TC 45

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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

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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 4663 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in November 1975.

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

Australia	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Italy	Sweden
Canada	Mexico	Turkey
Czechoslovakia	Netherlands	United Kingdom
France	Poland	U.S.A.
Germany	Portugal	U.S.S.R.

No member body expressed disapproval of the document.

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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 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 this method, the mass of the inertia member is supported by the test piece and the pendulum is set into free damped oscillation.

Method B : In this method, the mass of the inertia member is supported by a fine wire suspension and the pendulum is set into free damped oscillation.

Method C : This method 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, *Rubbers — Time-lapse between vulcanization and testing.*

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

ISO 3383, *Rubber — General directions for achieving elevated or sub-normal temperatures for tests.*

ISO 4648, *Rubber, vulcanized — Determination of dimensions of test pieces and products.* [1]

ISO 4661, *Rubber — Preparation of test pieces.*

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.

1) At present at the stage of draft.

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 $kg \cdot mm^2$ 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 ± 1 % in the region of rubber elasticity (in the transition range, an accuracy of ± 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 ± 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 %.

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 ± 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 °C to + 200 °C), within ± 1 °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 ± 0,2 mm

width : 8 ± 3 mm, 10 mm being the preferred value

length : 80 ± 40 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-lapse 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.

4.4.3 Test pieces shall be conditioned for not less than 3 h at one of the standard laboratory temperatures, as specified in ISO 471, immediately before testing.

5 PROCEDURE

5.1 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 value. The difference between the largest and smallest values of width and thickness respectively should not exceed 6 %; if it does, the test piece shall be rejected. As the thickness appears in the calculation to the third power, suitable care shall be taken when making these measurements.

NOTE — The measurement of 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 be checked at regular intervals by measurements on prepared test pieces.

5.2 Mechanical conditioning of test piece

The shear modulus of vulcanizates 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 not 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 is then advisable to use a separate test piece for each temperature of test.

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

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

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

5.3 Mounting of test piece

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

Place the two clamps in a vertical line, the centre lines forming the axis of rotation of the pendulum. The centre lines of the test piece after clamping shall coincide with the axis of rotation.

5.4 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, the test piece being in a straight but as far as possible unstrained condition. (see 3.1).

5.5 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 first and 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 thermostat is reached.

5.6 Testing

Start the oscillation by applying a slight twist to the movable clamp followed, in the case of the damping compensated instrument (method C), by switching on the compensating system.

Measure either the frequency and the decrement of the amplitude (methods A and B) or the frequency and the supplied mechanical compensating moment (method C).

6 STRAIN, FREQUENCY AND TEMPERATURE OF TEST

6.1 Strain

The shear strain in the rubber 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

$$\gamma = \frac{\alpha \times h}{L}$$

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°.

6.2 Frequency

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

6.3 Temperature

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

Measurements shall be made at 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.

NOTE — For crystallizing materials, the times used for conditioning and testing, or alternatively the rate of heating, may influence the degree of crystallization and thus the modulus values obtained. For that reason, this time shall be selected to suit the purpose of the test and shall be stated in the test report.

7 EXPRESSION OF RESULTS

7.1 Symbols

The following symbols are used in the calculation of results with their assigned meanings and units of measurement :

$|G^*|$ is the absolute value of the complex shear modulus, in pascals;

I is the moment of inertia of the inertia member and oscillating clamp, in kilograms metres squared;

f is the frequency of oscillation with the test piece, in hertz;

f_0 is the frequency of oscillation with no test piece, in hertz;

L is the free length of the test piece, in metres;

b is the width of the test piece, in metres;

h is the thickness of the test piece, in metres;

Λ is 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;

K is the compensating mechanical moment, in newton metres;

C is a correction factor $= (1 - 0{,}63\,h/b)$ 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 is to be taken into account when $\Lambda \geqslant 1$, and equated to 1 when $\Lambda < 1$.

7.2 Calculation of the absolute value of complex shear modulus

7.2.1 *Method A*

$$|G^*| = \frac{12\pi^2 L}{b\,h^3\,C} \cdot F_D \cdot I f^2$$

7.2.2 *Method B*

$$|G^*| = \frac{12\pi^2 L}{b\,h^3\,C} \cdot F_D \cdot I\,(f^2 - f_0^2)$$

7.2.3 *Method C*

$$|G^*| = \frac{12\pi^2 L}{b\,h^3\,C} \cdot I\,(f^2 - f_0^2)$$

7.3 Calculation of mechanical damping

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 δ by the expression

$$\tan\delta = \frac{\Lambda_R}{\pi}$$

7.3.1 *Method A*

The logarithmic decrement is given accurately by the formula

$$\Lambda_R = \log_e \frac{A_1}{A_2}$$

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 according to the formula

$$\Lambda_R = \frac{1}{n} \log_e \frac{A_0}{A_n}$$

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

7.3.2 *Method B*

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

$$\Lambda = \log_e \frac{A_1}{A_2}$$

See note to 7.3.1.

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

$$\Lambda_R = \Lambda - \Lambda_0 \frac{f_0}{f}$$

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 factor can usually be neglected.

7.3.3 *Method C*

The logarithmic decrement is given by the formula

$$\Lambda_R = \frac{K}{4\pi I f^2 A}$$

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

8 TEST REPORT

The test report shall include the following particulars

a) Sample details :

1) full description of the sample and its origin;

2) compound details and curing conditions, if known;

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

4) any relevant facts about the pre-test history of the test pieces;

5) width and thickness of test pieces as well as their free length between clamps.

b) Test method and test details :

1) number of this International Standard and corresponding national standard;

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

3) shear strain applied;

4) test frequencies;

5) whether mechanical conditioning was applied;

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

7) rate of heating or, alternatively, time for reaching temperature equilibrium used at each test temperature;

8) any non-standardized procedures adopted.

c) Test results .

Absolute value of the complex shear modulus $|G^*|$ and value of the logarithmic decrement Λ_R, together with the units used;

If values are taken over a large temperature interval they may preferably be presented as a smooth curve (shear modulus on logarithmic scale) to allow the detection of transition points.

d) Date of test.

ANNEX

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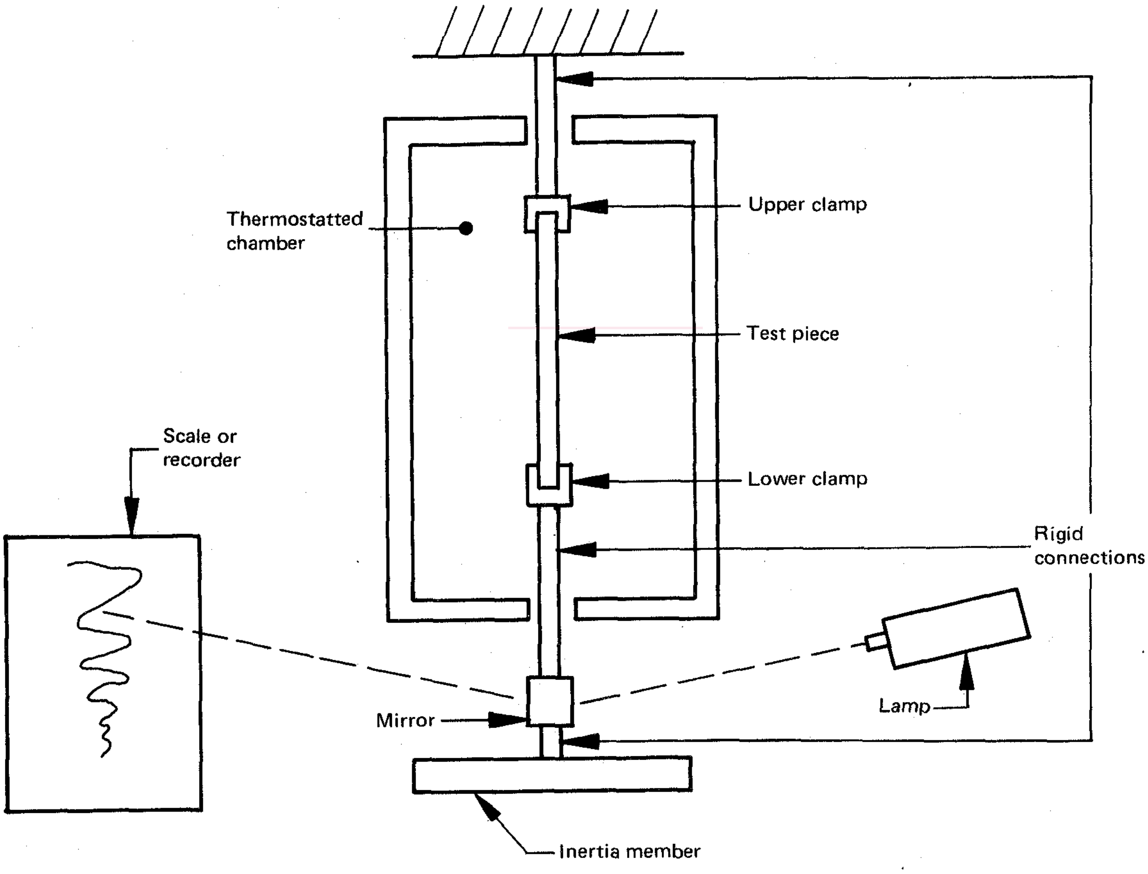

FIGURE 1 — **Uncompensated free oscillation apparatus with inertia member suspended below the test piece**

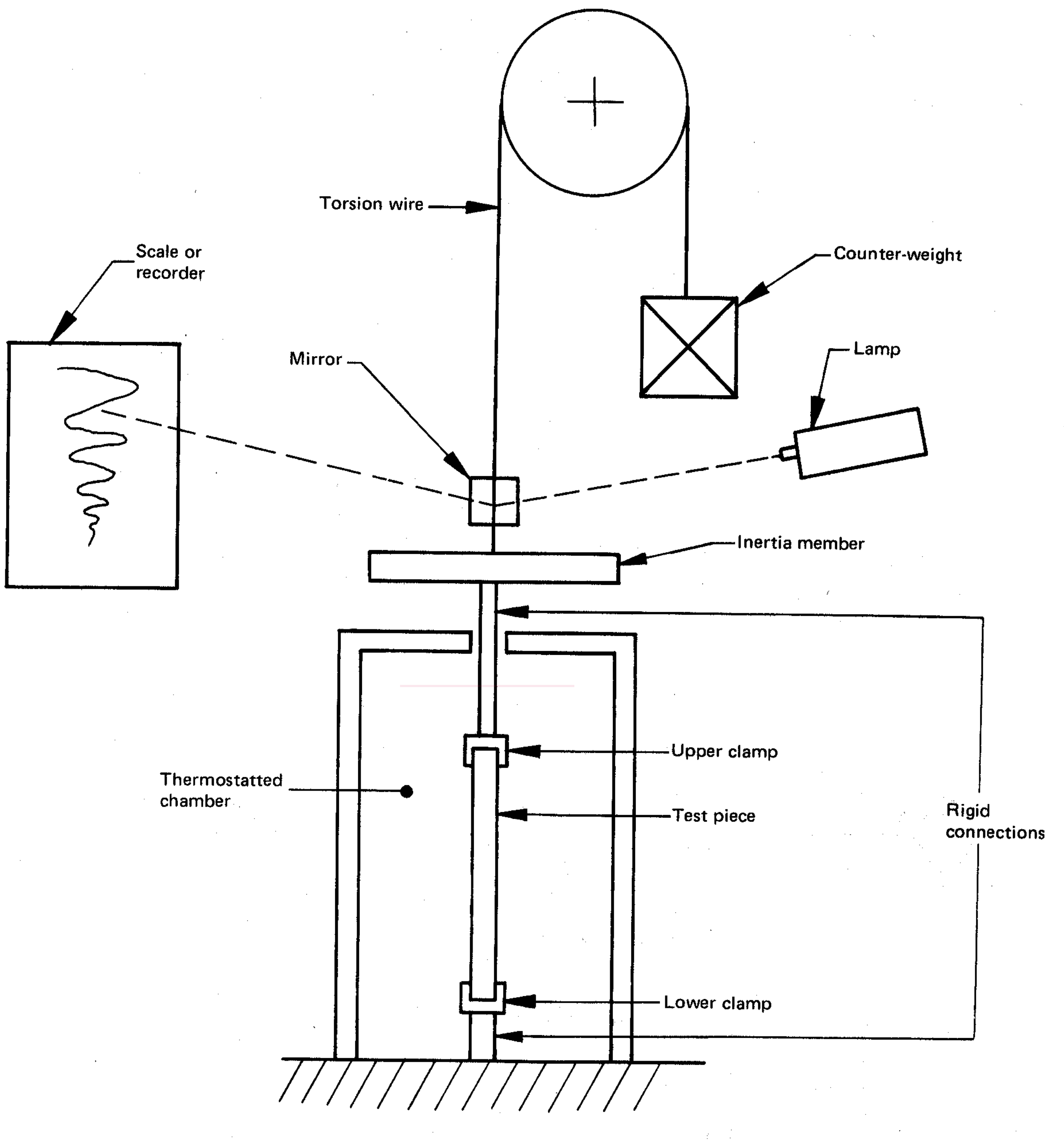

FIGURE 2 — **Uncompensated free oscillation apparatus with counter-weighted inertia member suspended above the test piece**

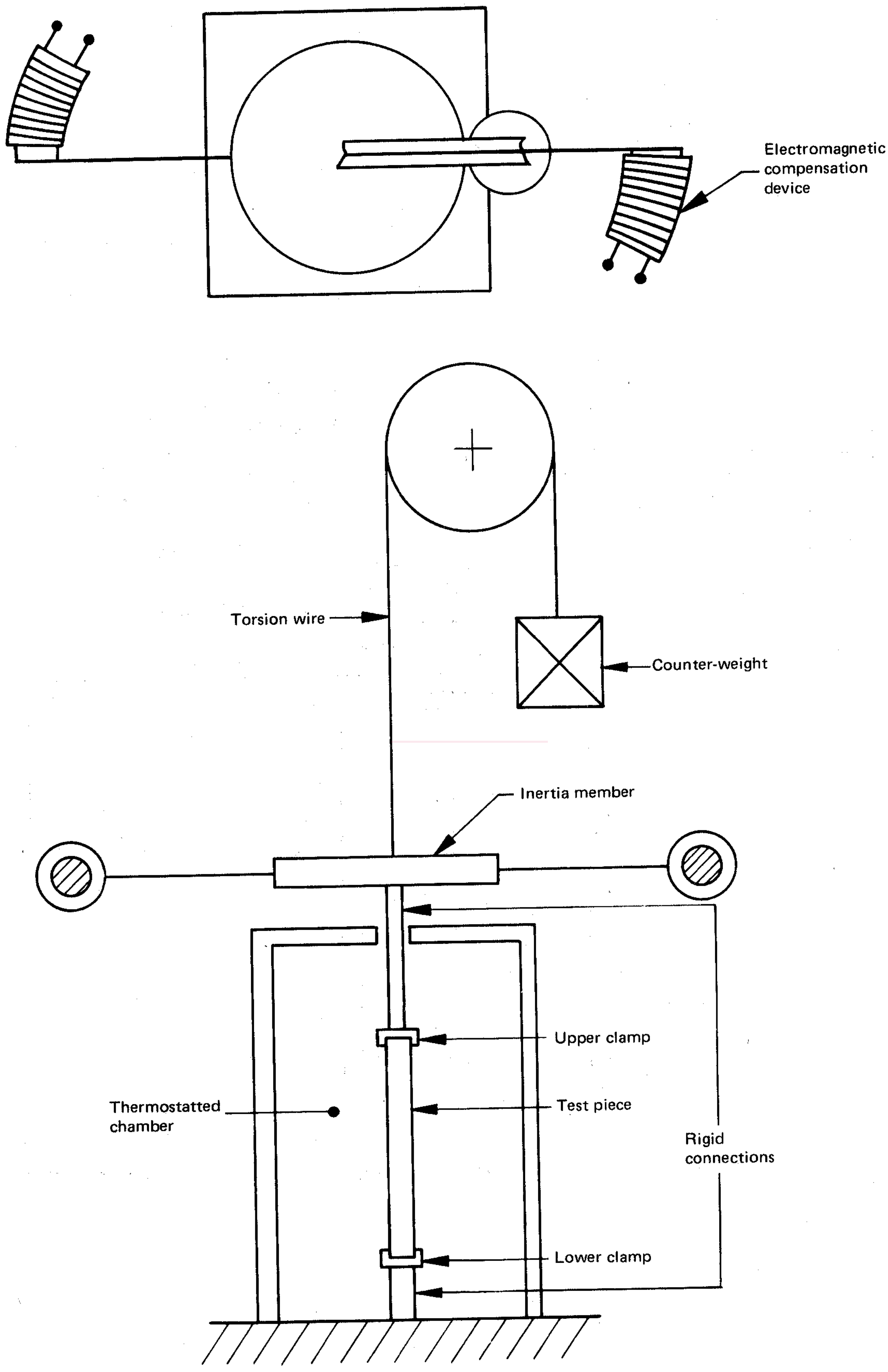

FIGURE 3 – **Forced resonance oscillation device**