भारतीय मानक

Indian Standard

IS 16848 (Part 1/Sec 2) : 2024
ISO 6502-2 : 2018
(Superseding IS 11720 (Part 2) : 2013/ ISO 3417 : 2008)

रबड — परीक्षण पद्धतियाँ

भाग 1 क्यूरिमीटर का उपयोग करके वल्कनीकरण विशेषताओं का मापन

अनुभाग 2 ऑसिलेटिंग डिस्क क्यूरिमीटर

Rubber — Methods of Test

Part 1 Measurement of Vulcanization Characteristics Using Curemeters

Section 2 Oscillating Disc Curemeter

ICS 83.060

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July 2024 **Price Group 8**

Methods of Test for Rubber and Rubber Products Sectional Committee, PCD 29

NATIONAL FOREWORD

This Indian Standard (Part 1/Sec 2) which is identical to ISO 6502-2 : 2018 ‘Rubber — Measurement of vulcanization characteristics using curemeters — Part 2: Oscillating disc curemeter’ issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on recommendation(s) of the Methods of Test for Rubber and Rubber Products Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

This standard was first published in 2018, which was identical to ISO 6502 : 2016. In 2018, ISO 6502 was technically revised and published into the following three parts, under the general title ‘Rubber — Measurement of vulcanization characteristics using curemeters’:

Part 1 Introduction

Part 2 Oscillating disc curemeter

Part 3 Rotorless curemeter

The Committee responsible for the formulation of the standard decided to adopt different parts of ISO 6502 as three different sections of Part 1 of IS 16848 under dual numbering system. The other sections are.

Part 1/Sec 1 Introduction

Part 1/Sec 3 Rotorless curemeter

While reviewing various test methods published by this Committee, it was further observed that IS 11720 (Part 2) : 2013 Methods of test for synthetic rubber : Part 2 Measurement of vulcanization characteristics with the oscillating disc curemeter is also available on the subject.

The Committee decided that this standard will supersede IS 11720 (Part 2) : 2013 Methods of test for synthetic rubber: Part 2 Measurement of vulcanization characteristics with the oscillating disc curemeter. After publication of this standard, IS 11720 (Part 2) : 2013 shall be treated as withdrawn.

The text of ISO standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words ‘International Standard’ appear referring to this standard, they should be read as ‘Indian Standard’; and

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to the following International Standard for which Indian Standard also exists. The corresponding Indian Standard which is to be substituted in its place is listed below along with its degree of equivalence for the edition indicated:

(*Continued on third cover*)

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Indian Standard

RUBBER — METHODS OF TEST

PART 1 MEASUREMENT OF VULCANIZATION CHARACTERISTICS USING CUREMETERS

SECTION 2 OSCILLATING DISC CUREMETER

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document 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 determine the applicability of any other restrictions.

WARNING 2 — Certain procedures specified in this document might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies a method for determining selected vulcanization characteristics of a rubber compound by means of an oscillating disc curemeter. The introduction to the use of curemeters is described in ISO 6502-1.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 6502-1:2018, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 1: Introduction*

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6502-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

4.1 A test piece of rubber is contained in a sealed test cavity under an initial positive pressure and maintained at an elevated temperature. A biconical disc is embedded in the test piece and is oscillated through a small rotary amplitude. This action exerts a shear strain on the test piece, and the torque required to oscillate the disc depends on the stiffness (shear modulus) of the rubber. The torque is recorded autographically as a function of time.

4.2 The stiffness of the rubber test piece increases as vulcanization proceeds. The curve is complete when the recorded torque rises either to an equilibrium value or to a maximum value (see ISO 6502-1). If the torque continues to increase, vulcanization is considered to be complete after a given time. The time required to obtain a vulcanization curve is a function of the test temperature and the characteristics of the rubber compound.

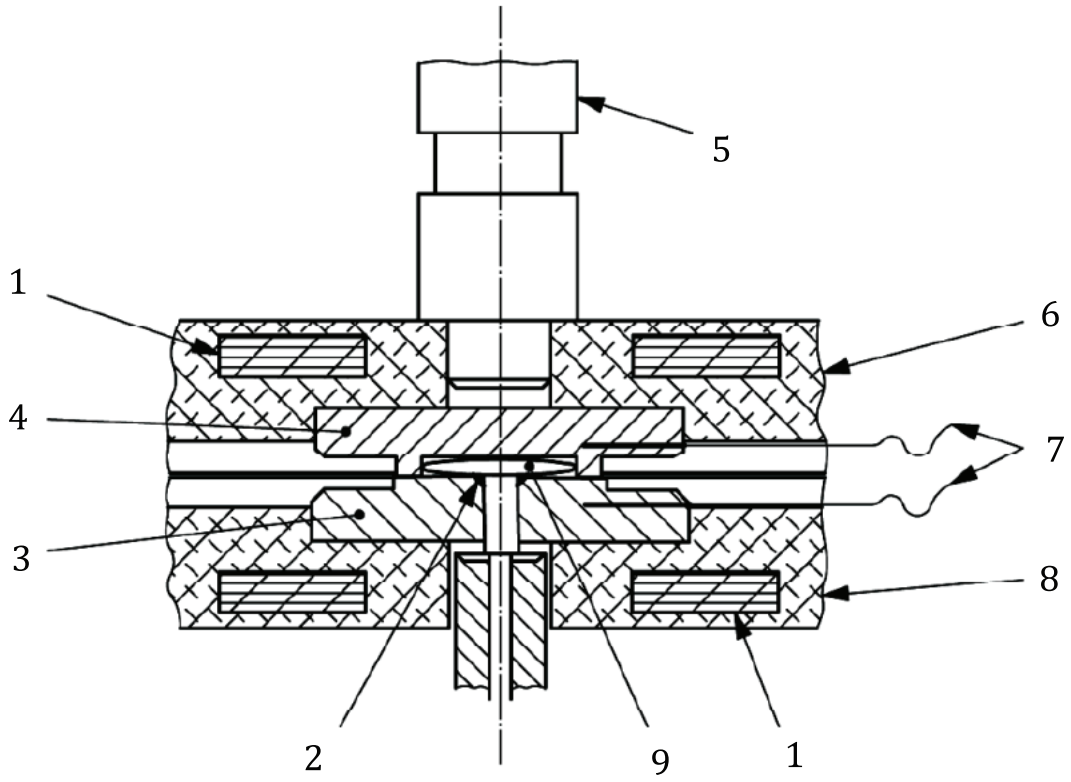
4.3 The parameters that can be measured from the recorded curve of torque as a function of time, i.e. $M = f(t)$, are presented in ISO 6502-1:2018, Clause 4.

5 Apparatus

5.1 General

The curemeter consists of a biconical disc in a temperature-controlled die cavity. The shaft of the disc is secured in a drive shaft and oscillated through a small rotary amplitude (see Figure 1).

The torque applied to the disc represents the resistance of the rubber test piece to deformation and is recorded autographically to yield a curve of torque versus time.



Key

1	heater	6	upper platen
2	seal	7	calibrated temperature sensors
3	lower die	8	lower platen
4	upper die	9	biconical disc
5	cylinder rod		

Figure 1 — Curemeter assembly

5.2 Dies

The dies shall be manufactured from a non-deforming tool steel having a minimum Rockwell hardness of 50 HRC or equivalent.

The geometry of the dies is shown in Figures 2 and 3. Suitable means shall be employed by the design of the dies or otherwise to apply pressure on the test piece throughout the test in order to minimize slippage between the disc and the rubber. Holes shall be drilled in both the upper and lower dies at the locations shown in Figures 2 and 3 to enable temperature sensors to be inserted. The surfaces of the die cavity shall contain rectangular-shaped grooves located at 20° intervals to minimize slippage. The lower die dimensions shall be as given in Figure 2. The upper die shall contain identically shaped grooves. The dimensions of the upper die shall be as given in Figure 3.

The form of the die cavity can be checked by measuring the dimensions of the vulcanized test piece after cutting.

The lower die shall have a hole in the centre to allow the insertion of disc stem. A suitable low constant friction seal shall be fitted in this hole to prevent material from leaking from the die cavity.

Dimensions in millimetres

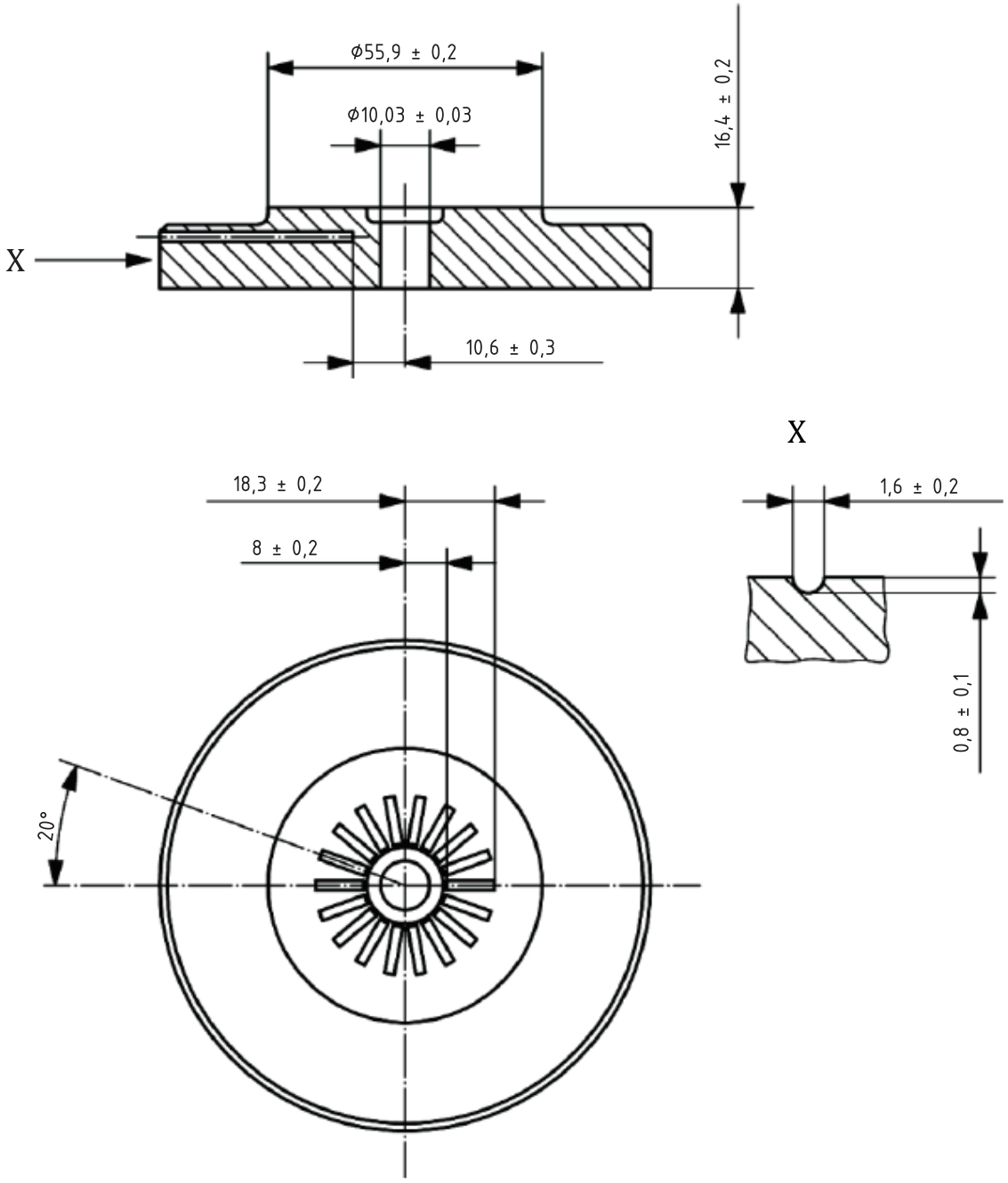


Figure 2 — Lower die

Dimensions in millimetres

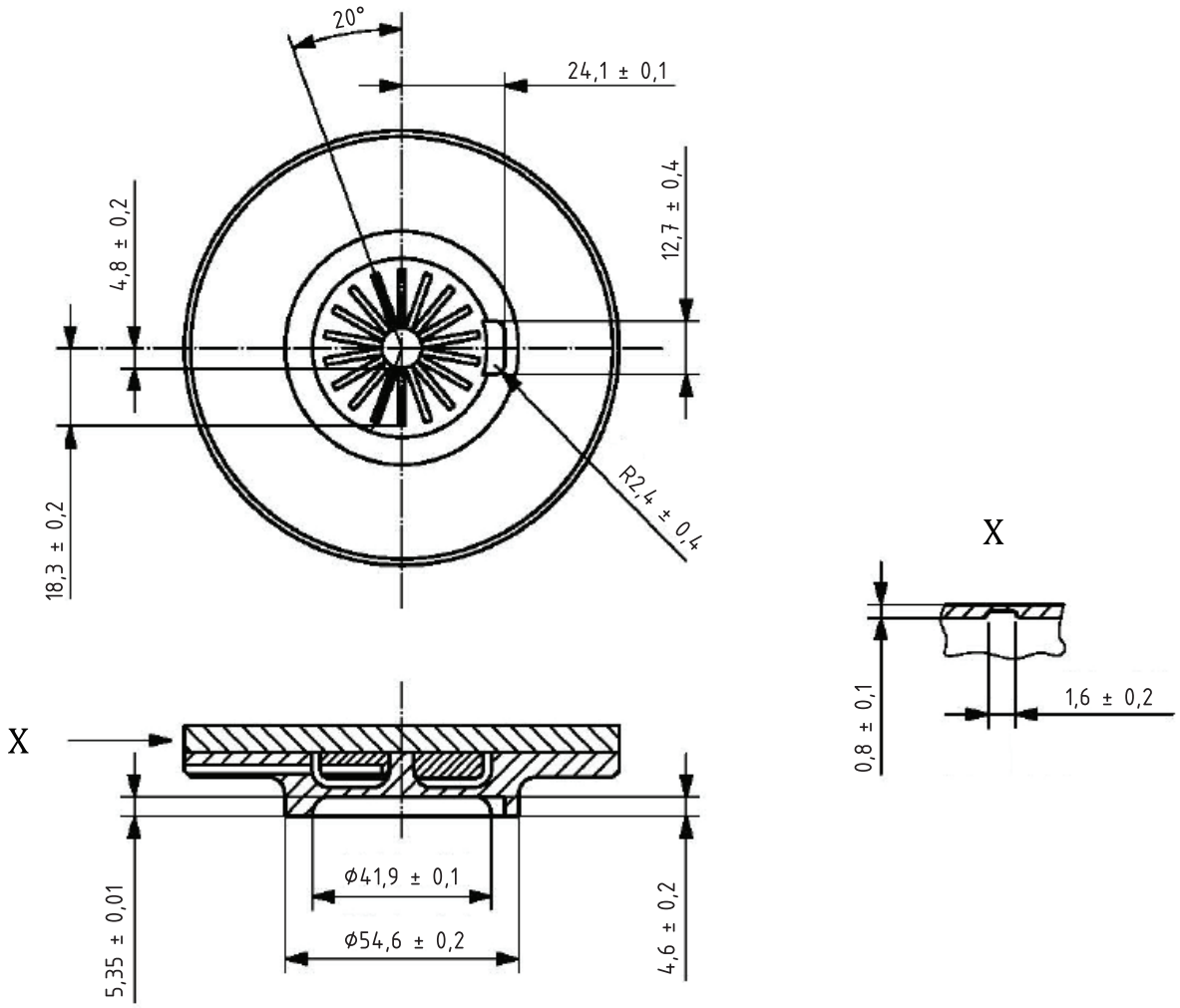


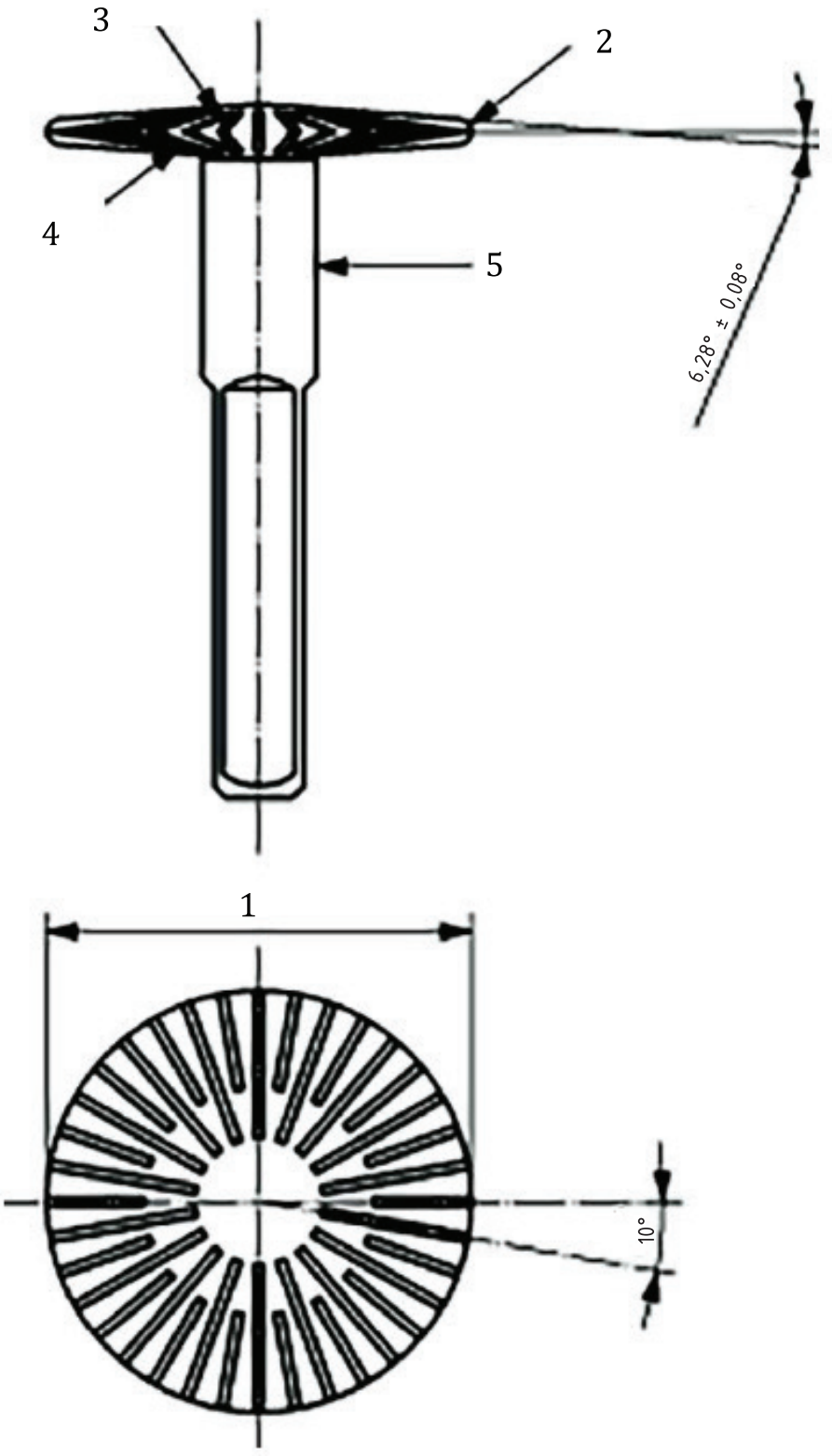
Figure 3 — Upper die

5.3 Die closure

The dies shall be closed, and held closed during the test, by a pneumatic cylinder exerting a force of 11,0 kN ± 0,5 kN.

5.4 Disc

The biconical disc shall be fabricated from a non-deforming tool steel having a minimum Rockwell hardness of 50 HRC or equivalent. The disc is shown in Figure 4, and the critical dimensions are given in Table 1.



Key

1 See Table 1.

Figure 4 — Biconical disc

Table 1 — Disc dimensions

Dimensions in millimetres

Reference	Dimension		Tolerance
1	Diameter	35,55	±0,01
2	Radius	0,80	±0,03
3[a]	Groove width	0,80	±0,05
	Groove depth	0,8	±0,1
	Groove lengths	7,5 minimum	
		12,5 minimum	
4	Groove width	0,80	±0,05
	Groove depth	0,8	±0,1
	Groove lengths	7,5 minimum	
		9,5 minimum	
5	Diameter	9,51	±0,01
	Length of the circular part of the disc shaft	20,0	±0,5
	Length of the square part of the disc shaft	35,0	±0,5

[a] The grooves on the top and bottom surfaces shall be staggered by 5°.

5.5 Disc oscillation

The frequency of the rotary oscillation of the disc shall be 1,7 Hz ± 0,1 Hz except for particular purposes when other frequencies in the range 0,05 Hz to 2 Hz may be used. The maximum angular displacement of the disc shall be 1,00° ± 0,02° about its central position (total amplitude 2°) when the die cavity is empty. If a torque is acting on the disc, the resulting decrease in the angle of oscillation with increasing torque shall be a linear function having a slope within the limits of 0,05°/N·m ± 0,002°/N·m.

Suitable devices shall be provided to verify both the initial amplitude of oscillation and the decrease in amplitude with applied torque.

Other amplitudes may be used when specified for particular purposes.

With different frequencies or amplitudes, different results will be obtained.

NOTE An initial amplitude of oscillation of 3° can be used in cases where danger of slippage between test piece and die cavity or disc can be excluded (first of all by regular cleaning of the rotor, see 10.2.3). A higher sensitivity in testing can be obtained at this amplitude, which is useful in production quality control.

5.6 Torque-measuring system

5.6.1 Measurement

A device which produces a signal that is directly proportional to the torque required to turn the disc shall be used to measure the torque acting on the disc.

5.6.2 Recording

A recorder shall be provided to continuously monitor the torque. It shall have a response time for full-scale deflection of 1 s or less. Automatic data acquisition and processing equipment is strongly recommended. The torque shall be recorded with an accuracy of ±1 % of the torque range. Three torque ranges from 0 N·m to 2,5 N·m, 0 N·m to 5 N·m and 0 N·m to 10 N·m shall be provided.

Although the procedure is written for a pen recorder with a paper feed, automatic data acquisition and processing equipment may also be used.

5.7 Heating and temperature control

5.7.1 The temperature-measuring systems shall enable the temperature of the dies to be measured to within ±0,1 °C over the range 100 °C to 200 °C. Calibrated thermocouples, or other suitable temperature sensors, inserted in the dies, shall be used for periodically checking the die temperatures.

5.7.2 The dies shall be mounted in electrically heated aluminium platens. Temperature controllers shall be used to control the temperature of each platen to within ±0,3 °C at steady state. After insertion of a test piece at 23 °C ± 5 °C, the temperature of the dies shall recover to within 0,3 °C of the test temperature within 3 min.

6 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in Annex A.

Provision shall be made for electronic verification of the recorder and torque transducer. One way of doing this is calibration by means of a resistor incorporated in the torque-measuring circuit and which simulates an applied torque of specified value.

The torque-measuring system shall be calibrated by means of masses or by a standard torque system such as a calibrated torsion spring.

NOTE In order to detect differences between curemeters or changes with use in a single curemeter, tests on reference compounds are useful. An adequate reference compound is a material with a shear modulus equal to or greater than the production compounds being tested, homogeneous and stable for several weeks. The procedure recommended is the following one:

— perform several tests on calibrated curemeter(s) in good condition and, from each curve, determine parameters such as M_H, M_L or t_c';

— define a confidence interval at a chosen statistical confidence level (95 % or 99 %) for each set of values obtained;

— small changes with use or small differences between curemeters do not require to be compensated for if the material parameters measured (M_H, M_L or t_c', for instance) are within the confidence intervals (in such a case, the differences observed are not statistically significant);

— determine the cause of large deviations (statistically significant variations observed when one of the parameters is no more within its confidence interval) and perform the necessary repairs.

7 Test piece

A test piece approximately 30 mm in diameter and 12,5 mm in thickness or of the equivalent volume shall be used for each test. Preferably, the test piece should be cut from a previously sheeted sample, which shall be as free from air as practical. A total volume of 8 cm^3 for the test piece is considered optimum.

NOTE Suitable test piece size is assured if a small amount of compound is allowed to extrude between the edges of the dies. Oversized test pieces cool the cavity excessively during the early part of the test cycle and invalidate the test.

8 Vulcanization

The vulcanization temperature is determined by the nature of the rubber compound or the application, but will normally be in the range 100 °C to 200 °C. The tolerances on the vulcanization temperature shall be ±0,3 °C.

9 Conditioning

The test piece shall be conditioned at 23 °C ± 5 °C, for a minimum of 3 h before testing. The test pieces cut from conditioned test samples may be tested immediately.

10 Procedure

10.1 Preparation for test

Bring the temperature of both dies (5.2) to the curing temperature, with the disc (5.4) in place and the die cavity in the closed position. With the disc in place and the dies closed, adjust the recorder pen to the zero-torque line on the chart. Position the pen at the zero-time position on the chart. Calibrate the recorder if needed (see Clause 6) and select the correct torque range (see 5.6.2).

10.2 Loading the curemeter

10.2.1 Open the dies, place the test piece on top of the disc, and close the dies within 5 s. When testing sticky compounds, insert some suitable thin-film material below the rotor and above the test piece to keep the compound from sticking to the dies.

NOTE Materials that have been found suitable include, for example, cellophane, polyester, nylon, high-density polyethylene (at 100 °C only), plain, uncoated fabric, and similar materials.

10.2.2 Time shall be counted from the instant the dies are closed. The disc may be oscillating (see 5.5) at zero time or started not later than 1 min after the dies are closed. The curve is complete when the recorded torque rises either to an equilibrium value or to a maximum value. If the torque continues to increase, vulcanization is considered to be complete after a given time.

10.2.3 A deposit of material from the rubber compounds under test can build up on the disc and dies. This might affect the final torque values. It is suggested that reference compounds be tested daily to detect this occurrence. If such contamination develops, it should be removed by very light blasting with a mild abrasive. Extreme care shall be used in this operation to retain sharpness of serrations and not change dimensions. Ultrasonic cleaning or cleaning with hot solvents or non-corrosive cleaning solutions can also remove the deposit. If solvent or solution cleaning is used, the first two sets of results after cleaning shall be rejected.

11 Expression of results

11.1 General

In accordance with ISO 6502-1, all or some of the cure characteristics indicated in 11.2 to 11.5 that are required for the purpose of the test shall be taken from the cure curve.

11.2 Torque values

M_L minimum torque, in N·m

M_{HF} plateau torque, in N·m

M_{HR} maximum torque (reverting curve), in N·m

M_H highest torque value attained, in N·m, in a curve where no plateau or maximum value is obtained after the specified time

11.3 Times to different percentages of full cure

Unless otherwise specified, it is recommended that the following specific parameters be used.

$t_c'(10)$ time, in minutes, for torque to reach $M_L + 0{,}1(M_H - M_L)$

$t_c'(50)$ time, in minutes, for torque to reach $M_L + 0{,}5(M_H - M_L)$

$t_c'(90)$ time, in minutes, for torque to reach $M_L + 0{,}9(M_H - M_L)$

11.4 Scorch time

The scorch time t_{sx} is the time required for the torque to increase by x units from M_L. A suitable value of x should be defined depending on the instrument used:

t_{s1} scorch time, in minutes, for torque to increase to 0,1 N·m above M_L.

If an amplitude of 3° is used instead of the standard 1°, t_{s2} shall be used in place of t_{s1}; i.e. the time, in minutes, for the torque to increase to 0,2 N·m above M_L.

11.5 Cure rate index

The cure rate index is the average slope of the rising curve and is given by $100 \Big/ \left[t_c'(y) - t_{sx} \right]$.

12 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 tested;

b) test method:

   1) a full reference to the test method used, i.e. the number of this document, ISO 6502-2;

   2) details of the curemeter used;

c) test details:

   1) the nominal amplitude of oscillation, reported as half the total displacement, i.e. 1° for a total displacement of 2°;

   2) the frequency of oscillation, in hertz, if other than 1,7 Hz (see 5.5);

   3) the vulcanization temperature, in degrees Celsius;

d) test results:

   1) the recorded vulcanization cure with torque and time scaled indicated, if required;

   2) the values of the parameters obtained from the vulcanization curve, as required;

e) the date of the test.

Annex A
(normative)

Calibration schedule

A.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded on any calibration report or certificate. It shall be reported whether calibration is made in the "as-received" condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

A.2 Schedule

Verification or calibration of the test apparatus is a normative part of this document. The frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory using ISO 18899 for guidance.

The calibration schedule given in Table A.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, part of that apparatus or an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure shall be carried out in accordance either with ISO 18899, another publication or a procedure particular to the test method which is detailed (whenever a more specific or detailed calibration procedure than that specified in ISO 18899 is available, it shall be used in preference).

The verification frequency for each parameter is given by a code letter.

The code letters used in the calibration schedule are:

— N Initial verification only;

— S Standard interval as given in ISO 18899.

Table A.1 — Calibration frequency schedule

Parameter	Requirement	Relevant subclause in ISO 18899:2013	Verification frequency guide	Notes
Surface hardness of the dies	≥50 HRC or equivalent	15.5	N	See ISO 6508-1
Dimensions of the dies	See 5.2	15.2	N	
Dimensions of the die grooves	See 5.2	15.2	N	
Die closure	11,0 kN ± 0,5 kN	21.3	S	
Surface hardness of the disc	≥50 HRC	15.5	N	
Dimensions of the disc	See 5.4	15.2	N	
Frequency of the rotary oscillation of the disc	1,7 Hz ± 0,1 Hz	23.3	S	
Angular displacement when the die cavity is empty	±1,00° ± 0,02°	15.9	S	
Slope of the linear function between the torque and the angle of oscillation	0,05°/N·m ± 0,002°/N·m	21.4 15.9	S	
Torque-measuring system	See 5.6	21.4	S	See the note in Clause 6
Temperature accuracy	±0,1 °C	18	S	
Temperature stability at steady-state	±0,3 °C	18	S	

Bibliography

[1] ISO 6508-1, *Metallic materials — Rockwell hardness test — Part 1: Test method*

(Continued from second cover)

In this adopted standard, reference appears to the following International Standard for which Indian Standard also exists. The corresponding Indian Standard which is to be substituted in its place is listed below along with its degree of equivalence for the edition indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 6502-1 Rubber — Measurement of vulcanization characteristics using curemeters — Part 1: Introduction	IS 16848 (Part 1/Sec 1) : 2024 ISO 6502-1 : 2018 Rubber — Methods of test: Part 1 Measurement of vulcanization characteristics using curemeters, Section 1 Introduction	Identical

The Committee has reviewed the provisions of the following International Standards referred in this adopted standard and has decided that it is acceptable for use in conjunction with this standard:

International Standard	*Title*
ISO 18899 : 2013	Rubber — Guide to the calibration of test equipment

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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Amendments Issued Since Publication

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