भारतीय मानक
Indian Standard

IS 16848 (Part 1/Sec 3) : 2024
ISO 6502-3 : 2023

रबड़ — परीक्षण पद्धतियाँ

भाग 1 क्यूरिमीटर का उपयोग करके वल्कनीकरण विशेषताओं का मापन

अनुभाग 3 रोटर रहित क्यूरिमीटर

Rubber — Methods of Test

Part 1 Measurement of Vulcanization Characteristics Using Curemeters

Section 3 Rotorless Curemeter

ICS 83.060

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

NATIONAL FOREWORD

This Indian Standard (Part 1/Sec 3) which is identical to ISO 6502-3 : 2023 'Rubber — Measurement of vulcanization characteristics using curemeters — Part 3: Rotorless 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 2 Oscillating disc curemeter

Futher, ISO 6502-3 : 2018 'Rubber — Measurement of vulcanization characteristics using curemeters — Part 3: Rotorless curemeter' has been technically revised at ISO and is now published as ISO 6502-3 : 2023 'Rubber — Measurement of vulcanization characteristics using curemeters — Part 3: Rotorless curemeter'.

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 given 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

(*Continued on third cover*)

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Indian Standard

RUBBER — METHODS OF TEST

PART 1 MEASUREMENT OF VULCANIZATION CHARACTERISTICS USING CUREMETERS

SECTION 3 ROTORLESS CUREMETER

WARNING 1 — Users of 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 applicability of any other restrictions.

WARNING 2 — Certain procedures specified in this document can 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 a rotorless curemeter. An introduction to the use of curemeters is given 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, *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 terminology databases for use in standardization at the following addresses:

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

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

4 Principle

A test piece of rubber is placed in a heated cavity formed by two dies, one of which is oscillated at a given frequency and amplitude. This action exerts a shear strain on the test piece and a shear torque which depends on the stiffness (shear modulus) of the rubber. The torque that increases as vulcanization proceeds is measured by a torque sensor incorporated in the other die member. The torque is recorded autographically as a function of time.

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.

The vulcanization characteristics are obtained from the recorded curve of torque as a function of time, in accordance with ISO 6502-1.

5 Apparatus

5.1 General

A rotorless curemeter consists of two dies that are heated and closed or almost closed, under a specified force, to form a test cavity that contains a test piece. One of the dies oscillates, and reaction torque on the stationary die, which is opposite to the moving die, can be measured at specified conditions of temperature, frequency and amplitude.

There are two types of assembly surrounding the test cavity formed by the upper and lower dies, one is unsealed or open type, and the other is sealed type. In the case of a sealed type die system, the die cavity is surrounded by seal plates and seal rings, and is completely closed. In the unsealed type the cavity is not completely closed.

Two kinds of cavity shape formed by the dies are used, namely, a biconical shape and a flat plate shape.

Three types of curemeter with different combination of sealing type and die cavity shape are available:

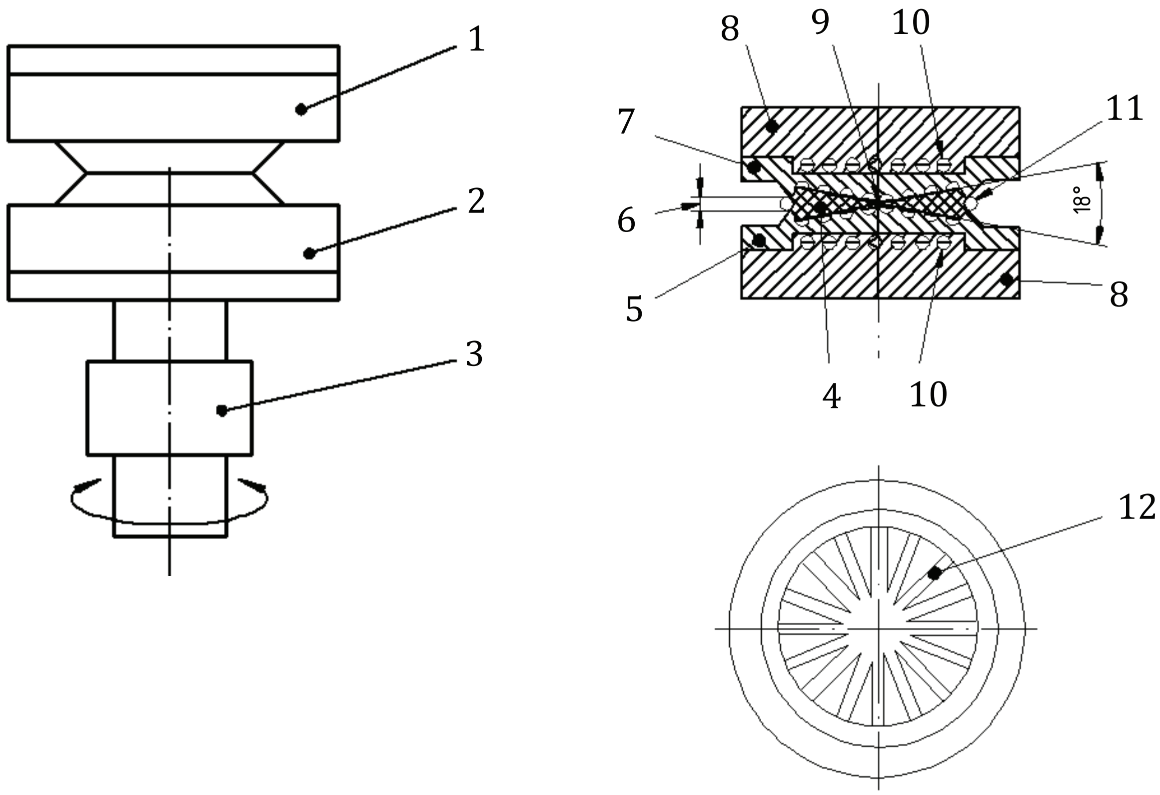
a) biconical die cavity with unsealed system;

b) biconical die cavity with sealed system; or

c) flat plate die cavity (with sealed system).

The general arrangements for rotorless curemeters are shown in Figure 1, Figure 2 and Figure 3, including typical machine dimensions.

Curemeters with different design can result in different torque responses and cure time (see ISO 6502-1:2018, A.2).

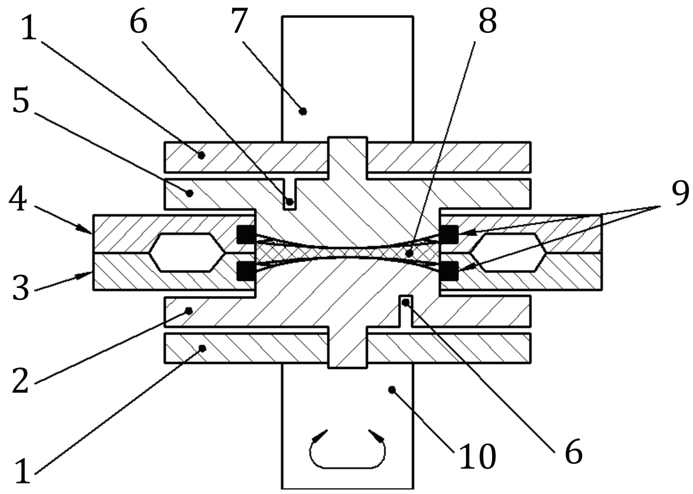


Key

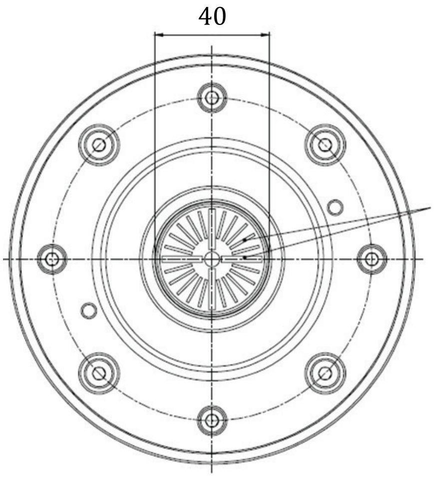
1	fixed die	7	upper die
2	oscillating die	8	temperature sensor
3	torque-measuring system	9	die gap
4	test piece	10	heater
5	lower die	11	spew
6	die gap	12	grooves

Figure 1 — Typical unsealed torsion-shear rotorless curemeter with biconical-die cavity

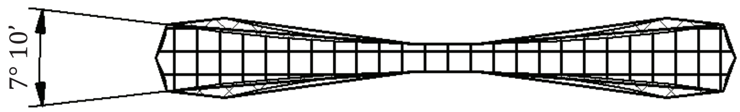
Dimensions in millimetres



a) Measurement principle



b) Die (upper and lower)



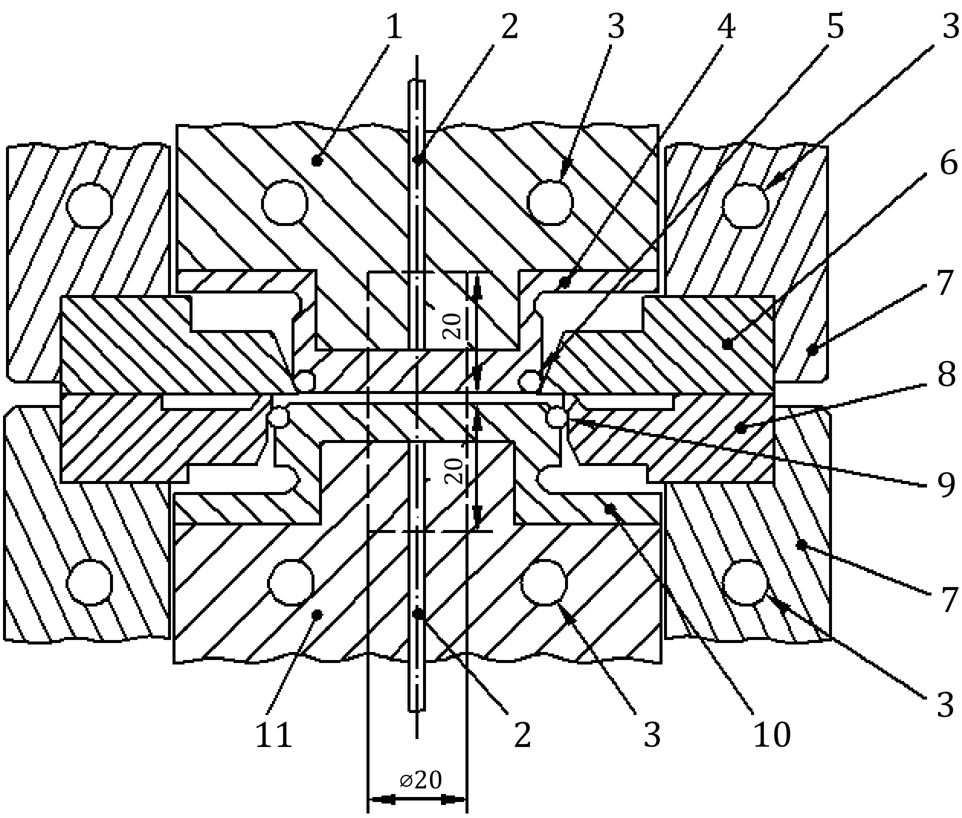
c) Test piece

Key

1 heater
2 lower die
3 lower seal plate
4 upper seal plate
5 upper die
6 temperature sensor
7 torque-measuring system
8 test piece
9 seals
10 oscillating-drive system
11 grooves

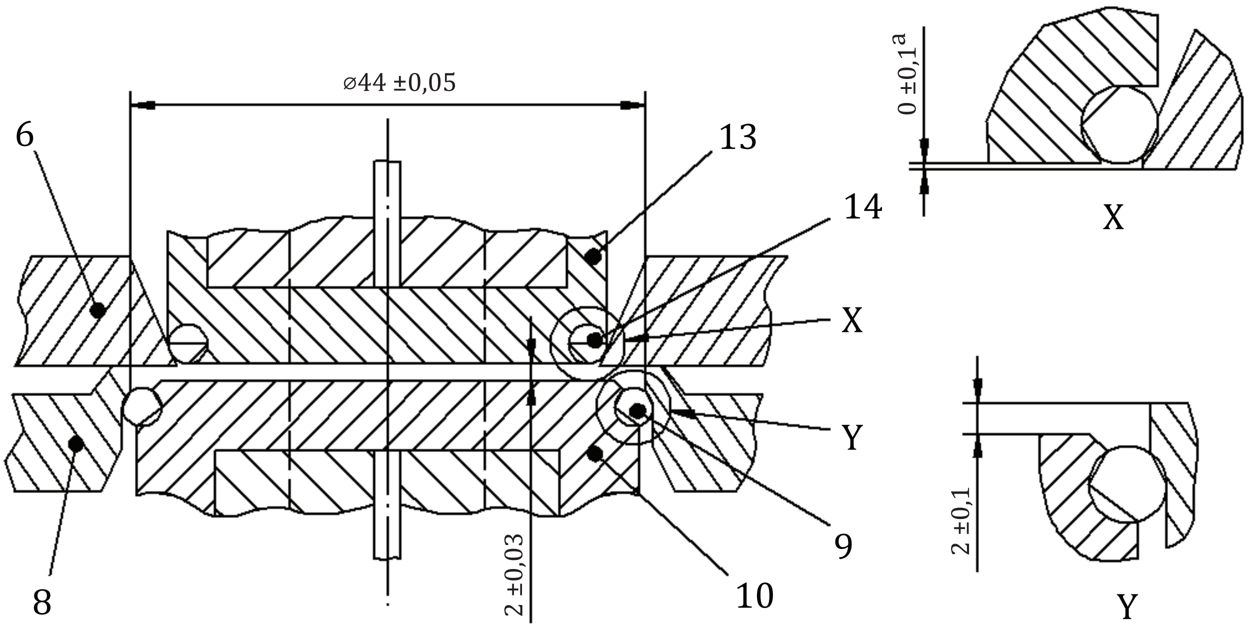
Figure 2 — Typical sealed torsion-shear rotorless curemeter with biconical-die cavity

Dimensions in millimetres



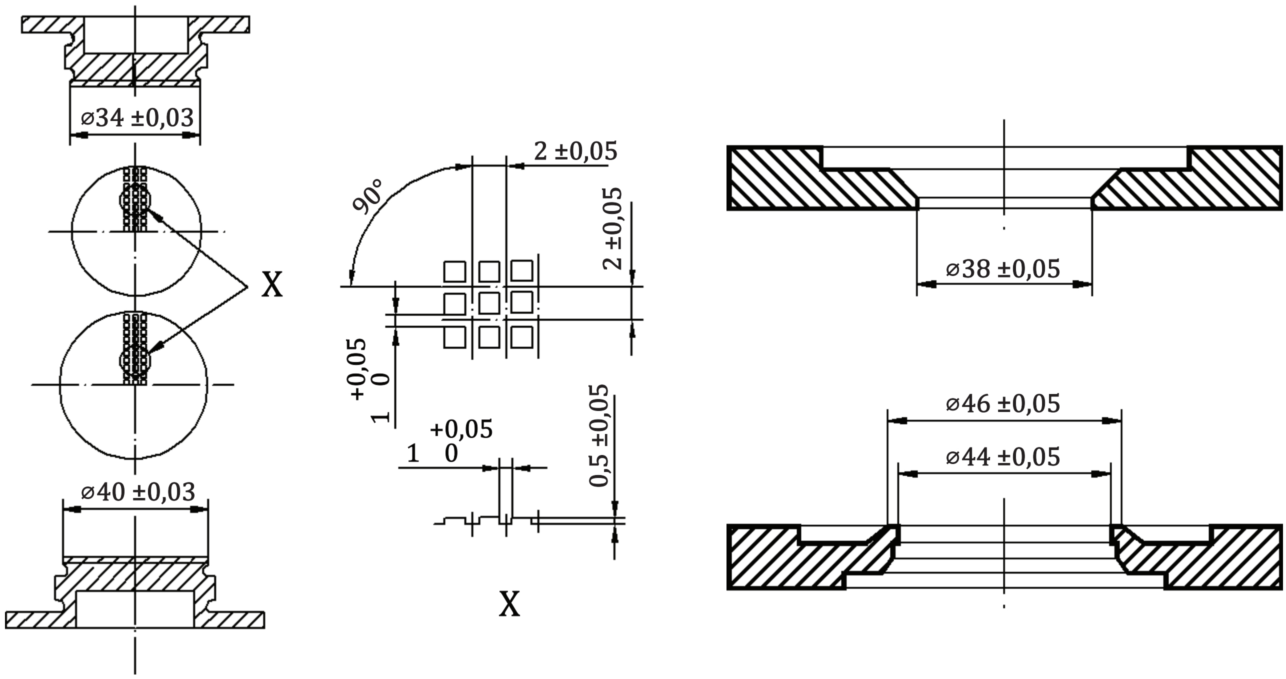
a) Measurement principle

Dimensions in millimetres



b) Details of dies and seal plates

Dimensions in millimetres



c) Shapes and dimensions of dies and seal plates

Key

1 shaft of torque-measuring system

2 temperature sensor

8 lower seal plate

9 lower seal

3 heater
4 upper die
5 upper seal
6 upper seal plate
7 platen
10 lower die
11 drive shaft
13 upper die
14 upper seal

a 0 ± 0,1 means that the difference between the bottom level of both parts (right and left) shall be within 0,1 mm.

Figure 3 — Typical sealed torsion-shear rotorless curemeter with flat-plate-die cavity

5.2 Dies

5.2.1 General

The dies shall be manufactured from a non-deforming, high stiffness and low thermal expansion material to minimize system compliance and prevent gap changes with temperature. The surface of the dies shall be treated to minimize the effect of test piece contamination if protective or carrying film are not used and it shall be hard enough to prevent wear. A minimum Rockwell hardness of 50 HRC, or equivalent, is recommended.

A die cavity is formed with a fixed lower die and a vertically-moving upper die, and the shape of the die cavity shall be biconical or flat-plate.

— A biconical die cavity is formed by two conically shaped dies with angle of separation ranging from 7° to 18°. The spacing between both dies facing each other differs by the distance on the periphery than the centre so as to be minimum at the central point, to increase toward the outer side from the central point.

— A flat plate die cavity is disc-shaped. It has same spacing all over the cavity.

Biconical dies are designed to provide a constant shear strain throughout the entire test piece, while flat plate dies give uniform temperature distribution in the test piece.

The top and bottom surfaces of the cavity shall have a pattern of grooves to prevent slippage of the rubber test piece. For the biconical type dies, radial grooves, for the flat plate type dies, checkered pattern grooves are recommended.

Suitable means shall be employed by design of dies or otherwise to apply pressure to the test piece.

The typical dimensions for the die cavity are shown in Table 1. The tolerances necessary on the dimensions of the dies depend on the particular design.

The dimensions of the die cavity can be checked by measuring the dimensions of the vulcanized test piece. For biconical type dies, particular attention should be paid to the thin central portion, the thickness of which depends on the die gap.

Table 1 — Typical dimensions of die cavity

	Biconical cavity	Flat-plate cavity
Diameter	40 mm ± 2 mm	44,00 mm ± 0,05 mm
Height	Angle of separation ranging from 7° to 18° In the centre of the dies, a separation equal to 0,5 mm plus the die gap	2,00 mm ± 0,03 mm

a The gap between the edges of the dies in the closed position.

b A level difference between the surfaces of the upper die and of the upper seal plate, or lower die and lower seal plate.

Table 1 *(continued)*

	Biconical cavity		Flat-plate cavity
Die gap[a]	Unsealed type	Sealed type	Level differences between die and seal plate[b] Upper: 0,0 mm ± 0,1 mm Lower: 2,0 mm ± 0,1 mm
	0,05 mm to 0,20 mm, preferably 0,1 mm	No gap	
Grooves	Radial grooves at 20° intervals	Radial grooves at 15° to 22,5° intervals	In a criss-cross pattern 16 grooves on the upper die 19 grooves on the lower die

[a] The gap between the edges of the dies in the closed position.

[b] A level difference between the surfaces of the upper die and of the upper seal plate, or lower die and lower seal plate.

5.2.2 Seal plate

For sealed type (biconical cavity or flat plate cavity) die systems, seal plates shall be provided around the upper and lower dies. The seal plates shall be fabricated from sufficiently rigid and abrasion resistant material. A minimum Rockwell hardness of 50 HRC, or equivalent, is recommended.

5.2.3 Seal

The sealed type (biconical cavity or flat plate cavity) curemeter shall have suitable low constant friction seals to prevent material leaking from the cavity. The material shall have adequate flexibility, high resistance to the test temperature and to wear. O-rings made from fluororubber, silicone rubber or tetrafluoroethylene resin are recommended.

The seals should be replaced periodically to prevent a lowering of the torque measuring accuracy or leakage of test piece by thermal degradation.

5.3 Die closure

The dies shall be held closed during the test by, for example, pneumatic cylinder.

The closing force shall be maintained at a minimum of 7 kN for dies of both biconical cavity and flat plate dies.

For sealed cavities, the contact of the die cavity edges shall be such as to form a perfectly sealed cavity.

NOTE For checking the sealing condition, place a piece of soft tissue paper not thicker than 0,04 mm between the mating surfaces and see if continuous pattern of uniform intensity is obtained when the dies are closed. A non-uniform pattern indicates incorrect adjustment of the die closure, worn or faulty mating surfaces, or distortion of the dies. Any of these conditions can result in leakage and erroneous results.

In unsealed rotorless instruments, the dies are not completely closed but a small clearance is left which shall be between 0,05 mm and 0,2 mm, preferably 0,1 mm.

5.4 Oscillation system

A torsional oscillating movement shall be applied to one of the dies (typically the lower one in the cavity).

The frequency of the rotary oscillation shall be 1,7 Hz ± 0,1 Hz except for particular purposes when other frequencies in the range of 0,05 Hz to 2,00 Hz may be used.

The range of oscillation amplitude shall be between ±0,1° and ±2,0°. For routine control purposes, ±0,5° or ±1,0° is preferable. The tolerance on amplitude shall be ±2 %, and the drive shall be sufficiently powerful and stiff to substantially maintain the amplitude under load.

NOTE Generally, greater sensitivity can be obtained with larger amplitudes but the amplitude that can be used in practice is restricted by the possibility of slippage between the test piece and the die surface.

When a torque is applied between the upper and lower dies, the resulting angular amplitude of oscillation decreases with increasing torque. For a flat plate type machine, the decrement of amplitude shall be a linear function having a slope of less than 0,01°/N·m.

5.5 Torque-measuring system

A suitable transducer capable of measuring the torque with an accuracy of ±1 % of the torque range shall be provided. This tolerance shall include any errors due to deformation of the measuring device and its coupling and of the output device.

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.

5.6 Protective films

One or more layers of protective film (approximately 0,025 mm thick) may be inserted between the test piece and the dies.

Film introduction can be useful to avoid frequent cavity cleaning, since material remaining in the die cavity can influence the test results.

The film shall not react with the test pieces. However, the presence of films can affect the test results, so if they are used, their specification (material, maximum temperature of use, thickness, stiffness) shall be reported.

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.

5.7 Heating and temperature control

Both dies are mounted on, or formed part of platens equipped with a heating device capable of maintaining the temperature of the dies to an accuracy of ±0,3 °C for the test duration.

The temperature-measuring system shall enable the temperature of the dies to be measured to a resolution of 0,1 °C over the range of 100 °C to 200 °C. Temperature sensors shall be provided in both the upper and lower dies and located for the best possible heat contact with the dies, i.e. heat gaps and other heat resistance shall be excluded.

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 for biconical-die rotorless instruments.

For flat-plate-die rotorless instruments, the recovery range shall be ±1 °C within 1,5 min at the test temperature of 150 °C.

6 Calibration

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

Where available, the procedures for calibration shall be in accordance with the manufacturer's instructions. The torque shall be determined at several points over the range(s) used but, additionally, it can be useful to have provision to make in-use checks.

Some practical examples of the calibration methods for torque and oscillation amplitude are described in Annex B.

Stable rubber reference compounds can also be tested periodically to check for consistent performance.

7 Test piece

Test pieces with appropriate thickness should be cut from a rubber sheet. The sheet shall be homogeneous and, as far as possible, free from trapped air.

The test piece should be circular with a diameter slightly smaller than the die cavity. The volume of test piece shall be slightly larger than the die cavity volume (typically 130 % to 150 % larger) so that a small amount of material is extruded between all edges of the dies when they are closed. Typical volume of test piece is between 3 cm^3 and 6 cm^3 for a biconical cavity, and approximately 5 cm^3 for a flat plate cavity. The pertinent volume shall be determined by preliminary tests, and test pieces of equal volume should be used to obtain reproducible results. The use of an appropriate device that ensures the production of test pieces of constant volume is recommended.

NOTE Undersized test pieces can cause low cavity pressure and low torque readings. Oversize test pieces can cool the cavity excessively during the early part of the test cycle.

8 Vulcanization temperature

The vulcanization temperature is chosen as that appropriate for the rubber compound being tested and intended processing. The normal range is 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

The temperature of both dies shall be brought to the test temperature with the cavity closed and allowed to stabilize. Bring the temperature of both dies to the curing temperature, with the cavity 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.

10.2 Loading the curemeter

The loading of the test piece and the closure of the dies shall be carried out as quickly as possible. The dies shall be closed immediately after insertion of the test piece. The whole cycle, from opening to closure, shall not exceed 20 s.

The vulcanization time shall be counted from the instant the dies are fully closed. Oscillation of the movable die shall be started before or at the instant of die closure.

After removal of the cured tested piece, a further test piece may be inserted immediately if the temperature of the dies remains within ±0,3 °C of the set value. If not, the dies shall be closed and the temperature be allowed to recover to the set value.

For low viscosity or sticky materials, a piece of protective film may be inserted between the test piece and the die surface for prevention of contamination and easy removal after the test. For biconical-die roterless curemeters of the sealed-cavity type, the use of protective film is recommended.

If film layers are not used, a deposit of material from the rubber compound can build up on the dies which can affect the final torque values. Reference compounds should be tested daily to detect this occurrence. If such contamination develops, it can be removed by very light blasting with a mild abrasive, ultrasonic cleaning or non-corrosive cleaning fluids. Great care should be taken with cleaning, and the manufacturer's advice should be followed. If fluids are used, the first two tests after cleaning shall be rejected. Running a natural rubber gum compound can be used to remove debris.

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 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. A typical example for a biconical cavity curemeter is 0,1 N·m at ±0,5° of oscillation amplitude, or in the case of a flat-plate cavity type, 0,04 N·m at ±1° of amplitude.

11.4 Time to a percentage of full cure

The time to a percentage of full cure from minimum torque, $t_c'(y)$ is the time taken for the torque to reach from M_L to $M_L + 0{,}01y(M_H - M_L)$.

— $t'_c(10)$ is a measure of the early stages of cure.

— $t'_c(50)$ can be determined accurately provided that the slope of the curve is greatest at this point.

11.5 Cure rate index

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

12 Precision

See Annex C.

13 Test report

The test report shall include the following information:

a) sample details:

1) full description of the sample and its origin;

   2) details of the compound tested.

b) test method:

   1) a reference to this document, i.e. ISO 6502-3;

   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;

   3) the vulcanization temperature, in degrees Celsius;

   4) details of any film layers used.

d) test results:

   1) the recorded vulcanization curve with torque and time scales indicated, if required;

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

e) date(s) 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 carried out 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/calibration of the test apparatus is a mandatory part of this document. However, 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, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a calibration procedure which is more specific or detailed than that 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 advised in ISO 18899:2013.

Table A.1 — Calibration frequency schedule

Parameter	Requirement	Subclause in ISO 18899: 2013	Verification frequency guide	Notes
Surface hardness of the dies	≥50 HRC or equivalent		N	See ISO 6508-1
Dimensions of the dies	±2 %	15.2	N	
Die grooves	See Figures 1, 2 and 3		N	
Die gap	See Table 1		S	
Die closing force	Biconical cavity: 7 kN Minimum Flat-plate cavity: 7 kN Minimum	21.3	S	
Tolerance on oscillation frequency	±0,1 Hz	23.3	S	

Table A.1 *(continued)*

Parameter	Requirement	Subclause in ISO 18899: 2013	Verification frequency guide	Notes
Tolerance on oscillation amplitude	±2 %		S	
Temperature resolution	±0,1 °C	18	N	
Temperature accuracy	±0,3 °C	18	S	
Temperature stability at steady-state	Within ±0,3 °C	18	S	
Torque transducer accuracy	±1 %	21.4	S	

In addition to the items listed in Table A.1, use of the following is implied, all of which shall be calibrated in accordance with ISO 18899:2013:

— instruments for determining the dimensions of the dies;

— a load cell for checking the die-closing force;

— a thermometer for monitoring the conditioning.

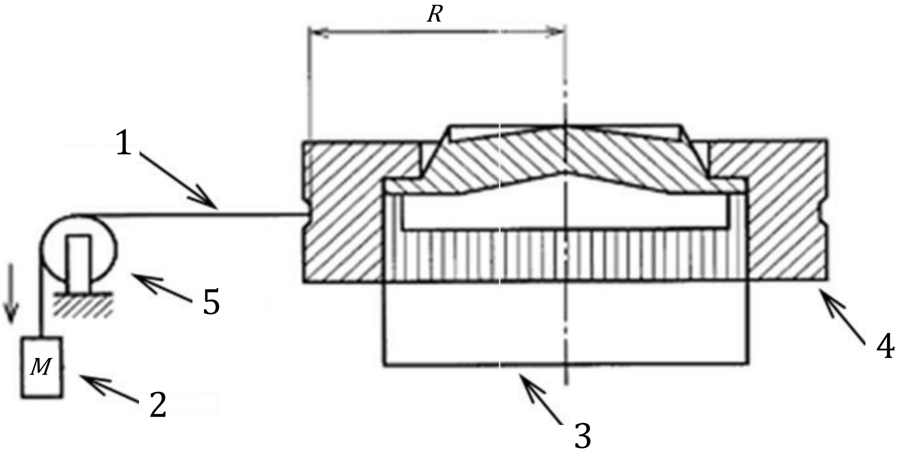
Annex B
(informative)

Practical examples of calibration for the various curemeters

B.1 Examples of calibration for torque

B.1.1 Wire-mass method

A calibration pulley is fitted instead of the upper die, a wire is fastened to the pulley and load is applied to the pulley by a weight attached to the other end of the wire. Figure B.1 provides an example of calibration for biconical die, and Figure B.2 provides an example for flat plate die.



Key

1 wire

2 weight

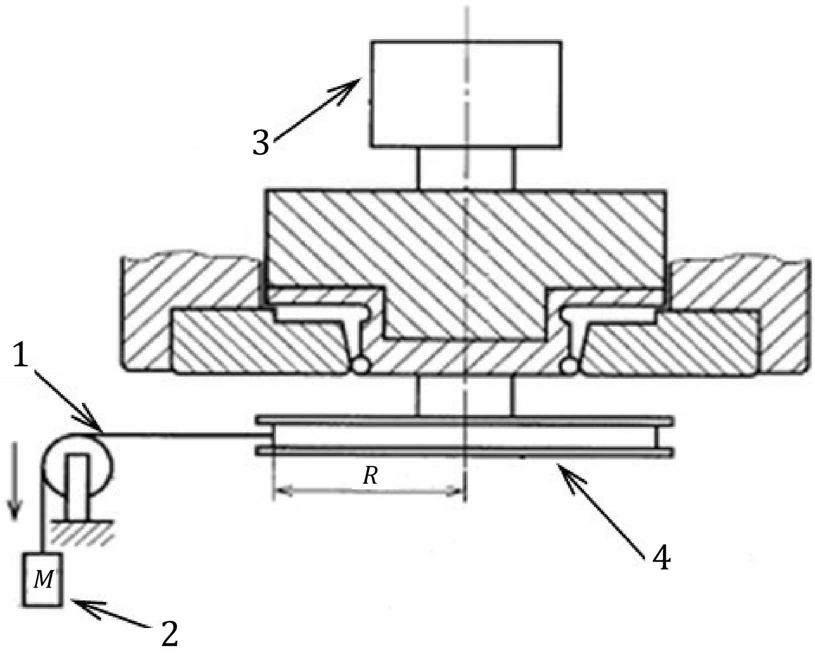
3 torque measuring system

4 calibration pulley

5 pulley

M mass of the weight

Figure B.1 — Biconical die



Key

1 wire

2 weight

3 torque measuring system

4 calibration pulley

R effective radius of the pulley

M mass of the weight

Figure B.2 — Flat plate die

The torque T can be calculated using the following equation:

$$T = R \times M \times g$$

where

R is effective radius of the pulley;

M is mass of the weight;

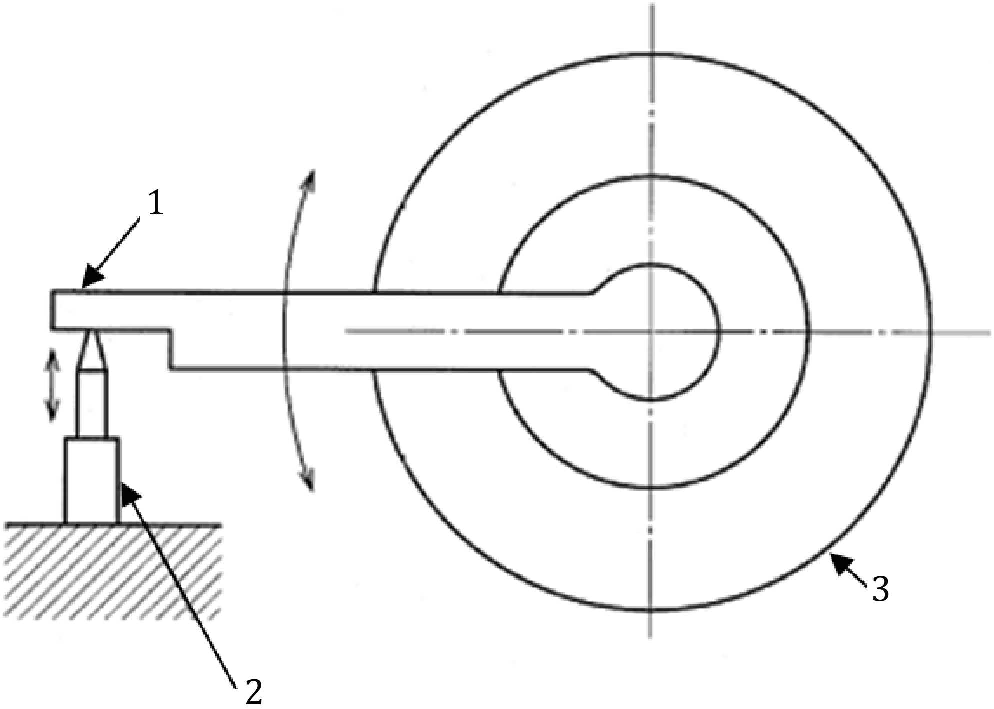
g is the gravitational acceleration.

B.1.2 Torsional spring rod method

A standard spring rod can be used to calibrate the torque measuring system. The standard spring rod is installed between the oscillating die and the torque measuring dies, and it checks the torque measurement at the selected angular displacement.

B.2 Oscillation angle

A calibration rod is fixed to the oscillation driving unit, and a displacement transducer is coupled by a knife-edge bearing in contact with the rod. The calibration is carried out without inserting a test piece. Figure B.3 provides an example of calibration for oscillation angles.



Key

1 calibration arm

2 displacement gauge

3 lower die

Figure B.3 — Example of calibration for oscillation angles

Annex C
(informative)

Precision

C.1 General

The precision calculations to provide repeatability and reproducibility values were performed in accordance with ISO/TR 9272, the guidance document for ISO/TC 45 test methods. See ISO/TR 9272 for precision concepts and nomenclature.

C.2 Programme details

An interlaboratory test programme (ITP) was organized in 2010. 27 laboratories from 10 nations participated.

Formulation of compounds tested (L, M, and H) is reported in Table C.1.

Table C.1 — Formulation of compounds

Material	**Compound**		
	L	M	H
SBR 1500	100,00	100,00	100,00
N234 Black	30,00	40,00	50,00
Stearic Acid	1,00	1,00	1,00
Zinc Oxide	3,00	3,00	3,00
TBBS	2,20	2,50	2,80
Sulfur	1,50	1,50	1,50
Total	137,70	148,00	158,30

ITP results were divided into 2 groups. Group No. 1 was for the result of biconical type curemeters, 22 laboratories from 10 nations participated. Group No. 2 was for flat-plate type curemeters, 5 laboratories from 1 nation participated.

The calculation of the precision figures was carried according to ISO/TR 9272 and, in line with definitions this ITP is classified as “Level 1” (3 materials, 2 repetition in 2 different days; the chosen option for outliers' treatment was Option 1, i.e. deletion of results identified as outlier at 5 % and 2 % significance level).

Since the precision was determined directly on the target material with a limited preparation before the analysis, this ITP generated a “Type 1 precision”.

C.3 Precision results

The precision results for the biconical curemeters are given in Table C.2, those for the flat-plate curemeters in Table C.3.

For the results of biconical curemeters, after outlier’s deletion, the total number of labs represents a significant population for a pertinent statistical data analysis, while for the results of flat-plate curemeters, after the outlier’s deletion, numbers of labs are under minimum statistical significance level, so the data suggested for indication only.

The symbols used in Table C.2 and Table C.3 are defined as follows:

— s_r: within-laboratory standard deviation (in measurements units);

— r: repeatability (in measurements units);

— (r): repeatability (in per cent of mean level);

— s_R: between-laboratory standard deviation (for total between-laboratory variation, in measurement units);

— R: reproducibility (in measurements units);

— (R): reproducibility (in per cent of mean level).

Table C.2 — Precision data determined for biconical curemeters

		Intralaboratory			Interlaboratory			Number of laboratories[a]
	Average	s_r	r	(r)	s_R	R	(R)	
				$t'_c(10)$				
L	4,83 min	0,195	0,55	11,4	0,355	1,01	20,8	18
M	4,7 min	0,146	0,41	8,8	0,256	0,72	15,4	17
H	4,74 min	0,086	0,24	5,1	0,268	0,76	16	16
				$t'_c(50)$				
L	7,14 min	0,36	1,02	14,3	0,488	1,38	19,3	20
M	6,98 min	0,28	0,79	11,3	0,368	1,04	14,9	19
H	7,05 min	0,246	0,7	9,9	0,451	1,28	18,1	20
				$t'_c(90)$				
L	10,89 min	0,434	1,23	11,3	0,68	1,92	17,7	21
M	10,41 min	0,373	1,06	10,2	0,482	1,36	13,1	20
H	10,47 min	0,371	1,05	10	0,634	1,8	17,1	21
				M_L				
L	1,61 dNm	0,011	0,03	2	0,059	0,17	10,3	18
M	2,25 dNm	0,023	0,07	2,9	0,054	0,15	6,8	17
H	3,27 dNm	0,026	0,07	2,3	0,074	0,21	6,4	16
				M_H				
L	16,17 dNm	0,215	0,61	3,8	0,534	1,51	9,3	21
M	21 dNm	0,2	0,57	2,7	0,74	2,09	10	20
H	25,5 dNm	0,255	0,72	2,8	1,099	3,11	12,2	21

Oscillation frequency: 1,666 Hz

Temperature: 160 °C ± 0,3 °C

Oscillation angle: 0,5°

[a] The final number of laboratories in the revised database after deletion of outliers (option 1 of ISO/TR 9272:2005).

Table C.3 — Precision data determined for flat die curemeters

		Intralaboratory			Interlaboratory			Number of laboratories[a]
	Average	s_r	r	(r)	s_R	R	(R)	
				$t'_c(10)$				
L	5,3 min	0,125	0,35	6,7	1,293	3,66	69	4
M	5,39 min	0,262	0,74	13,8	0,202	0,57	10,6	3
H	5,28 min	0,122	0,34	6,5	1,023	2,9	54,9	4
				$t'_c(50)$				
L	7,37 min	0,181	0,51	7	1,715	4,85	65,8	4
M	7,18 min	0,258	0,73	10,2	1,587	4,49	62,5	4
H	7,29 min	0,17	0,48	6,6	1,421	4,02	55,1	4
				$t'_c(90)$				
L	9,96 min	0,143	0,4	4,1	1,472	4,17	41,8	4
M	9,64 min	0,297	0,84	8,7	0,212	0,6	6,2	3
H	9,4 min	0,158	0,45	4,8	0,114	0,32	3,4	2
				M_L				
L	1,92 dNm	0,022	0,06	3,3	0,055	0,16	8,1	4
M	2,56 dNm	0,019	0,05	2,1	0,055	0,15	6	4
H	3,63 dNm	0,035	0,1	2,7	0,093	0,26	7,3	4
				M_H				
L	14,44 dNm	0,214	0,61	4,2	0,674	1,91	13,2	5
M	18,46 dNm	0,227	0,64	3,5	0,648	1,83	9,9	5
H	22,23 dNm	0,291	0,82	3,7	0,517	1,46	6,6	5

Oscillation frequency: 1,666 Hz

Temperature: 160 °C ± 0,3 °C

Oscillation angle: 0,5°

[a] The final number of laboratories in the revised database after deletion of outliers (option 1 of ISO/TR 9272:2005).

Bibliography

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

[2] ISO/TR 9272:2005,[1] *Rubber and rubber products — Determination of precision for test method standards*

1) Cancelled and replaced by ISO 19983.

(*Continued from second cover*)

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*)'.

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