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Rubber, vulcanized or thermoplastic — Determination of hardness —

Part 4: Indentation hardness by durometer method (Shore hardness)

Caoutchouc vulcanisé ou thermoplastique - Détermination de la dureté —

Partie 4: Dureté par pénétration par la méthode au duromètre (dureté Shore)

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This first edition of ISO 48-4 cancels and replaces ISO 7619-1:2010 which has been technically revised.

The main changes compared to the previous edition are as follows:

— a new standard number has been given.

— in the Introduction, an explanation of the purpose of the grouping work has been added.

— 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.

A list of all parts in the ISO 48 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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].

Rubber, vulcanized or thermoplastic — Determination of hardness —

Part 4: Indentation hardness by durometer method (Shore hardness)

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.

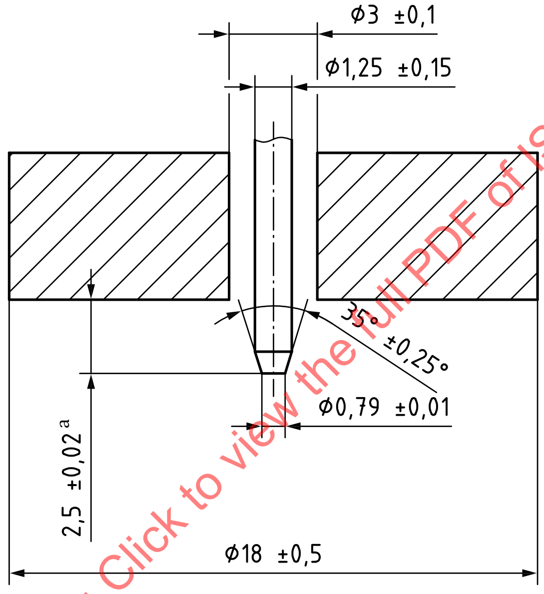
— For type AO durometers:

$F = 550 + 75H_{AO}$

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.

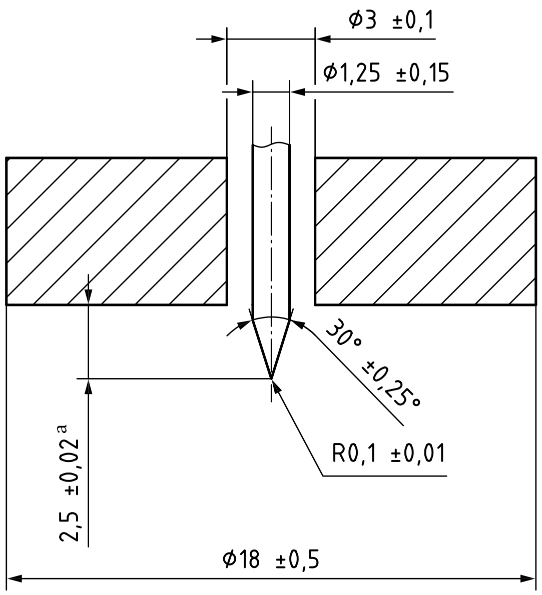
Dimension in millimetres



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

Figure 1 — Indentor for type A durometer

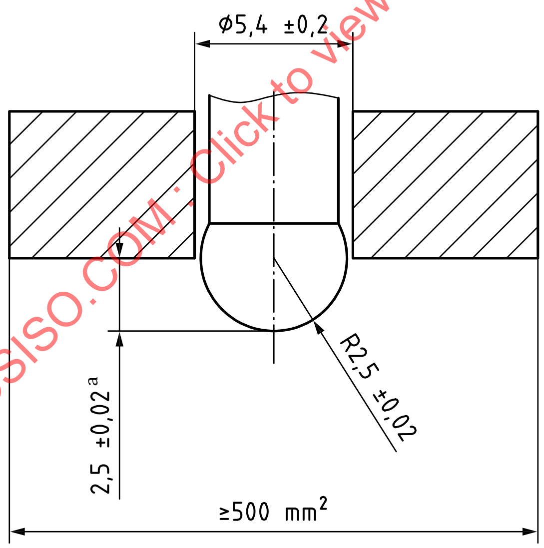
Dimension in millimetres



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

Figure 2 — Indentor for type D durometer

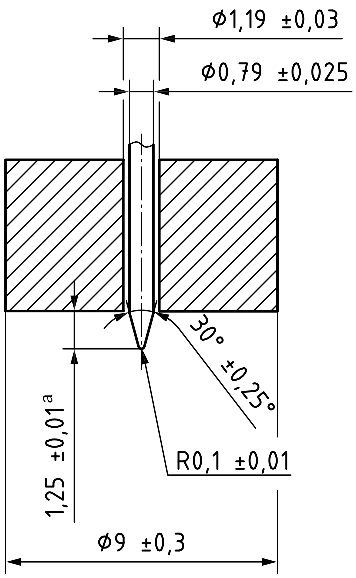
Dimension in millimetres



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.