INTERNATIONAL STANDARD

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Metallic materials — Knoop hardness test —

Part 3: Calibration of reference blocks

Matériaux métalliques — Essai de dureté Knoop —

Partie 3: Étalonnage des blocs de référence

Reference number
ISO 4545-3:2005(E)

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

ISO 4545-3 cancels and replaces the first edition of ISO 4547:1993, 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 3: Calibration of reference blocks

1 Scope

This part of ISO 4545 specifies the method for the calibration of reference blocks to be used for the indirect verification of Knoop hardness testing machines, as specified in ISO 4545-2.

The method is applicable only for indentations with long 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 376:2004, *Metallic materials — Calibration of force-proving instruments used for the verification of uniaxial testing machines*

ISO 4287:1997, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

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

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

3 Manufacture of the block

3.1 The block shall be specially manufactured for use as a hardness-reference block.

NOTE Attention is drawn to the need to use a manufacturing process which will give the necessary homogeneity, stability of structure and uniformity of surface hardness.

3.2 The reference block thickness shall be greater than twenty times the depth of indentation made with the certified test force.

3.3 The reference blocks shall be free of magnetism.

3.4 The maximum deviation in flatness of the surfaces shall not exceed 0,005 mm.

3.5 The maximum error in parallelism shall not exceed 0,010 mm/50 mm.

3.6 The test surface shall be free from scratches which interfere with the measurement of the indentations. The surface roughness Ra shall not exceed 0,1 µm for the test surface. The sampling length L shall be 0,80 mm (see ISO 4287:1997, 3.1.9).

3.7 To verify that no material is subsequently removed from the reference block, the thickness at the time of calibration shall be marked on it, to the nearest 0,1 mm, or an identifying mark shall be made on the test surface [see 8.1.e)].

4 Calibration machine

4.1 In addition to fulfilling the general requirements specified in ISO 4545-2, the calibration machine shall also meet the requirements given in 4.2 to 4.7.

NOTE Examples of procedures for adjustment of illumination systems are given in Annex A.

4.2 The machine shall have been directly verified in intervals not exceeding 12 months.

Direct verification involves:

a) calibration of the test force;

b) verification of the indenter;

c) calibration of the measuring device;

d) verification of the testing cycle; if this is not possible, at least the force versus time behaviour.

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

4.4 Each test force shall be measured three times using an elastic proving device (of ISO 376:2004, Class 0,5 or better), or by another method having the same or better accuracy. Each measurement shall agree with the nominal value to within ± 0,5 %.

4.5 The indenter shall meet the following requirements:

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

b) The angles α and β (see ISO 4545-1:2005, Figure 1), between opposite edges at the vertex of the diamond pyramid, shall be (172,5 ± 0,1)° and (130 ± 0,1)°.

The angle between the axis of the diamond pyramid and the axis of the indenter holder (normal to the seating surface) shall not exceed 0,3°. The four faces shall meet at a point; the length of any common junction between opposite faces being less than 0,3 µm.

4.6 The device for measuring the diagonal of the indentation shall permit estimation of the length of the diagonal to within ± 0,1 µm.

The measuring device shall be calibrated against an accurately ruled line-scale (object micrometer) or a device of equivalent accuracy. The errors of the line-scale shall be known within an uncertainty of 0,02 µm.

The maximum permissible error of the measuring device shall be ± 0,08 % or 0,3 µm, whichever is greater.

4.7 The maximum allowable vibrational acceleration reaching the machine shall be less than 0,005 g_n [g_n being the acceleration due to gravity (g_n = 9,806 65 m/s^2)].

5 Calibration procedure

The reference blocks shall be calibrated in a calibration machine as specified in Clause 4, at a temperature of (23 ± 5) °C, using the general procedure described in ISO 4545-1.

During calibration, the thermal drift should not exceed 1 °C.

The time from the initial application of force until the full test force is reached shall be between 5 s and 7 s. The approach velocity of the indenter shall be within the range 15 µm/s to 70 µm/s. The duration of the test force shall be from 13 s to 15 s.

6 Number of indentations

On each reference block, a minimum of five indentations shall be made, uniformly distributed over the test surface.

To reduce the measurement uncertainty, more than 5 indentations should be made. It is recommended to make 10, 15 or 25 indentations distributed over 5 locations on the reference block.

7 Uniformity of hardness

7.1 In the case of 25 indentations, let d_1, d_2, ..., d_{25} be the values of the measured diagonals of the 25 calibration indentations, arranged in increasing order of magnitude, and let

$$\bar{d} = \frac{d_1 + d_2 + \ldots + d_{25}}{25} \quad (1)$$

The non-uniformity, U, of the block under the particular conditions of calibration is characterized by

$$U = d_{25} - d_1 \quad (2)$$

and is expressed as a percentage U_{rel} of $\bar{d}$ as:

$$U_{rel} = \frac{100\left(d_{25} - d_1\right)}{\bar{d}} \quad (3)$$

7.2 The uniformity of the reference block is satisfactory if $U \leqslant 0{,}001$ mm. If $U > 0{,}001$ mm, the uniformity of the reference block is satisfactory when U_{rel} is less than or equal to the percentages indicated in Table 1.

7.3 The determination of the uncertainty of measurement of hardness-reference blocks is given in Annex B.

Table 1

Hardness range of reference blocks	Test force N	Maximum permissible U_{rel} %
100 ⩽ HK ⩽ 200	0,098 07 ⩽ F ⩽ 0,980 7	8
200 ⩽ HK ⩽ 250		5
250 ⩽ HK ⩽ 650		4
HK > 650		3
100 ⩽ HK ⩽ 250	0,980 7 < F ⩽ 4,903	7
250 < HK ⩽ 650		4
HK > 650		3
100 ⩽ HK ⩽ 250	4,903 < F ⩽ 19,614	4
250 < HK ⩽ 650		3
HK > 650		2

8 Marking

8.1 Each reference block shall be marked with the following particulars:

a) arithmetic mean of the hardness values found in the calibration test, for example 249 HK 1, if possible;

b) name or mark of the supplier or manufacturer;

c) serial number;

d) name or mark of the calibrating agency;

e) thickness of the block, or an identifying mark on the test surface (see 3.7);

f) year of calibration, if not indicated in the serial number.

8.2 Any mark put on the side of the block shall be the right way up when the test surface is the upper face.

8.3 Each delivered reference block shall be accompanied with a document giving at least the following information:

a) a reference to this part of ISO 4545;

b) the identity of the block;

c) the date of calibration;

d) the arithmetic mean of the hardness values and the value characterizing the non-uniformity of the block;

e) information about the location of the reference indentation and the value of the long diagonal.

9 Validity

The reference block is only valid for the scale for which it was calibrated.

The calibration validity should be limited to a duration of 5 years. Attention is drawn to the fact that, for Al- and Cu-alloys, the calibration validity could be reduced to 2-3 years.

Annex A
(informative)

Adjustment of Kohler illumination systems

A.1 General

While some optical systems are permanently aligned, others have means of minor adjustments. To gain the utmost in resolution, the following adjustments shall be made.

A.2 Kohler illumination

Focus, to critical sharpness, the surface of a flat polished specimen.

Centre the illuminating source.

Centrally align the field and aperture diaphragms.

Open the field diaphragm so that it just disappears from the field of view.

Remove the eyepiece and examine the rear focal plane of the objective. If all the components are in their proper places, the source of illumination and the aperture diaphragm will appear in sharp focus.

A full-aperture diaphragm is preferred for maximum resolving power. If glare is excessive, reduce the aperture; but never use less than 3/4 of the opening, since resolution would be decreased and diffraction phenomena could lead to false measurements.

If the light is too strong for eye comfort, reduce the intensity by using of an appropriate neutral density filter or rheostat control.

Annex B
(informative)

Uncertainty of mean hardness value of hardness-reference blocks

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

B.1 Direct verification of the hardness-calibration machine

B.1.1 Calibration of the test force

See ISO 4545-2:2005, Annex B.

B.1.2 Calibration of the optical measuring device

See ISO 4545-2:2005, Annex B.

B.1.3 Verification of the indenter

See ISO 4545-2:2005, Annex B.

B.1.4 Verification of the test cycle

See ISO 4545-2:2005, Annex B.

B.2 Indirect calibration of the hardness-calibration machine

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

The uncertainty of measurement of the indirect calibration of the hardness-calibration machine follows from the equation:

$$u_{CM} = \sqrt{u_{CRM-P}^2 - u_{xCRM-1}^2 - u_{CRM-D}^2 - u_{ms}^2} \qquad (B.1)$$

where

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

u_{xCRM-1} is the standard deviation of the hardness-calibration machine due to its repeatability;

u_{CRM-D} is the hardness change of the primary hardness-reference block since its last calibration due to drift;

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

EXAMPLE

Primary hardness-reference block:	402,1 HK1
Uncertainty of measurement of the primary hardness-reference block (k = 1)	u_{CRM} = ± 6,0 HK
Time drift of the primary hardness-reference block	$u_{CRM\text{-}D}$ = 0
Resolution of the optical measuring device	R_{ms} = 0,1 µm

Table B.1 — Results of the indirect verification

No.	Measured indentation diagonal d mm	Calculated hardness value H HK[a]
1	0,188 0	402,6
2	$0{,}187\ 5_{min}$	$404{,}7_{max}$
3	0,187 9	403,0
4	0,188 4	400,9
5	$0{,}188\ 9_{max}$	$399{,}2_{min}$
Mean value $\overline{H}$	0,188 1	402,1
Standard deviation $s_{x\text{CRM-1}}$	0,000 50	2,1
Standard uncertainty of measurement $u_{x\text{CRM-1}}$	0,000 26	1,08

[a] HK: Knoop hardness.

$$u_{x\text{CRM-1}} = \frac{t \cdot s_{x\text{CRM-1}}}{\sqrt{n}} = 1{,}08 \qquad \text{(B.2)}$$

(t = 1,14 for n = 5)

Table B.2 — 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 $u_i(H)$ HK
u_{CRM}	402,1	6,0 HK	Normal	1,0	6,0
$u_{x\text{CRM-1}}$	0	1,08 HK	Normal	1,0	1,08
u_{ms}	0	0,000 029 mm	Rectangular	4275,4 [a]	0,12
$u_{CRM\text{-}D}$	0	0 HK	Triangular	1,0	0
Combined uncertainty of measurement u_{CM}					6,1

HK: Knoop hardness.

[a] The sensitivity coefficient follows from:

$$c = \partial H / \partial d = 2(H/d) \qquad \text{(B.3)}$$

for H = 402,1 HK, d = 0,1881 mm

B.3 Uncertainty of measurement of hardness-reference blocks

The uncertainty of measurement of hardness-reference blocks follows from the equation:

$$u_{\mathrm{CRM}} = \sqrt{u_{\mathrm{CM}}^2 + u_{x\mathrm{CRM\text{-}2}}^2} \tag{B.4}$$

where

u_{CRM} calibration uncertainty of hardness-reference blocks;

$u_{x\mathrm{CRM\text{-}2}}$ standard deviation due to the inhomogeneity of the hardness distribution on the hardness-reference block;

u_{CM} see Equation B.1.

Table B.3 — Determination of the inhomogeneity of the hardness-reference block

No.	Measured indentation diagonal d mm	Calculated hardness value H_{CRM} HK
1	0,188 1	402,2
2	0,187 6_{min}	$404{,}3_{\mathrm{max}}$
3	0,188 2	401,7
4	0,188 5_{max}	$400{,}5_{\mathrm{min}}$
5	0,187 6	404,3
Mean value $\bar{H}$	0,188 0	402,6
Standard deviation $s_{x\mathrm{CRM\text{-}2}}$	0,000 39	1,69
HK: Knoop hardness.		

Standard uncertainty of CRM

$$u_{x\mathrm{CRM\text{-}2}} = \frac{t \cdot s_{x\mathrm{CRM\text{-}2}}}{\sqrt{n}} \tag{B.5}$$

with $t = 1{,}14$ and $n = 5$:

$u_{x\mathrm{CRM\text{-}2}} = 0{,}86$ HK

Table B.4 — Uncertainty of measurement of the hardness-reference block

Hardness of hardness-reference block H_{CRM} HK	Inhomogeneity of the hardness-reference block $u_{x\mathrm{CRM\text{-}2}}$ HK	Uncertainty of measurement of primary hardness-calibration machine u_{CM} HK	Expanded calibration uncertainty of hardness-reference block U_{CRM} HK
402,1	0,86	6,1	12,3
HK: Knoop hardness.			

with

$$U_{\mathrm{CRM}} = 2\sqrt{u_{\mathrm{CM}}^2 - u_{x\mathrm{CRM\text{-}2}}^2} \tag{B.6}$$

Bibliography

[1] A. SAWLA: *Uncertainty of measurement in the verification and calibration of the force measuring systems of testing machines*, Proceedings of the Asia-Pacific symposium on measurement of force, mass and torque (APMF), Tsukuba, Japan, November 2000

[2] A. WEHRSTEDT, I. PATKOVSZKY: *News in the field of standardization about verification and calibration of materials testing machines*, May 2001, EMPA Academy 2001

[3] W. GABAUER: *Manual 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] T. POLZIN, D. SCHWENK: Method for Uncertainty Determination of Hardness Testing; PC File for Determination, *Materialprüfung* 44 (2002) 3, pp. 64-71

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