INTERNATIONAL STANDARD

ISO 6507-3

Third edition
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Metallic materials — Vickers hardness test —

Part 3: Calibration of reference blocks

Matériaux métalliques — Essai de dureté Vickers —

Partie 3: Étalonnage des blocs de référence

Reference number
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Contents

Page

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 6507-3 was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

This third edition cancels and replaces the second edition (ISO 6507-3:1997), which has been technically revised.

ISO 6507 consists of the following parts, under the general title *Metallic materials — Vickers hardness test*:

— *Part 1: Test method*

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

— *Part 3: Calibration of reference blocks*

— *Part 4: Tables of hardness values*

Metallic materials — Vickers hardness test —

Part 3:
Calibration of reference blocks

1 Scope

This part of ISO 6507 specifies a method for the calibration of reference blocks to be used for the indirect verification of Vickers hardness testing machines, as specified in ISO 6507-2.

The method is applicable 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 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 6507-1:2005, *Metallic materials — Vickers hardness test — Part 1: Test method*

ISO 6507-2:2005, *Metallic materials — Vickers hardness test — Part 2: Verification and calibration of testing machines*

3 Manufacture of reference blocks

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 Each metal block to be calibrated shall be of a thickness not less than 5 mm.

3.3 The reference blocks shall be free of magnetism. It is recommended that the manufacturer shall ensure that the blocks, if made of steel, have been demagnetized at the end of the manufacturing process.

3.4 The maximum deviation in flatness of the test and support surfaces shall not exceed 0,005 mm. The maximum error in parallelism shall not exceed 0,010 mm in 50 mm.

3.5 The test surface shall be free from scratches which interfere with the measurement of the indentations. The surface roughness R_a shall not exceed 0,000 05 mm for the test surface and 0,000 8 mm for the bottom surface. The sampling length l shall be 0,80 mm (see ISO 4287:1997, 3.1.9).

3.6 To verify that no material has been subsequently removed from the reference block, its thickness at the time of calibration shall be marked on it, to the nearest 0,01 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 6507-2, the calibration machine shall also meet the requirements of 4.2 to 4.7.

4.2 The calibration machine shall be verified directly at intervals not exceeding 12 months.

Direct verification involves

a) verification of the test force;

b) verification of the indenter;

c) verification 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 using an elastic proving device (or ISO 376:2004, Class 0.5 or better), or by another method having the same or better accuracy. This measurement shall agree with the nominal value to within ± 0,1 % for normal and low-force hardness, and to within ± 0,5 % for microhardness.

4.5 The indenter shall meet the following requirements:

a) The four faces of the square-based diamond pyramid shall be highly polished, free from surface defects, and flat within 0,000 3 mm.

b) The angle between the opposite faces of the vertex of the diamond pyramid shall be 136° ± 0,1°.

The angle between the axis of the diamond pyramid and the axis of the indenter-holder (normal to the seating surface) shall be less than 0,3°.

c) The point of the diamond indenter shall be examined with a high-power measuring microscope or preferably with an interference microscope and, if the four faces do not meet at a point, the line of junction between opposite faces shall comply with the values in Table 1.

Table 1

Ranges of test force, F N	Maximum permissible length of the line of junction, a mm
$F \geqslant 49{,}03$	0,001
$1{,}961 \leqslant F < 49{,}03$	0,000 5
$0{,}098\,07 \leqslant F < 1{,}961$	0,000 25

d) It shall be verified that the quadrilateral which would be formed by the intersection of the faces with a plane perpendicular to the axis of the diamond pyramid has angles of 90° ± 0,2° (see Figure 1).

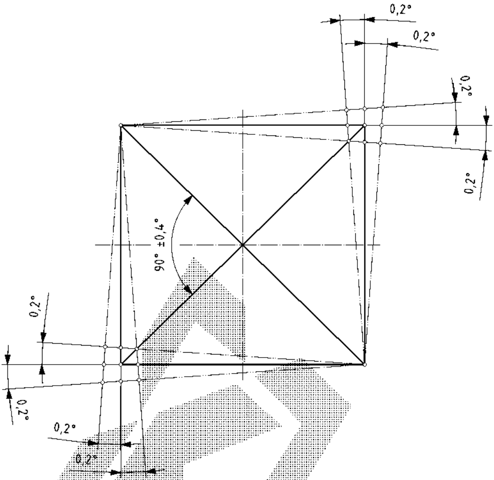

Figure 1 — Permissible difference of the sectional planes of the square form

4.6 The resolution required of the measuring device depends on the size of the smallest indentation to be measured.

The scale of the measuring device shall be graduated to permit estimation of the diagonals of the indentation in accordance with Table 2.

Table 2

Diagonal length, d mm	**Resolution of the measuring device**	**Maximum permissible error**
$d \leqslant 0{,}040$	0,000 1 mm	0,000 2 mm
$0{,}040 < d \leqslant 0{,}200$	0,25 % of d	0,5 % of d
$d > 0{,}200$	0,000 5 mm	0,000 1 mm

The measuring device shall be verified by measurements made on an object micrometer at a minimum of five intervals over each working range.

The maximum permissible error shall not exceed the values given in Table 2.

5 Calibration procedure

The reference blocks shall be calibrated in a calibration machine as described in Clause 4, at a temperature of (23 ± 5) °C, using the general procedure specified in ISO 6507-1.

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, and the approach velocity of the indenter, shall meet the requirements given in Table 3.

The duration of application of the test force shall be 13 s to 15 s.

For microhardness testing, the maximum allowable vibrational acceleration reaching the calibration machine shall be 0,005 g_n (g_n equals the standard acceleration of free fall: $g_n = 9{,}806\ 65\ \text{m/s}^2$).

Table 3

Ranges of test force, F N	Time for application of the test force s	Approach velocity of the indenter mm/s
$F < 1{,}961$	$\leqslant 10$	0,05 to 0,2
$1{,}961 \leqslant F < 49{,}03$	$\leqslant 10$	0,05 to 0,2
$F \geqslant 49{,}03$	6 to 8	0,05 to 1

6 Number of indentations

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

For microhardness tests and to reduce the measurement uncertainty, more than 5 indentations should be made. It is recommended to make 10, 15 or 25 indentations on 5 locations on the reference block.

7 Uniformity of hardness

7.1 Let d_1, d_2, d_3, d_4, d_5, be the arithmetic mean values of the measured diagonals, arranged in increasing order of magnitude, and

$$\bar{d} = \frac{d_1 + d_2 + d_3 + d_4 + d_5}{5} \tag{1}$$

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

$$U = d_5 - d_1 \tag{2}$$

and is expressed as a percentage U_{rel}, as

$$U_{rel} = \frac{100 \times (d_5 - d_1)}{\bar{d}} \tag{3}$$

7.2 The maximum permissible value of non-uniformity U_{rel} of a reference block is given in Table 4.

Table 4

Hardness of block	Maximum permissible value of non-uniformity U_{rel}, %		
	< HV 0,2	⩽ HV 0,2 to ⩽ HV 5	HV 5 to HV 100
⩽ 225 HV [a]	4,0 or 0,001 mm [b]	3,0	2,0
> 225 HV		2,0	1,0

[a] For hardness values < 150 HV, the maximum permissible value of non-uniformity shall be 8 % or 0,001 mm, whichever is greater.

[b] Whichever is greater.

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

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 HV 30;

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 3.6);

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 6507;

b) identity of the block;

c) date of calibration;

d) 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, together with the mean measured diagonal length.

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)

Uncertainty of the mean hardness value of hardness-reference blocks

The metrological chain necessary to define and disseminate hardness scales is shown in Figure D.1 in ISO 6507-1:2005.

A.1 Direct verification of the hardness-calibration machine

A.1.1 Calibration of the test force

See ISO 6507-2:2005, Annex C.

A.1.2 Calibration of the optical measuring device

See ISO 6507-2:2005, Annex C.

A.1.3 Verification of the indenter

See ISO 6507-2:2005, Annex C.

A.1.4 Verification of the test cycle

See ISO 6507-2:2005, Annex C.

A.2 Indirect calibration of the hardness-calibration 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 the 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:

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

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: 400,1 HV 30

Uncertainty of measurement of the primary hardness-reference block ($k = 1$) $u_{CRM} = \pm 2,5$ HV

Time drift of the primary hardness-reference block $u_{CRM-D} = 0$

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

Table A.1 — Results of the indirect verification

No.	Measured indentation diagonal d mm	Calculated hardness value H HV [a]
1	$0,373\,4_{max}$	$399,0_{min}$
2	0,373 0	399,9
3	$0,372\,5_{min}$	$400,9_{max}$
4	0,372 8	400,3
5	0,372 9	400,3
Mean value	0,372 92	400,1
Standard deviation s_{xCRM-1}	0,000 33	0,70
Standard uncertainty of measurement u_{xCRM-1}	0,000 17	0,36

[a] HV: Vickers hardness.

$$u_{xCRM-1} = \frac{t \cdot s_{xCRM-1}}{\sqrt{n}} = 0,36 \quad \text{(A.2)}$$

($t = 1,14$ for $n = 5$)

Table A.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 $u_i(H)$
u_{CRM}	400,1 HV 30	2,5 HV	Normal	1,0	2,5 HV
u_{xCRM-1}	0 HV	0,36 HV	Normal	1,0	0,36 HV
u_{ms}	0 HV	0,1 µm = 0,000 1 mm	Rectangular	2 146,0 [a]	0,06 HV
u_{CRM-D}	0 HV	0 HV	Triangular	1,0	0 HV
Combined uncertainty of measurement u_{CM}					2,53 HV

HV: Vickers hardness.

[a] The sensitivity coefficient follows from:

$$c = \partial H / \partial d = 2(\mathrm{HV}/d) \quad \text{(A.3)}$$

for $H = 400,1$ HV, $d = 0,372\,92$ mm.

A.3 Uncertainty of measurement of hardness-reference blocks

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

$$u_{CRM} = \sqrt{u_{CM}^2 + u_{xCRM-2}^2} \quad (A.4)$$

where

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

u_{xCRM-2} is the standard deviation due to the inhomogeneity of the hardness distribution on the hardness-reference block;

u_{CM} see Equation (A.1).

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

No.	Measured indentation diagonal d mm	Calculated hardness value H_{CRM} HV [a]
1	$0{,}373\,6_{max}$	$398{,}6_{min}$
2	0,373 1	399,6
3	$0{,}372\,3_{min}$	$401{,}4_{max}$
4	0,372 5	400,9
5	0,373 1	399,6
Mean value	0,372 92	400,0
Standard deviation s_{xCRM-2}	0,000 52	1,12

[a] HV: Vickers hardness.

Standard uncertainty of CRM:

$$u_{xCRM-2} = \frac{t \cdot s_{xCRM-2}}{\sqrt{n}} \quad (A.5)$$

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

$u_{xCRM-2} = 0{,}58$ HV

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

Hardness of hardness-reference block H_{CRM} HV [a]	Inhomogeneity of the hardness-reference block u_{xCRM-2} HV	Uncertainty of measurement of primary hardness-calibration machine u_{CM} HV	Expanded calibration uncertainty of hardness-reference block U_{CRM} HV
400,1	0,57	2,53	5,18

[a] HV: Vickers hardness.

with

$$U_{CRM} = 2\sqrt{u_{CM}^2 + u_{xCRM-2}^2} \quad (A.6)$$

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