भारतीय मानक
Indian Standard

IS 3400 (Part 2/Sec 4) : 2022
ISO 48-4 : 2018

[*Superseding IS 3400* (*Part 23/Sec 1*) *: 2018*]

रबड़, वल्कनीकृत या थर्मोप्लास्टिक के परीक्षण की पद्धतियाँ

भाग 2 कठोरता ज्ञात करना

अनुभाग 4 डयूरोमीरटर विधि (आधार कठोरता) वारा इंडेंटेशन हार्डनेस

(दूसरा पुनरीक्षण)

Methods of Test for Rubber, Vulcanized or Thermoplastic

Part 2 Determination of Hardness

Section 4 Indentation Hardness by Durometer Method (Shore Hardness)

(Second Revision)

ICS 83.060

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

NATIONAL FOREWORD

This Draft Indian Standard (Part 2/Sec 4) (Second Revision) which is identical with ISO 48-4 : 2018 'Rubber, vulcanized or thermoplastic — Determination of hardness — Part 4: Indentation hardness by durometer method (Shore hardness)' 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.

The standard, was published in 2002 as IS 3400 (Part 23) under dual numbering with ISO 7619 : 1997. Since ISO 7619 has been revised in 2010 and split in to two parts, the Sectional Committee decided to align IS 3400 (Part 23) with the latest International Standards available on the subject. As this Indian Standard is already part of series of standards, it was further decided to publish the two different parts of ISO 7016 as two sections of standard namely IS 3400 (Part 23/Sec 1) and IS 3400 (Part 23/Sec 2).

Earlier, three Indian Standards, namely IS 3400 (Part 2) : 2014/ISO 48 : 2010 'Methods of test for vulcanized rubber — Part 2: Rubber vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD) (Fourth Revision)', IS 3400 (Part 23/Sec 1) : 2018/ISO 7619-1 : 2010 'Methods of test for vulcanized rubbers: Part 23 Rubber — Determination of indentation hardness by means of pocket hardness meters, Sec 1 Durometer method (Shore hardness) (First Revision)' and IS 3400 (Part 23/Sec 2) : 2018/ISO 7619-2 : 2010 'Methods of test for vulcanized rubbers — Part 23 Rubber — Determination of indentation hardness by means of pocket hardness meters, Sec 2 IRHD pocket meter method (First Revision)' were published for the determination of hardness under dual numbering system.

ISO has now published nine parts of ISO 48 Standard for the determination of hardness, which cancels and replaces the following ISO Standards:

ISO 48-1	ISO 48-6	ISO 48	ISO 7267-2
ISO 48-2	ISO 48-7	ISO 27588	ISO 7267-3
ISO 48-3	ISO 48-8	ISO 7619-1	ISO 18898
ISO 48-4	ISO 48-9	ISO 7619-2	
ISO 48-5	ISO 18517	ISO 7267-1	

Therefore, the Committee had decided to adopt all parts of ISO 48 available on the subjects as nine different sections of IS 3400 (Part 2), under dual numbering system.

Second revision of this standard is being carried out to align it with the latest published International Standard ISO 48-4 : 2018. This revision of standard will supersede IS 3400 (Part 23/Sec 1) : 2018 Methods of Test for Vulcanized Rubbers — Part 23 Rubber — Determination of Indentation Hardness by Means of Pocket Hardness Meters, Section 1 Durometer method (Shore hardness) *(first revision)*.

The Committee also decided to modify the common title of all the Indian Standards under IS 3400 series as 'Methods of test for rubber, vulcanized or thermoplastics' for the uniformity in the title in line with the ISO Standards.

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Introduction

ISO/TC 45/SC 2 established a principle that it would be helpful for users if standards on the same subject but covering different aspects or methods were grouped together, preferably with an introductory guidance standard, rather than being scattered throughout the numbering system. This has been achieved for some subjects, for example curemeters (ISO 6502) and dynamic properties (ISO 4664).

In 2017, it was decided to group standards for hardness and, subsequently, it was agreed that they would be grouped under the ISO 48 number. The new standards together with the previously numbered standards are listed below.

— ISO 48-1: former ISO 18517

— ISO 48-2: former ISO 48

— ISO 48-3: former ISO 27588

— ISO 48-4: former ISO 7619-1

— ISO 48-5: former ISO 7619-2

— ISO 48-6: former ISO 7267-1

— ISO 48-7: former ISO 7267-2

— ISO 48-8: former ISO 7267-3

— ISO 48-9: former ISO 18898

The hardness of rubber, as measured by a durometer (Shore hardness) or an IRHD pocket meter, is determined from the response of the rubber to an applied indentation. The response is complex and will depend on:

a) the elastic modulus of the rubber;

b) the viscoelastic properties of the rubber;

c) the thickness of the test piece;

d) the geometry of the indentor;

e) the pressure exerted;

f) the rate of increase of pressure;

g) the interval after which the hardness is recorded.

Because of these factors, it is inadvisable to relate results using a durometer (Shore hardness) directly to IRHD values, although correlations have been established for some individual rubbers and compounds.

Durometers were originally portable hand-held instruments that have proved to be particularly convenient for making measurements on products. By now a lot of laboratories also use them on a stand with a weight applied to the pressure foot in order to improve precision significantly.

NOTE ISO 48-2 specifies hardness measurements for determination of hardness between 10 IRHD and 100 IRHD. Further information on the relationship between the durometer values and IRHD values is given in References [5][6][7].

Indian Standard

METHODS OF TEST FOR RUBBER, VULCANIZED OR THERMOPLASTIC

PART 2 DETERMINATION OF HARDNESS

Section 4 Indentation hardness by durometer method (Shore hardness)

(Second Revision)

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 the indentation hardness (Shore hardness) of vulcanized or thermoplastic rubber using durometers with the following scales:

— the A scale for rubbers in the normal-hardness range;

— the D scale for rubbers in the high-hardness range;

— the AO scale for rubbers in the low-hardness range and for cellular rubbers;

— the AM scale for thin rubber test pieces in the normal-hardness range.

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 48-9, *Rubber, vulcanized or thermoplastic — Determination of hardness — Part 9: Calibration and verification of hardness testers*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

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 https://www.electropedia.org/

4 Principle

An indentor of specified dimensions is pressed into a test piece under a specified load and the depth of indentation measured. This indentation is converted to a hardness value by means of a specified relation.

5 Choice of durometer

When using durometers, the scale should be chosen as follows:

— for values less than 20 with a type D durometer: type A;

— for values less than 20 with a type A durometer: type AO;

— for values over 90 with a type A durometer: type D;

— for thin test pieces (less than 6 mm thick): type AM.

6 Apparatus

6.1 Durometer types A, D and AO

These durometers consist of the components specified in 6.1.1 to 6.1.5.

6.1.1 Pressure foot

The pressure foot for types A and D shall have a diameter of 18 mm ± 0,5 mm and a central hole of diameter 3 mm ± 0,1 mm. For type AO, the pressure foot shall have a minimum area of 500 mm^2 with a central hole of diameter 5,4 mm ± 0,2 mm. The tolerances on the dimension of the central hole and the requirement for the size of the pressure foot only apply to instruments used on a stand.

6.1.2 Indentor

The indentor shall be formed from a hardened-steel rod of diameter 1,25 mm ± 0,15 mm to the shape and dimensions shown in Figure 1 for type A durometers and Figure 2 for type D durometers. Type AO durometers shall have a round indentor with a radius of 2,5 mm ± 0,02 mm in accordance with Figure 3.

6.1.3 Indicating device

This is a device for allowing the extent of protrusion of the point of the indentor beyond the face of the pressure foot to be read. It shall be calibrated directly in terms of units ranging from 0 for the maximum protrusion of 2,50 mm ± 0,02 mm to 100 for zero protrusion obtained by placing the pressure foot and indentor in firm contact with a suitable flat, hard surface (e.g. glass).

6.1.4 Calibrated spring

This is used to apply a force, F, expressed in millinewtons, to the indentor in accordance with one of the following formulae:

— For type A durometers:

$$F = 550 + 75H_A$$

where H_A is the hardness reading taken from the type A durometer.

— For type D durometers:

$F = 445H_D$

where H_D is the hardness reading taken from the type D durometer.

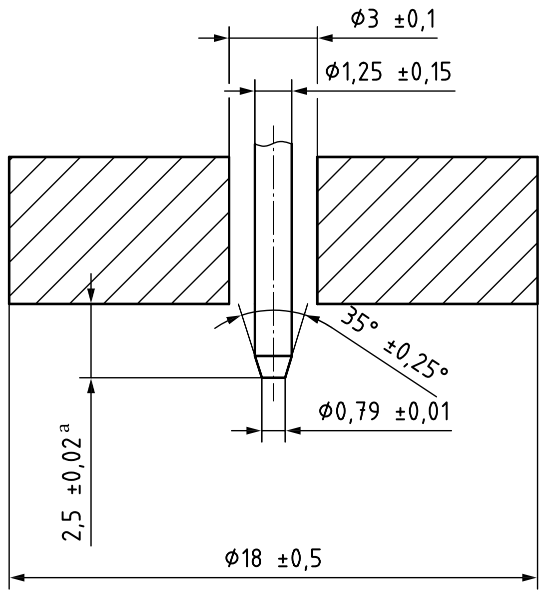
— For type AO durometers:

$F = 550 + 75H_{AO}$

where H_{AO} is the hardness reading taken from the type AO durometer.

NOTE The rubber industry uses the term equation for the relationships herein termed formula. The term formula is used to describe the table of ingredients in a rubber compound.

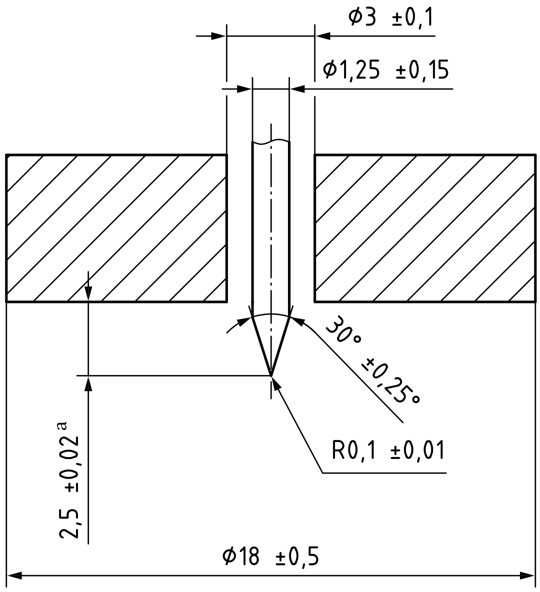
Dimension in millimetres



a The protrusion shown is valid for a reading of 0.

Figure 1 — Indentor for type A durometer

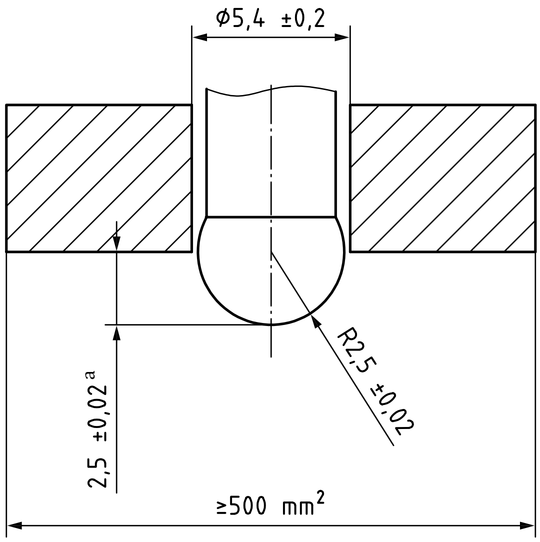
Dimension in millimetres



a The protrusion shown is valid for a reading of 0.

Figure 2 — Indentor for type D durometer

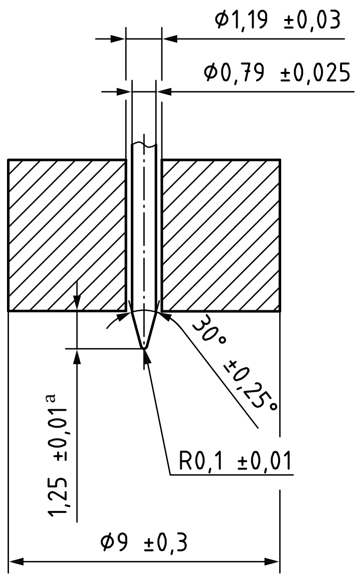
Dimension in millimetres



a The protrusion shown is valid for a reading of 0.

Figure 3 — Indentor for type AO durometer

Dimension in millimetres



a The protrusion shown is valid for a reading of 0.

Figure 4 — Indentor for type AM durometer

6.1.5 Automatic timing device (optional)

The timer shall be automatically activated when the pressure foot is in contact with the test piece and shall indicate the end of the test time or lock the test value at its completion. Use of a timing device for the test time improves precision. When the instrument is used on a stand, the time tolerance shall be ±0,3 s.

6.2 Durometer type AM

This durometer consists of the components specified in 6.2.1 to 6.2.5.

6.2.1 Pressure foot

The pressure foot shall have a diameter of 9 mm ± 0,3 mm and a central hole of diameter 1,19 mm ± 0,03 mm.

6.2.2 Indentor

The indentor shall be formed from a hardened-steel rod of diameter 0,79 mm ± 0,025 mm to the shape and dimensions shown in Figure 4.

6.2.3 Indicating device

This is a device for allowing the extent of protrusion of the point of the indentor beyond the face of the pressure foot to be read. It shall be calibrated directly in terms of units ranging from 0 for the maximum protrusion of 1,25 mm ± 0,01 mm to 100 for zero protrusion obtained by placing the pressure foot and indentor in firm contact with a suitable flat, hard surface (e.g. glass).

6.2.4 Calibrated spring

This is used to apply a force, *F*, expressed in millinewtons, to the indentor in accordance with the formula:

$$F = 324 + 4{,}4H_{AM}$$

where H_{AM} is the hardness reading taken from the type AM durometer.

NOTE The rubber industry uses the term equation for the relationships herein termed formula. The term formula is used to describe the table of ingredients in a rubber compound.

6.2.5 Automatic timing device (optional)

The timer, if used, shall be automatically activated when the pressure foot is in contact with the test piece and shall indicate the end of the test time or lock the test value at its completion. Use of a timing device for the test time improves precision. The time tolerance shall be ±0,3 s.

6.3 Stand

6.3.1 The original concept of a durometer was a portable instrument that could be used, for example, on a product in service. However, better precision can be expected by using a stand with a weight centred on the axis of the indentor to apply the pressure foot to the test piece. Durometer types A, D and AO may be used either as hand-held instruments or mounted on a stand. Type AM durometers shall always be mounted on a stand. Clearly, when a stand is used, the portability is lost.

NOTE As a general trend, it can be expected that the precision will follow the order:

Hand held durometer < Hand held durometer equipped with force-calibrated hand-grip < Durometer using a stand < Durometer equipped with a timer and using a stand.

Precision is affected by a number of factors, including parallax error, time of load application, speed of applying the load to the foot and orientation of the test piece. A detailed study of the reproducibility of rubber hardness tests can be found in Reference [8].

6.3.2 The operating stand shall be capable of supporting the pressure-foot surface of the durometer parallel to the test piece support table.

6.3.3 The stand shall be capable of applying the test piece to the indentor, or vice versa, without shock.

NOTE A maximum speed of 3,2 mm/s has been found to be suitable.

6.3.4 The total mass of the durometer and extra mass to overcome the spring force shall be:

— $1^{+0,1}_{0}$ kg for types A and AO;

— $5^{+0,5}_{0}$ kg for type D;

— $0,25^{+0,05}_{0}$ kg for type AM.

6.4 Durometer spring force calibration

The force values shall be in accordance with Table 1.

Table 1 — Durometer spring forces

Indicated durometer value	Spring force mN		
	Type AM	Type A and AO	Type D
0	324	550	—
10	368	1 300	4 450
20	412	2 050	8 900
30	456	2 800	13 350
40	500	3 550	17 800
50	544	4 300	22 250
60	588	5 050	26 700
70	632	5 800	31 150
80	676	6 550	35 600
90	720	7 300	40 050
100	764	8 050	44 500
Millinewtons (mN) **per unit**	4,4	75	445
Spring force tolerance	±8,8	±37,5	±222,5

7 Test pieces

7.1 General

Test pieces shall be prepared in accordance with ISO 23529.

7.2 Thickness

For the determination of hardness using type A, D and AO durometers, the thickness of the test piece shall be at least 6 mm.

For the determination of hardness using type AM durometers, the thickness of the test piece shall be at least 1,5 mm.

For sheets thinner than 6 mm and 1,5 mm (see above), the test piece may be composed of not more than three layers, in order to obtain the necessary thickness. However, determinations made on such test pieces might not agree with those made on single-layer test pieces.

For comparative-test purposes, the test pieces shall be similar.

NOTE Measurements made on thin test pieces of soft rubber will be influenced by the support table and will give a result which is too high.

7.3 Surface

The other dimensions of the test piece shall be sufficient to permit measurements at least 12 mm away from any edge for types A and D, and 15 mm and 4,5 mm away from any edge for type AO and type AM, respectively.

The surface of the test piece shall be flat and parallel over an area sufficient to permit the pressure foot to come into contact with the test piece over an area having a radius of at least 6 mm from the indentor point for types A and D, 9 mm for type AO and 2,5 mm for type AM.

Satisfactory hardness determinations cannot be made on rounded, uneven or rough surfaces using durometers. However, their use in certain specialized applications is recognized, e.g. ISO 48-7 for the determination of the hardness of rubber-covered rolls. In such applications, the limitations to their use shall be clearly identified.

8 Conditioning and test temperature

For all test purposes, the minimum time between vulcanization and testing shall be 16 h. Where practical, test pieces shall be conditioned immediately before testing for a minimum period of 1 h at one of the standard laboratory temperatures specified in ISO 23529. The same temperature shall be used throughout any single test or series of tests intended to be comparable.

9 Procedure

9.1 General

Place the test piece on a flat, hard, rigid surface (e.g. glass). Apply the pressure foot to the test piece, or vice versa, as rapidly as possible but without shock, keeping the foot parallel to the surface of the test piece and ensuring that the indentor is normal to the rubber surface.

9.2 Test time

Apply a force in accordance with 6.3.3 and 7.3 sufficient only to obtain firm contact between the pressure foot and the test piece and take the reading at the specified time after the pressure foot is in firm contact with the test piece. The standard test time is 3 s for vulcanized rubber and 15 s for thermoplastic rubber. Other test times may be used, provided they are stated in the test report. Rubbers of unknown types should be treated as vulcanized.

NOTE 1 A test time of 15 s has been introduced for thermoplastic rubber because the hardness value continues to decrease over a longer period of time than for vulcanized rubber, this test time being the same as that specified for plastics in ISO 868.

NOTE 2 If a flow curve is recorded for at least 15 s (hardness every 0,1 s) for an unknown material, the appropriate measuring time (3 s or 15 s) can be selected instead of treating it as vulcanized.

9.3 Measurements

Make three (known material) or five (unknown material) measurements of hardness at different positions on the test piece at least 6 mm apart for types A, D and AO and at least 0,8 mm apart for type AM, and determine the median value.

If the time interval after which each reading was taken is different from 3 s, record the individual values of the indentation hardness together with the time interval after which each reading was taken, and determine the median value and the maximum and minimum values obtained.

10 Calibration and checking

10.1 Calibration

The instrument shall be calibrated regularly using suitable instruments for measuring force, indentation depth and indenter geometry in accordance with ISO 48-9.

NOTE Abrasive material can strongly wear the indenter. Therefore, recalibration or replacement of the indenter can be needed more often than the standard frequency.

10.2 Checking using standard rubber blocks[1)]

Press the instrument against a suitable flat, hard surface (e.g. glass) and adjust, where possible, the reading on the scale to give a value of 100.

Continue the instrument check using a set of standard rubber blocks covering the measurement range. All adjustments shall be made in accordance with the manufacturer's instructions. The set of standard rubber blocks used shall consist of at least three reference blocks in a suitable covered container away from light, heat, oil and grease. The reference blocks shall be recalibrated once a year, or if the mean value deviates more than one unit compared with the certificate of the reference blocks. The calibration of the reference blocks needs to be done with a reference measurement device of an accredited laboratory. Instruments in regular use shall be checked at least each week against standard rubber blocks.

11 Precision

See Annex A.

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) a description of the test piece, including its thickness and, in the case of a composite test piece, the number of layers;

b) the test method used, i.e. the reference number of this document (ISO 48-4:2018);

c) test details:

   1) the temperature of test, and the relative humidity when the hardness of the material is dependent on the humidity;

   2) the type of instrument used, including whether it was hand-held or on a stand;

   3) number of measurements;

   4) the time which elapsed between the preparation of the test piece and the measurement of its hardness;

   5) details of any deviation from the standard procedure;

   6) details of any operations not specified in this document and any incidents likely to have had an influence on the result;

d) the test result, expressed as a whole number, together with the scale used, e.g. 75 Shore A (A75);

e) the date of the test.

1) Standard rubber hardness blocks are available from a number of instrument manufacturers and accredited test laboratories.

Annex A
(informative)

Precision

A.1 General

Interlaboratory test programmes (ITPs) for precision evaluation for hardness tests were conducted in 1985, 2004 and 2007.

NOTE ISO/TR 9272:1986[2)] was used for the ITP carried out in 1985, but ISO/TR 9272:2005 was used for the 2004 and the 2007 programmes.

A.2 ITP in 1985

A.2.1 The first ITP was organized and conducted by Statens Provningsanstalt (Sweden) in late 1985. Cured test pieces of four rubber compounds (materials) were prepared in one laboratory and sent to all participants. The nominal hardness values were 30, 45, 65 and 85. Thirty-two laboratories participated for measurement of the Shore meter hardness. On each of 2 days, 1 week apart, five determinations (measurements) of hardness were made on each compound. The median of the five values was used as a "test result" for the precision analysis.

A.2.2 The precision assessment is a type 1 (cured prepared test pieces circulated) and the time for repeatability and reproducibility is on a scale of days.

A.2.3 The precision results for the Shore hardness meter are given in Table A.1.

Table A.1 — Type 1 precision for a Shore hardness meter

Material	Average value	Within-laboratory		Between laboratories	
		Repeatability (absolute) r[a]	Repeatability (relative) (r)[b]	Reproducibility (absolute) R[a]	Reproducibility (relative) (R)[b]
A	32,7	2,67	8,15	6,41	19,6
B	47,2	1,65	3,49	4,80	10,2
C	65,6	1,53	2,34	2,83	4,31
D	80,2	1,64	2,04	5,19	6,46
Pooled values	56,2	1,93	3,42	5,03	8,94

[a] In measurement units.

[b] In percent of mean level.

A.3 ITP in 2004 (Precision results for the type AM durometer compared to that of Micro IRHD testing)

A.3.1 The precision was determined for a type AM durometer. The results are given in Tables A.2 and A.3. Although Micro IRHD testing is not specified in this test method, the precision for Micro International

2) Withdrawn standard replaced by ISO/TR 9272:2005.

Rubber Hardness Degrees (Micro IRHD) was also evaluated for the purposes of comparison. ISO 48-2 includes the additional set of precision results for IRHD[3].

The *repeatability*, or local domain precision, for each of the hardness test methods was established for each material as the values found in Table A.2. Two individual test results (obtained by the proper use of this document) that differ by more than the tabulated values of *r* (in measurement units) and (*r*) (in percent) should be considered as suspect, i.e. as having come from different populations, and should suggest that some appropriate investigative action be taken.

The *reproducibility*, or global domain precision, for the type AM durometer hardness test method was established for each material as the values found in Table A.2. Two individual test results obtained in different laboratories (by the proper use of this document) that differ by more than the tabulated values of *R* (in measurement units) and (*R*) (in percent) should be considered as suspect, i.e. as having come from different populations, and should suggest that some appropriate investigative action be taken.

A.3.2 A type 1 precision was evaluated (for both tests) using cured test pieces prepared from four different rubber compounds, A, B, C and D (with a range of hardness values), supplied to each of the six laboratories participating in the ITP. On each of two test days, two weeks apart, the following test sequence was carried out. Three test pieces were furnished for each compound, and the median value of five hardness measurements on each of the three test pieces was obtained for each of two operators. For each operator, the median value was selected for all three test pieces. The two median values were then averaged to obtain a single value designated as the test result for that test day. Shore AM measurements were made on one side of the test piece and IRHD measurements were made on the reverse side. The precision analysis was based on test result data, i.e. two test result values per laboratory.

The ISO/TR 9272:2005 option 2 outlier treatment procedure, outlier replacement, was adopted since the ITP had the minimum number of six participating laboratories. This option 2 procedure replaces each outlier declared as significant with a value that is consistent with the data value distribution for the non-outlier data for that material. See ISO/TR 9272:2005 for the rationale behind this concept and for other details.

The precision results as determined by this ITP may not be applied to acceptance or rejection testing for any group of materials or products without documentation that the results of this precision evaluation actually apply to the materials or products tested.

A.3.3 The precision results for type AM durometer measurements are given in Table A.2, with the materials listed in order of increasing hardness. The results are given in terms of both absolute precision, *r* and *R*, and relative precision (*r*) and (*R*). General statements for the use of the precision results are given above.

A.3.4 The precision results for Micro IRHD testing are given in Table A.3, with the materials listed in order of increasing hardness. The results are given in terms of both absolute precision, *r* and *R*, and relative precision (*r*) and (*R*). General statements for the use of the precision results are given above.

A.3.5 The results of the precision analyses in Tables A.2 and A.3 indicate that there is no pronounced trend for *r* or *R* versus hardness level over the 46 to 74 range. The repeatability parameters for the type AM durometer [*r* = 0,88, (*r*) = 1,47] and for Micro IRHD [*r* = 1,14, (*r*) = 2,04] are reasonably similar. However, the reproducibility of the two hardness measurement methods is substantially different: for Shore AM, *R* = 5,08 and (*R*) = 8,98, and for IRHD, *R* = 2,20 and (*R*) = 3,85. The reproducibility parameters

3) This is to ensure that optimum use is made of the results on IRHD from the ITP for ISO 7619-1 at that time, given that these results might otherwise be discarded. Having precision in both documents (ISO 48-2 and ISO 48-4) for IRHD expands the precision knowledge base for IRHD and provides more precision information on this method.

R and (*R*) for IRHD are 43 % of the value(s) for Shore AM, indicating much better between-laboratory agreement for the IRHD measurements.

Table A.2 — Precision for ISO 7619-1 (current ISO 48-4) hardness — Type AM durometer

Material	Mean level	Within-laboratory			Between laboratories			Number of laboratories[d]
		Standard deviation	Repeatability (absolute)	Repeatability (relative)	Standard deviation	Reproducibility (absolute)	Reproducibility (relative)	
		s_r[a]	r[a]	(r)[b]	s_R[c]	R[a]	(R)[b]	
B (2)	47,9	0,276	0,772	1,61	2,32	6,5	13,57	6
C (3)	55,2	0,223	0,623	1,13	1,85	5,17	9,35	6 (1)
A (1)	62,8	0,404	1,13	1,8	1,95	5,45	8,68	6
D (4)	73,9	0,357	1	1,35	1,14	3,2	4,33	6 (1)
Average		—	0,881 25	1,472 5	—	5,08	8,982 5	—

NOTE See A.3.5 for a discussion of the precision results in this table.

a In measurement units.

b In percent of mean level.

c For total between-laboratory variation, in measurement units.

d Number of option 2 outlier laboratory replacement values given in parentheses.

Table A.3 — Precision for ISO 48 (current ISO 48-2) hardness — Micro IRHD

Material	Mean level	Within-laboratory			Between laboratories			Number of laboratories[d]
		Standard deviation	Repeatability (absolute)	Repeatability (relative)	Standard deviation	Reproducibility (absolute)	Reproducibility (relative)	
		s_r[a]	r[a]	(r)[b]	s_R[c]	R[a]	(R)[b]	
B (2)	45,6	0,404	1,13	2,48	0,954	2,67	5,85	6
C (3)	53,9	0,469	1,31	2,43	0,583	1,63	3,03	6 (1)
A (1)	63,7	0,605	1,7	2,66	0,728	2,04	3,2	6
D (4)	74	0,149	0,416	0,57	0,875	2,45	3,31	6 (1)
Average		—	1,139	2,035	—	2,197 5	3,847 5	—

NOTE See A.3.5 for a discussion of the precision results in this table.

a In measurement units.

b In percent of mean level.

c For total between-laboratory variation, in measurement units.

d Number of option 2 outlier laboratory replacement values given in parentheses.

A.4 ITP in 2007

A.4.1 Programme details

A.4.1.1 An ITP for the evaluation of the precision of Shore A and D as well as IRHD N, M, and L hardness tests was conducted in 2007, using the procedures and guidelines described in ISO/TR 9272:2005. Precision for IRHD, which are covered by ISO 48-2, was determined for the purpose of comparison with Shore precision. See ISO 48-2 for more details on hardness testing using IRHD test methods.

A.4.1.2 A type 1 precision was evaluated, using cured test pieces prepared from seven different reference materials or compounds (RM) designated as RM 121, 122, 123, 124, 125, 126 and 128. These materials had a range of hardness levels from low to high. See the actual precision tables (Tables A.4 and A.5) for the actual hardness levels with Shore A and Shore D test methods.

A.4.1.3 The number of laboratories volunteering to participate was as follows: 26 laboratories for Shore A; 18 laboratories for Shore D.

However, some of the laboratories that initially volunteered did not participate in the testing. The number of laboratories on which each type of hardness method is based is given in the tables of precision results (Tables A.4 and A.5). The number of participating laboratories as noted in these tables is the final number after certain laboratory values were deleted as outliers (for each of the five different types of test) using the procedures as given in ISO/TR 9272:2005.

A.4.1.4 For each RM or compound and for each laboratory, two test pieces (designated as a and b) were furnished and five hardness measurements were made on each of the two test pieces on each of two test days (Monday and Friday) in a given test week. This process was repeated on alternating test weeks for a total of four "test" weeks that covered a total time span of eight calendar weeks.

A.4.1.5 For each of the two data sets (a and b pieces) of five measurements each day (of each week), a median value was selected. The two median values (a and b) for each test day were then averaged to obtain a single value designated as the "combined test result" value for any given test day and test week. Statistical analysis for precision was then conducted on each of these day 1 and day 2 "pooled combined test result" values. A separate precision analysis was conducted for each of the four test weeks and to generate the final precision tables, the precision parameters (*r*, *R*, etc.) were averaged to obtain a pooled precision parameter, i.e. an all-four-week value.

A.4.1.6 The participating laboratories were encouraged to use two equally competent operators (if available) for this ITP: Operator 1 for test weeks 1 and 3 and Operator 2 for test weeks 2 and 4. The decision to use different test pieces and different operators as well as the use of four test weeks was based on the desire to include such normal variation sources in the final or pooled combined database. Thus the precision values, as listed in Tables A.4 and A.5, represent more reliable or realistic values compared to the usual ITP results which constitute a "single point in time" estimate of precision.

A.4.1.7 The precision results as determined by this ITP should not be applied to acceptance or rejection testing for any group of materials or products without documentation that the results of this precision evaluation actually apply to the products or materials tested.

A.4.2 Precision results

A.4.2.1 The precision results for Shore hardness tests obtained for the 2007 ITP are given in Tables A.4 and A.5 and the ones for IRHD hardness tests are given in ISO 48-2. Precision is given for Shore A in Table A.4 and precision for Shore D in Table A.5. The results are given in terms of both the absolute precision, *r* or *R*, and the relative precision, (*r*) and (*R*).

A.4.2.2 The precision results showed that the precision of IRHD N was substantially better than IRHD M. IRHD L appeared to be roughly equivalent to IRHD N but caution was advised since the IRHD L precision was based on only four laboratories. IRHD N precision was essentially equal to Shore A, but Shore D precision was the worst of all methods.

A.4.2.3 Bias is the difference between a measured average test result and a reference or true value for the measurement in question. Reference values do not exist for this test method and therefore bias cannot be evaluated.

Table A.4 — Precision data for the Shore A test method

Material	Mean level	Within-laboratory			Between laboratories			Number of laboratories[d]
		Standard deviation s_r[a]	Repeatability (absolute) r[a]	Repeatability (relative) (r)[b]	Standard deviation s_R[c]	Repeatability (absolute) R[a]	Repeatability (relative) (R)[b]	
RM 122	35,6	0,199	0,560	1,57	0,613	1,72	4,83	19
RM 124	57,5	0,263	0,720	1,28	0,720	2,02	3,51	20
RM 126	79,3	0,473	1,320	1,67	0,821	2,30	2,90	20
Average[e]		0,312	0,867	1,51	0,718	2,01	3,75	

[a] In measurement units.

[b] In percent of mean level.

[c] For total between-laboratory variation, in measurement units.

[d] Average number of laboratories after outliers have been deleted.

[e] Simple mean values for comparison.

Table A.5 — Precision data for the Shore D test method

Material	Mean level	Within-laboratory			Between laboratories			Number of laboratories[d]
		Standard deviation s_r[a]	Repeatability (absolute) r[a]	Repeatability (relative) (r)[b]	Standard deviation s_R[c]	Repeatability (absolute) R[a]	Repeatability (relative) (R)[b]	
RM 126	24,4	0,369	1,030	4,22	0,756	2,12	8,66	15
RM 128	43,4	0,617	1,730	3,98	1,040	2,92	6,73	14
Average[e]		0,493	1,380	4,10	0,898	2,52	7,70	

[a] In measurement units.

[b] In percent of mean level.

[c] For total between-laboratory variation, in measurement units.

[d] Average number of laboratories after outliers have been deleted.

[e] Simple mean values for comparison.

Bibliography

[1] ISO 48-2, *Rubber, vulcanized or thermoplastic — Determination of hardness — Part 2: Hardness between 10 IRHD and 100 IRHD*

[2] ISO 48-7, *Rubber, vulcanized or thermoplastic — Determination of hardness — Part 7: Apparent hardness of rubber-covered rollers by Shore-type durometer method*

[3] ISO 868, *Plastics and ebonite — Determination of indentation hardness by means of a durometer (Shore hardness)*

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

[5] Brown, R.P., *Physical testing of rubber*, Chapman and Hall, London, 2006

[6] Oberto, S., *Rubber Chemistry Technology*, 1955, 28, 1054

[7] Juve, A.E., *Rubber Chemistry Technology*, 1957, 30, 367

[8] Brown, R.P., *Polymer Testing*, 1991, 10, 117

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The main changes in this revision are as follows:

- The title and designation of the standard have been modified.
- In **6.3.1**, the description has been improved to distinguish hand-held instruments and on-a-stand instruments more clearly.
- In Clause **8**, the description for the required time for conditioning has been improved for better understanding.
- In **9.2**, the use of talcum powder has been removed.
- In Annex A, precision results from ITPs that were carried out in 1985 and 2007 have been included.

Other standards which are being published in this series as sections are as follows,

Sec 1	Introduction and guidance
Sec 2	Hardness between 10 IRHD and 100 IRHD (*fifth revision*)
Sec 3	Dead-load hardness using the very low rubber hardness (VLRH) scale
Sec 5	Indentation hardness by IRHD pocket meter method (*second revision*)
Sec 6	Apparent hardness of rubber-covered rollers by IRHD method
Sec 7	Apparent hardness of rubber-covered rollers by Shore-type durometer method
Sec 8	Apparent hardness of rubber-covered rollers by Pusey and Jones method
Sec 9	Calibration and verification of hardness testers

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'.

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 certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their respective places, are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 48-9 Rubber, vulcanized or thermoplastic — Determination of hardness — Part 9: Calibration and verification of hardness testers	IS 3400 (Part 2/Sec 9) :2022/Doc No.: PCD 29 (17589) Methods of Test for Vulcanized Rubber, Vulcanized or Thermoplastic — Part 2 Determination of Hardness, Sec 9 Calibration and Verification of Hardness Testers (*under publication*)	Identical with ISO 48-9 : 2018
ISO 23529 Rubber — General procedures for preparing and conditioning test pieces for physical test methods	IS 13867 : 2021 Rubber — General procedures for preparing and conditioning test pieces for physical test methods (*first revision*)	Identical with ISO 23529 : 2016

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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This Indian Standard has been developed from Doc No.: PCD 29 (17583).

Amendments Issued Since Publication

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