61

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Plastics — Testing with the torsion pendulum

Plastiques — Essai au pendule de torsion

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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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 537 was prepared by Technical Committee ISO/TC 61, *Plastics.*

This second edition cancels and replaces the first edition (ISO 537 : 1980), of which it constitutes a technical revision.

Annexes A and B of this International Standard are for information only.

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Plastics — Testing with the torsion pendulum

1 Scope

This International Standard specifies two methods (A and B) for determining, as a function of temperature, dynamic mechanical properties (shear modulus and mechanical damping) of plastics for small deformations within the frequency range from 0,1 Hz to 10 Hz. The values of shear modulus and mechanical damping as measured by one of these methods may not be in agreement with the values as measured by the other. The temperature dependence of these quantities over a sufficiently broad range of temperatures (for example from − 50 °C to + 150 °C for the majority of commercially available plastics) allows the transition regions (for example the "glassy/rubbery" transition) of the polymer to be determined. It also gives information concerning the onset of plastic flow. Methods A and B described here are not applicable to non-symmetrical laminates, for which attention is drawn to ISO 6721.

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 investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291 : 1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 293 : 1986, *Plastics — Compression moulding test specimens of thermoplastic materials.*

ISO 294 : 1975, *Plastics — Injection moulding test specimens of thermoplastic materials.*

ISO 472 : 1988, *Plastics — Vocabulary.*

ISO 2856 : 1981, *Elastomers — General requirements for dynamic testing.*

ISO 4593 : 1979, *Plastics — Film and sheeting — Determination of thickness by mechanical scanning.*

ISO 6721 : 1983, *Plastics — Determination of damping properties and complex modulus by bending vibration.*

3 Definitions

For the purposes of this International Standard, the following definitions, as well as those given in ISO 472, ISO 2856 and ISO 6721, apply.

3.1 shear modulus, G: For a body under shear, the shear modulus G is the ratio of the shear stress and the shear strain. The shear modulus is determined by measuring the frequency of free torsional oscillations of small amplitude. Defined in this way, the shear modulus is a measure of the stiffness of the material under test.

3.2 logarithmic decrement of damping, Λ: For an oscillating body, the logarithmic decrement of damping Λ is the natural logarithm of the ratio of the amplitudes of two successive oscillations in the same direction. For a test specimen made of a particular material, the logarithmic decrement is a measure of the internal damping properties of the material.

3.3 mechanical loss factor, d: A parameter (see annex A) represented to a good approximation by the formula

$$d \approx \frac{\Lambda}{\pi} \qquad \ldots (1)$$

4 Principle

A test specimen of rectangular cross-section is clamped between a fixed clamp and a rotatable clamp. The rotatable clamp is connected to an inertial member (disc) by a connecting rod in such a way that, when the disc is set in oscillatory motion about its axis, the specimen is induced to perform torsional oscillations with it.

The driving disc may be suspended, by means of the rod, from the specimen (method A), in which case the oscillating system comprises the disc, rod and specimen (see figure 1), or it may be positioned above the specimen (method B), supported by a counterweight to which it is linked by a wire, in which case the wire also forms part of the oscillating system (see figure 2).

5 Apparatus

5.1 Torsion pendulum

Two types of torsion pendulum apparatus are specified in this International Standard, namely:

— driving disc suspended from test specimen, lower end of specimen rotatable (method A, figure 1);

— test specimen suspended from driving disc (with a wire and counterweight), upper end of specimen rotatable (method B, figure 2).

In both cases, the torsion pendulum apparatus consists of the driving disc, two clamps for gripping the specimen (one of which is connected to the disc by a rod and is able to rotate freely with the disc) and a temperature-controlled chamber surrounding the specimen and the clamps. For method B, a counterweight supporting the disc with the aid of a wire is also necessary.

5.1.1 Driving disc

5.1.1.1 Method A (see figure 1)

The total mass of the disc (which may be made of aluminium, for example), the lower clamp and the rod connecting them shall be such that the tensile stress acting on the specimen does not exceed 0,1 MPa. The moment of inertia I of the disc shall be selected as a function of the stiffness of the specimen and the total mass of the disc, clamp and rod so that the natural frequency of the system, which is temperature-dependent, is between 0,1 Hz and 10 Hz.

For testing standard specimens (see 6.2), a value of I of about 3×10^{-5} kg·m^2 is recommended. If, however, a nearly constant frequency over a broad temperature range is desired, several discs with different values of I may be used, provided that the tensile stress on the specimen does not become too high (see above).

NOTE — For certain materials, e.g. filled polymers and semi-crystalline polymers, a value of I of about 5×10^{-5} kg·m^2 may be necessary.

5.1.1.2 Method B (see figure 2)

The total weight of the disc, the upper clamp and the rod shall be compensated by a suitable counterweight in such a way that the tensile stress on the specimen does not exceed 0,1 MPa. Discs with a broad range of moments of inertia may be used, however, depending on the purpose of the test. In the case of method B, not only the specimen, upper clamp, rod and disc, but also the wire supporting these parts, belong to the oscillating system.

5.1.2 Clamps

The clamps shall be designed to prevent slippage of the gripped parts of the specimen. They shall be self-aligning, so that the specimen axis is maintained coincident with the axis of rotation, and be able to adequately secure the test piece over the temperature range of interest without distortion, thus allowing the free length to be accurately known.

The clamp at the free end of the specimen shall be of low mass and negligible moment of inertia. If the latter is not the case, the moment of inertia of the whole system (consisting of this clamp, the disc and the rod connecting them) shall be determined experimentally.

In order to prevent heat conduction between the specimen and the space outside the temperature-controlled chamber, the rod connecting the clamp at the free end of the specimen with the disc shall be designed so that it is sufficiently thermally insulating.

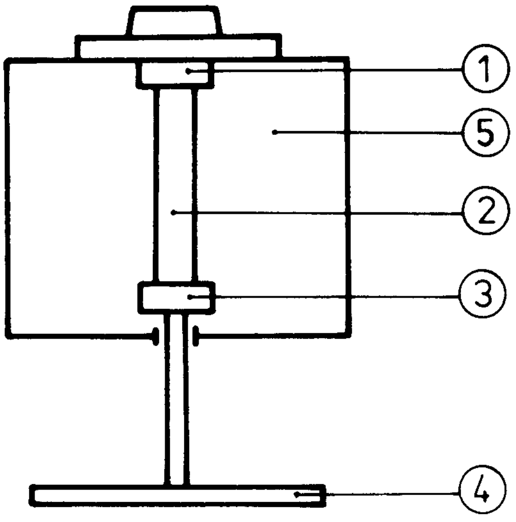
5.1.3 Temperature-controlled chamber

The test specimen and the clamps shall be enclosed in a temperature-controlled chamber. The chamber shall contain either air or inert gas, depending on the purpose of the test. When inert gas is used, it shall be under slight pressure, i.e. a stream of gas shall be passed through the chamber at a rate of about 1 200 ml/h.

The rod connecting the disc with the rotating clamp shall not be in contact with the wall of the chamber, but the distance between the rod and the wall shall be just sufficient to allow the oscillating system to move freely.

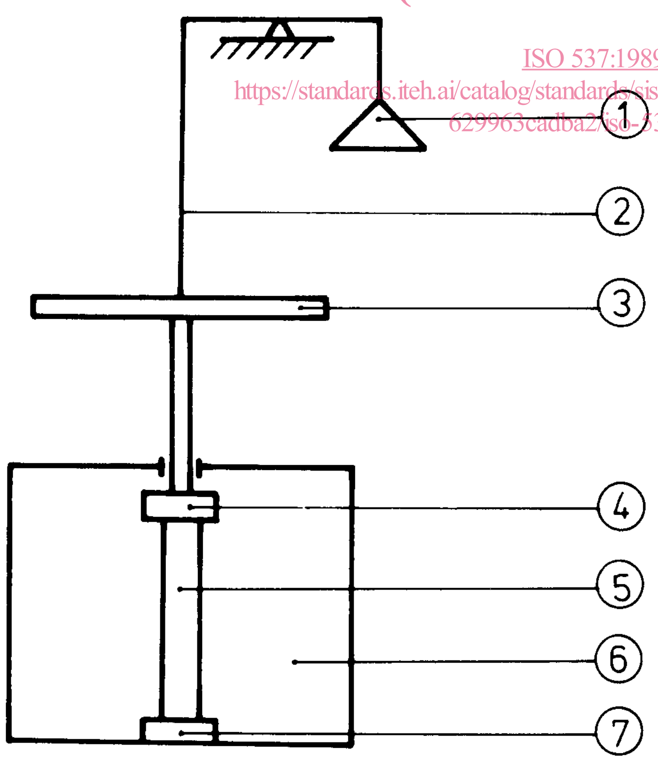
The temperature-controlled chamber shall be designed so that its temperature can be adjusted between limits wide enough apart (for instance −100 °C and 300 °C or −60 °C and 300 °C) to permit the selection of a temperature range which is suitable for the particular material under test and the purpose of the test. It is recommended that the chamber be provided with temperature-programming facilities.

The temperature in the vicinity of the specimen shall be uniform to within ± 1 K along the whole length of the specimen. If the constant-temperature procedure is used (see 7.3.1), the temperature variation shall not exceed ± 1 K during the test. When a constant rate of increase (or decrease) in temperature is to be applied (see 7.3.2), the rate shall be not greater than 120 K/h and the temperature shall not vary by more than ± 0,25 K in the close vicinity of the specimen during a single measurement (a series of free oscillations after one twist impulse has been applied to the oscillating system).



1 Upper (fixed) clamp
2 Test specimen
3 Lower (rotatable) clamp
4 Disc
5 Temperature-controlled chamber

Figure 1 — Apparatus for method A



1 Counterweight
2 Wire
3 Disc
4 Upper (rotatable) clamp
5 Test specimen
6 Temperature-controlled chamber
7 Lower (fixed) clamp

Figure 2 — Apparatus for method B

5.2 Device for setting the oscillating system in motion

The device for setting the oscillating system in motion shall be capable of applying to the oscillating system a single twist impulse such that the angle of twist of the resultant oscillatory motion does not exceed 1,5° in either direction.

When testing low-modulus materials (similar to elastomers), the angle of twist shall not exceed 3° in either direction.

5.3 Devices for recording the oscillatory motion and for measuring the oscillation frequency and the logarithmic decrement

Optical, electrical or other recording equipment may be used provided no additional damping is caused by them. If a chart recorder is employed, the chart strip shall move with a speed not varying by more than ± 1 %. The devices for measuring the oscillation frequency and the logarithmic decrement shall be capable of measuring these quantities to ± 1 % (within the transition region ± 5 %).

5.4 Devices for measuring the test specimen dimensions

The device for measuring the thickness of the specimen shall be capable of determining the thickness to ± 0,003 mm (see also ISO 4593).

The device for measuring the width and the length of the specimen shall be capable of determining these quantities to ± 0,05 mm and ± 0,1 mm respectively.

5.5 Device for measuring temperature

The device for measuring the temperature in the vicinity of the specimen shall be capable of determining the temperature to ± 0,1 K. The use of an electronic thermometer with low-inertia sensor is recommended.

6 Test specimens

The quantities measured by this method (the frequency and amplitude of oscillation) are sensitive to the dimensional uniformity of the specimen, and to its physical state (e.g. the degree of crystallinity, internal stresses) as well as to its temperature. These facts shall be considered when choosing for example the dimensions and dimensional tolerances, the method of preparation and the conditioning procedure for a particular material.

6.1 Preparation

Depending on the purpose of the test, i.e. determining the properties of a material or of a particular finished product, test specimens shall be machined from compression-moulded plates (see ISO 293) or from the finished product. Exceptionally, the specimens may be injection moulded (see ISO 294), if agreed between the interested parties.

6.2 Shape and dimensions

It is recommended that rectangular test specimens with the following dimensions be used:

— free length (distance between clamps), L: 40 mm to 120 mm, preferably 50 mm;

— width, b: 5 mm to 11 mm, preferably 10 mm;

— thickness, h: 0,15 mm to 2 mm, preferably 1 mm.

Specimens of rectangular cross-section whose thickness and/or width varies along the main axis of the specimen by more than 3 % of the mean value shall not be used. When comparing specimens made of different materials, the dimensions shall be identical. In the case of rectangular specimens whose dimensions differ from the preferred dimensions given above, the dimensions of the specimens shall be chosen, as far as possible, so that the ratios L/b and b/h are the same as for the preferred dimensions.

Specimens of other shapes (for example cylindrical or tubular) may be used. In such cases, the dimensions and dimensional tolerances shall be agreed upon by the interested parties.

Specimens having a thickness of less than 0,15 mm (down to 0,05 mm) may also be used, provided that the damping effect of the measuring system without the specimen can be calculated or neglected.

6.3 Number

For quality-control purposes, one specimen is sufficient. In other cases, two specimens shall be tested.

6.4 Conditioning

Before measuring their dimensions, the test specimens shall be conditioned as required by the specification for the material, or as agreed by the interested parties.

6.4.1 For environmental conditioning, the most appropriate set of conditions from ISO 291 shall be used, preferably 23 °C/50 % RH for 16 h.

6.4.2 For mechanical conditioning, if required, the test specimens shall be twisted by not less than 5°, but not more than 90°, in both directions and then brought back to their normal positions.

7 Procedure

7.1 Measuring the cross-section of a rectangular specimen

Before the test, measure the thickness and width of the specimen at not less than five places along its length. All specimens that have visible irregularities or that show a variation in thickness and/or width greater than 3 % of the average value shall be rejected.

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

7.2 Clamping and mounting of the test specimen

Clamp the test specimen between the upper and lower clamps. The longitudinal axis of the test specimen shall be coincident with the axis of rotation of the oscillating system. Any eccentricity will cause lateral oscillations and thus interfere with the normal oscillation process.

After clamping the test specimen, measure the distance between the clamps (the free length) to ± 0,1 mm. When mounting the oscillating system in the chamber, take care that the test specimen is not stressed.

After checking that the oscillating system is centrally mounted, start the heating (or cooling) (see 7.3) unit of the temperature-controlled enclosure.

7.3 Heating (or cooling) during the test

The determination may be carried out at constant temperature or at continuously increasing (or decreasing) temperature. In the former case, the dependence of the dynamic mechanical properties on temperature is determined by making measurements at various temperatures.

7.3.1 Constant temperature (see also 5.1.3)

Set the temperature-controlled chamber to the chosen temperature and wait for the whole apparatus to come to temperature equilibrium before setting the oscillating system in motion.

7.3.2 Continuously increasing (or decreasing) temperature (see also 5.1.3)

Set the temperature-controlled chamber to the chosen starting temperature. Allow temperature equilibrium to be established and then set the required rate of temperature change. The rate of change of temperature shall not be greater than 120 K/h.

7.4 Setting the oscillating system in motion

Induce the oscillating system to execute free oscillations either by twisting the lower end of the specimen (method A) or by rotating the disc (method B), depending upon the design of the apparatus (see 5.2). The angle of twist shall not exceed 1,5° in either direction.

Record the amplitude of the oscillations during the test. Determine the frequency of oscillation from this record.

8 Expression of results

8.1 Symbols for variables and correction factors

b width, in metres, of a rectangular specimen;

h thickness, in metres, of a rectangular specimen;

L free length, in metres, of a rectangular specimen;

I moment of inertia, in kilogram square metres ($kg \cdot m^2$), of the driving disc (if necessary inclusive of the rotatable clamp and the connecting rod);

f_A natural frequency, in hertz, of the oscillating system used in method A (specimen + driving disc + rod);

f_B natural frequency, in hertz, of the oscillating system used in method B (specimen + driving disc + rod + wire);

f_o natural frequency, in hertz, of the oscillating system used in method B less the test specimen (driving disc + rod + wire);

Λ logarithmic decrement of the oscillating system consisting of the specimen and the driving disc (including the connecting rod), in which damping of the system is caused only by the damping properties of the specimen;

Λ_B logarithmic decrement of the oscillating system used in method B, consisting of the specimen, the driving disc (including the connecting rod) and the wire (natural frequency f_B);

Λ_o logarithmic decrement of the oscillating system used in method B less the test specimen, consisting of the driving disc (including the connecting rod) and the wire (natural frequency f_o);

F_g is a term related to the dimensions of a test specimen of rectangular cross-section for which $b/h > 3$. It is given by the equation

$$F_g = \frac{3L}{b \cdot h^3 \left(1 - 0{,}63 \frac{h}{b}\right)} = \frac{3L}{b \cdot h^3 \cdot C} \quad \ldots (2)$$

where $C = 1 - 0{,}63 \frac{h}{b}$

F_{di} is a correction factor given by the equation

$$F_{di} = 1 + \frac{\Lambda_i^2}{4\pi^2} \quad \ldots (3)$$

where $\Lambda_i = \Lambda$, Λ_B or Λ_o

k is the torsional modulus, in kilogram square metres per second ($kg \cdot m^2/s$), of the wire used in method B. it is given by the equation

$$k = 4\pi^2 I \cdot f_o^2 \quad \ldots (4)$$

G is the shear modulus, in pascals, of the specimen.

NOTE — For reasons given in annex B, the correction term S_E used in the first edition of this International Standard may be neglected.

8.2 Logarithmic decrement

The logarithmic decrement Λ (method A) or Λ_B (method B) is calculated either from two successive oscillations in the same direction, as in the following equation:

$$\Lambda \text{ (or } \Lambda_B) = \ln \left(\frac{A_n}{A_{n+1}}\right) \quad \ldots (5)$$

where A_n and A_{n+1} are the amplitudes of two successive oscillations in the same direction;

or from any two oscillations in the same direction, as in the following equation:

$$\Lambda \text{ (or } \Lambda_B) = \frac{1}{n-m} \ln \left(\frac{A_m}{A_n}\right) \qquad \ldots (6)$$

where

A_m is the amplitude of the mth oscillation;

A_n is the amplitude of the nth oscillation;

$n > m$

NOTE — The following alternative equation for calculating the logarithmic decrement Λ or Λ_B can be used in the case of amplitudes that cannot be measured accurately with respect to the centreline of the damped sinusoidal curve:

$$\Lambda \text{ (or } \Lambda_B) = \ln \left(\frac{A_n^*}{A_{n+1}^*}\right) = \frac{1}{n-m} \ln \left(\frac{A_m^*}{A_n^*}\right) \qquad \ldots (7)$$

where A_n^*, A_{n+1}^* and A_m^* are the distances between successive positive and negative peaks of the curve.

8.2.1 Method A

The logarithmic decrement Λ for the specimen is given directly by equation (5), (6) or (7), as applicable.

8.2.2 Method B

With this method, equations (5), (6) and (7) give the logarithmic decrement Λ_B. The logarithmic decrement Λ for the specimen is calculated from Λ_B using equation (9) derived as shown below:

$$\Lambda = \frac{f_B \Lambda_B - f_o \Lambda_o}{\left[f_B^2 - f_o^2 + \frac{f_o \Lambda_o}{2\pi^2} (f_B \Lambda_B - f_o \Lambda_o) \right]^{1/2}} \qquad \ldots (8)$$

Provided that $\Lambda_o \approx 0$ (which is practically always the case), then

$$\Lambda \approx \Lambda_B \left(\frac{f_B^2}{f_B^2 - f_o^2} \right)^{1/2} \qquad \ldots (9)$$

8.3 Shear modulus G

The shear modulus of a test specimen of rectangular cross-section is calculated from equation (10) or (11), as applicable.

8.3.1 Method A

$$G = 4\pi^2 I \cdot f_A^2 F_d \cdot F_g$$

If $\Lambda < 1$, then $F_d \approx 1$ and

$$G \approx \frac{12\pi^2 I \cdot f_A^2 \cdot L}{b \cdot h^3 \cdot C} \qquad \ldots (10)$$

[see equation (2)].

8.3.2 Method B

$$G = 4\pi^2 I (f_B^2 \cdot F_{dB} - f_o^2 \cdot F_{do}) F_g$$

If $\Lambda_o \ll 1$, then $F_{do} \approx 1$

If, furthermore, $\Lambda_B < 1$, then $F_{dB} \approx 1$ and

$$G \approx \frac{12\pi^2 I (f_B^2 - f_o^2) L}{b \cdot h^3 \cdot C} \qquad \ldots (11)$$

[see equation (2)].

8.4 Mechanical loss factor d

The mechanical loss factor d is calculated from equation (1) (see 3.3), i.e.

$$d \approx \frac{\Lambda}{\pi}$$

(see also annex A).

8.5 Maximum shear angle

In order to be able to check that the maximum permissible shear angle γ has not been exceeded, this angle is calculated from the equation

$$\gamma = \alpha \frac{h}{L} \qquad \ldots (12)$$

where α is the angle of twist, in radians, between the clamps.

9 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) a complete description of the material tested;

c) the test method used (A or B);

d) details of the test specimen:

1) preparation,

2) temperature/time history before testing (conditioning),

3) shape, dimensions and dimensional tolerances,

4) number (if more than one);

e) details of the apparatus and the test procedures:

1) description (particularly the moment of inertia and mass) of the driving disc,

2) maximum shear angle,

3) frequency range,

4) whether air or inert gas was used,

5) the temperature programme used (constant, rising or falling temperature);

f) the individual values or temperature plots of the

1) frequency,

2) shear modulus,

3) logarithmic decrement,

4) mechanical loss factor (if required).

If included in the form of a temperature plot, the shear modulus values shall be plotted on a logarithmic scale.

Annex A
(informative)

Mechanical loss factor

The relationships between the variables measured by the torsion pendulum test described here (shear modulus G and logarithmic decrement of damping Λ) and those measured using forced vibrations (real part G' of the complex shear modulus G^*, the absolute value $|G^*|$ of G, and the mechanical loss factor d) cannot be expressed exactly with any certainty because there are different mathematical treatments of the torsion pendulum test in the literature, which lead to different results.

NOTE — NIELSEN [*Rev. Sci. Inst.* **22**/**9** (1951), p. 690] has published the relationships:

$$|G^*| = G \qquad \text{and} \qquad d = \frac{\Lambda}{\pi\left[1 - \left(\frac{\Lambda}{2\pi}\right)^2\right]}$$

whereas STAVERMAN and SCHWARZL [Stuart (editor): *Die Physik der Hochpolymeren,* Vol. 4, Springer Verlag, Berlin, Goettingen, Heidelberg (1956)] found that

$$G' = G \qquad \text{and} \qquad d = \frac{\Lambda}{\pi\left[1 - \left(\frac{\Lambda}{2\pi}\right)^2\right]}$$

At a later date, STRUIK [*Rheol. Acta* **6** (1967), p. 119] showed that both of the formulae given above lead to wrong results. In his paper, he also estimated the error caused by the Nielsen and the Staverman and Schwarzl treatments.

Therefore at this time only the following approximate relationships can be used:

$$G \approx G' \approx |G^*| \qquad \text{and} \qquad d \approx \frac{\Lambda}{\pi}$$