International Standard ISO 4666/3

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION●МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ●ORGANISATION INTERNATIONALE DE NORMALISATION

Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing — Part 3 : Compression flexometer

Caoutchouc vulcanisé — Détermination de l'élévation de température et de la résistance à la fatigue dans les essais aux flexomètres — Partie 3 : Flexomètre à compression

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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 4666/3 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in September 1979.

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

Australia	India	Spain
Austria	Italy	Sweden
Belgium	Korea, Rep. of	Switzerland
Czechoslovakia	Libyan Arab Jamahiriya	Turkey
Denmark	Malaysia	United Kingdom
Egypt, Arab Rep. of	Netherlands	USA
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The member bodies of the following countries expressed disapproval of the document on technical grounds :

Brazil
Canada

Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing — Part 3 : Compression flexometer

0 Introduction

One major consequence of the internal heat generation of rubber under a flexing compression is the development of an elevated temperature in the rubber. This International Standard provides for the measurement of the temperature rise.

Under particularly severe heat generation and temperature rise conditions, internal rupture of the test piece may occur with fatigue failure. Provision is also made for the measurement of resistance to this type of fatigue.

The test is conducted under conditions of a selected static pre-stress or compression and a selected cyclic strain of constant maximum amplitude imposed upon the pre-stressed test piece.

1 Scope and field of application

This part of ISO 4666 specifies the compression flexometer test for the determination of the temperature rise and resistance to fatigue of vulcanized rubbers. The flexometer specified is known as the Goodrich flexometer, but any other apparatus giving equivalent performance may be used.

It gives directions for carrying out measurements which make possible predictions regarding the durability of rubbers in finished articles (tyres, bearings, supports, V-belts, cable-pulley insert rings and similar products subject to dynamic flexing in service). However, owing to the wide variations in service conditions, no simple correlation between the accelerated tests described in this International Standard and service performance can be assumed.

The method is not recommended for rubber having a hardness greater than 85 IRHD.

2 References

ISO 48, *Vulcanized rubbers — Determination of hardness (Hardness between* 30 *and* 85 IRHD*).*

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

ISO 4666/1, *Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing — Part 1 : Basic principles.*

3 Definitions

The terms and concepts used in connection with this test and in evaluating the test results are dealt with in ISO 4666/1.

4 Principle

Application of a specified compressive load to a test piece through a lever system having high inertia, while imposing on the test piece an additional high-frequency cyclic compression of specified amplitude. Measurement of the increase in temperature at the base of the test piece with a thermocouple to provide a relative indication of the heat generated in flexing the test piece, and measurement of the number of cycles to produce fatigue breakdown.

Subjection of the test pieces to a constant applied load, or to a constant initial compression, and continuous measurement of the change in height of the test piece during the test. Measurement of the permanent set after testing.

5 Test piece

The test piece, prepared from vulcanized rubber, shall be cylindrical in shape, having a diameter of 17,8 ± 0,15 mm and a height of 25 ± 0,25 mm.

The standard method of preparing the test piece shall be the direct moulding of the cylinder. It is suggested, for purposes of uniformity and closer tolerances in the moulded test piece, that the dimensions of the mould be specified and shrinkage compensated for therein. A plate cavity of thickness 25,4 ± 0,05 mm and diameter 18,00 ± 0,05 mm, having overflow cavities at both top and bottom when combined with two end plates, will provide one type of a suitable mould.

An optional method of preparing the test piece may be cutting from a laboratory slab. The vulcanized thickness shall be such that buffing is not required.

The circular die used for cutting the test piece shall have an inside diameter of 17,8 ± 0,03 mm. In cutting the test piece, the die shall be suitably rotated in a drill press or similar device and lubricated by means of a soap solution. A minimum distance of 13 mm shall be maintained between the cutting edge of the die and the edge of the slab. The cutting pressure shall be as light as possible to minimize cupping or taper in the diameter of the test piece.

NOTE — It should be recognized that an equal time and temperature used for both moulded and slab test pieces will not produce an equivalent state of vulcanization in the two types of test piece. A higher degree of vulcanization will be obtained in the moulded test piece. Adjustments, preferably in the time of cure, must be taken into consideration if comparisons between the two types of test piece are to be considered valid.

6 Apparatus

6.1 Flexometer

6.1.1 General description

The essential parts of the apparatus are shown in figure 1.

The test piece is placed between anvils faced with a thermal-insulating material. The top anvil is connected to an adjustable eccentric usually driven at an oscillation rate of 30 ± 0,2 Hz.

The load is applied by means of a lever resting on a knife edge. The moment of inertia of the lever system is increased, and its natural frequency reduced by suspending masses of approximately 24 kg at each end of the lever system. The lower anvil may be raised or lowered relative to the lever by means of a calibrated micrometer device. This device permits the lever system to be maintained in a horizontal position during the test as determined by a pointer and a reference mark on the end of the bar.

The increase in temperature at the base of the test piece is determined by means of a thermocouple placed at the centre of the bottom anvil.

6.1.2 Detailed description

The apparatus (see figure 1) consists of a balance beam (6) which can be locked in its horizontal position by means of a steel pin. The beam is provided with masses of 24 kg (8) at both ends. The distance between the knife edge supporting the beam and the edges supporting the masses is 288 ± 0,5 mm. An equivalent inertial system may be used.

The test piece (2) is placed upon a plate (3) on one arm of the balance beam. The distance of the test piece support (10) from the fulcrum is 127 ± 0,5 mm. On the other side of the balance beam, additional weights (7) are placed in order to apply a load to the test piece. The desired weights are 11 kg or 22 kg which correspond to a prestress of 1,0 ± 0,03 MPa or 2,0 ± 0,06 MPa, respectively.

The test piece (2) is placed between the plates (1 and 3), which are made of a thermal-insulating material having a thermal conductivity of not more than 0,28 W/m·K or, equivalently, 0,24 kcal/h·m·°C. Phenolic hardpaper may be used for this purpose. In the centre of the lower plate, a thermocouple, for example iron-constantan, is attached for temperature measurement. The sensing point of the thermocouple shall be in contact with the test piece. The sensitivity of the thermocouple shall be ± 0,5 °C.

Means shall be provided for measuring the decrease in height of the test piece, as the test proceeds, with an accuracy of 0,1 mm. For this purpose, the distance between the lower and upper plates can be varied by means of a calibrated micrometer device until it returns to the horizontal position, which can be recognized by a mark on the balance beam and a pointer (5) on the casing. The adjustment device consists of a micrometer screw (4) which, by means of a chain and sprocket-wheel drive, moves the screw (9) up or down without rotating the lower plate (3). The degree of adjustment is read from the micrometer screw (4). The centre point of the upper plate (1) remains in the same position. The upper plate (1) is connected through a guide bearing to an eccentric which can be set to the desired stroke in a range from 4,45 to 6,35 mm and is driven by a motor at 30 ± 0,2 Hz.

The test piece with the supporting plates is located in a chamber, the temperature of which can be controlled to within ± 1 °C of a standard test temperature generally in the range from 40 to 100 °C. The chamber should have the following dimensions :

— width 100 mm;

— depth 130 mm;

— height 230 mm.

The bottom of the chamber should be situated 25 ± 2 mm above the balance beam.

A thermocouple of the same type as that used in the lower plate should be used for measurement of the temperature in the chamber. The thermocouple should be positioned at a distance of 6 to 9 mm towards the right hand side behind the rear edge of the plate and at a height midway between the plates. A length of at least 100 mm of the thermocouple wire should be within the chamber.

The air circulation within the chamber is provided by a radial fan of 75 mm diameter, operating at a rotational frequency of 25 to 28 Hz. The air intake should have a diameter of 60 mm. The air outlet should measure 40 mm × 45 mm. The grid shelf for supporting test pieces during conditioning should be fitted 10 ± 2 mm above the bottom of the chamber. A diagram of the heating chamber is shown in figure 2.

6.2 Measuring gauge

The gauge for measuring the height and diameter of test pieces shall conform to the requirements of ISO 4648. A dial gauge having a circular foot probe of diameter 10 mm and exerting a pressure of 22 ± 5 kPa is suitable.

6.3 Timer

A stopwatch or other similar device shall be used.

7 Procedure

7.1 Preparation of flexometer

Locate the machine on a firm foundation. Adjust the levelling screws in the base to bring the machine into a level position in all directions at a point just to the rear of the fulcrum of the loading lever. With the loading lever locked in place with the pin, place a level on the lever bar and verify the level setting.

Adjust the eccentric to give a stroke or a double amplitude of 4,45 ± 0,03 mm. This is best accomplished by means of a dial micrometer resting on either the cross bar of the upper anvil or by means of adapters attached to the loading arm of the eccentric.

NOTE — The 4,45 mm stroke is selected as the standard for calibration purposes. When strokes other than 4,45 mm are to be used, the displacement of the lower anvil should be maintained within the tolerance specified for its height above the loading lever. The tolerance for all stroke settings shall be ± 0,03 mm.

Raise the top anvil as far as the eccentric will permit by its rotation. Place a calibrating block of height 25,0 ± 0,01 mm on the lower anvil.

NOTE — A suitable block may be made from brass having a diameter of 17,8 mm. The end to be placed on the lower anvil should be counterbored for clearance of the thermocouple disc.

Raise the anvil by means of the micrometer until the bottom side of the metal cup holding the thermocouple is 67 ± 3 mm above the top of the loading lever. The loading lever shall be in the locked position.

Adjust the cross bar of the upper anvil, maintaining a parallel setting with the lower anvil and a firm contact with the calibrating block. The micrometer should now be set at zero. This may require disengagement of the gear train nearest the vernier scale of the micrometer.

Remove the calibrating block and recheck the stroke or double amplitude for 4,45 mm setting. Set the pointer on the mark at the end of the lever bar. This establishes the level position.

Remove the locking pin from the loading lever and gently oscillate the lever system to determine the point of rest. If the bar does not come to rest in approximately the level position, slowly return it to its level position and release. If movement from the level position is observed, add or remove a weight to or from the required inertia weight to obtain a balance.

7.2 General test operation

Check the machine for proper adjusment (see 7.1) and the required test conditions (see clause 8). Place the necessary weights on the rear hanger to give the desired load.

If a stroke other than 4,45 mm is desired, a new zero setting will be required on the micrometer after adjusting the eccentric to the new stroke. Proceed as outlined in 7.1 to obtain the zero setting.

For elevated temperatures requiring the use of the heating chamber, allow a minimum of 2 h for preheating of the apparatus and the attainment of equilibrium prior to the start of test. Maintain the lower anvil at the zero setting, that is, 67 mm above the loading lever during the conditioning period.

Measure and record the height of the test piece. Then measure its hardness in accordance with ISO 48.

When the heating chamber is to be used, place the test piece in the chamber on the platform. Condition for a minimum of 30 min before the start of test.

Before starting the test, the lower platen temperature and the ambient test temperature shall be equal. With the upper anvil or cross bar in its highest position, lower the bottom anvil and quickly position the test piece thereon, inverting its position from that used during the warm-up period.

Raise the lower anvil by means of the micrometer until a firm contact is established with the upper anvil or hammer. Remove the locking pin and apply the load. Then advance the micrometer until the beam is again restored to its original level position as determined by the indicator.

If the test piece had an original height of exactly 25,0 mm, then the micrometer reading may be used without correction for the compression height.

When the original height of the test piece is less than 25,0 mm, then the difference shall be subtracted from the micrometer reading. For a test piece of height greater than 25,0 mm, the difference shall be added to the micrometer reading.

For a smooth start, restore the pin to the locked position of the loading lever, and back off the micrometer three to four turns. Then loosen the pin, start the machine, and remove the pin completely. Immediately restore the beam to the level position by means of the micrometer and record the reading. Subject this reading to the same corrections as used for the static measurements.

CAUTION — If the initial running deflection is less than one half of the impressed stroke or does not exceed this value within 1 or 2 min of the start, an unreliable and misleading temperature rise will be obtained. The loading lever must be maintained in a level position throughout the test.

NOTE — The thermocouple in the lower anvil will stabilize at a temperature approximately 6 °C lower in an ambient chamber temperature of 100 °C. This is the base temperature above which the temperature rise is measured. Any momentary drop in the base temperature at the start of the test should be disregarded.

If a recorder is not used to obtain a continuous temperature rise curve, obtain a series of measurements using a suitable potentiometer. Plot the readings and draw the temperature rise curve.

7.2.1 Determination of temperature rise and of compression set

To determine the temperature rise and the compression set, continue the test for the recommended test duration of 25 min, provided that no premature failure occurs in the test piece. Longer test durations may be required if steady state conditions are always to be attained. At the end of the test, remove the test piece from the apparatus and after storage for 1 h for cooling to standard laboratory temperature, measure the height h_e.

7.2.2 Determination of fatigue resistance

To determine the fatigue life, continue the test until breakdown occurs. Incipient breakdown is revealed by an irregularity in the temperature curve (sudden temperature rise), by a marked increase in creep and by the onset of internal porosity.

If no breakdown occurs, more severe testing conditions should be selected (see clause 8).

7.2.3 Determination of creep

To determine creep, measure the test piece height 6 s after the start of the cyclic loading and then after a specified test duration.

8 Test conditions

The conditions specified in the following table are normally employed in tests with this compression flexometer.

Condition	Nominal value
Chamber temperature	55 ± 1 °C or 100 ± 1 °C
Stroke (double amplitude)	4,45, 5,71 or 6,35 mm
Pre-stress on test piece[1)]	1,0 or 2,0 MPa

1) A pre-stress of 1,0 MPa is equivalent to a weight on the balance beam of 11 kg; a pre-stress of 2,0 MPa is equivalent to a weight of 22 kg.

Tests with the heating chamber removed are referred to as "room temperature" tests, or tests at standard laboratory temperature. These may be selected for special test purposes if desired. The standard laboratory temperature used shall be specified in the test report.

For the measurement of temperature rise a chamber temperature of either 55 °C or 100 °C may be selected with a stroke of 4,45 mm or 5,71 mm. Any of these choices of temperature and stroke may be used with either the 1,0 or 2,0 MPa pre-stress on the test piece. These choices ordinarily give a temperature rise that is essentially at equilibrium after the normal test duration of 25 min. However, test times longer than 25 min may be selected, if desired, for special test purposes.

For measurement of the fatigue properties of rubber, more severe test conditions are needed. Specifically, strokes of 5,71 mm and 6,35 mm are recommended with the higher pre-stress or force on the balance beam. Selection of the more severe conditions avoids excessive test durations for each test piece.

In general, for medium hardness rubbers that have ordinary temperature rise characteristics, a pre-stress of 1,0 MPa, a stroke of 5,71 mm and a chamber temperature of 55 °C or 100 °C is recommended.

The same test conditions shall be maintained throughout a series of tests intended for comparison of a group of compounds.

9 Evaluation of test data

9.1 Temperature rise

If the temperature in the bottom area of the test piece is θ_0 at the beginning of the test and θ_{25} after 25 min of testing, then the temperature rise, $\Delta\theta$, expressed in degrees Celsius, is given by the formula

$$\Delta\theta = \theta_{25} - \theta_0 \qquad \ldots (1)$$

9.2 Creep

The creep, F_t, expressed as a percentage, is defined as the difference between the test piece height h_6, determined 6 s after the start of the cyclic loading, and the test piece height h_t, determined after the test duration t, referred to the original height h_0 in the unloaded condition, i.e.

$$F_t = \frac{h_6 - h_t}{h_0} \times 100 \qquad \ldots (2)$$

The test piece height shall be measured as previously described. The balance beam shall be set to its zero position after not more than 6 s of running time. For the original height h_0, the nominal value $h_0 = 25$ mm shall be used, since the differences must be within the tolerance range of ± 0,2 mm.

NOTE — The expression in equation (2) must be distinguished from the usual definition of creep in other cases, where creep is expressed relative to the initial deformation :

$$F_t = \frac{h_6 - h_t}{h_0 - h_6} \qquad \ldots (3)$$

The definition given in equation (2) has the advantage that there is no need to recalculate with reference to the height h_6 in the loaded condition. The quantity $(h_6 - h_t)/h_0$ can also be easily read on the micrometer or recorded by an automatic compensator, since the original height h_0 is considered to be constant (within the tolerance limits ± 0,2 mm).

9.3 Compression set

The compression set, S, is given by the difference between the original height h_0 and the final height h_e after 1 h of cooling in an unloaded condition, referred to the original height h_0, and is expressed as a percentage, all measurements being made at standard laboratory temperature, i.e.

$$S = \frac{h_0 - h_e}{h_0} \times 100 \qquad \ldots (4)$$

9.4 Fatigue life

The fatigue life is expressed as the number of cycles, N, to breakdown or failure of the test piece. Breakdown or the failure criterion must be clearly defined and the definition cited.

10 Quality control check of flexometer operation

It has been found that the use of a standard rubber formulation, prepared and vulcanized as outlined below, allows for a rapid check of the operating condition of the compression flexometer.

Standard formulation

Material	Parts (m/m)
SBR 1500	100
Zinc oxide	5
Carbon black (N 330)	45
Stearic acid	1
Tetramethylthiuram disulphide	3

This compound should be vulcanized at 150 °C for 50 ± 1 min. Test pieces cut from vulcanized block should give a temperature rise of 27 ± 1 °C when tested using a stroke of 4,45 mm, a pre-stress of 1,0 MPa and a chamber temperature of 100 ± 1 °C.

11 Test report

The test report shall include the following particulars :

a) a reference to this International Standard;

b) sample details :

1) full description of the sample and its origin,

2) compound details and vulcanizing conditions, if known;

c) test piece details :

1) method of preparation, for example whether moulded or cut or taken from finished products,

2) original height and, in case of deviations from standard dimensions, the diameter,

3) hardness;

d) test conditions :

1) pre-stress, in megapascals,

2) stroke, in millimetres,

3) heating chamber temperature, in degrees Celsius;

e) test results :

1) for the measurement of temperature rise : the test duration, the individual values and the mean value,

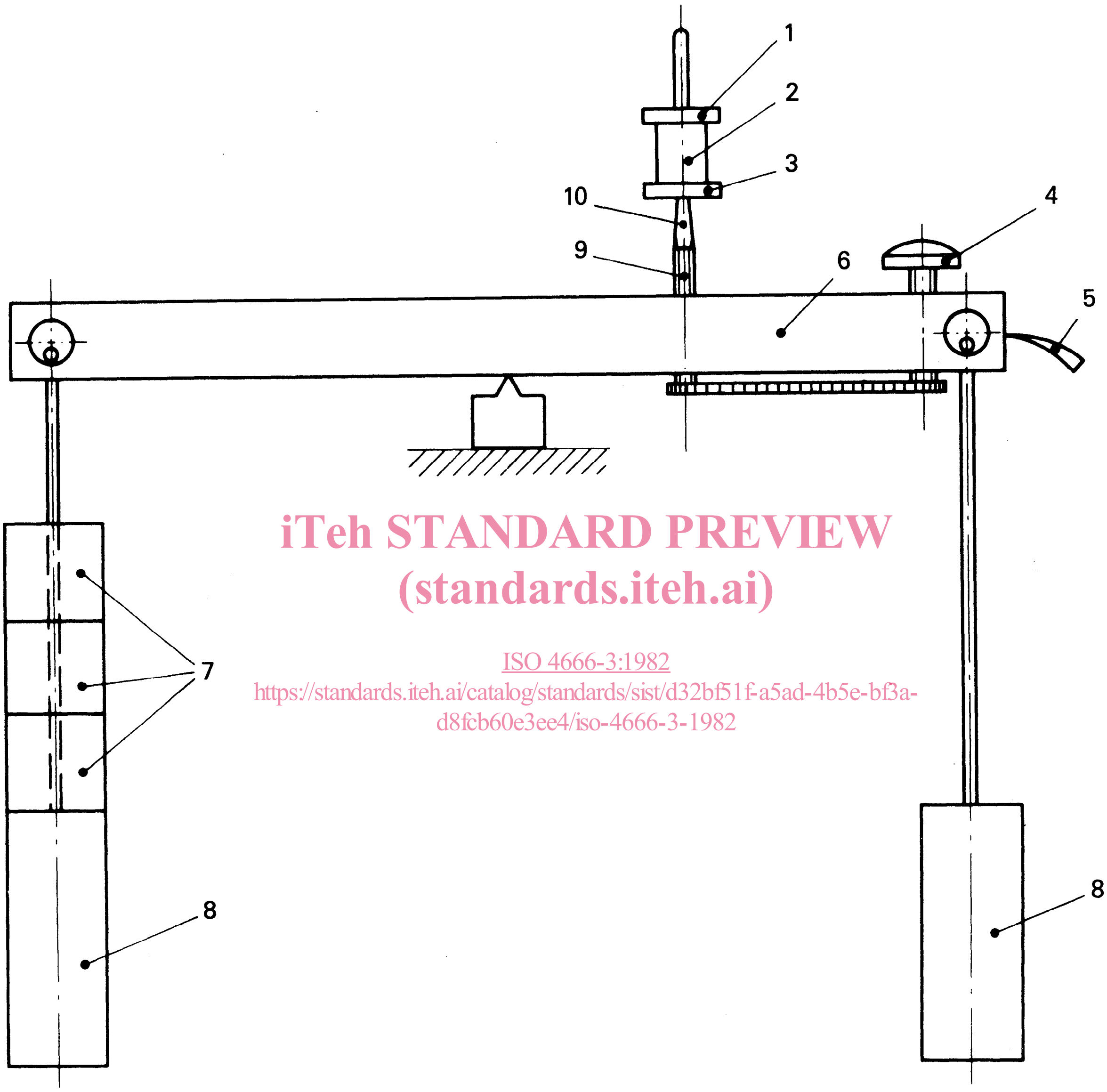
2) for the measurement of creep : the test duration, the individual values and the mean value, as a percentage,

3) for the measurement of compression set : the test duration, the individual values and the mean value, as a percentage,

4) for the measurement of fatigue resistance : the number of test pieces used, the criterion of fatigue failure, and the number of cycles to this selected failure point, expressed as individual values and the mean value;

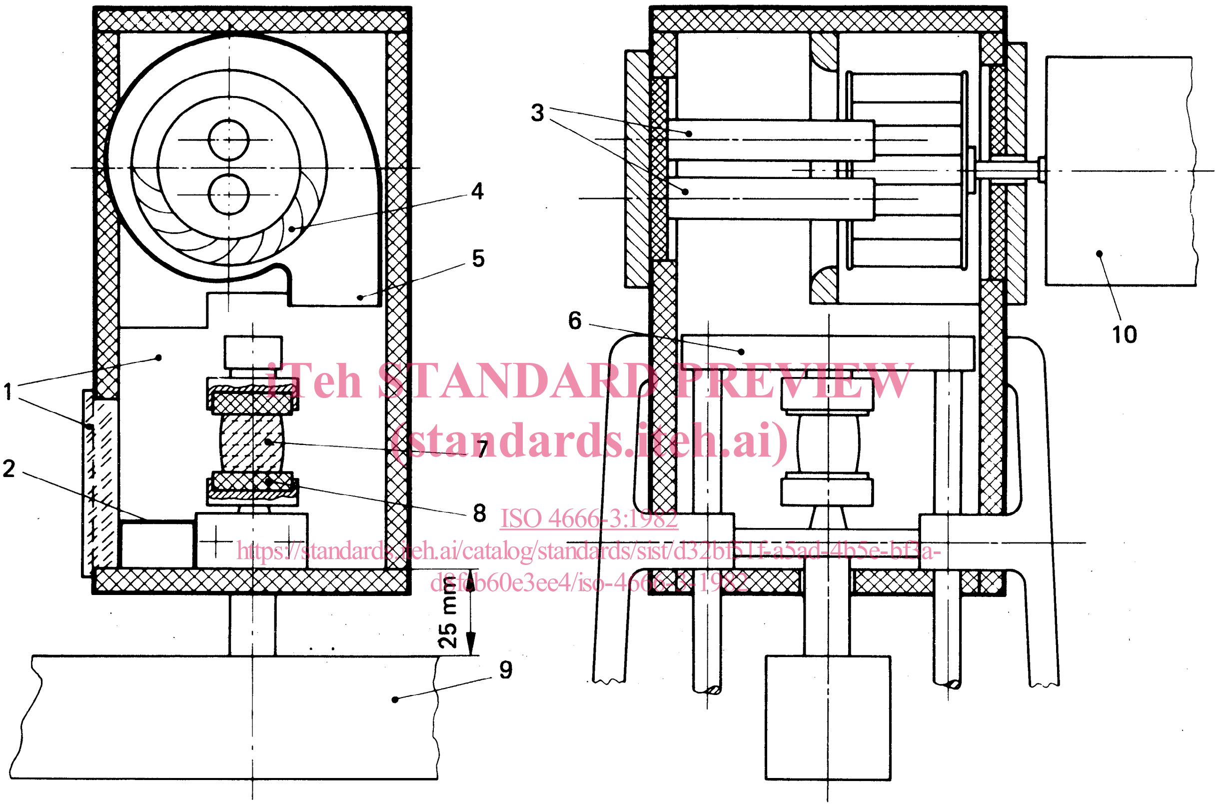
f) any departures from the procedures specified in this International Standard;

g) the date of the test.



1 Upper plate
2 Test piece
3 Lower plate
4 Micrometer screw
5 Pointer
6 Balance beam
7 Additional weights
8 Weights
9 Screw
10 Test piece support

Figure 1 — Compression flexometer — General arrangement



1 Heating chamber with door
2 Grid shell for supporting test pieces during conditioning
3 Heating elements
4 Radial fan
5 Air outlet
6 Crossbar with lifting rods and upper plate
7 Test piece
8 Lower plate with thermocouple
9 Balance beam
10 Motor of radial fan

Figure 2 — Heating chamber for compression flexometer