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Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)

Caoutchouc vulcanisé ou thermoplastique — Détermination de la dureté (dureté comprise entre 10 DIDC et 100 DIDC)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 48 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fifth edition cancels and replaces the fourth edition (ISO 48:2007), of which it constitutes a minor revision intended to update the precision statements in Annex B. It also incorporates the Technical Corrigendum ISO 48:2007/Cor.1:2009.

Introduction

The hardness test specified in this International Standard is intended to provide a rapid measurement of rubber stiffness, unlike hardness tests on other materials which measure resistance to permanent deformation.

Hardness is measured from the depth of indentation of a spherical indentor, under a specified force, into a rubber test piece. An empirical relationship between depth of indentation and Young's modulus for a perfectly elastic isotropic material has been used to derive a hardness scale which can conveniently be used for most rubbers.

When it is required to determine the value of Young's modulus itself, it is expected that an appropriate test method be used, for example that described in ISO 7743.

The guide to hardness testing, ISO 18517, can also be a useful reference.

Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 ensure compliance with any national regulatory conditions.

CAUTION — Certain procedures specified in this International Standard may 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 International Standard specifies four methods for the determination of the hardness of vulcanized or thermoplastic rubbers on flat surfaces (standard-hardness methods) and four methods for the determination of the apparent hardness of curved surfaces (apparent-hardness methods). The hardness is expressed in international rubber hardness degrees (IRHD). The methods cover the hardness range from 10 IRHD to 100 IRHD.

These methods differ primarily in the diameter of the indenting ball and the magnitude of the indenting force, these being chosen to suit the particular application. The range of applicability of each method is indicated in Figure 1.

This International Standard does not specify a method for the determination of hardness by a pocket hardness meter, which is described in ISO 7619-2.

This International Standard specifies the following four methods for the determination of standard hardness.

— Method N (normal test) is appropriate for rubbers with a hardness in the range 35 IRHD to 85 IRHD, but can also be used for hardnesses in the range 30 IRHD to 95 IRHD.

— Method H (high-hardness test) is appropriate for rubbers with a hardness in the range 85 IRHD to 100 IRHD.

— Method L (low-hardness test) is appropriate for rubbers with a hardness in the range 10 IRHD to 35 IRHD.

— Method M (microtest) is essentially a scaled-down version of the normal test method N, permitting the testing of thinner and smaller test pieces. It is appropriate for rubbers with a hardness in the range 35 IRHD to 85 IRHD, but can also be used for hardnesses in the range 30 IRHD to 95 IRHD.

NOTE 1 The value of the hardness obtained by method N within the ranges 85 IRHD to 95 IRHD and 30 IRHD to 35 IRHD might not agree precisely with that obtained using method H or method L, respectively. The difference is not normally significant for technical purposes.

NOTE 2 Because of various surface effects in the rubber and the possibility of slight surface roughness (produced, for example, by buffing), the microtest might not always give results agreeing with those obtained by the normal test.

This International Standard also specifies four methods, CN, CH, CL and CM, for the determination of the apparent hardness of curved surfaces. These methods are modifications of methods N, H, L and M, respectively, and are used when the rubber surface tested is curved, in which case there are two possibilities:

a) the test piece or product tested is large enough for the hardness instrument to rest upon it; or

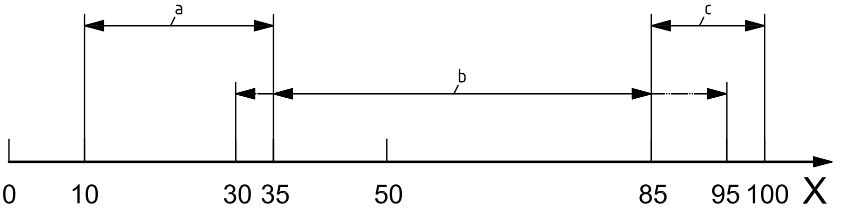
b) the test piece or product tested is small enough for both the test piece and the instrument to rest upon a common support.

A variant of b) would be where the test piece rests upon the specimen table of the instrument.

Apparent hardness can also be measured on non-standard flat test pieces using methods N, H, L and M.

The procedures described cannot provide for all possible shapes and dimensions of test piece, but cover some of the commonest types, such as O-rings.

This International Standard does not specify the determination of the apparent hardness of rubber-covered rollers, which is specified in ISO 7267 (all parts).



Key

X hardness (IRHD)

[a] Method L and method CL.

[b] Methods N and M and methods CN and CM.

[c] Method H and method CH.

Figure 1 — Range of applicability

2 Normative references

The following referenced documents are indispensable for the application 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 18898, *Rubber — 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

For the purposes of this document, the following terms and definitions apply.

3.1
international rubber hardness degree scale
IRHD scale
hardness scale chosen so that 0 represents the hardness of material having a Young's modulus of zero and 100 represents the hardness of a material of infinite Young's modulus

NOTE The following characteristics applying over most of the normal range of hardnesses:

a) one international rubber hardness degree always represents approximately the same proportional difference in Young's modulus;

b) for highly elastic rubbers, the IRHD and Shore A scales are comparable.

3.2
standard hardness
hardness obtained using the procedures described in methods N, H, L and M on test pieces of the standard thickness and not less than the minimum lateral dimensions specified

NOTE Standard hardness is reported to the nearest whole number in IRHD.

3.3
apparent hardness
hardness obtained using the procedures described in methods N, H, L and M on test pieces of non-standard dimensions, as well as hardness values obtained using methods CN, CH, CL and CM

NOTE 1 Apparent hardness is reported to the nearest whole number in IRHD.

NOTE 2 Values obtained by methods CN, CH, CL and CM are always given as apparent hardnesses since tests are commonly made on the complete article where the thickness of the rubber can vary and, in many cases, the lateral dimensions might not provide the minimum distance between the indentor and the edge necessary to eliminate edge effects. Thus the readings obtained do not in general coincide with readings obtained on standard test pieces as defined in methods N, H, L and M or on a flat parallel-faced slab of the same thickness as the article. Moreover, the readings might depend appreciably on the method of support of the article and whether or not a presser foot is used. Therefore, results obtained on curved surfaces are arbitrary values applicable only to test pieces or articles of one particular shape and of particular dimensions, and supported in one particular way, and in extreme cases such values can differ from the standard hardness by as much as 10 IRHD. Furthermore, surfaces that have been buffed or otherwise prepared to remove cloth markings, etc., can give slightly different hardness values from those with a smooth, moulded finish.

4 Principle

The hardness test consists in measuring the difference between the depths of indentation of a ball into the rubber under a small contact force and a large (indenting) force. From this difference, multiplied when using the microtest by the scale factor 6, the hardness in IRHD is obtained from Tables 3 to 5 or from graphs based on these tables or from a scale, reading directly in IRHD, calculated from the tables and fitted to the indentation-measuring instrument. These tables and curves are derived from the empirical relationship between indentation depth and hardness given in Annex A.

5 Apparatus

5.1 General

Calibration and verification of the apparatus shall be performed in accordance with ISO 18898.

5.2 Methods N, H, L and M

The essential parts of the apparatus are as specified in 5.2.1 to 5.2.6, the appropriate dimensions and forces being shown in Table 1.

5.2.1 Vertical plunger, having a rigid ball or spherical surface on the lower end, and **means for supporting the plunger** so that the spherical tip is kept slightly above the surface of the annular foot prior to applying the contact force.

5.2.2 Means for applying a contact force and an additional indenting force to the plunger, making allowance for the mass of the plunger, including any fittings attached to it, and for the force of any spring acting on it, so that the forces actually transmitted through the spherical end of the plunger are as specified.

5.2.3 Means for measuring the increase in depth of indentation of the plunger caused by the indenting force, either in metric units or reading directly in IRHD.

The gauge employed may be mechanical, optical or electrical.

5.2.4 Flat annular foot, normal to the axis of the plunger and having a central hole for the passage of the plunger.

The foot rests upon the test piece and exerts a pressure on it of 30 kPa ± 5 kPa provided that the total load on the foot does not fall outside the values given in Table 1. The foot shall be rigidly connected to the indentation-measuring device, so that a measurement is made of the movement of the plunger relative to the foot (i.e. the top surface of the test piece), not relative to the surface supporting the test piece.

Table 1 — Forces and dimensions of apparatus

Test	Diameters mm	Force on ball Contact N	Indenting N	Total N	Force on foot N
Method N (normal test)	Ball 2,50 ± 0,01 Foot 20 ± 1 Hole 6 ± 1	0,30 ± 0,02	5,40 ± 0,01	5,70 ± 0,03	8,3 ± 1,5
Method H (high hardness)	Ball 1,00 ± 0,01 Foot 20 ± 1 Hole 6 ± 1	0,30 ± 0,02	5,40 ± 0,01	5,70 ± 0,03	8,3 ± 1,5
Method L (low hardness)	Ball 5,00 ± 0,01 Foot 22 ± 1 Hole 10 ± 1	0,30 ± 0,02	5,40 ± 0,01	5,70 ± 0,03	8,3 ± 1,5
Method M (microtest)	**Diameters** mm	**Contact** mN	**Indenting** mN	**Total** mN	**Force on foot** mN
	Ball 0,395 ± 0,005 Foot 3,35 ± 0,15 Hole 1,00 ± 0,15	8,3 ± 0,5	145 ± 0,5	153,3 ± 1,0	235 ± 30

NOTE 1 In the microtest, when using instruments in which the test piece table is pressed upwards by a spring, the values of the foot pressure and the force on the foot are those acting during the period of application of the total force. Before the indenting force of 145 mN is applied, the force on the foot is greater by this amount, and hence equals 380 mN ± 30 mN.

NOTE 2 Not all possible combinations of dimensions and forces given in this table will meet the pressure requirements of 5.2.4.

5.2.5 Means for gently vibrating the apparatus, for example an electrically operated buzzer, to overcome any slight friction.

(This may be omitted in instruments where friction is effectively eliminated.)

5.2.6 Chamber for the test piece, when tests are made at temperatures other than a standard laboratory temperature.

This chamber shall be equipped with a means of maintaining the temperature within 2 °C of the desired value. The foot and vertical plunger shall extend through the top of the chamber, and the portion passing through the top shall be constructed from a material having a low thermal conductivity. A sensing device shall be located within the chamber near or at the location of the test piece, for measuring the temperature (see ISO 23529).

5.3 Methods CN, CH, CL and CM

The apparatus used shall be essentially that described in 5.2 but differing in the following respects.

5.3.1 Cylindrical surfaces of radius greater than 50 mm

The base of the instrument shall have a hole below the plunger, allowing free passage of the annular foot such that measurement may be made above or below the base.

The lower surface of the base shall be in the form of two cylinders parallel to each other and the plane of the base. The diameter of the cylinders and their distance apart shall be such as to locate and support the instrument on the curved surface to be tested. Alternatively, the modified base may be fitted with feet movable in universal joints so that they adapt themselves to the curved surface.

5.3.2 Surfaces with double curvature of large radius greater than 50 mm

The instrument with adjustable feet described in 5.3.1 shall be used.

5.3.3 Cylindrical surfaces of radius 4 mm to 50 mm or small test pieces with double curvature

On surfaces too small to support the instrument, the test piece or article shall be supported by means of special jigs or V-blocks so that the indentor is vertically above the test surface. Wax may be used to fix small items to the test piece table.

In general, an instrument as described for method M should be used only where the thickness of the rubber tested is less than 4 mm.

NOTE Instruments for method M in which the test piece table is pressed upwards by a spring are not suitable for use on large test pieces or articles with a large radius of curvature.

5.3.4 Small O-rings and articles of radius of curvature less than 4 mm

These shall be held in suitable jigs or blocks or secured by wax to the instrument table. Measurements shall be made using the instrument for method M.

No test shall be made if the smallest radius is less than 0,8 mm.

6 Test pieces

6.1 General

Test pieces shall be prepared in accordance with ISO 23529.

6.2 Methods N, H, L and M

6.2.1 General

The test pieces shall have their upper and lower surfaces flat, smooth and parallel to one another.

Tests intended to be comparable shall be made on test pieces of the same thickness.

6.2.2 Thickness

6.2.2.1 Methods N and H

The standard test piece shall be 8 mm to 10 mm thick and shall be made up of one or more layers of rubber, the thinnest of which shall not be less than 2 mm thick. All surfaces shall be flat and parallel.

Non-standard test pieces may be either thicker or thinner, but not less than 4 mm thick.

6.2.2.2 Method L

The standard test piece shall be 10 mm to 15 mm thick and shall be made up of one or more layers of rubber, the thinnest of which shall not be less than 2 mm thick. All surfaces shall be flat and parallel.

Non-standard test pieces may be either thicker or thinner, but not less than 6 mm thick.

6.2.2.3 Method M

The standard test piece shall have a thickness of 2 mm ± 0,5 mm. Thicker or thinner test pieces may be used, but in no case less than 1 mm thick. Readings made on such test pieces do not in general agree with those obtained on the standard test piece.

6.2.3 Lateral dimensions

6.2.3.1 Methods N, H and L

The lateral dimensions of both standard and non-standard test pieces shall be such that no test is made at a distance from the edge of the test piece less than the appropriate distance shown in Table 2.

Table 2 — Minimum distance of point of contact from test piece edge

Dimensions in millimetres

Total thickness of test piece	Minimum distance from point of contact to edge of test piece
4	7,0
6	8,0
8	9,0
10	10,0
15	11,5
25	13,0

6.2.3.2 Method M

The lateral dimensions shall be such that no test is made at a distance from the edge of less than 2 mm.

When test pieces thicker than 4 mm are tested on the microtest instrument because the lateral dimensions or the available flat area do not permit testing on a normal instrument, the test shall be made at a distance from the edge as great as possible.

6.3 Methods CN, CH, CL and CM

The test piece shall be either a complete article or a piece cut therefrom. The underside of a cut piece shall be such that it can be properly supported during the hardness test. If the surface on which the test is to be made is cloth-marked, it shall be buffed prior to testing. Test pieces shall be allowed to recover at a standard laboratory temperature (see ISO 23529) for at least 16 h after buffing and shall be conditioned in accordance with Clause 8. The conditioning period may form part of the recovery period.

7 Time interval between vulcanization and testing

Unless otherwise specified for technical reasons, the following requirements shall be observed (see ISO 23529):

— For all normal test purposes, the minimum time between vulcanization and testing shall be 16 h. In cases of arbitration, the minimum time shall be 72 h.

— For non-product tests, the maximum time between vulcanization and testing shall be 4 weeks and, for evaluations intended to be comparable, the tests, as far as possible, shall be carried out after the same time interval.

— For product tests, whenever possible, the time between vulcanization and testing shall not exceed 3 months. In other cases, tests shall be made within 2 months of the date of receipt by the purchaser of the product.

8 Conditioning of test pieces

8.1 When a test is made at a standard laboratory temperature (see ISO 23529), the test pieces shall be maintained at the conditions of test for at least 3 h immediately before testing.

8.2 When tests are made at higher or lower temperatures, the test pieces shall be maintained at the conditions of test for a period of time sufficient to reach temperature equilibrium with the testing environment, or for the period of time required by the specification covering the material or product being tested, and then immediately tested.

9 Temperature of test

The test shall normally be carried out at standard laboratory temperature (see ISO 23529). When other temperatures are used, these shall be selected from the list of preferred temperatures specified in ISO 23529.

10 Procedure

Condition the test piece as specified in Clause 8.

Lightly dust the upper and lower surfaces of the test piece with dusting powder. Place the test piece on a horizontal rigid surface. Bring the foot into contact with the surface of the test piece. Press the plunger and indenting ball for 5 s on to the rubber, the force on the ball being the contact force.

NOTE Talc has been found to be a suitable dusting powder.

If the gauge is graduated in IRHD, adjust it to read 100 at the end of the 5 s period; then apply the additional indenting force and maintain it for 30 s, when a direct reading of the hardness in IRHD is obtained.

If the gauge is graduated in metric units, note the differential indentation D (in hundredths of a millimetre) of the plunger caused by the additional indenting force, applied for 30 s. Convert this (after multiplying by the scale factor of 6 when using the apparatus for the microtest) into IRHD, using Tables 3 to 5 or a graph constructed therefrom.

During the loading periods, vibrate the apparatus gently unless it is essentially free of friction.

11 Number of readings

Make one measurement at each of a minimum of three different points distributed over the test piece and separated from each other by a minimum of 6 mm, and take the median of the results when these are arranged in increasing order.

12 Expression of results

Express the hardness, to the nearest whole number, as the median of the individual measurements in IRHD, indicated by the degree sign (°) followed by:

a) either the letter S indicating that the test piece was of the standard thickness or, for tests on non-standard test pieces, the actual test piece thickness and the smaller lateral dimension (in millimetres) (the result then being an apparent hardness);

b) the code-letter for the method, i.e. N for the normal test, H for the high-hardness test, L for the low-hardness test and M for the microtest;

c) for tests on curved surfaces, the prefix letter C.

EXAMPLE 1 58°, SN

EXAMPLE 2 16°, 8 × 25 mm, L

EXAMPLE 3 90°, CH

13 Precision

Precision results of interlaboratory test programmes (ITPs) are given in Annex B.

Table 3 — Conversion of values of differential indentation, D, to IRHD for use in method N using a 2,5 mm indentor

Dimensions of D in hundredths of a millimetre

D	IRHD	D	IRHD	D	IRHD	D	IRHD	D	IRHD	D	IRHD
0	100,0	31	82,9	62	64,5	93	51,2	124	41,7	155	34,6
1	100,0	32	82,2	63	64,0	94	50,9	125	41,4	156	34,4
2	99,9	33	81,5	64	63,5	95	50,5	126	41,1	157	34,2
3	99,8	34	80,9	65	63,0	96	50,2	127	40,9	158	34,0
4	99,6	35	80,2	66	62,5	97	49,8	128	40,6	159	33,8
5	99,3	36	79,5	67	62,0	98	49,5	129	40,4	160	33,6
6	99,0	37	78,9	68	61,5	99	49,1	130	40,1	161	33,4
7	98,6	38	78,2	69	61,1	100	48,8	131	39,9	162	33,2
8	98,1	39	77,6	70	60,6	101	48,5	132	39,6	163	33,0
9	97,7	40	77,0	71	60,1	102	48,1	133	39,4	164	32,8
10	97,1	41	76,4	72	59,7	103	47,8	134	39,1	165	32,6
11	96,5	42	75,8	73	59,2	104	47,5	135	38,9	166	32,4
12	95,9	43	75,2	74	58,8	105	47,1	136	38,7	167	32,3
13	95,3	44	74,5	75	58,3	106	46,8	137	38,4	168	32,1
14	94,7	45	73,9	76	57,9	107	46,5	138	38,2	169	31,9
15	94,0	46	73,3	77	57,5	108	46,2	139	38,0	170	31,7
16	93,4	47	72,7	78	57,0	109	45,9	140	37,8	171	31,6
17	92,7	48	72,2	79	56,6	110	45,6	141	37,5	172	31,4
18	92,0	49	71,6	80	56,2	111	45,3	142	37,3	173	31,2
19	91,3	50	71,0	81	55,8	112	45,0	143	37,1	174	31,1
20	90,6	51	70,4	82	55,4	113	44,7	144	36,9	175	30,9
21	89,8	52	69,8	83	55,0	114	44,4	145	36,7	176	30,7
22	89,2	53	69,3	84	54,6	115	44,1	146	36,5	177	30,5
23	88,5	54	68,7	85	54,2	116	43,8	147	36,2	178	30,4
24	87,8	55	68,2	86	53,8	117	43,5	148	36,0	179	30,2
25	87,1	56	67,6	87	53,4	118	43,3	149	35,8	180	30,0
26	86,4	57	67,1	88	53,0	119	43,0	150	35,6		
27	85,7	58	66,6	89	52,7	120	42,7	151	35,4		
28	85,0	59	66,0	90	52,3	121	42,5	152	35,2		
29	84,3	60	65,5	91	52,0	122	42,2	153	35,0		
30	83,6	61	65,0	92	51,6	123	41,9	154	34,8		

Table 4 — Conversion of values of differential indentation, D, into IRHD for use in method H using a 1 mm indentor

Dimensions of D in hundredths of a millimetre

D	IRHD	D	IRHD	D	IRHD	D	IRHD	D	IRHD	D	IRHD
0	100,0	8	99,3	16	97,0	24	93,8	32	90,2	40	86,6
1	100,0	9	99,1	17	96,6	25	93,4	33	89,7	41	86,1
2	100,0	10	98,8	18	96,2	26	92,9	34	89,3	42	85,7
3	99,9	11	98,6	19	95,8	27	92,5	35	88,8	43	85,3
4	99,9	12	98,3	20	95,4	28	92,0	36	88,4	44	84,8
5	99,8	13	98,0	21	95,0	29	91,6	37	87,9		
6	99,6	14	97,6	22	94,6	30	91,1	38	87,5		
7	99,5	15	97,3	23	94,2	31	90,7	39	87,0		

Table 5 — Conversion of values of differential indentation, D, into IRHD for use in method L using a 5 mm indentor

Dimensions of D in hundredths of a millimetre

D	IRHD	D	IRHD	D	IRHD	D	IRHD	D	IRHD	D	IRHD
110	34,9	146	26,8	182	21,1	218	17,0	254	13,8	290	11,5
112	34,4	148	26,4	184	20,8	220	16,8	256	13,7	292	11,4
114	33,9	150	26,1	186	20,6	222	16,6	258	13,5	294	11,3
116	33,4	152	25,7	188	20,3	224	16,4	260	13,4	296	11,2
118	32,9	154	25,4	190	20,1	226	16,2	262	13,3	298	11,1
120	32,4	156	25,0	192	19,8	228	16,0	264	13,1	300	11,0
122	31,9	158	24,7	194	19,6	230	15,8	266	13,0	302	10,9
124	31,4	160	24,4	196	19,4	232	15,6	268	12,8	304	10,8
126	30,9	162	24,1	198	19,2	234	15,4	270	12,7	306	10,6
128	30,4	164	23,8	200	18,9	236	15,3	272	12,6	308	10,5
130	30,0	166	23,5	202	18,7	238	15,1	274	12,5	310	10,4
132	29,6	168	23,1	204	18,5	240	14,9	276	12,3	312	10,3
134	29,2	170	22,8	206	18,3	242	14,8	278	12,2	314	10,2
136	28,8	172	22,5	208	18,0	244	14,6	280	12,1	316	10,1
138	28,4	174	22,2	210	17,8	246	14,4	282	12,0	318	9,9
140	28,0	176	21,9	212	17,6	248	14,3	284	11,8		
142	27,6	178	21,6	214	17,4	250	14,1	286	11,7		
144	27,2	180	21,3	216	17,2	252	14,0	288	11,6		

14 Test report

The test report shall include the following particulars:

a) a reference to this International Standard (ISO 48:2010);

b) test piece details:

   1) the dimensions of the test piece,

   2) the number of layers and the thickness of the thinnest layer,

   3) in the case of curved or irregularly shaped test pieces, a description of the test piece,

   4) the method of preparation of the test piece from the sample, for example moulded, buffed, cut out,

   5) details of the compound and the cure, where appropriate;

c) test method:

   1) the method used,

   2) for curved test pieces, the way in which the test piece was mounted;

d) test details:

   1) the time and temperature of conditioning prior to testing,

   2) the temperature of test, and the relative humidity, if necessary,

   3) any deviation from the procedure specified;

e) test results:

   1) the number of test pieces,

   2) the individual test results,

   3) the median of the individual results, expressed as in Clause 12;

f) the date of the test.

Annex A
(informative)

Empirical relationship between indentation and hardness

The relationship between the differential indentation and the hardness expressed in IRHD is based on the following.

a) The known relationship [10], for a perfectly elastic isotropic material, between indentation D, expressed in hundredths of a millimetre, and Young's modulus E, expressed in megapascals, is given by the following equation:

$$D = 61{,}5R^{-0{,}48}\left[\left(\frac{F_{\text{in}}}{E}\right)^{0{,}74} - \left(\frac{F_{\text{c}}}{E}\right)^{0{,}74}\right]$$

where

F_{in} is the total indenting force, in newtons;

F_{c} is the contact force, in newtons;

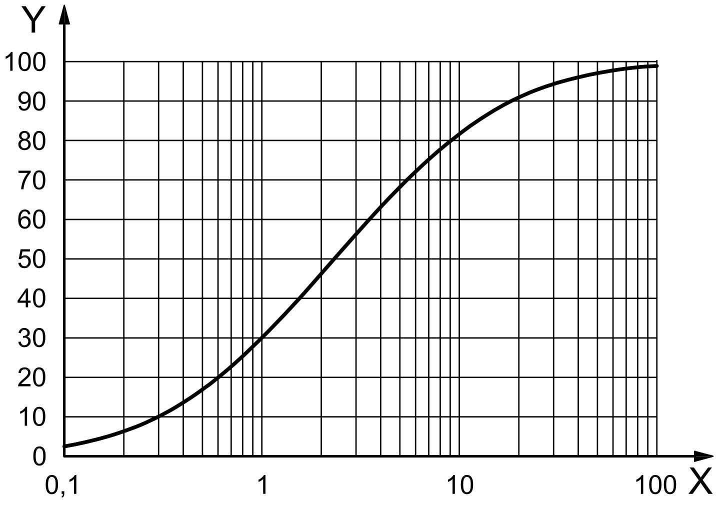
R is the radius of the ball, in millimetres.

b) The use of a probit (integrated normal error) curve to relate $\log_{10}E$ to the hardness in IRHD. This curve is defined in terms of

1) the value of $\log_{10}E$ corresponding to the midpoint of the curve: 0,364 (E being expressed in megapascals);

2) the maximum slope: 57 IRHD per unit increase in $\log_{10}E$.

Figures A.1 to A.3 show the relationship between E, in MPa, and IRHD as defined in 1) and 2).

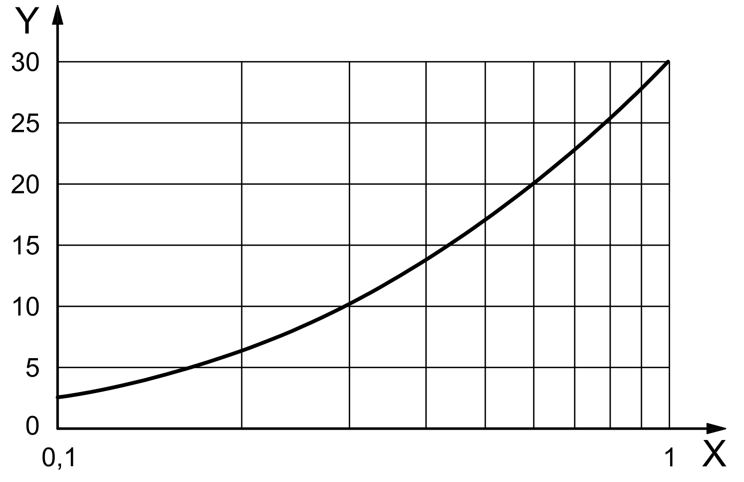


Key

X E, in MPa

Y IRHD

Figure A.1 — Relationship between E and hardness in IRHD from 3 to 100

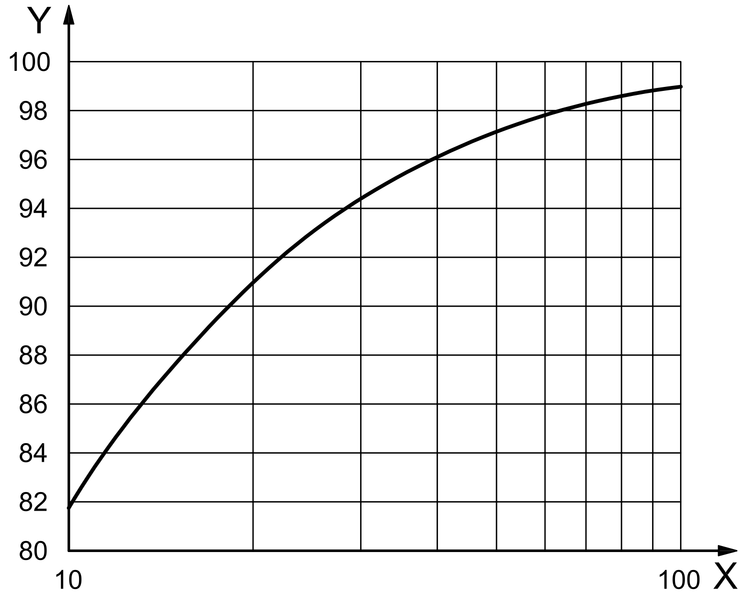


Key

X E, in MPa

Y IRHD

Figure A.2 — Relationship between E and hardness in IRHD from 3 to 30



Key

X E, in MPa

Y IRHD

Figure A.3 — Relationship between E and hardness in IRHD from 80 to 100

Annex B
(informative)

Precision results from interlaboratory test programmes

B.1 General

The following interlaboratory test programmes (ITPs) were initially carried out between 1985 and 2007.

a) Five ITPs were carried out between 1985 and 1989 (see B.2).

b) A further ITP was carried out specifically for method M (the microtest method) in 2004 (see B.3).

c) A very comprehensive precision evaluation (ITP) was conducted in 2007 where a complete series of tests was repeated for four test weeks with each test week separated by an intervening week, thus giving four separate estimates of precision, one for each of the four weeks (see B.4).

This rather intensive multi-year series of precision evaluation programmes (1985 to 2007) was carried out because hardness is a very frequently used test in the rubber industry. Thus it is important to fully evaluate this type of testing.

All calculations to provide repeatability and reproducibility values were performed in accordance with ISO/TR 9272. Precision concepts and nomenclature are also given in ISO/TR 9272.

Annex C gives guidance on the use of repeatability and reproducibility values.

NOTE The 1986 edition of ISO/TR 9272 was used for the ITPs carried out between 1985 and 1989, but the later or current edition was used for the 2004 and the 2007 programmes.

B.2 Precision results from the ITPs carried out between 1985 and 1989

B.2.1 Programme details

B.2.1.1 Five ITPs were organized and conducted by Statens Provningsanstalt (Sweden) between 1985 and 1989. Cured test pieces were prepared in one laboratory and sent to all the participants. The details of the five ITPs are as follows.

a) **Medium-hardness rubbers (method N)**. Four rubber compounds, nominal hardness range 30 IRHD to 85 IRHD, 26 laboratories. Three determinations (measurements) of hardness on each compound on each of two days, one week apart, using method N. The median of the three was used as the “test result” for the precision analysis.

b) **Medium-hardness rubbers (method M)**. Four rubber compounds, nominal hardness range 30 IRHD to 85 IRHD, 26 laboratories. Three determinations (measurements) of hardness on each of two days, one week apart, using method M. The median of the three was used as the “test result” for the precision analysis.

c) **High-hardness rubbers (method N)**. Three rubber compounds, nominal hardness range 85 IRHD to 100 IRHD, 12 laboratories. Five determinations (measurements) of hardness on each of two days, one week apart, using method N. The median of the five was used as the “test result” for the precision analysis.

d) **High-hardness rubbers (method H)**. Three rubber compounds, nominal hardness range 85 IRHD to 100 IRHD, 12 laboratories. Three determinations (measurements) of hardness on each of two days, one week apart, using method H. The median of the three was used as the “test result” for the precision analysis.

e) **Low-hardness rubber (method L)**. One rubber compound of nominally low hardness, five laboratories. Three determinations (measurements) of hardness on each of two days, one week apart, using method L. The median of the three was used as the “test result” for the precision analysis.

B.2.1.2 The precision assessments are type 1 (cured, prepared test pieces circulated) and the time for repeatability and reproducibility is on a scale of days. For the low-hardness rubber, method L, due to the small number of laboratories in the precision evaluation programme the tabulated precision results should be used with caution.

B.2.2 Precision results (1985 to 1989)

B.2.2.1 The precision results are given in Table B.1 for medium-hardness rubbers using method N, Table B.2 for medium-hardness rubbers using method M, Table B.3 for high-hardness rubbers using method N, Table B.4 for high-hardness rubbers using method H, and Table B.5 for the low-hardness rubber using method L.

B.2.2.2 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.

Table B.1 — Type 1 precision, medium-hardness rubbers, method N

Material	Average value	Within lab		Between labs	
		r	(*r*)	*R*	(*R*)
A	31,5	1,29	4,08	2,98	9,47
B	47,1	1,23	2,61	2,68	5,68
C	66,6	1,65	2,48	4,47	6,71
D	86,5	2,32	2,68	3,49	4,03
Pooled values	58,3	1,68	2,89	3,49	5,99

Explanation of symbols:

r = absolute repeatability, in measurement units;

(*r*) = relative repeatability, in percent;

R = absolute reproducibility, in measurement units;

(*R*) = relative reproducibility, in percent.

Table B.2 — Type 1 precision, medium-hardness rubbers, method M

Material	Average value	Within lab		Between labs	
		r	(r)	R	(R)
A	36,6	1,57	4,29	5,82	15,9
B	50,9	2,31	4,55	5,44	10,7
C	64,9	4,89	7,54	7,47	11,5
D	88,6	4,76	5,38	6,80	7,68
Pooled values	60,3	3,71	6,16	6,43	10,7

Explanation of symbols:

r = absolute repeatability, in measurement units;

(r) = relative repeatability, in percent;

R = absolute reproducibility, in measurement units;

(R) = relative reproducibility, in percent.

Table B.3 — Type 1 precision, high-hardness rubbers, method N

Material	Average value	Within lab		Between labs	
		r	(r)	R	(R)
A	85,8	0,78	0,91	3,53	4,11
B	93,4	1,11	1,19	2,96	3,17
C	98,5	0,33	0,34	1,45	1,47
Pooled values	92,6	0,81	0,87	2,86	3,09

Explanation of symbols:

r = absolute repeatability, in measurement units;

(r) = relative repeatability, in percent;

R = absolute reproducibility, in measurement units;

(R) = relative reproducibility, in percent.

Table B.4 — Type 1 precision, high-hardness rubbers, method H

Material	Average value	Within lab		Between labs	
		r	(r)	R	(R)
A	87,0	0,96	1,03	3,12	3,41
B	94,2	1,00	1,07	2,15	2,31
C	98,7	0,71	0,76	1,03	1,10
Pooled values	93,3	0,75	0,90	2,29	2,46

Explanation of symbols:

r = absolute repeatability, in measurement units;

(r) = relative repeatability, in percent;

R = absolute reproducibility, in measurement units;

(R) = relative reproducibility, in percent.

Table B.5 — Type 1 precision, low-hardness rubber, method L

Material	Average value	Within lab		Between labs	
		r	(r)	R	(R)
A	33,0	0,20	0,61	2,00	6,04

Explanation of symbols:

r = absolute repeatability, in measurement units;

(r) = relative repeatability, in percent;

R = absolute reproducibility, in measurement units;

(R) = relative reproducibility, in percent.

B.3 Precision results from the ITP carried out in 2004

B.3.1 Programme details

B.3.1.1 An ITP for the evaluation of the precision of micro hardness tests was conducted in 2004, using the procedures and guidelines described in ISO/TR 9272:2005. Precision for the microtest method was determined for the purposes of comparison with Shore AM hardness determined in accordance with ISO 7619-1.

B.3.1.2 A type 1 precision was evaluated (for both methods), using cured test pieces prepared from four different rubber compounds, A, B, C and D (with a range of hardnesses), 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.

B.3.1.3 For each compound, three test pieces were furnished and five hardness measurements were made on each of the three test pieces by each of two operators. For each operator, a median value was selected for the 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 the test result data, i.e. two test result values per laboratory.

B.3.1.4 The ISO/TR 9272:2005 option 2 outlier treatment procedure, outlier replacement, was used since the ITP had only the minimum number of participating laboratories (six). 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 of this concept and for other details.

B.3.1.5 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.

B.3.2 Precision results (2004)

B.3.2.1 The precision results obtained for the IRHD microtest method are given in Table B.6, with the materials listed in increasing order of hardness. The results are given in terms of both the absolute precision, r or R, and the relative precision, (r) and (R).

B.3.2.2 The results of the precision analysis given in Table B.6 for the IRHD microtest method and those given in ISO 7619-1:2004/Amd.1 for the Shore AM method indicate that there is no pronounced trend for r or R against hardness level over the IRHD 46 to IRHD 74 range. The repeatabilities found for the Shore AM method, $r = 0{,}88$ and $(r) = 1{,}47$, and for the IRHD microtest method, $r = 1{,}14$ and $(r) = 2{,}04$, are reasonably similar. However, the reproducibility of the two hardness measurement methods is substantially different. For Shore AM, $R = 5{,}08$ and $(R) = 8{,}98$, whereas for the IRHD microtest method $R = 2{,}20$ and $(R) = 3{,}85$.

B.3.2.3 The reproducibility parameters R and (R) for IRHD are 43 % of the values for Shore AM, indicating much better between-laboratory agreement for the IRHD measurements.

Table B.6 — Precision data for the IRHD microtest method

Material	Mean level	Within lab			Between labs			No. of labs[a]
		s_r	r	(r)	s_R	R	(R)	
B	45,6	0,404	1,13	2,48	0,954	2,67	5,85	6 (1)
C	53,9	0,469	1,31	2,43	0,583	1,63	3,03	6 (1)
A	63,7	0,605	1,7	2,66	0,728	2,04	3,2	6
D	74	0,149	0,416	0,57	0,875	2,45	3,31	6
Average			1,139	2,035		2,197 5	3,847 5	

Explanation of symbols:

s_r = within-laboratory standard deviation (in measurement units);

r = repeatability (in measurement units);

(r) = repeatability (in percent of mean level);

s_R = between-laboratory standard deviation (for total between-laboratory variation in measurement units);

R = reproducibility (in measurement units);

(R) = reproducibility (in percent of mean level).

[a] Number of option 2 outlier lab replacement values given in brackets.

B.4 Precision results from the ITP carried out in 2007

B.4.1 Programme details

B.4.1.1 An ITP for the evaluation of the precision of IRHD N, M, and L hardness tests as well as Shore A and D was conducted in 2007, using the procedures and guidelines described in ISO/TR 9272:2005. Precision for Shore A and D methods, which are covered by ISO 7619, was determined for the purposes of comparison with IRHD precision. See ISO 7619 for more details on hardness testing using Shore A and D procedures.

B.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 B.7 to B.11) for the actual hardness levels with the various hardness methods (IRHD and Shore).

B.4.1.3 The number of laboratories volunteering to participate was as follows for each test method (IRHD and Shore): 26 laboratories for IRHD N and Shore A; 15 laboratories for IRHD M; 18 laboratories for Shore D; and 7 laboratories for IRHD L.

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 B.7 to B.11). 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 TR 9272:2005.

B.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.

B.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.

B.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 B.7 to B.11, represent more reliable or realistic values compared to the usual ITP results which constitute a "single point in time" estimate of precision.

B.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.

B.4.2 Precision results (2007)

B.4.2.1 The precision results obtained for the 2007 ITP are given in Tables B.7 to B.11. Precision is given for IRHD N in Table B.7; precision for IRHD M in Table B.8; precision for IRHD L in Table B.9; precision for Shore A in Table B.10 and precision for Shore D in Table B.11. The precision for IRHD L is to be used with caution since it is based on only four laboratories. The results are given in terms of both the absolute precision, r or R, and the relative precision, (r) and (R).

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

B.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 B.7 — Precision data for the IRHD N test method

Material	Mean level	Within lab			Between labs			No. of labs[a]
		s_r	r	(r)	s_R	R	(R)	
RM 123	45,0	0,197	0,550	1,22	0,717	2,01	4,46	14
RM 124	58,2	0,233	0,650	1,12	0,654	1,83	3,15	13
RM 126	84,1	0,541	1,520	1,80	0,916	2,56	3,05	14
Average[b]		0,324	0,907	1,38	0,762	2,13	3,55	

Explanation of symbols:

s_r = within-laboratory standard deviation (in measurement units);

r = repeatability (in measurement units);

(r) = repeatability (in percent of mean level);

s_R = between-laboratory standard deviation (for total between-laboratory variation in measurement units);

R = reproducibility (in measurement units);

(R) = reproducibility (in percent of mean level).

a Average number of laboratories after outliers have been deleted.

b Simple mean values for comparison.

Table B.8 — Precision data for the IRHD M test method

Material	Mean level	Within lab			Between labs			No. of labs[a]
		s_r	r	(r)	s_R	R	(R)	
RM 122	34,08	0,331	0,930	2,72	0,683	1,91	5,61	11
RM 124	58,09	0,605	1,690	2,92	1,068	2,99	5,15	11
RM 125	80,54	1,264	3,540	4,40	2,21	6,20	7,70	11
Average[b]		0,733	2,053	3,35	1,32	3,70	6,15	

Explanation of symbols:

s_r = within-laboratory standard deviation (in measurement units);

r = repeatability (in measurement units);

(r) = repeatability (in percent of mean level);

s_R = between-laboratory standard deviation (for total between-laboratory variation in measurement units);

R = reproducibility (in measurement units);

(R) = reproducibility (in percent of mean level).

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

[b] Simple mean values for comparison.

Table B.9 — Precision data for the IRHD L test method

Material	Mean level	Within lab			Between labs			No. of labs[a]
		s_r	r	(r)	s_R	R	(R)	
RM 121	34,0	0,221	0,62	1,82	0,310	0,87	2,55	4

Explanation of symbols:

s_r = within-laboratory standard deviation (in measurement units);

r = repeatability (in measurement units);

(r) = repeatability (in percent of mean level);

s_R = between-laboratory standard deviation (for total between-laboratory variation in measurement units);

R = reproducibility (in measurement units);

(R) = reproducibility (in percent of mean level).

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

Table B.10 — Precision data for the Shore A test method

Material	Mean level	Within lab			Between labs			No. of labs[a]
		s_r	r	(r)	s_R	R	(R)	
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[b]		0,312	0,867	1,51	0,718	2,01	3,75	

Explanation of symbols:

s_r = within-laboratory standard deviation (in measurement units);

r = repeatability (in measurement units);

(r) = repeatability (in percent of mean level);

s_R = between-laboratory standard deviation (for total between-laboratory variation in measurement units);

R = reproducibility (in measurement units);

(R) = reproducibility (in percent of mean level).

a Average number of laboratories after outliers have been deleted.
b Simple mean values for comparison.

Table B.11 — Precision data for the Shore D test method

Material	Mean level	Within lab			Between labs			No. of labs[a]
		s_r	r	(r)	s_R	R	(R)	
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[b]		0,493	1,380	4,10	0,898	2,52	7,70	

Explanation of symbols:

s_r = within-laboratory standard deviation (in measurement units);

r = repeatability (in measurement units);

(r) = repeatability (in percent of mean level);

s_R = between-laboratory standard deviation (for total between-laboratory variation in measurement units);

R = reproducibility (in measurement units);

(R) = reproducibility (in percent of mean level).

a Average number of laboratories after outliers have been deleted.
b Simple mean values for comparison.

Annex C
(informative)

Guidance for using precision results

C.1 The general procedure for using precision results is as follows, with the symbol $|x_1 - x_2|$ designating a positive difference in any two measurement values (i.e. without regard to sign).

C.2 Enter the appropriate precision table (for whatever test parameter is being considered) at an average value (of the measured parameter) nearest to the “test” data average under consideration. This line will give the applicable r, (r), R or (R) for use in the decision process.

C.3 With these r and (r) values, the following general repeatability statements may be used to make decisions.

a) For an absolute difference: the difference $|x_1 - x_2|$ between two test (value) averages, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability r on average not more than once in 20 cases.

b) For a percentage difference between two test (value) averages, the percentage difference is calculated as follows:

$$\frac{|x_1 - x_2|}{\frac{1}{2}(x_1 + x_2)} \times 100$$

between two test values, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability (r) on average not more than once in 20 cases.

C.4 With these R and (R) values, the following general reproducibility statements may be used to make decisions.

a) For an absolute difference: the absolute difference $|x_1 - x_2|$ between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility R not more than once in 20 cases.

b) For a percentage difference between two test (value) averages, the percentage difference is calculated as follows:

$$\frac{|x_1 - x_2|}{\frac{1}{2}(x_1 + x_2)} \times 100$$

between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility (R) not more than once in 20 cases.

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