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Rubber, vulcanized or thermoplastic — Determination of dynamic properties —

Part 2:
Torsion pendulum methods at low frequencies

Caoutchouc vulcanisé ou thermoplastique — Détermination des propriétés dynamiques —

Partie 2: Méthodes du pendule de torsion à basses fréquences

Reference number
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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.

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The main task of technical committees is to prepare International Standards. 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.

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.

ISO 4664-2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

It cancels and replaces ISO 4663:1986, of which it constitutes a technical revision.

ISO 4664 consists of the following parts, under the general title *Rubber, vulcanized or thermoplastic — Determination of dynamic properties*:

— *Part 1: General guidance*

— *Part 2: Torsion pendulum methods at low frequencies*

Rubber, vulcanized or thermoplastic — Determination of dynamic properties —

Part 2: Torsion pendulum methods at low frequencies

WARNING — Persons using this part of ISO 4664 should be familiar with normal laboratory practice. This part of ISO 4664 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 4664 specifies methods, using a torsion pendulum, of determining the dynamic properties in shear, that is the shear modulus and mechanical damping, of vulcanized or thermoplastic rubbers over a wide temperature range at low frequencies in the range 0,1 Hz to 10 Hz and at comparatively low strains of less than 5×10^{-4}.

2 Normative references

The following referenced documents are indispensable for the application 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 4664-1, *Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 1: General guidance*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4664-1 apply.

4 Principle

This test is primarily intended for the determination of the temperature at which the test piece shows transitions in its visco-elastic properties, by plotting observed values of modulus and damping as a function of temperature. The methods are not particularly accurate for the determination of absolute values of the modulus.

In the torsion pendulum, a strip test piece of uniform cross-section constitutes the elastic member of the pendulum. The test piece is clamped at both ends. One clamp is fixed to a rigid frame while the other one is provided with an appropriate inertial mass, for example a flywheel.

Three methods of using the torsion pendulum are specified:

— **method A**, in which the mass of the inertial member is supported by the test piece and the pendulum is set in free damped oscillation;

— **method B**, in which the mass of the inertial member is supported by a fine suspension wire and the pendulum is set in free damped oscillation;

— **method C**, which is similar to method B except that the oscillations are maintained at constant amplitude by supplying energy to the system.

5 Apparatus

5.1 Test piece holder

The test piece shall be held between clamps, one of which is fixed and the other attached to the inertial member. The length of the test piece between the clamps shall be between 30 mm and 100 mm, 50 mm being the preferred length. Provision shall be made for the measurement of the length between the clamps to an accuracy of 0,5 mm.

In order to obtain a constant temperature over the length of test pieces, the parts of the clamp protruding from the thermostatted test chamber (5.4) shall be made of material having low thermal conductivity.

Care shall be taken to ensure that the test piece is free to expand or retract as a result of changes in temperature without changing the initial stress or tension in the test piece.

5.2 Inertial member

The inertial member may be a disc or a symmetrically supported rod having a moment of inertia such that the frequency of oscillation of the pendulum and test piece is between 0,1 Hz and 10 Hz at standard laboratory temperature. In the case of method A, the mass of the inertial member is limited by the longitudinal stress (see 5.3.1). The moment of inertia of a disc or rod of about 30 $kg{\cdot}mm^2$ has been found to be suitable.

Means shall be attached to the inertial member to enable a torsional disturbance to be applied to the pendulum in order to start the system oscillating. Low angles of deformation shall be used, such that the shear strain in the rubber is below 5×10^{-4}.

Means shall be provided for measuring the frequency of oscillation to an accuracy of ± 1 % in the region of rubber elasticity. In the transition range, an accuracy of ± 5 % is permissible.

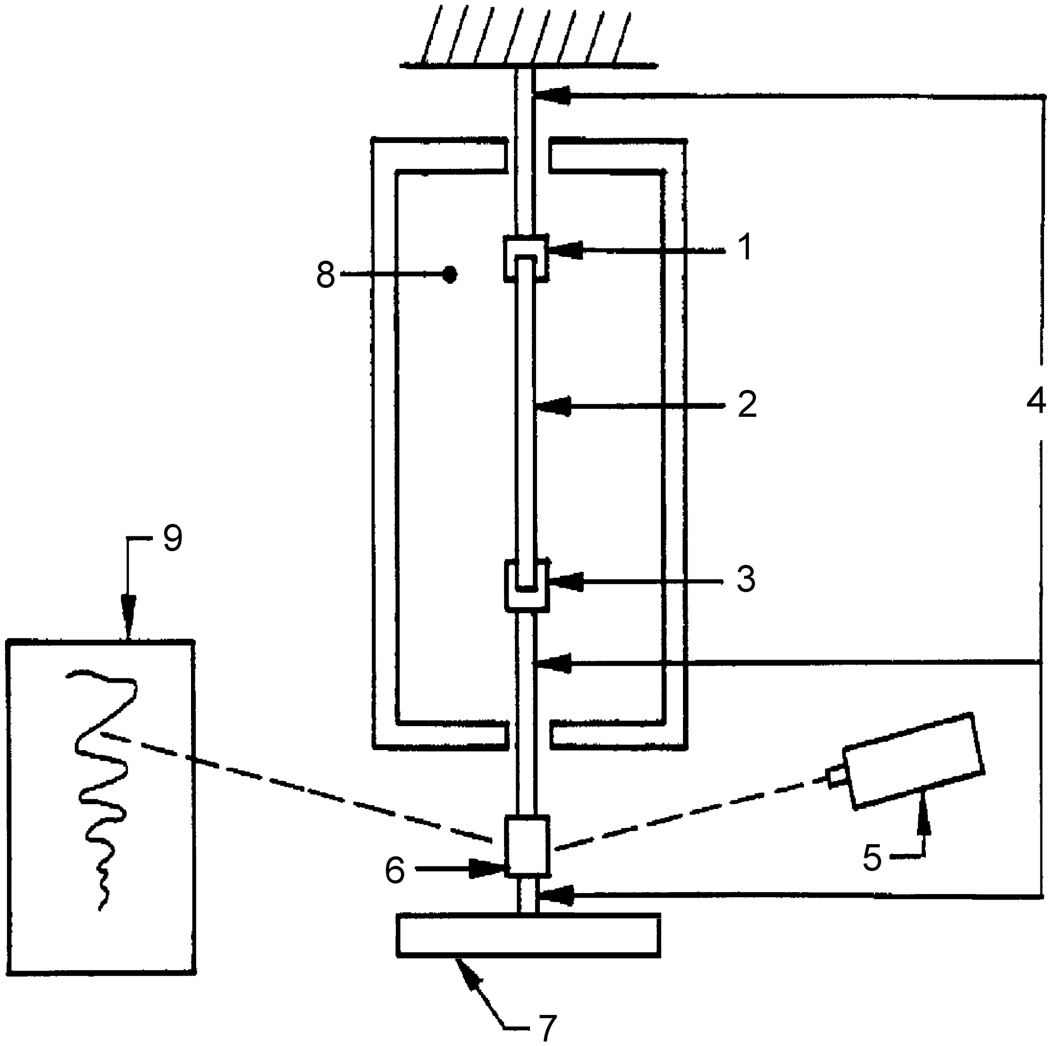
5.3 Torsion pendulum

5.3.1 Method A

The inertial member shall be freely suspended below the test piece as shown in Figure 1. The mass of the inertial member shall be such that the longitudinal stress in the test piece is less than 30 kPa.

The method of measurement shall permit the determination of the amplitudes of deformation to an accuracy of ± 1 %. When recorders are used, the recording strip shall move with a speed which is known to within ± 1 %, and with a linearity within ± 1 %.

NOTE If a lamp and mirror system is used to measure amplitude, a distance of at least 2 m is needed between the mirror and scale to achieve the required precision.



Key

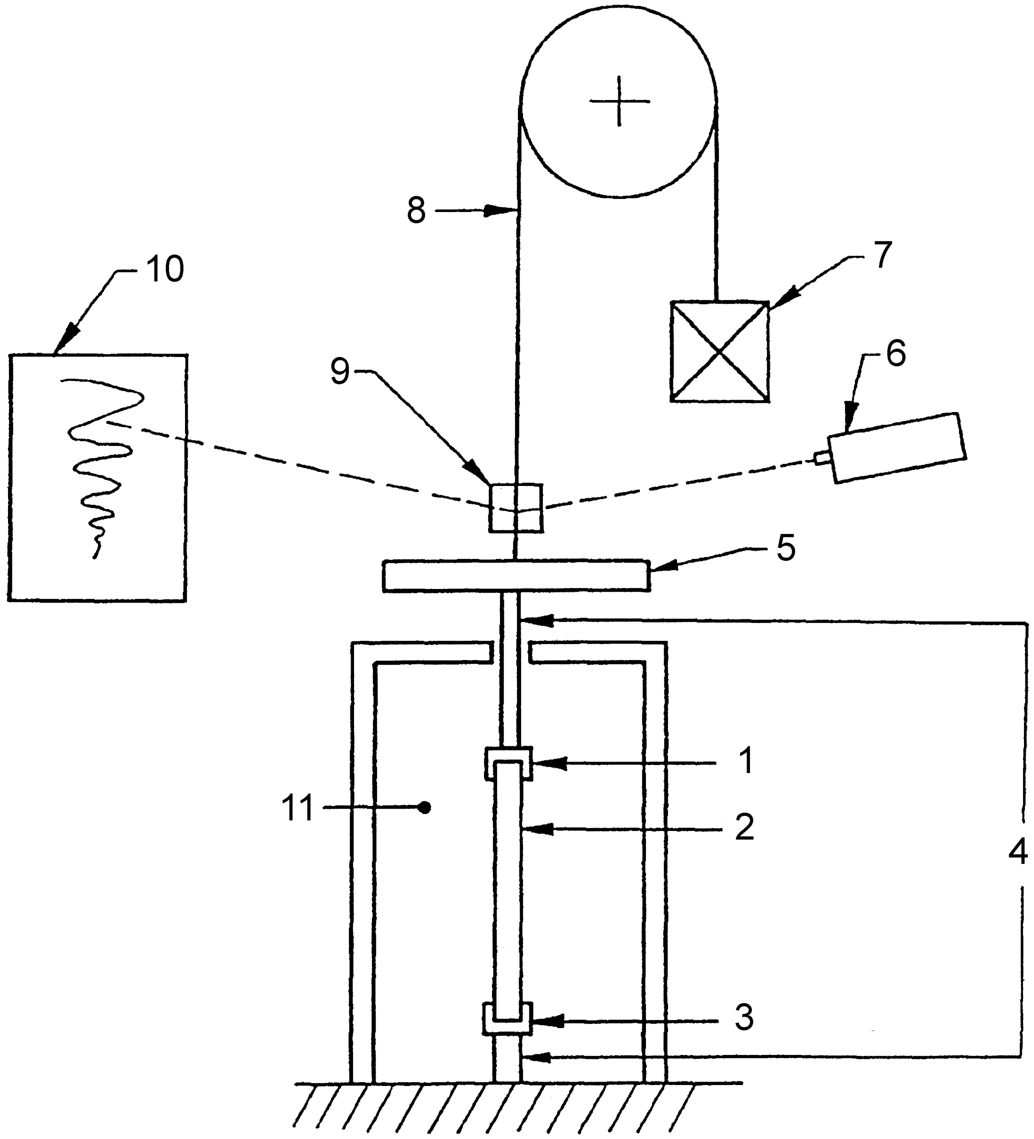
1 upper clamp
2 test piece
3 lower clamp
4 rigid connections
5 lamp
6 mirror
7 inertial member
8 thermostatted chamber
9 scale or recorder

Figure 1 — Uncompensated free-oscillation apparatus with counterweighted inertial member suspended below the test piece

5.3.2 Method B

The torsion pendulum shall be constructed according to the principles shown in Figure 2. The inertial member shall be supported from above by a fine wire suspension and the test piece shall be attached below. The length and diameter of the wire shall be chosen so that the restoring torque due to the wire suspension is not greater than 25 % of the restoring torque in the test piece plus the suspension.

The measurement system shall conform to that specified for method A.



Key

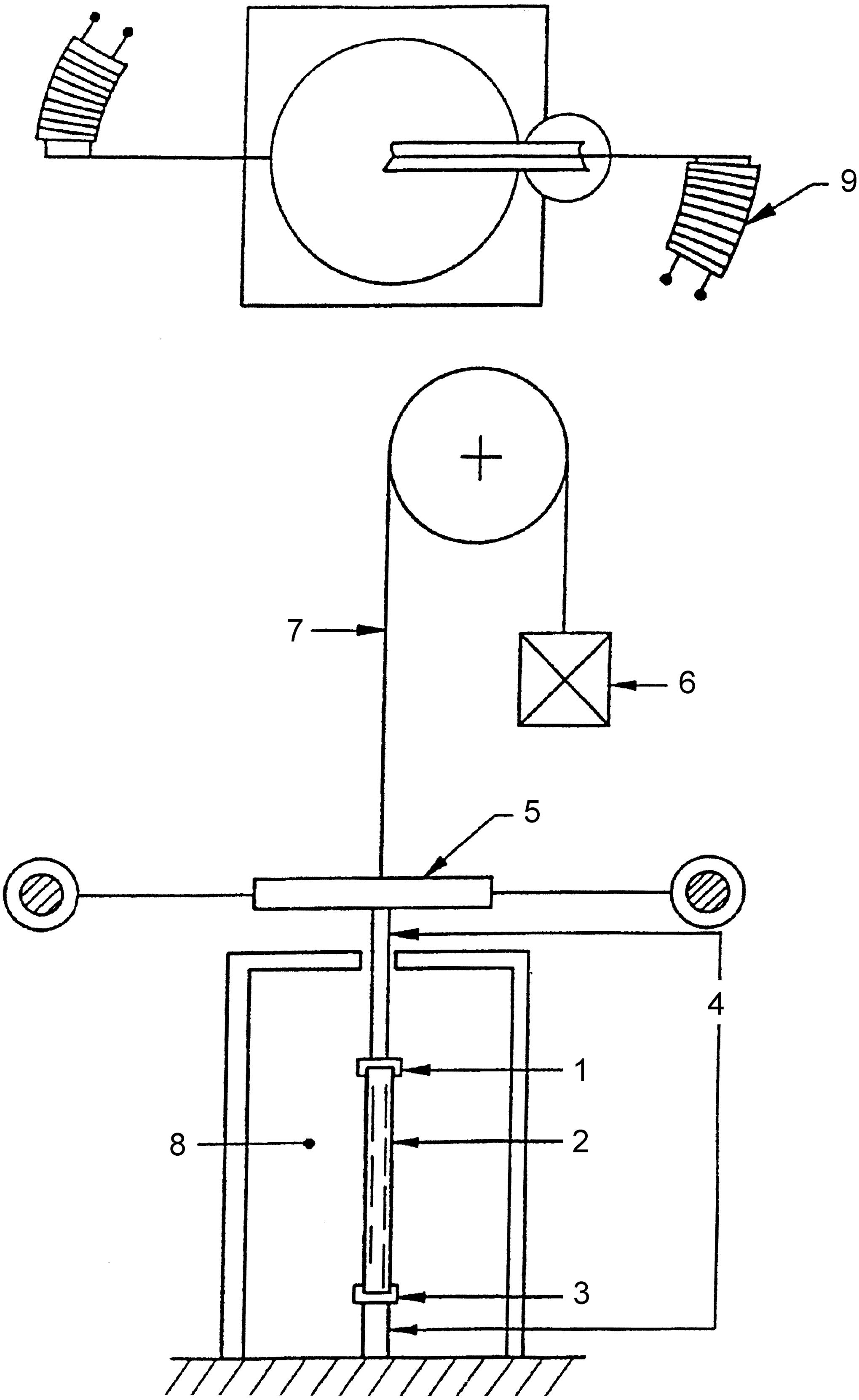
1 upper clamp
2 test piece
3 lower clamp
4 rigid connections
5 inertial member
6 lamp
7 counterweight
8 torsion wire
9 mirror
10 scale or recorder
11 thermostatted chamber

Figure 2 — Uncompensated free-oscillation apparatus with counterweighted inertial member suspended above the test piece

5.3.3 Method C

The torsion pendulum for this forced-vibration method is the same as that described for method B, with the addition of means to exert a friction-free mechanical moment on the pendulum system. A suitable system is shown in Figure 3 in which the mechanical moment is exerted electromagnetically. The applied moment shall be equal in magnitude but opposite in sign to the mechanical moment produced by damping in the test piece. In this way, a constant amplitude of oscillation can be maintained in the test piece.

A suitable amplitude-monitoring system and means of measuring the restoring moment with an accuracy of $\pm$ 2 % shall be incorporated.



Key

1 upper clamp
2 test piece
3 lower clamp
4 rigid connections
5 inertial member
6 counterweight
7 torsion wire
8 thermostatted chamber
9 electromagnetic compensation device

Figure 3 — Forced-resonance oscillation device

5.4 Thermostatted test chamber

The thermostatted chamber, with a gaseous heat-transfer medium, shall be in accordance with ISO 23529. During the actual test period, it shall not be possible for any draughts to act on the test piece.

The temperature in the vicinity of the test piece shall be maintained within the desired temperature range, for example − 100 °C to + 200 °C, to within ± 1 °C of the desired value. Suitable temperature sensors are thermocouples or resistance elements.

5.5 Devices for measurement of test piece dimensions

Suitable devices in accordance with ISO 23529 shall be used for the measurement of test piece width and thickness.

6 Test pieces

6.1 Dimensions

Each test piece shall be a strip of material of uniform cross-section within the following dimensions:

thickness: (1,0 ± 0,2) mm;

width: 5 mm to 11 mm, with 10 mm being the preferred value;

length: 40 mm to 120 mm, chosen to fit the clamping device and to give the desired free length (see 5.1).

Individual test pieces shall comply with the tolerances given in 8.1. For comparative tests, test pieces of the same nominal dimensions shall be used.

6.2 Preparation

Test pieces shall be prepared in accordance with ISO 23529.

6.3 Number

A minimum of three test pieces shall be tested.

6.4 Conditioning

The time interval between vulcanization and testing shall be in accordance with ISO 23529.

Samples and test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing.

Test pieces shall be conditioned for not less than 3 h at the appropriate standard laboratory temperature, as specified in ISO 23529, immediately before testing.

7 Strain, frequency and temperature of test

7.1 Strain

The shear strain in the rubber during the test shall not exceed 5×10^{-4}.

NOTE For a strip of length L, width b and thickness h, where $b/h > 3$, the maximum shear strain γ is approximately related to the torsion angle α, in radians, by the equation $\gamma = \alpha h/L$

For the preferred test piece dimensions of h = 1 mm and L = 50 mm, the prescribed maximum shear strain of 5×10^{-4} corresponds to a torsion angle α of 1,5°.

7.2 Frequency

The frequency of oscillation shall be between 0,1 Hz and 10 Hz in the region of rubber elasticity of the material under test.

7.3 Temperature

The temperature or temperature range of the test shall be chosen according to the material tested and the information desired.

Measurements shall be made at maximum intervals of (10 ± 1) °C as the temperature is raised from the lowest to the highest value.

Closer intervals shall be used in the transition regions where the shear modulus and mechanical damping are changing rapidly with change of temperature.

For crystallizing materials, the conditioning time and the test time, i.e. the rate of heating, may influence the degree of crystallization and thus the modulus values obtained. For that reason, these times shall be selected to suit the purpose of the test and shall be stated in the test report.

8 Procedure

8.1 Measurement of dimensions of test piece

Before the test, determine the dimensions of the test piece. Make the measurements at the standard laboratory temperature.

Measure the width b to an accuracy of 0,1 mm at five places.

Measure the thickness h to an accuracy of 0,01 mm, at five places.

Record the mean values. The difference between the largest and smallest values of width and thickness shall not exceed 6 %; if it does, the test piece shall be rejected. As the thickness appears in the calculation of the shear modulus (see 9.2) to the third power, suitable care shall be taken when making these measurements.

The measurement of the width may be omitted if the test pieces are cut with a die from a sheet, in which case the distance between the parallel cutting edges may be taken as the width of the test piece. The correctness of this procedure shall, however, be checked at regular intervals by measurements on prepared test pieces.

8.2 Mechanical conditioning of test piece

The shear modulus of rubbers containing reinforcing fillers, especially carbon blacks, is markedly dependent on the degree of filler structure present. Some improvement in reproducibility of results may be obtained by breaking down this structure by the mechanical conditioning described below before measuring the shear modulus. However, if service conditions are such that filler structure is not broken down (i.e. if there is no substantial deformation), the mechanical conditioning shall be omitted and care taken not to deform the test piece, because this will cause a partial but indefinite degree of structure breakdown; it will then be advisable to use a separate test piece for each test temperature.

If mechanical conditioning is required, it shall be carried out at standard laboratory temperature immediately before determining the shear modulus, as follows:

a) twist the test piece to a deflection of 90° in both directions, then return it to approximately zero deflection;

b) repeat the procedure described in a) to give a total of five double deformations.

8.3 Mounting of test piece

Mount the test piece in suitable clamps so that the free length of the test piece is between 30 mm and 100 mm, 50 mm being the preferred value.

Place the two clamps in a vertical line, with their centrelines along the axis of rotation of the pendulum. The centreline of the test piece after clamping shall also coincide with the axis of rotation.

8.4 Measurement of test piece free length

Measure the free length L of the test piece between the clamps to an accuracy of 0,5 mm, with the test piece held between the clamps, ready to start the test (see 5.1).

8.5 Temperature conditioning of test piece

Cool the test piece to the lowest test temperature.

After temperature equilibrium has been reached, the test piece may be heated by either of two methods.

The preferred method is to heat the test piece at a rate that shall not exceed 1 °C/min and to take pendulum oscillation measurements under non-equilibrium temperature conditions.

An alternative method is to bring the test piece to the desired test temperature and to take pendulum oscillation measurements after temperature equilibrium between test piece and thermostatted enclosure has been reached.

NOTE Where automatic recording allows measurements to be made in less than 20 s, a uniform rate of heating not exceeding 1 °C/min may be used. However, with this procedure the test piece temperature can differ considerably from the air temperature, but may be determined by using a fine-wire thermocouple inserted into a dummy test piece of the same material and approximately the same dimensions and mounted inside the thermostatted enclosure.

8.6 Testing

Start the oscillation by applying a slight twist to the movable clamp. In the case of the damping-compensated instrument (method C), switch on the damping-compensation system.

Measure either the frequency of oscillation and the amplitude decrement (methods A and B) or the frequency of oscillation and the applied mechanical compensating moment (method C). For further details, see the Bibliography.

9 Expression of results

9.1 Symbols

G' is the elastic shear modulus (in-phase modulus), in Pa

I is the moment of inertia of the inertial member and the oscillating clamp, in kg·m^2

f is the frequency of oscillation with the test piece, in Hz

f_0 is the frequency of oscillation without the test piece, in Hz

L is the free length of the test piece, in m

b is the width of the test piece, in m

h is the thickness of the test piece, in m

Λ is the the logarithmic decrement

A is the amplitude of oscillation, expressed in scale divisions, for methods A and B and converted to radians for method C

M is the compensating mechanical moment, in N·m

C is a correction factor equal to (1 – 0,63h/b) for $b/h > 3$

$$F_D = 1 + \frac{\Lambda^2}{4\pi^2}$$

The factor F_D incorporates the influence of the damping on the shear modulus. It shall be taken into account when $\Lambda \geqslant 1$ and is equal to 1 when $\Lambda < 1$.

9.2 Calculation of the elastic shear modulus (in-phase modulus)

9.2.1 Method A

$$G' = (4\pi^2 L/bh^3 C) \times F_D I f^2$$

9.2.2 Method B

$$G' = (4\pi^2 L/bh^3 C) \times F_D I (f^2 - f_0^2)$$

9.2.3 Method C

$$G' = (4\pi^2 L/bh^3 C) \times F_D I (f^2 - f_0^2)$$

9.3 Calculation of mechanical damping

9.3.1 General

The mechanical damping of the material shall be expressed in terms of the logarithmic decrement Λ_R of the test piece.

NOTE For small values, Λ_R is related to the mechanical loss factor $\tan\delta$ by the expression

$$\tan\delta = \Lambda_R/\pi$$

9.3.2 Method A

The logarithmic decrement is given accurately by the equation

$$\Lambda_R = \log_e(A_1/A_2)$$

where A_1 and A_2 are the amplitudes of successive oscillations in the same direction.

NOTE It is recommended for low-damping materials that Λ be determined using several cycles and then calculated from the equation

$$\Lambda_R = (1/n) \times \log_e(A_0/A_n)$$

where A_0 is the amplitude of initial oscillation and A_n is the amplitude of the nth oscillation in the same direction.

9.3.3 Method B

The logarithmic decrement of the complete system is given by the equation

$$\Lambda = \log_e(A_1/A_2)$$

See the Note to 9.3.2.

The damping due to the rubber alone is then given by the equation

$$\Lambda_R = \Lambda - \Lambda_0 f_0/f$$

where Λ_0 is the damping of the pendulum without a test piece, and is due to losses in the wire suspension system. For a well-designed pendulum, this correction can usually be neglected.

9.3.4 Method C

The logarithmic decrement is given by the equation

$$\Lambda_R = M/(4\pi I f^2 A)$$

NOTE This expression assumes that any correction required due to the suspension is negligible.

10 Test report

The test report shall include the following information:

a) sample details:

   1) a full description of the sample and its origin,

   2) details of the compound and the curing conditions, if known,

   3) details of the preparation of the test pieces, for example whether moulded or cut,

   4) the width and thickness of the test pieces as well as their free length between the clamps;

b) test method and test details:

   1) a reference to this part of ISO 4664,

   2) the type of pendulum used, i.e. method A, B or C, its inertial mass and the mass of the lower clamp in the case where this was loading the test piece,

   3) the shear strain applied,

   4) the test frequencies,

   5) whether mechanical conditioning was carried out,

   6) the time and temperature of conditioning of the test pieces prior to testing,

   7) the rate of heating or, alternatively, the time to reach temperature equilibrium at each test temperature,

   8) details of any procedures not specified in this part of ISO 4664;

c) test results:

   1) the number of test pieces tested,

   2) the individual and mean values of the elastic shear modulus (in-phase modulus) G' and the logarithmic decrement Λ_R, together with the units used [if the values were taken over a wide temperature interval, they should preferably be presented as a smooth curve (with shear modulus plotted on a logarithmic scale) to allow the detection of transition points];

d) date of test.

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Source for method A

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