± 0.5%, identify a different region of the specimen or choose another specimen.

9.3 PERFORMING THE TEST

9.3.1 Method A: Immersion method

With the specimen absent from the ultrasonic beam, identify a reference point on the received pulse that may be used to accurately record an arrival time for the pulse. A point where the pulse amplitude passes through zero volts early in the pulse is recommended for this purpose as shown in figure 3. Record the reference point time (see note 4).

(Note 4. With viscoelastic or multiphase materials, changes in pulse shape after transmission through the specimen can arise because of dispersion or scattering. This leads to an error in transit-time measurement which is difficult to quantify. This error can be minimised by