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Metallic materials — Knoop hardness test —

Part 2:
Verification and calibration of testing machines

Matériaux métalliques — Essai de dureté Knoop —

Partie 2: Vérification et étalonnage des machines d'essai

Reference number
ISO 4545-2:2005(E)

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
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Foreword

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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 4545-2 was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

ISO 4545-2 cancels and replaces ISO 4546:1993, 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*

Metallic materials — Knoop hardness test —

Part 2: Verification and calibration of testing machines

1 Scope

This part of ISO 4545 specifies the method of verification of testing machines for determining Knoop hardness for metallic materials in accordance with ISO 4545-1-1. It covers test forces from 0,098 07 N to 19,614 N. The method is recommended only for indentations with diagonals ⩾ 0,020 mm.

It specifies a direct verification method for checking the main functions of the machine, and an indirect verification method suitable for the overall checking of the machine. The indirect verification method may be used on its own for periodic routine checking of the machine in service.

If a testing machine is also to be used for other methods of hardness testing, it should be verified independently for each method.

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 376:2004, *Metallic materials — Calibration of force-proving instuments used for the verification of uniaxial testing machines*

ISO 4545-1:2005, *Metallic materials — Knoop hardness test — Part 1: Test method*

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

3 General conditions

Before a Knoop hardness testing machine is verified, it shall be checked to ensure that it is properly set up in accordance with the manufacturer's instructions.

Especially, it should be checked that:

a) the mount holding the indenter is capable of moving freely without any friction or excess side play;

b) the indenter is firmly mounted in the mount;

c) the test force can be applied and removed without shock or vibration and in such a manner that the readings are not influenced;

d) the measuring system is integral with the machine:

- the change in mode from the application and removal of the test force to the measuring mode does not influence the readings,
- illumination does not affect the readings,
- the centre of the indentation is near the centre of the field of view.

4 Direct verification

4.1 General

4.1.1 Direct verification should be carried out at a temperature of (23 ± 5) °C. If the verification is carried out at a temperature outside this range, it shall be noted in the verification report.

4.1.2 The instruments used for verification and calibration shall be traceable to national standards.

4.1.3 Direct verification involves:

a) calibration of the test force;

b) verification of the indenter;

c) calibration of the measuring system;

d) verification of the testing cycle.

4.2 Calibration of the test force

4.2.1 Each test force used (see Table 2 in ISO 4545-1:2005), within the working range of the testing machine, shall be measured.

4.2.2 The test force shall be measured by one of the following two methods:

- by means of an elastic proving device in accordance with ISO 376:2004, class 1, or
- by balancing against a force, accurate to ± 0,2 %, applied by means of calibrated masses or another method with the same accuracy.

4.2.3 Three readings shall be taken for each test force. Immediately before each reading is taken, the indenter shall be moved in the same direction as during the test. All readings shall be within the tolerances defined in Table 1.

Table 1 — Test-force tolerances

Test force, F N	**Tolerance** %
$0{,}098\,07 \leqslant F < 1{,}961$	± 1,5
$1{,}961 \leqslant F \leqslant 19{,}614$	± 1,0

4.3 Verification of the indenter

4.3.1 The four faces of the diamond pyramid shall be polished and free from surface defects.

4.3.2 Verification of the shape of the indenter can be made by direct measurement or optical measurement. The device used for the verification shall be accurate to within ± 0,07°.

4.3.3 The angle α between the opposite edges at the vertex of the diamond pyramid shall be (172,5 ± 0,1)° (see Figure 1).

4.3.4 The angle β between the opposite edges at the vertex of the diamond pyramid shall be (130 ± 1,0)° (see Figure 1).

4.3.5 The indenter constant c (see ISO 4545-1:2005, Table 1) shall be within 1,0 % of the ideal value 0,070 28, (0,069 58 $\leqslant c \leqslant$ 0,070 98).

NOTE To achieve the tolerances for the indenter constant c, the values of angle α and/or angle β may be kept to closer tolerances than given above.

4.3.6 The angle between the axis of the diamond pyramid and the axis of the indenter holder (normal to the seating surface) shall be within ± 0,5°.

4.3.7 The four faces shall meet at a common point. The maximum permissible length of the line of conjunction between opposite faces shall be less than 1,0 µm (see Figure 2).

NOTE For indents less than 0,020 mm, the maximum permissible length of the line of conjunction should be proportionally less. The line of conjunction may be determined by measuring an indentation.

4.4 Calibration of the measuring system

4.4.1 The system for measuring the long diagonal of the indentation shall be calibrated at each magnification to be used against an accurately ruled line scale (object micrometer) or system of equivalent accuracy. The errors of the line scale shall be known within an uncertainty of 0,1 µm or 0,05 %, whichever is greater.

4.4.2 The measuring system shall be verified by measurements made on a stage micrometer at a minimum of five intervals over each working range.

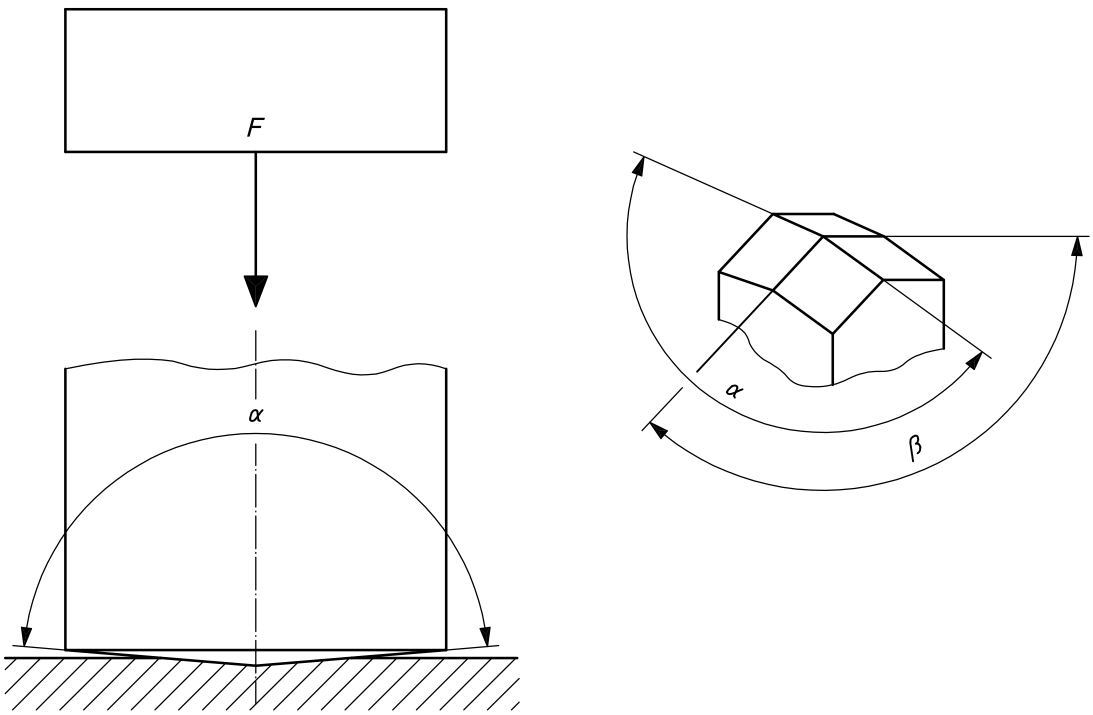

Figure 1 — Principle of the test and indenter geometry

Dimensions in µm

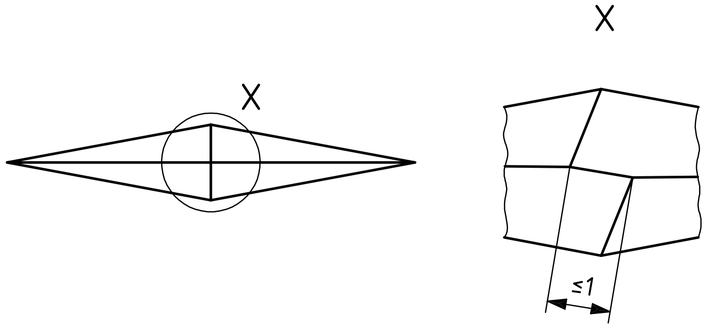

Figure 2 — Line of conjunction on the top of the indenter (schematic)

4.4.3 The maximum permissible error of the measuring system shall be ± 0,5 % or 0,4 µm, whichever is greater. If necessary, a calibration factor can be applied to comply with this tolerance.

4.5 Verification of the testing cycle

The testing cycle shall be timed with an uncertainty of 1 s and shall conform to the testing cycle of ISO 4545-1.

5 Indirect verification

5.1 Indirect verification should be carried out at a temperature of (23 ± 5) °C by means of reference blocks calibrated in accordance with ISO 4545-3. If the verification is carried out at a temperature outside this range, it shall be noted in the verification report.

5.2 On each reference block, measure the reference indentation. For each block, the difference between the mean measured value and the certified long diagonal shall not exceed the greater of 0,5 % and 0,4 µm.

5.3 When verifying testing machines used for several test forces, all used forces shall be chosen. One of the forces shall be the lowest force used and the other force shall be chosen within the upper half of those used. For each test force chosen, two different reference blocks shall be chosen within the range for which the machine is used. The ratio of the hardness values for the two blocks shall be equal to or greater than 2.

5.4 When verifying testing machines used for only one test force, three reference blocks shall be used, uniformly distributed over the range of the machine.

5.5 On each reference block, five indentations shall be made and measured. The tests shall be carried out in accordance with ISO 4545-1.

5.6 For each reference block, let d_1, d_2, …, d_5 be the values of the measured diagonals of the indentations, arranged in increasing order of magnitude, and

$$\bar{d} = \frac{d_1 + d_2 + \ldots + d_5}{5} \quad (1)$$

5.7 The repeatability r of the testing machine, under the particular verification conditions, is calculated as:

$$r = d_5 - d_1 \quad (2)$$

The repeatability, expressed as a percentage of $\bar{d}$, is calculated as:

$$r_{rel} = 100 \frac{d_5 - d_1}{\bar{d}} \quad (3)$$

The repeatability of the testing machine is satisfactory if $r \leqslant 0{,}001$ mm. If $r > 0{,}001$ mm, the repeatability of the testing machine is satisfactory when r_{rel} is less than or equal to the percentages indicated in Table 2.

Table 2 — Relative repeatability

Hardness range of standardized test blocks	Test force N	Maximum permissible r_{rel} %
$100 \leqslant$ HK $\leqslant 250$ $250 <$ HK $\leqslant 650$ HK > 650	$0{,}098\ 07 \leqslant F \leqslant 4{,}903$	9 5 4
$100 \leqslant$ HK $\leqslant 250$ $250 <$ HK $\leqslant 650$ HK > 650	$4{,}903 < F \leqslant 19{,}614$	8 5 4
HK: Knoop hardness		

5.8 The error, E, of the testing machine under the particular verification conditions is calculated by the following formula:

$$E = \bar{d} - d_c \quad (4)$$

The percent error, E_{rel}, is calculated by the following equation:

$$E_{rel} = 100 \frac{\bar{d} - d_c}{d_c} \quad (5)$$

where d_c is the reported certified mean diagonal length for the reference block, in millimetres.

The error of the testing machine is satisfactory if $E \leqslant \pm\ 0{,}000\ 5$ mm. If $E > 0{,}000\ 5$ mm, the error of the testing machine is satisfactory when $E_{rel} \leqslant \pm\ 2$ %.

5.9 The determination of the uncertainty of measurement of the calibration results of the hardness testing machine is given in Annex B.

6 Intervals between verifications

The specifications for the direct verifications of hardness testing machines are given in Table 3.

Indirect verification shall be performed at least once every 12 months and after a direct verification has been performed.

Table 3 — Direct verifications of hardness testing machines

Requirements of verification	Force	Measuring system	Test cycle	Indenter [a]
before setting to work first time	x	x	x	x
after dismantling and reassembling, if force, measuring system or test cycle are affected	x	x	x	
failure of indirect verification[b]	x	x	x	
indirect verification > 14 months ago	x	x	x	

[a] In addition, it is recommended that the indenter be directly verified after 2 years of use.

[b] Direct verification of these parameters may be carried out sequentially (until the machine passes indirect verification) and is not required if it can be demonstrated (e.g. by tests with a reference indenter) that the indenter was the cause of the failure.

7 Verification report/calibration certificate

The verification report/calibration certificate shall contain the following information:

a) a reference to this part of ISO 4545;

b) method of verification (direct and/or indirect);

c) identification data of the hardness testing machine;

d) means of verification (reference blocks, elastic proving devices, etc.);

e) test force(s) used;

f) hardness values of standardized blocks used;

g) verification temperature, if it is outside the range specified in Clause 4;

h) the result obtained;

i) date of verification and reference to the verification institution;

j) uncertainty of the verification result.

Annex A
(informative)

Notes on diamond indenters

Experience has shown that a number of initially satisfactory indenters can become defective after use for a comparatively short time. This is due to small cracks, pits or other flaws in the surface. If such faults are detected in time, regrinding may reclaim many indenters. If not, any small defects on the surface rapidly worsen and make the indenter useless.

Therefore,

— the condition of indenters should be monitored by visually checking the appearance of the indentation on a reference block, each day the testing machine is used;

— the verification of the indenter is no longer valid when the indenter shows defects;

— reground or otherwise repaired indenters shall meet all of the requirements of 4.3.

Annex B
(informative)

Uncertainty of measurement of the calibration results of the hardness testing machine

The metrological chain necessary to define and disseminate hardness scales is shown in Figure B.1 in ISO 4545-1:2005.

B.1 Direct calibration of the hardness testing machine

B.1.1 Calibration of the test force

The combined relative standard uncertainty of the test force calibration is calculated according to the following equation:

$$u_F = \sqrt{u_{FRS}^2 + u_{FHTM}^2} \quad \text{(B.1)}$$

where

u_{FRS} is the relative uncertainty of measurement of the force transducer (from calibration certificate);

u_{FHTM} is the relative standard uncertainty of the test force generated by the hardness testing machine.

The uncertainty of measurement of the reference instrument, force transducer, is indicated in the corresponding calibration certificate. The influence quantities, like

— temperature dependence,

— long-term stability, and

— interpolation deviation,

should be considered for critical applications. Depending on the design of the force transducer, the rotational position of the transducer related to the indenter axis of the hardness testing machine should be considered.

EXAMPLE

Uncertainty of measurement of the force transducer (from calibration certificate): U_{FRS} = 0,24 % (k = 2)

Calibration value of the force transducer F_{RS} = 9,806 7 N

Table B.1 — Results of the test force calibration

Number of height position for test force calibration	Series 1, F_1 N	Series 2, F_2 N	Series 3, F_3 N	Mean value $\bar{F}$, N	Relative deviation ΔF_{rel}, %	Relative standard measurement uncertainty u_{FHTM}, %
1	9,809	9,815	9,822	9,815	0,08	0,04

where

$$\Delta F_{\text{rel}} = \frac{F_{\text{RS}} - \overline{F}}{\overline{F}} \quad \text{(B.2)}$$

$$u_{\text{FHTM}} = \frac{s_{\text{F}i}}{\overline{F}} \cdot \frac{1}{\sqrt{n}}, (n = 3) \quad \text{(B.3)}$$

$s_{\text{F}i}$ is the standard deviation of the test-force indication values in the i-th height position

Table B.2 — Calculation of the uncertainty of measurement of the test force

Quantity X_i	Estimated value x_i	Relative limit values a_i	Distribution type	Relative standard measurement uncertainty $u(x_i)$	Sensitivity coefficient c_i	Relative uncertainty contribution $u_i(H)$
u_{FRS}	294,2 N		Normal	$1,2 \times 10^{-3}$	1	$1,2 \times 10^{-3}$
u_{FHTM}			Normal	$4,0 \times 10^{-4}$	1	$4,0 \times 10^{-4}$
Relative combined standard uncertainty $u(F)$						$1,26 \times 10^{-3}$
Relative expanded uncertainty of measurement $U(F)$ ($k = 2$)						$2,5 \times 10^{-3}$

Table B.3 — Calculation of the maximum relative deviation of the test force including the uncertainty of measurement of the reference instrument

Relative deviation of test force ΔF_{rel}, %	Expanded relative measurement uncertainty of test force U_{F}, %	Max. relative deviation of test force including measurement uncertainty of reference instrument ΔF_{max}, %
0,08	0,25	0,33

where

$$\Delta F_{\text{max}} = \left|\Delta F_{\text{rel}}\right| + U_{\text{F}} \quad \text{(B.4)}$$

The result of the example means that the deviation of the test forces including the uncertainty of measurement of the reference instrument specified in 4.2.3, amounting to ± 1,0 % is complied with.

B.1.2 Calibration of the optical measuring system

The combined relative standard uncertainty of the reference instrument for the measuring system is calculated as follows:

$$u_{\text{L}} = \sqrt{u_{\text{LRS}}^2 + u_{\text{ms}}^2 + u_{\text{LHTM}}^2} \quad \text{(B.5)}$$

where

u_{LRS} is the relative uncertainty of measurement of the object micrometer (reference standard) from the calibration certificate for $k = 1$;

u_{ms} is the uncertainty of measurement due to the resolution of the measuring system;

u_{LHTM} is the relative standard uncertainty of measurement of the hardness testing machine.

The uncertainty of measurement of the reference instrument for the optical measuring system, the object micrometer, is indicated in the corresponding calibration certificate. The influence quantities, for example,

- temperature dependence,
- longterm stability, and
- interpolation deviation,

do not exert an essential influence on the uncertainty of measurement of the object micrometer.

EXAMPLE

Uncertainty of measurement of the object micrometer: $U_{\mathrm{LRS}} = 0{,}000\,5$ mm ($k = 2$)

Resolution of the measuring system $\delta_{\mathrm{ms}} = 0{,}1$ µm

Table B.4 — Results of the calibration of the measuring system

Indication value of the object micrometer L_{RS}, mm	Series 1, L mm	Series 2, L_2 mm	Series 3, L_3, mm	Mean value, $\bar{L}$ mm	Relative deviation ΔL_{rel}, %	Relative standard measurement uncertainty u_{LHTM}, %
0,05	0,050 1	0,050 0	0,050 1	0,050 1	0,13	0,07
0,10	0,100 2	0,100 0	0,100 1	0,100 1	0,10	0,06
0,20	0,200 1	0,199 5	0,200 1	0,199 9	–0,05	0,10
0,30	0,299 7	0,300 1	0,300 1	0,300 0	–0,01	0,01
0,40	0,400 2	0,400 9	0,400 7	0,400 6	0,15	0,05

where

$$u_{\mathrm{LHTM}} = \frac{s_{\mathrm{L}i}}{\bar{L}} \cdot \frac{1}{\sqrt{n}}, \ (n = 3) \tag{B.6}$$

$$\Delta L_{\mathrm{rel}} = \frac{\bar{L} - L_{\mathrm{RS}}}{L_{\mathrm{RS}}} \tag{B.7}$$

$s_{\mathrm{L}i}$ is the standard deviation of the length indication values for the i-th indication value of the object micrometer.

Table B.5 — Calculation of the uncertainty of measurement of the measuring system

Quantity X_i	Estimated value x_i	Limit value a_i	Distribution type	Relative standard measurement uncertainty $u(x_i)$	Sensitivity coefficient c_i	Relative uncertainty contribution $u_i(H)$
$u_{\mathrm{LRS\ rel}}$	0,40 mm		Normal	$6{,}25 \times 10^{-4}$	1	$6{,}25 \times 10^{-4}$
$u_{\mathrm{ms\ rel}}$		$3{,}5 \times 10^{-4}$	Rectangular	$0{,}7 \times 10^{-4}$	1	$0{,}7 \times 10^{-4}$
u_{LHTM}	0,40 mm		Normal	$10{,}0 \times 10^{-4}$	1	$10{,}0 \times 10^{-4}$
Relative combined uncertainty of measurement u_{L}, %						0,12
Relative expanded uncertainty of measurement U_{L} ($k = 2$), %						0,24

Table B.6 — Calculation of the maximum relative deviation of the measuring system including the uncertainty of measurement of the length reference instrument

Test length L_{RS}	Relative deviation of the measuring system ΔL_{rel}, %	Expanded relative uncertainty of measurement U_L, %	Max. relative deviation of measuring system including measurement uncertainty of length reference instrument ΔL_{max}, %
0,40 mm	0,15	0,24	0,39

where

$$\Delta L_{max} = |\Delta L_{rel}| + U_L \tag{B.8}$$

The result of the example means that the deviation of the measuring system, including the uncertainty of measurement of the length reference instrument specified in 4.4.3 amounting to ± 0,5 % is complied with.

B.1.3 Verification of the indenter

The indenter, consisting of indenter tip and holder, cannot be verified, respectively calibrated, in-site. A valid calibration certificate of an accredited calibration laboratory shall exist which confirms the geometrical deviations of the indenter (see 4.3).

B.1.4 Verification of the test cycle

In 4.4 the permissible deviation for every section of the test cycle is stipulated as ± 0,5 s. While measuring with a usual time measuring system (stopwatch), the uncertainty of measurement can be indicated as 0,1 s. Therefore, an estimation of the uncertainty of measurement is not necessary.

B.2 Indirect verification of the hardness testing machine

NOTE In this Annex the index “CRM (Certified Reference Material)” means, according to the definitions of the hardness testing standards, “Hardness Reference Block”.

By indirect verification with hardness reference blocks, the overall function of the hardness testing machine is checked and the repeatability, as well as the deviation of the hardness testing machine from the real hardness value, are determined.

The uncertainty of measurement of the indirect verification of the hardness testing machine follows from the equation:

$$u_{HTM} = \sqrt{u_{CRM}^2 + u_{CRM\text{-}D}^2 + u_H^2 + u_{ms}^2} \tag{B.9}$$

where

u_{CRM} is the calibration uncertainty of the hardness reference block according to the calibration certificate for $k = 1$;

$u_{CRM\text{-}D}$ is the hardness change of the hardness reference block since its last calibration due to drift (negligible for use of the hardness reference block complying with the standard);

u_H is the standard uncertainty of hardness testing machine when measuring CRM;

u_{ms} is the uncertainty due to the resolution of the hardness testing machine.

EXAMPLE

Hardness of the hardness reference block	H_{CRM} = (802,7 ± 12,0) HK1
Uncertainty of measurement of the hardness reference block	U_{CRM} = ± 12,0 HK1
Resolution of the hardness testing machine	δ_{ms} = 0,1 µm

Table B.7 — Results of the indirect verification

No.	Measured indentation diagonal, d mm	Calculated hardness value, H HK[a]
1	0,133 2	802,0
2	0,133 3	800,8
3	0,133 5_{max}	$798{,}4_{min}$
4	0,133 0_{min}	$804{,}4_{max}$
5	0,133 1	803,2
Mean value $\bar{H}$	0,133 2	801,7
Standard deviation s_H		2,3

[a] HK: Knoop hardness

$$\begin{aligned}\bar{b} &= \bar{H} - H_{CRM} \\ &= 801{,}7 - 802{,}7 = -1{,}0 \text{ HK}\end{aligned} \tag{B.10}$$

$$u_H = \frac{t \cdot s_H}{\sqrt{n}} \tag{B.11}$$

For t = 1,14, n = 5 and s_H = 2,3 HK follows:

u_H = 1,18 HK

B.3 Budget of uncertainty of measurement

Table B.8 — Budget of uncertainty of measurement

Quantity X_i	Estimated value x_i	Standard uncertainty of measurement $u(x_i)$	Distribution type	Sensitivity coefficient c_i	Uncertainty contribution $u_i(H)$
u_{CRM}	402,7 HK	6,0 HK	Normal	1,0	6,0 HK
u_H	0 HK	1,18 HK	Normal	1,0	1,18 HK
u_{ms}	0 HK	0,000 029 mm	Rectangular	4 284,6[a]	0,00 HK
u_{CMR-D}	0 HK	0 HK	Triangular	1,0	0 HK
Combined uncertainty of measurement u_{HTM}					6,12 HK
Expanded uncertainty of measurement U_{HTM} (k = 2)					12,2 HK

HK: Knoop hardness

[a] $c = \partial H / \partial d = 2(H/d)$ for H = 801,7 HK1 and d = 0,133 1 mm

Table B.9 — Maximum deviation of the hardness testing machine including the uncertainty of measurement

Measured hardness on the hardness testing machine H HK	**Expanded uncertainty of measurement** U_{HTM} HK	**Deviation of the testing machine when calibrating with the reference block** $\lvert\bar{b}\rvert$ HK	**Maximum deviation of the testing machine including uncertainty of measurement** ΔH_{HTMmax} HK
801,7 HK1	12,2	1,0	13,2
HK: Knoop hardness			

where

$$\bar{b} = \bar{H} - H_{CRM} \tag{B.12}$$

$$\Delta H_{HTMmax} = U_{HTM} + \Delta H_{HTM} = 12{,}2 + 1{,}0 = 13{,}2 \text{ HK}$$

The result of the example above means that the permissible limit deviation of the testing machine, including the uncertainty of measurement of the testing machine specified in 5.8 amounting to ± 2 %, is complied with.

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