
International Standard



4663

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Rubber — Determination of dynamic behaviour of vulcanizates at low frequencies — Torsion pendulum method

Caoutchouc — Détermination du comportement dynamique des vulcanisats à basses fréquences — Méthode du 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.

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 4663 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 4663-1984), of which it constitutes a minor revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Rubber — Determination of dynamic behaviour of vulcanizates at low frequencies — Torsion pendulum method

1 Scope and field of application

This International Standard specifies a method of determining the dynamic behaviour, that is the shear modulus and mechanical damping, of vulcanized rubbers over a wide temperature range at low frequencies in the range of 0,1 to 10 Hz, and comparatively low strain, less than 5×10^{-4} in shear, with the aid of a torsion pendulum. The test method and definitions used are in accordance with ISO 2856. The theory behind the test is also described in ISO 2856.

The test is primarily intended for the determination of the temperature at which the test piece shows transitions in the visco-elastic properties by plotting observed values of modulus and damping as a function of temperature. The method is not particularly accurate for the determination of absolute values of 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 the frame while the other one is provided with an appropriate inertial mass, for example a flywheel.

Three methods of using a torsion pendulum are specified :

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

method B, in which the mass of the inertia member is supported by a fine wire suspension and the pendulum is set into 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.

NOTE — The various methods and instruments may apply various conditions of frequency, amplitude, etc. which may influence test results. Comparison of values obtained from different methods is to be avoided.

2 References

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1826, *Rubber, vulcanized — Time-interval between vulcanization and testing — Specification.*

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

ISO 3383, *Rubber — General directions for achieving elevated or subnormal temperatures for test purposes.*

ISO 4648, *Rubber, vulcanized — Determination of dimensions of test pieces and products for test purposes.*

ISO 4661/1, *Rubber, vulcanized — Preparation of samples and test pieces — Part 1: Physical tests.*

3 Apparatus

3.1 Test piece holder

The test piece shall be held between clamps, one of which is fixed and the other attached to the inertia member. The length of the test piece between clamps shall be between 30 and 100 mm, 50 mm being the preferred length. Provision shall be made for the measurement of the length between 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 (3.4) 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.

3.2 Inertia member

The inertia 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 and 10 Hz. In the case of method A, the mass of the inertia member is limited by the longitudinal stress (see 3.3.1). A moment of inertia of the disc or rod of about 30 kg·mm² has been found suitable.

Means shall be attached to the inertia 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 $\pm 1\%$ in the region of rubber elasticity (in the transition range, an accuracy of $\pm 5\%$ is permissible).

3.3 Torsion pendulum

3.3.1 Method A

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

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

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

3.3.2 Method B

The torsion pendulum shall be constructed according to the principles shown in figure 2. The inertia 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 measuring system shall conform to that specified for method A.

3.3.3 Method C

The torsion pendulum for this forced resonance method is the same as that described for method B, with the addition of a 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 detection system, and means of measuring the supplied energy with an accuracy of $\pm 2\%$ shall be incorporated.

3.4 Thermostatted test chamber

The thermostatted chamber, with gaseous heat-transfer medium, shall be in accordance with ISO 3383. The temperature in the vicinity of the test piece shall be maintained, within the desired temperature range (for example $-100\text{ }^{\circ}\text{C}$ to $+200\text{ }^{\circ}\text{C}$), within $\pm 1\text{ }^{\circ}\text{C}$ of the desired value.

It is advisable to pass a stream of dry inert gas at the test temperature into the chamber along the test piece at a rate of several cubic centimetres per minute. This shall be done in such a way as to prevent any draught during the actual test period. Suitable temperature-sensing elements are thermocouples or resistance elements.

3.5 Devices for measurement of test piece dimensions

Suitable devices for measurement of test piece width and thickness shall be in accordance with ISO 4648.

4 Test piece

4.1 Dimensions

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

thickness : $1,0 \pm 0,2\text{ mm}$

width : $8 \pm 3\text{ mm}$, 10 mm being the preferred value

length : $80 \pm 40\text{ mm}$, chosen to fit the clamping device and to give the desired free length (see 3.1)

Individual test pieces shall comply with the tolerances given in 5.1. For comparative tests, the same nominal dimensions of test pieces shall be used.

4.2 Preparation

Test pieces shall be prepared in accordance with ISO 4661.

4.3 Number

For each test, one or more test pieces may be used.

4.4 Conditioning

4.4.1 The time interval between vulcanization and testing shall be in accordance with ISO 1826.

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