TC 45

INTERNATIONAL STANDARD ISO 4664

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Rubber — Determination of dynamic properties of vulcanizates for classification purposes (by forced sinusoidal shear strain)

Caoutchouc — Détermination des propriétés dynamiques des caoutchoucs vulcanisés aux fins de classification (par application d'une force sinusoïdale de cisaillement)

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

International Standard ISO 4664 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in September 1975.

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

Australia	Hungary	Sweden
Belgium	India	Turkey
Brazil	Italy	United Kingdom
Bulgaria	Mexico	U.S.A.
Canada	Netherlands	U.S.S.R.
Czechoslovakia	Poland	Yugoslavia
France	Romania	
Germany	Sri Lanka	

No member body expressed disapproval of the document.

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Rubber — Determination of dynamic properties of vulcanizates for classification purposes (by forced sinusoidal shear strain)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method of determining the dynamic properties of vulcanized rubbers in the hardness range 30 to 80 IRHD using a testing machine operating with forced sinusoidal shear strain. A limited set of conditions are suggested for the test, chosen to agree with typical service conditions of rubber in mechanical components operating under dynamic strain, for example vibration isolators. The method of test is mainly intended for practical comparison of dynamic properties of vulcanizates in such applications, for example for classification purposes.

The test method and definitions used are in general accordance with ISO 2856, which should also be consulted for the theoretical background of the test.

NOTE — This test method operates with a limited set of conditions, including a single frequency and a single shear strain amplitude, to make a simple test and a simple testing machine possible. For full assessment of the dynamic properties of rubber, testing over a wide range of temperatures, frequencies and amplitudes may be necessary. In such cases use the standard conditions of ISO 2856.

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 4661, *Rubber — Preparation of test pieces.*[1)]

3 TESTING MACHINE

3.1 General principles of construction

The testing machine used shall be in general agreement with the requirements of ISO 2856. It shall be of sturdy construction to give accurate and reproducible results under the operating conditions described below. Care shall be taken to avoid resonance phenomena under operating conditions.

Any driving mechanism to give forced dynamic sinusoidal displacements, determined with an accuracy of ± 2,5 %, in agreement with the conditions prescribed may be used, for example mechanical (eccentric), servo-hydraulic or electro-dynamic.

3.2 Force capability and measurement

The testing machine shall have an adequate range for the materials to be tested and test pieces and deformations used. A device shall be supplied enabling the force to be read with an accuracy of ± 2,5 %. A calibration device shall also be supplied, for example steel springs, by which force readings over the range utilized can be calibrated with an accuracy of ± 2,5 %.

3.3 Cycling capability

The testing machine shall be able to operate with a frequency of 10 ± 0,5 Hz and with the required dynamic displacement amplitude.

The deformation cycles shall be in the form of a continuous wave train of sinusoidal shape with less than 10 % harmonic content.

3.4 Damping measurement device

The testing machine shall be fitted with a device to enable the loss factor of the material under test to be determined to an accuracy of ± 5 % or 0,02 in $\tan\delta$, whichever is the greater.

3.5 Thermostatic chamber

The testing machine shall be supplied with a thermostatic chamber with gaseous heat-transfer medium in accordance with ISO 3383. Care shall be taken to reduce heat conduction from the test piece through the test piece holders to the outside of the chamber.

Accurate temperature control is of great importance owing to the generally high temperature-dependence of dynamic properties of rubber.

1) At present at the stage of draft.

3.6 Devices for measurement of test piece dimensions

3.6.1 Micrometer dial gauge having a scale graduated in unit divisions of 0,01 mm, for the measurement of test piece thickness.

3.6.2 Suitable device for measurement of test piece cross-sectional dimensions, capable of measuring accurately to the nearest 0,1 mm.

4 TEST PIECE

4.1 Preparation

Test pieces shall be prepared in accordance with ISO 4661.

4.2 Dimensions

The test piece shall be of double sandwich construction (see the figure) containing two parallellepipedic rubber elements with a ratio of side to thickness of 4 : 1. The preferred thickness of the elements is 4 mm. In no case shall the thickness of the elements be less than 3 mm or more than 7 mm.

The elements shall be firmly attached to metal end plates. Bonding to end plates is best done during the curing operation starting from uncured rubber compound. When test pieces are cut from the product, bonding with a suitable adhesive, for example a cyanoacrylate adhesive, may be used.

The size of the test piece is selected to suit the force and amplitude range of the testing machine available.

4.3 Number of test pieces

Three test pieces shall be used.

4.4 Conditioning

4.4.1 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 before testing at one of the standard laboratory temperatures specified in ISO 471.

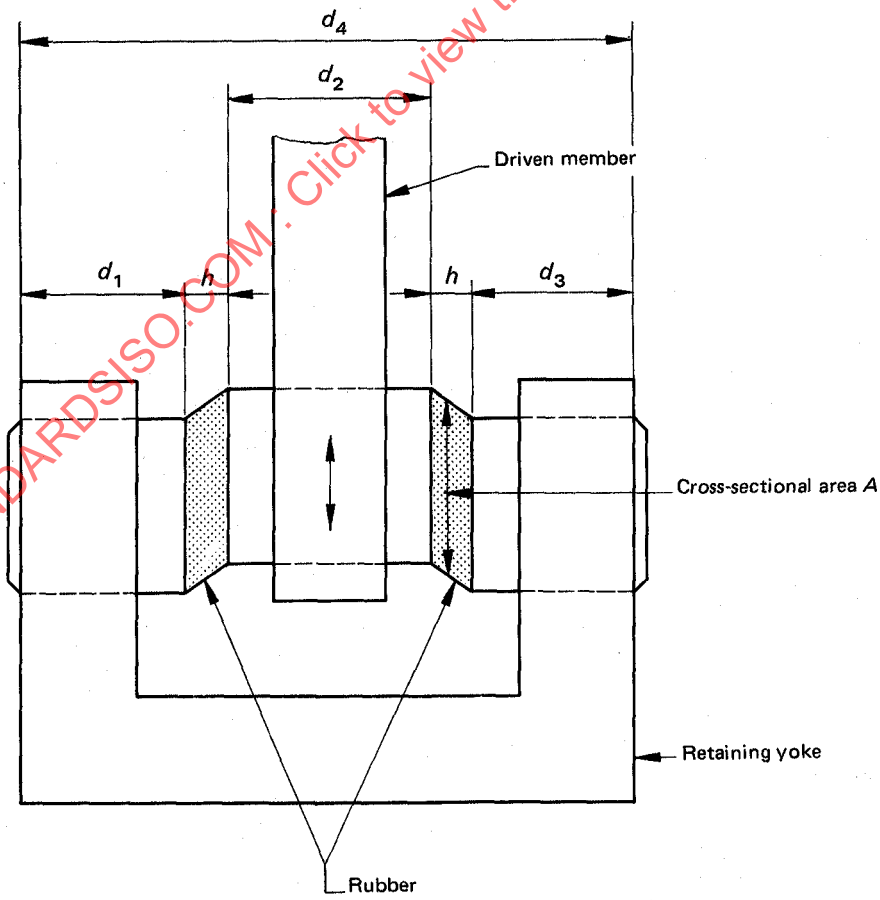

FIGURE — **Structure and dimensions of the test piece**

5 PROCEDURE

5.1 Measurement of dimensions of test piece (see the figure)

5.1.1 Measure all thicknesses to the nearest 0,01 mm and all cross-section dimensions to the nearest 0,1 mm.

Carry out all measurements at standard laboratory temperature.

5.1.2 For test pieces prepared by bonding previously vulcanized rubber elements to metal end pieces, the thickness h and cross-sectional area A shall be measured before bonding. The mean values for h and A shall be calculated.

5.1.3 For test pieces prepared by moulding, measure the thicknesses d_1, d_2 and d_3 before moulding, and the overall dimension d_4 after moulding; from these values, the mean value of h can be determined as :

$$h = \frac{1}{2}[d_4 - (d_1 + d_2 + d_3)]$$

The cross-sectional area A shall be measured after bonding.

5.2 Conditioning

When measurements are to be made at 70 ± 1 °C test pieces shall be conditioned at that temperature in accordance with ISO 3383. This can be done in the thermostatic chamber connected to the testing machine or, if suitable, in an adjacent conditioning chamber operating as specified in ISO 3383. Test piece holders and instruments used for handling test pieces shall also be conditioned for at least 60 min at the test temperature before testing.

5.3 Clamping of test pieces

Firmly attach the test piece so that no slippage occurs during testing.

5.4 Testing

Start the dynamic vibration device. After at least six cycles of running, record the measurements of force amplitude, displacement and damping. Measurements shall be made within the first 60 s of running. Preferably, measurements of force and displacement amplitudes should be made before those of damping.

5.5 Control of test piece homogeneity

Test pieces that have been used for the test shall be cut through their cross-section after testing; if any internal defects such as gas bubbles are found, the test result shall be discarded.

6 STRAIN, FREQUENCY AND TEMPERATURE OF TEST

6.1 Strain

The shear strain amplitude shall be 0,06 ± 0,006.

NOTE — The actual strain must be known to within ± 0,001 5 in order to ensure that the error is less than ± 2,5 %.

6.2 Frequency

The test shall be run with a frequency of 10 ± 0,5 Hz.

6.3 Temperature

Standard laboratory temperature shall be used as the temperature of test. If, however, an elevated temperature is desired to comply with some operating conditions, 70 ± 1 °C shall be used as the temperature of test.

7 EXPRESSION OF RESULTS

7.1 General

For accuracy, all values of test piece areas and strain shall be calculated from measured values of unstrained test piece dimensions.

7.2 Calculation

Calculate the absolute value of complex shear modulus $|G^*|$, in pascals, by means of the formula

$$|G^*| = \frac{F}{2A \times \gamma}$$

where

F is the shear force amplitude, in newtons;

A is the test cross-sectional area, in square metres;

γ is the shear strain amplitude.

The formula applies for the double sandwich test piece and the ratio of side to thickness of 4 : 1 (see ISO 2856-1975, sub-clause 7.2.3.1) and assumes linearity.

7.3 Damping

Depending on the design of the testing machine, different measures of damping may be obtained, for example loss modulus, loss factor or loss angle. These shall be transformed to and reported as loss factor, $\tan\delta$ (see ISO 2856).

7.4 Calculation of average

Calculate the averages of $|G^*|$ and $\tan\delta$ for the three pieces; if any single value deviates more than 10 % from the average, the test shall be repeated using three additional test pieces, and the average for all six test pieces reported.

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) method of attaching metal plates to test pieces;

5) any relevant facts about its pre-test history.

b) Test method and test details :

1) number of International Standard and corresponding national standard;

2) testing machine used;

3) mechanical conditioning (see 5.4) prior to recording data;

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

5) test temperature;

6) any non-standardized procedures adopted.

c) Test results :

1) number of test pieces tested;

2) dimensions of the rubber elements of test pieces;

3) mean absolute value of complex shear modulus and mean $\tan \delta$ of the test pieces tested, together with the appropriate units used;

4) date of test.