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# International Standard



# 537

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## Plastics — Testing with the torsion pendulum

*Plastiques — Essai au pendule de torsion*

First edition — 1980-03-15

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UDC 678.5/.8 : 620.178.322.4

Ref. No. ISO 537-1980 (E)

**Descriptors** : plastics, tests, mechanical tests, torsion tests, torsion pendulums, test equipment.

Price based on 5 pages

## Foreword

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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.

International Standard ISO 537 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in June 1978.

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It has been approved by the member bodies of the following countries :

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Canada	Italy	Sweden
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The member body of the following country expressed disapproval of the document on technical grounds :

United Kingdom

This International Standard cancels and replaces ISO Recommendation R 357-1966, of which it constitutes a technical revision.

# Plastics — Testing with the torsion pendulum

## 1 Scope and field of application

This International Standard specifies a method for determining dynamic mechanical properties (shear modulus and mechanical damping) of plastics in the range of small deformations, as a function of temperature, within the frequency range 0,1 to 10 Hz. Shear modulus and mechanical damping are quantities the values of which are independent of the design of the apparatus as well as of the shape of the test specimen. The dependence of these quantities on temperature in a sufficiently broad range of temperature (for example from  $-50\text{ }^{\circ}\text{C}$  to  $+150\text{ }^{\circ}\text{C}$  for the majority of commercially available polymers) determines the transition regions (for example "glassy-rubbery" transition) of the polymer. It also gives information concerning the commencement of plastic flow.<sup>1)</sup>

## 2 Definitions

**2.1 shear modulus ( $G$ )** : The quotient of the shearing stress and the resulting angular deformation of the test specimen measured in the range of small recoverable deformations.

**2.2 logarithmic decrement of damping ( $\lambda$ )** : The natural logarithm of the ratio of the amplitudes of two successive oscillations. It is a measure of the damping of the free torsional oscillations.

**2.3 mechanical loss factor ( $d$ )** : The quantity defined by the formula :

$$d = \frac{\lambda/\pi}{1 + \lambda^2/4\pi^2} \quad \dots(1)$$

For  $\lambda < 2$ ,  $d \approx \lambda/\pi$ .

## 3 Testing atmosphere

Unless otherwise agreed upon by the interested parties, the test shall be made at a relative humidity of  $(50 \pm 5)\%$ . For temperature control, see 5.2.3.

## 4 Test specimen

**4.1** The test specimen shall be prepared and conditioned according to the requirements of the specification for the material to be tested. In the absence of such requirements, the method of preparation and conditioning shall be agreed upon by the interested parties.

**4.2** It is recommended that the specimen be of the following dimensions :

length ( $L$ ) : 60 mm

width ( $b$ ) : 10 mm

thickness ( $h$ ) : 1 mm

**4.3** Other dimensions are permissible as required by the design of the apparatus (see notes below).

It is recommended not to use specimens with a thickness less than 0,05 mm. The thickness range 0,05 to 0,15 mm is acceptable when the oscillating system is in vacuum.

Tolerances for the dimensions of specimens shall be agreed upon by the interested parties. It is recommended not to use specimens of rectangular cross-section the thickness of which varies along the longitudinal axis by more than 3 % of the mean thickness value.

### NOTES

1 Alternative shapes of specimens may be used (cylindrical or tubular); in such cases, dimensions and tolerances shall be agreed upon by the interested parties.

2 In the case of rectangular specimens whose dimensions differ from those recommended in 4.2, preference shall be given to dimensions in the ratios  $L/b$  and  $b/h$  of the recommended specimen, keeping  $b$  within the range of 5 to 25 mm.

3 Quantities measured by this method (frequency and amplitude) are sensitive to dimensional uniformity of the specimen, and to the physical state of the specimen (degree of crystallinity, internal stresses, etc.) as well as to the temperature of the specimen; these facts should be considered when choosing the dimensions and tolerances, method of preparation, conditioning procedure, etc., for a particular material.

1) For physical description of the viscoelastic behaviour of polymers, see *Plastics — Terminology for characterizing the damping properties of solid polymers*, document prepared by ISO/TC 61/WG 2 (September 1975), available from TC 61 secretariat as item 374.

## 5 Apparatus

### 5.1 Torsion pendulum

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

- test specimen supporting inertia member (method A);
- test specimen not supporting inertia member (method B).

### 5.2 Torsion pendulum for method A (see figure 1)

#### 5.2.1 Disc

The dimensions and mass of the light metal (for example aluminium) disc shall correspond to a moment of inertia  $I$  of about  $30 \text{ kg}\cdot\text{mm}^2$  for testing specimens of dimensions according to 4.2, provided that the dimensions and mass of the lower axis and clamps do not influence the value of  $I$ . The total mass of the disc, lower clamp and lower axis shall be such that the tensile stress of the specimen does not exceed  $0,1 \text{ MPa}^*$ . The moment of inertia shall be adjusted according to the stiffness of the specimen, as well as to the total mass of disc clamp and axis. It shall be agreed upon by the interested parties.

#### 5.2.2 Clamps

The clamps shall ensure perfect positioning of the specimen in the axis of the oscillating system. In accordance with 5.2.1, the lower clamp shall be of low mass and negligible moment of inertia. Self-tightening clamps shall be used for testing at low temperatures.

#### 5.2.3 Temperature control chamber

The test specimen and clamps shall be enclosed in a temperature chamber. According to the purpose of the test, the temperature chamber (space around the specimen) shall contain air or inert gas. When inert gas is used as an environment, it shall be under slight pressure, i.e. a stream of gas shall be passed through at a rate of about  $1\ 200 \text{ ml/h}$ .

The axis of the disc shall not be in contact with the wall of the chamber. The space between the axis and the wall shall be just sufficient to allow a free twisting of the oscillating system. The upper clamp with the upper "fixed" axis is mounted into the upper wall of the chamber so that no gas from inside can pass through the opening. A slight force shall be necessary to twist the upper axis. Any twisting of the upper axis during the oscillating period of the system shall be prevented.

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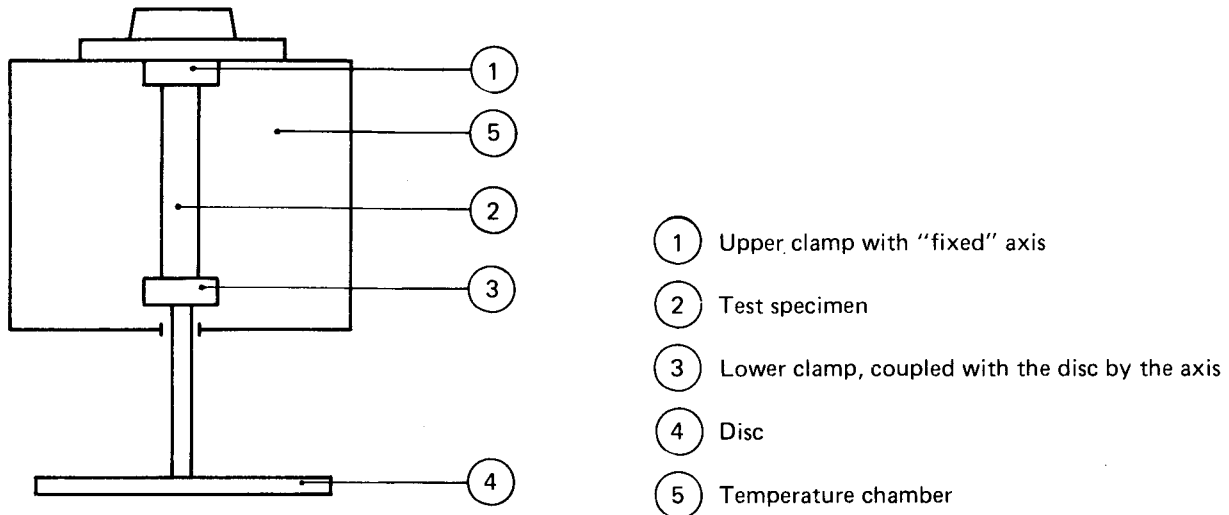


Figure 1

\*  $1 \text{ MPa} = 1 \text{ N/mm}^2$

The temperature in the vicinity of the specimen shall be uniform along the whole length of the specimen. The temperature is measured by a thermocouple or by another sensitive element, and controlled in such a way that, using the constant-temperature procedure (see 6.2), the variation does not exceed the range of  $\pm 1\text{ }^{\circ}\text{C}$ .

It is recommended that the temperature chamber be designed for the temperature range of  $-60\text{ }^{\circ}\text{C}$  to  $+300\text{ }^{\circ}\text{C}$ .

It is recommended that equipment be used for programming the temperature during the measurement. When a constant rate of increase in temperature is to be applied, the rate shall be not greater than  $50\text{ }^{\circ}\text{C/h}$ , and the permitted variation of temperature shall be agreed upon by the interested parties.

The whole system shall be so designed that the temperature in the close vicinity of the test specimen will not vary by more than  $\pm 0,25\text{ }^{\circ}\text{C}$  during a single measurement (a series of free oscillations after one twisting impulse).

**5.3 Torsion pendulum for method B** (see figure 2)

**5.3.1 Disc**

Discs of a broad range of moments of inertia may be used. The

mass and dimensions of the disc shall be based on the purpose of the test.

**5.3.2 Counterweight**

The counterweight shall balance the system so that the maximum tensile stress on the test specimen is  $0,1\text{ MPa}^*$ .

**5.3.3 Clamps**

Clamps shall be designed to prevent movement of the gripped volume of specimens; they shall be self-centering to maintain the specimen axis coincident with the axis of rotation and self-tightening to allow for specimen contraction during low-temperature measurements. When the moment of inertia of the upper clamp is not negligible, the moment of inertia of the system "disc, upper axis and clamp" shall be experimentally determined.

**5.3.4 Temperature control chamber**

See 5.2.3. The difference from method A is that the lower clamp is fixed and the opening for the disc is in the upper wall of the chamber.

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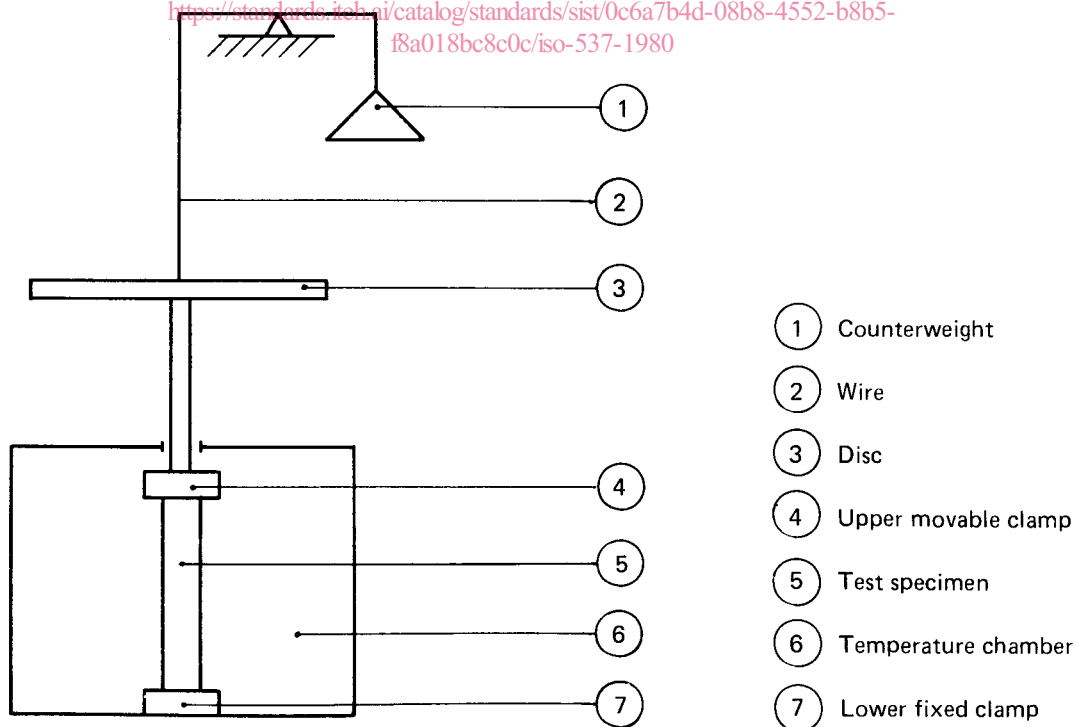


Figure 2

\*  $1\text{ MPa} = 1\text{ N/mm}^2$

## 5.4 Apparatus for recording the oscillations

Optical, electrical, or other recording systems may be used if no additional damping is caused by them. If recorders are employed, the recording strip shall move with a speed not varying by more than  $\pm 1\%$ .

## 5.5 Devices for measuring test specimen dimensions

The device for measuring the dimensions of rectangular test specimens shall be suitable for measuring the thickness to  $\pm 0,003$  mm, the width to  $\pm 0,05$  mm and the length between the clamps to  $\pm 0,1$  mm. The test specimen shall not be deformed during the measurement. Precision for measuring dimensions of specimens of other shapes shall be agreed upon by the interested parties.

NOTE — To make sure that the testing device works satisfactorily, the apparatus can be checked by testing a reference specimen (for example piano wire with a diameter of 0,5 mm) the torsion modulus of which is already known (see 7.2).

## 6 Procedure

### 6.1 Measurement of the cross-section of the rectangular test specimen

Measure the thickness and width of the test specimen before the test at not less than five places along its length. All specimens that have visible defects or that show a variation of thickness greater than 3 % from the average value shall be rejected. It is recommended that test specimens whose width varies by more than 0,2 mm should not be used.

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

### 6.2 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 in 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 length) to  $\pm 0,1$  mm. When mounting the movable system into the chamber, take care that the test specimen is not stressed.

After having checked the central mounting of the oscillating system, start the heating. The measurement can be carried out at continuously increasing temperature of the test specimen (see 5.2.3), or at constant temperature. In the second case the dependence of the dynamic mechanical properties on the temperature is ascertained by determinations at various temperatures.

## 6.3 Torsional oscillation test

Depending upon the design of the apparatus, bring the movable system into the state of free oscillations, either

- by the twisting of the upper axis (method A), or
- by the twisting of the disc (method B).

The angle of twist shall not exceed  $3^\circ$  in either direction. The frequency and the amplitude shall be measured.

## 7 Expression of results

### 7.1 Logarithmic decrement $\lambda$

#### 7.1.1 Method A

The logarithmic decrement of damping  $\lambda$  shall be calculated using the equation

$$\lambda = \ln \frac{A_1}{A_2} \quad \dots(2)$$

or, in general,

$$\lambda = \ln \frac{A_n}{A_{n+1}} \quad \dots(3)$$

where  $A_1, A_2, \dots, A_n, A_{n+1}$  are the amplitudes of successive oscillations in the same direction.

For calculation of  $\lambda$ , the amplitudes of any two oscillations in the same direction may be used. A suitable equation is then

$$\lambda = \frac{1}{n-m} \ln \frac{A_m}{A_n} \quad \dots(4)$$

where

$A_m$  is the  $m$ th amplitude;

$A_n$  is the  $n$ th amplitude;

$n > m$ .

NOTE — The following alternative equation for calculating the logarithmic decrement can be used in the case of amplitudes which cannot be measured accurately with respect to the centre of the damped sinusoid :

$$\lambda = \ln \frac{A_1^*}{A_2^*} = \frac{1}{q} \ln \frac{A_p^*}{A_{p+q}^*}$$

where  $A_1^*, A_2^*, \dots, A_p^*, \dots, A_{p+q}^*$  are the distances between successive positive and negative amplitude peaks of the oscillation.

#### 7.1.2 Method B

The logarithmic decrement  $\lambda$  shall be calculated from the equation given in 7.1.1.

For method B calculate the logarithmic decrement  $\lambda_s$  of the specimen using the equation

$$\lambda_s = \lambda \frac{f^2}{f^2 - f_0^2}$$

where

$\lambda$  is the logarithmic decrement of damping (of the system with the test specimen);

$f$  is the frequency of the oscillations of the system with the test specimen;

$f_0$  is the frequency of the oscillations of the system without the specimen.

## 7.2 Shear modulus

The shear modulus for the rectangular cross-section of the test specimen shall be calculated from equation (5) or (6) as appropriate (see notes 1 and 2 below) :

### a) Method A

$$G = I f^2 \left( \frac{12 \pi^2 L}{b h^3 C} \right) - \frac{(m_A g b)}{4 h^3 C} \quad \dots(5)$$

$F_{gA}$                        $S_{EA}$

### b) Method B

$$G = (4 \pi^2 I f^2 - K) \left( \frac{3 L}{b h^3 C} \right) - \frac{(m_B g b)}{4 h^3 C} \quad \dots(6)$$

$F_{gB}$                        $S_{EB}$

where

$G$  is the shear modulus, in newtons per square millimetre;

$I$  is the moment of inertia of the disc, in kilogram square millimetres (see note 3 below);

$f$  is the frequency of the oscillations of the system with the test specimen, in hertz;

$b$  is the width of the specimen, in millimetres;

$h$  is the thickness of the specimen, in millimetres;

$L$  is the distance between the clamps ("length" of the specimen), in millimetres;

$m_A$  is the mass of the disc, including the lower clamp and the lower axis (which provide the tensile force), in kilograms;

$m_B$  is the mass of the counterweight minus the mass of a disc including the upper clamp and upper axis, in kilograms;

$g$  is the acceleration of free fall, in millimetres per second squared;

$C$  is a correction factor (see note 4 below);

$K$  is the torsion constant of the wire suspending the inertia member, in newton millimetres ( $= 4 \pi^2 I f_0^2$ ) (see note 5 below),  $f_0$  being the frequency of the oscillations of the system without the specimen, in hertz.

## NOTES

1 When  $\lambda > 1,0$ , then  $F_{gA}$  shall be multiplied by the factor  $F_d$ , where

$$F_d = 1 + \frac{\lambda^2}{4 \pi^2} \quad \dots(7)$$

2 For specimens of cross-section other than rectangular (see note 1 in 4.3), appropriate equations [for use instead of equations (5) and (6)] can be found in the literature.

3 In the case of separate determination of the moment of inertia of the system "disc, clamp and axis" as mentioned in 5.3.3,  $I$  is the moment of inertia of this system.

4  $C = 3 \mu / 16$ , when  $\mu$  is the shape factor which is defined by the ratio  $b/h$ . The values of  $\mu$  are to be calculated from the equation

$$\mu = 5,33 - 3,36 \frac{h}{b} \left( 1 - \frac{h^4}{12 b^4} \right) \quad \dots(8)$$

If  $h/b < 0,4$ , then  $C = 1 - (0,632 h/b)$ .

5 In the case of method B,  $K$  may be ignored if  $4 \pi^2 I f^2 > 100 K$ .

## 8 Test report

The test report shall include the following particulars :

- a) reference to this International Standard;
- b) designation of the material;
- c) test method (A or B);
- d) preparation of the test specimen;
- e) conditioning of the test specimen;
- f) dimensions of the test specimen;
- g) values of  $\lambda$  and  $G$  if the test was carried out only at one or a few temperatures;
- h) plots of  $\lambda$  and  $G$  versus temperature;
- j) frequencies at which the individual values of  $\lambda$  and  $G$  were measured.

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