INTERNATIONAL STANDARD

**ISO
4545-1**

Metallic materials — Knoop hardness test —

Part 1:
Test method

Matériaux métalliques — Essai de dureté Knoop —

Partie 1: Méthode d'essai

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Contents

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 4545-1 was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

ISO 4545-1 cancels and replaces ISO 4545:1993, which has been technically revised.

ISO 4545 consists of the following parts, under the general title *Metallic materials — Knoop hardness test*:

— *Part 1: Test method*

— *Part 2: Verification and calibration of testing machines*

— *Part 3: Calibration of reference blocks*

— *Part 4: Table of hardness values*

Introduction

The periodic checking of the testing machine described in informative Annex A is good metrological practice. It is intended to make this annex normative in the next revision of this part of ISO 4545.
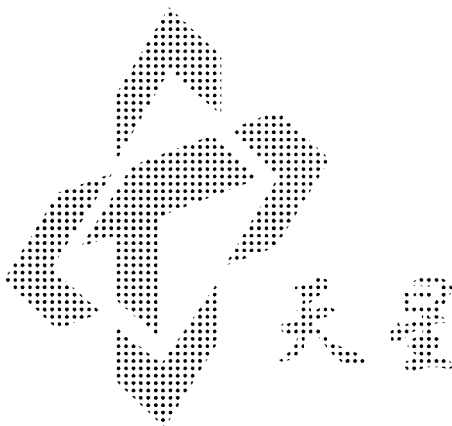
Metallic materials — Knoop hardness test —

Part 1:
Test method

1 Scope

This part of ISO 4545 specifies the Knoop hardness test method for metallic materials, for test forces from 0,098 07 N to 19,614 N. The method is recommended only for indentations with diagonals ⩾ 0,020 mm.

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 4545-2:2005, *Metallic materials — Knoop hardness test — Part 2: Verification and calibration of testing machines*

ISO 4545-3:2005, *Metallic materials — Knoop hardness test — Part 3: Calibration of reference blocks*

ISO 4545-4, *Metallic materials — Knoop hardness test — Part 4: Table of hardness values*

3 Principle

A diamond indenter, in the form of a rhombic-based pyramid with angles α and β between opposite faces respectively equal to 172,5° and 130° at the vertex, is forced into the surface of a test piece followed by measurement of the long diagonal, d, of the indentation remaining in the surface after removal of the test force, F (see Figures 1 and 2).
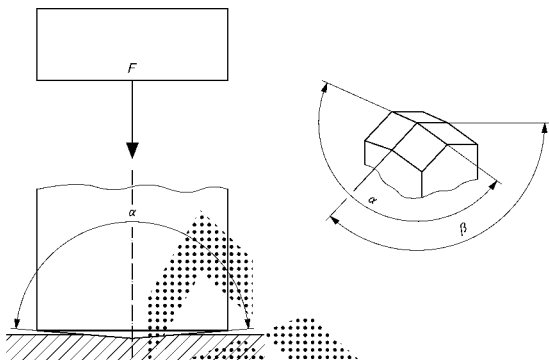
Figure 1 — Principle of the test and indenter geometry
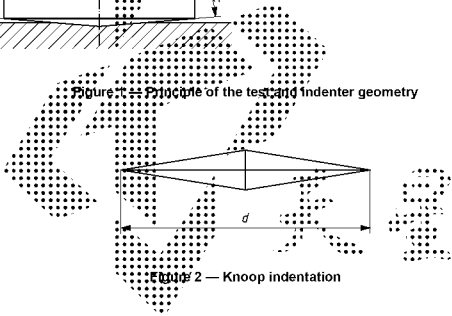
Figure 2 — Knoop indentation

The Knoop hardness is proportional to the quotient obtained by dividing the test force by the projected area of the indentation, which is assumed to be a rhombic-based pyramid, and having at the vertex the same angles as the indenter.

4 Symbols and abbreviated terms

4.1   See Table 1 and Figures 1 and 2.

4.2   The following is an example of the designation of Knoop hardness.

EXAMPLE

- Duration time of test force (20 s) if not within the specified range (10 s to 15 s)
- approximate kgf equivalent value of applied test force, where 1 kgf = 9,806 65 N
- hardness symbol
- Knoop hardness value
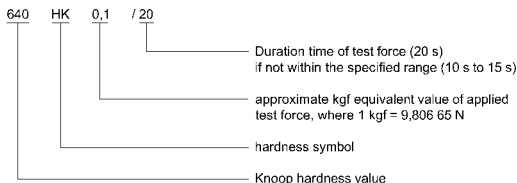
Table 1 — Symbols and abbreviated terms

| Symbol/ abbreviated term	Designation
F	Test force, in newtons
d	Length of the long diagonal, in millimetres
c	Indenter constant, relating projected area of the indentation to the square of the length of the long diagonal
	Indenter constant $c = \dfrac{\tan\dfrac{\beta}{2}}{2\tan\dfrac{\alpha}{2}}$, ideally $c = 0,070\,28$
	where α and β are the angles between the opposite edges at the vertex of the diamond pyramid (see Figure 1)
HK	Knoop hardness = Constant × $\dfrac{\text{Test force}}{\text{Projected area of indentation}}$
	$= 0,102 \times \dfrac{F}{c\,d^2} = 1,451\,\dfrac{F}{d^2}$
NOTE	Constant $= 0,102 = \dfrac{1}{9,806\,65}$, where 9,806 65 is the conversion factor from kgf to N.

5 Apparatus

5.1 Testing machine, capable of applying a predetermined force or forces from 0,098 07 N to 19,614 N, in accordance with ISO 4545-2.

5.2 Indenter, a diamond in the shape of a rhombic-based pyramid, as specified in ISO 4545-2.

5.3 Measuring system, as specified in ISO 4545-2.

The optical portion of the measuring system should have Kohler illumination. See Annex A of ISO 4545-3:2005.

Magnifications should be provided so that the diagonal can be enlarged to greater than 25 % but less than 75 % of the field of view.

The measuring system should report the diagonal lengths in 0,1 μm increments.

NOTE   A suggested procedure for checking the testing machine by the user is given in Annex A.

6 Test piece

6.1 The test shall be carried out on a polished surface, which is smooth and even, free from oxide scale and foreign matter and, in particular, completely free from lubricants, unless otherwise specified in product standards The finish of the surface shall permit accurate determination of the diagonal length of the indentation.

6.2 Preparation shall be carried out in such a way that any alteration of the surface hardness, due to excessive heating or cold work, for example, is minimized.

6.3 Due to the small depth of Knoop hardness indentations, it is essential that special precautions be taken during preparation. It is recommended to use a polishing/electropolishing technique that is adapted to the material to be measured.

6.4 No deformation shall be visible at the back of the test piece after the test.

6.5 For test pieces of small cross-section or irregular shape, it may be necessary to provide some form of additional support, for example, mounting in plastic material. The test piece shall be adequately supported by the mounting medium so that the test piece does not move during the force application.

7 Procedure

7.1 The test is normally carried out at a temperature of $23 \pm 5 °C$. If the test is carried out at a temperature outside this range, it shall be noted in the test report.

7.2 The test forces given in Table 2 are recommended.

7.3 The test piece shall be placed on a rigid support. The support surfaces shall be clean and free from foreign matter (scale, oil, dirt, etc.). It is important that the test piece lies firmly on the support so that displacement cannot occur during the test.

7.4 Focus the measuring microscope so that the specimen surface can be observed.

7.5 Bring the indenter into contact with the test surface and apply the test force in a direction perpendicular to the surface, without shock or vibration, until the applied force attains the specified value. The approach velocity of the indenter shall be within the range of 15 µm/s to 70 µm/s. The time from the initial application of the force until the full test force is reached shall not exceed 10 s.

7.6 The duration of the test force shall be from 10 s to 15 s unless otherwise specified. For certain materials, a longer time for maintaining the force may be necessary to obtain consistent results; this time shall be applied with a tolerance of ± 2 s.

7.7 Throughout the test, the apparatus shall be protected from shock or vibration.

7.8 The minimum distance between the limit of any indentation and the edge of the test piece shall be at least 3 times the short diagonal of the indentation.

Table 2 — Test forces

Hardness symbol	Test force value, F	
	N	approximate kgf [a] equivalent
HK 0,01	0,098 07	0,010
HK 0,02	0,196 1	0,020
HK 0,025	0,245 2	0,025
HK 0,05	0,490 3	0,050
HK 0,1	0,980 7	0,100
HK 0,2	1,961	0,200
HK 0,3	2,942	0,300
HK 0,5	4,903	0,500
HK 1	9,807	1,000
HK 2	19,614	2,000
[a] Not an SI unit.		

7.9 The minimum distance between the limits of two adjacent indentations, oriented side-by-side, shall be at least 2,5 times the length of the short diagonal. For indentations oriented end-to-end, the minimum distance between the limits of two adjacent indentations shall be at least once the length of the long diagonal. If two indentations differ in size, the minimum spacing shall be based on the short diagonal of the larger indentation.

7.10 The length of the long diagonal shall be measured and used for the calculation of the Knoop hardness. For all tests, the perimeter of the indentation shall be clearly defined in the field of view of the microscope.

NOTE   In general, decreasing the test force increases the scatter of the results of the measurements. This is particularly true for low force Knoop hardness tests where the principal limitation will arise in the measurement of the long diagonal of the indentation. For Knoop hardness, the accuracy of the determination of the long diagonal length is unlikely to be better than ± 0,001 mm.

Magnifications should be provided so that the diagonal can be enlarged to greater than 25 %, but less than 75 % of the field of view.

7.11 The Knoop hardness value shall be calculated by the formula given in Table 1, or by using the tables given in ISO 4545-4.

7.12 If one half of the long diagonal differs by more than 10 % from the other half, check the parallelism between the supporting plane and the measuring plane of the specimen and eventually the alignment of the indenter to the specimen. Test results with deviations greater than 10 % should be discarded.

8   Uncertainty of the results

A complete evaluation of the uncertainty should be done according to the *Guide to the expression of uncertainty in measurement* (GUM) [1].

Independent of the type of sources, for hardness there are two possibilities for the determination of the uncertainty.

— One possibility is based on the evaluation of all relevant sources appearing during a direct calibration. As a reference, an EA guideline [2] is available.

— The other possibility is based on indirect calibration using a hardness reference block [abbreviated as CRM (certified reference material)] (see [3-5] in the Bibliography). A guideline for the determination is given in Annex B.

It may not always be possible to quantify all the identified contributions to the uncertainty. In this case, an estimate of type A standard uncertainty may be obtained from the statistical analysis of repeated indentations into the test piece. Care should be taken if standard uncertainties of type A and B are summarised, that the contributions are not counted twice (see Clause 4 of GUM:1993).

9   Test report

The test report shall include the following information:

a)   a reference to this part of ISO 4545;

b)   all information necessary for identification of the test sample;

c)   the result obtained;

d)   all operations not specified in this part of ISO 4545 or regarded as optional;

e)   details of any circumstances, which may have affected the result;

f)   the temperature of the test, if it is outside the range specified in 7.1.

There is no general process of accurately converting Knoop hardness values into other scales of hardness or into tensile strength. Such conversions therefore should be avoided, unless a reliable basis for conversion can be obtained by comparison tests.

NOTE   A strict comparison of hardness values is only possible at identical test forces.

Annex A
(informative)

Procedure for periodic checking of the testing machine by the user

A check of the machine should be carried out on each day that the machine is used, at approximately each hardness level and for each range or scale that is to be used.

Prior to making the check, the measuring system should be indirectly verified (for each range/scale and hardness level) using a reference indenter on a hardness reference block, calibrated in accordance with ISO 4545-3. The measured dimension should agree with the certified value to within the greater of 0,5 % and 0,4 μm. If the measuring system fails the test, appropriate action should be taken.

The check involves at least one indentation being made on a hardness reference block, calibrated in accordance with ISO 4545-3. If the difference between the mean measured hardness and the block's certified value is within the permissible error limits given in 5.8 of ISO 4545-2:2005, the machine may be regarded as satisfactory. If not, an indirect verification should be performed.

A record of these results should be maintained over a period of time, and used to measure reproducibility and monitor drift of the machine.

Annex B
(informative)

Uncertainty of the measured hardness values

B.1 General requirements

The approach for determining uncertainty presented in this annex considers only those uncertainties associated with the overall measurement performance of the hardness testing machine with respect to the hardness reference blocks (abbreviated as CRM below). These performance uncertainties reflect the combined effect to all the separate uncertainties (indirect verification). Because of this approach, it is important that the individual machine components are operating within the tolerances. It is strongly recommended that this procedure be applied for a maximum of one year after the successful passing of a direct verification.

Figure B.1 shows the four-level structure of the metrological chain necessary to define and disseminate hardness scales. The chain starts at the **international level** using international definitions of the various hardness scales to carry out international intercomparisons. A number of *primary hardness standard machines* at the **national level** "produce" *primary hardness reference blocks* for the calibration laboratory level. Naturally, direct calibration and the verification of these machines should be at the highest possible accuracy.
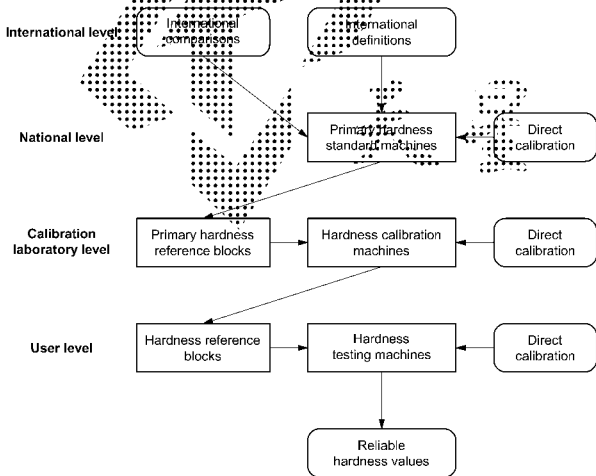

Figure B.1 — Structure of the metrological chain for the definition and dissemination of hardness scales

B.2 General procedure

The procedure calculates a combined uncertainty u_1 by the Root-Squared-Sum-Method (RSS) out of the different sources given in Table B.1. The expanded uncertainty, U, is derived from u_1 by multiplying with the coverage factor $k = 2$. Table B.1 contains all symbols and their designation.

B.3 Bias of the machine

The bias, b, of a hardness testing machine (also named error) which is derived from the difference between

— the mean value of the five indentations during calibration of the hardness testing machine, and

— the calibration value of the hardness reference block,

can be implemented in different ways into the determination of uncertainty.

B.4 Procedures for calculating uncertainty: Hardness measurement values

B.4.1 Procedure without bias (method 1)

Method 1 (abbreviated as M1) is a simplified method which can be used without considering the systematic error of the hardness testing machine.

In M1, the error limit, that means the range in which the machine is allowed to differ from the reference standard is used to define the source u_E of the uncertainty. There is no correction of the hardness values with respect to the error.

The procedure for the determination of U is explained in Table B.1 (see [1, 2] in the Bibliography).

$$U_{corr} = k \cdot \sqrt{u_E^2 + u_{CRM}^2 + u_{\bar{x}}^2 + u_{ms}^2}$$ (B.1)

Where the result of the measurement is given by

$$\bar{X} = \bar{x} \pm U$$ (B.2)

B.4.2 Procedure with bias (method 2)

As an alternative to (M1), method 2 may be used, which is correlated with the conduct of a control chart. M2 may lead to smaller values of uncertainty.

The error b (step 10) can be expected to be a systematic effect. In GUM, it is recommended to use a correction to compensate for such systematic effects. This is the base of M2. The error is no longer in the calculation of the uncertainty but all determined hardness values have to be corrected by b or U_{corr} has to be increased by b. The procedure for the determination of U_{corr} is explained in Table B.1 (see [4, 5] in the Bibliography).

$$U_{corr} = k \cdot \sqrt{u_{CRM}^2 - u_H^2 + u_{\bar{x}}^2 - u_{ms}^2 + u_b^2}$$ (B.3)

Where the result of the measurement is given by

$$\bar{X}_{corr} = (\bar{x} + \bar{b}) \pm U_{corr}$$ (B.4)

or by

$$\overline{X}_{\text{ucorr}} = \overline{x} = \left(U_{\text{corr}} + \left|\overline{b}\right| \right)$$ (B.5)

depending on whether the bias (error) $\overline{b}$ is thought to be part of the mean value or of the uncertainty.

B.5 Expression of the result of measurement

For the expression of the result of measurement, the method used should be indicated. In general, as a result of the measurement, method 1 [Equation (B.2)] should be used (see also Table B.1, step 12).
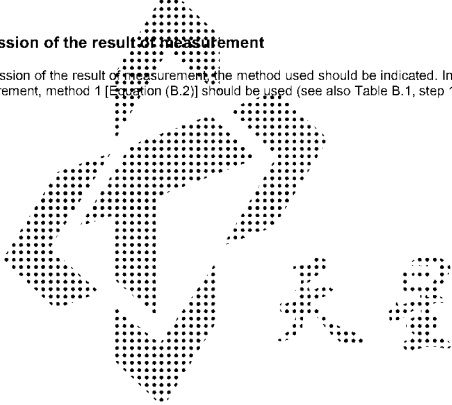
Table B.1 — Determination of the expanded uncertainty according to methods M1 and M2

Step Method	Sources of uncertainty	Symbols	Formula	Literature/Certificate	Example [.] = HV1
1 M1	Standard uncertainty according to the maximum permissible error	u_E	$u_E = \dfrac{2\pi_{E,2} \cdot \bar{X}_{CRM}}{2.8}$	Permissible error $\pi_{E,2}$ according to ISO 4545-2, related to d. $\bar{X}_{CRM}$ from calibration certificate. See Note 1.	$u_E = \dfrac{2 \times 0.02 \times 396.8}{2.8} = 5.67$
2 M1 M2	Standard uncertainty and mean value of hardness of CRM (for detailed calculation see ISO 4545-3:2005, Table B.4)	u_{CRM}, $\bar{X}_{CRM}$	$u_{CRM} = \dfrac{U_{CRM}}{2}$	u_{CRM}, $\bar{X}_{CRM}$ according to calibration certificate of CRM. See Note 2.	$u_{CRM} = \dfrac{12.00}{2} = 6.00$ $\bar{X}_{CRM} = 396.8$
3 M1 M2	Mean value and standard deviation of the measurement on CRM	$\bar{H}$, s_H	$\bar{H} = \dfrac{\sum\limits_{i=1}^{n} H_i}{n}$ $s_H = \sqrt{\dfrac{1}{n-1}\sum\limits_{i=1}^{n}(H_i - \bar{H})^2}$	$n = 5$ according to ISO 4545-2:2005 Clause 4. For the calculation of s_H the larger value of s_{H1} and s_{H2} will be taken.	Single measurements: *1) 402.6 – 404.7 – 403 – 400.9 – 399.2 $\bar{H}_1 = 402.1; s_{H1} = 2.1$ *2) 406.5 – 403 – 400.9 – 403.4 – 397.5 $\bar{H}_2 = 402.3; s_{H2} = 3.3$
4 M1 M2	Standard uncertainty of hardness testing machine when measuring CRM	u_H	$u_H = \dfrac{t \cdot s_H}{\sqrt{n}}$		$u_H = \dfrac{1.14 \times 3.3}{\sqrt{5}} = 1.70$
5 M1 M2	Mean value and standard deviation of the testing of a test piece	$\bar{x}$, s_x	$\bar{x} = \dfrac{\sum\limits_{j=1}^{n} x_j}{n}$ $s_x = \sqrt{\dfrac{1}{n-1}\sum ...}$	$n = 5$ 5 measurements on the test piece. See Note 3. If $r < s_x \to 0$. The certificate should state that the uncertainty applies only to the specific reading, not to the testpiece as a whole.	Single values 391 – 409 – 421 – 413 – 416 $\bar{x} = 410.0$ $s_x = 11.5$
6 M1 M2	Standard uncertainty when measuring a test piece	u_x	$u_x = \dfrac{t \cdot s_x}{\sqrt{n}}$	$t = 1.14$ for $n = 5$	$u_x = \dfrac{1.14 \times 11.5}{\sqrt{5}} = 5.86$

Table B.1 *(continued)*

Step Method	Sources of uncertainty	Symbols	Formula	Literature/Certificate	Example $LJ = HV1$
7 M2	Standard uncertainty according to the resolution of the length measuring system	u_{ms}	$u_{ms} = \dfrac{2H}{d} \cdot \dfrac{\delta_{ms}}{2\sqrt{3}}$	$\delta_{ms} = 0.0005$ mm $H = 410.0$ HK $d = 0.186$ mm	$u_{ms} = \dfrac{2 \times 410.0}{0.186} \cdot \dfrac{0.0005}{2 \times \sqrt{3}} = 0.64$
8 M2	Deviation of hardness testing machine from calibration value	$\bar{b}$	$\bar{b} = \bar{H} \cdot \bar{X}_{CRM}$	Steps 2 and 3. See Note 4.	$b_1 = 402.1 - 396.8 = 5.3$ $b_2 = 402.3 - 396.8 = 5.5$
9 M2	Standard deviation of the deviation b	s_b	$\bar{b} = \dfrac{\sum b_m}{2}$ $s_b = \sqrt{\dfrac{\sum (b_1 - \bar{b})^2}{n-1}}$	Step 8	$\bar{b} = 5.4$ $s_b = 0.13$
10 M2	Standard uncertainty of the determination of b. Can be determined only after the second series of measurements	u_b	$u_b = \dfrac{t \cdot s_b}{\sqrt{n \cdot m}}$	$t = 1.84$ for $n-2$ See Note 5.	$u_b = \dfrac{1.84 \times 0.13}{\sqrt{2}} = 0.17$
11 M1	Determination of the expanded uncertainty	U	$U = k \cdot \sqrt{u_E^2 + u_{CRM}^2 + u_{ms}^2 + u_{rms}^2 + u_h^2}$	Steps 1 to 11 $k = 2$	$U = 2 \times \sqrt{5.87^2 + 6.00^2 - 1.70^2 + 5.86^2 - 0.64^2}$ $U = 20.56$ HK
12 M1	Result of the measurement	$\bar{X}$	$\bar{X} = \bar{X} - U$	Steps 5 and 11	$\bar{X} = 410.0 - 20.6$ HK (M1)
13 M2	Determination of the corrected expanded uncertainty	U_{corr}	$U_{corr} = k \cdot \sqrt{u_{CRM}^2 + u_H^2 + u_{ms}^2 + u_{rms}^2 + u_b^2}$	Step 2 to 7 and 10 $k = 2$	$U_{corr} = 2 \times \sqrt{6.0^2 + 1.70^2 - 5.86^2 + 0.64^2 - 0.17^2}$ $U_{corr} = 17.16$ HK
14 M2	Result of the measurement with corrected mean value	$\bar{X}_{corr}$	$\bar{X}_{corr} = (\bar{X} + \bar{b}) \pm U_{corr}$	Steps 5, 8 and 13	$\bar{X}_{corr} = (415.4 \pm 17.2)$ HK (M2)

Table B.1 (continued)

Step Method	Sources of uncertainty	Symbols	Formula	Literature/Certificate	Example [.] = HV1
15 M2	Result of the measurement with corrected uncertainty	$\bar{X}_{w\,corr}$	$\bar{X}_{w\,corr} - \bar{x} \pm (U_{\,corr} \ldots)$	Steps 5, 8 and 13	$\bar{X}_{w\,corr} - (410,0 = 22,6)$ HK (M2)

NOTE 1 The factor 2,8 is derived from the determination of the standard uncertainty for a rectangular distribution.

NOTE 2 If necessary, the hardness change of the CRM has to be considered.

NOTE 3 If between the measurement of CRM and test piece the ... may influence ... be considered.

NOTE 4 If $0,8\,u_{F_{\bar{p},\gamma s}} < h < 1,0\,n_{F_{\gamma s}}$, the relationship of hardness values ... CRM ... considered.

NOTE 5 Because, for $n_m - 2$, the influence ... of the long-term change of b is not ... for critical applications ... may be necessary to raise the number of measurements n_m.

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[1] BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML, *Guide to the Expression of Uncertainty in Measurement (GUM)*, 1993[1)]

[2] EA 10-16, *Guidelines on the Estimation of Uncertainty in Hardness Measurements*, 2001

[3] GABAUER W., *Manual of Codes of Practice for the Determination of Uncertainties in Mechanical Tests on Metallic Materials*, The Estimation of Uncertainties in Hardness Measurements, Project, No. SMT4-CT97-2165, UNCERT COP 14: 2000

[4] GABAUER W., BINDER O., *Abschätzung der Messunsicherheit in der Härteprüfung unter Verwendung der indirekten Kalibriermethode*, DVM Werkstoffprüfung, Tagungsband 2000, S. pp. 255-261

[5] POLZIN T., SCHWENK D., *Estimation of Uncertainty of Hardness Testing; PC file for the determination*, Materialprüfung, 3, 2002 (44), pp. 64-71

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1) Corrected and reprinted: 1995.